Unified Patent Court
Paris Local Division

Docket No. 2015/01

Claimant: 3 ABRASIVE (3A) (A company with its registered office located in the USA)
Defendant: La Toilemeri SA (A company with its registered office located in France)

INDEX

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Defendant: La Toilemeri SA (A company with its registered office located in France)

RI – Report of inspection

- RI1 – Distribution contract
- RI2 – Test report (confidential)
- RI3 – Durations (confidential)

APM – Application for provisional measures

- APM1 – Patent EP 2 900 000
- APM2 – Claim 1 of EP 2 900 000
- APM3 – Report of inspection
- APM4 – D1 FR 2 565 870 and translation
- APM5 – Opposition Division Decision (September 3, 2014)
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OPM – Objections to provisional measures

- OPM1 – Declaration Duck (Annex DEF 1)
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- OPM3 – XPS Study (Annex DEF 3)

Axel CASALONGA
March 30, 2015

SYNOPSIS
MOCK TRIAL April 2, 2015

The main procedural questions which will be developed during this Mock Trial are the following:

- the Application for an order to preserve evidence and to inspect premises (Rules 192, 195, 197 and 199): what should be indicated in the Application; how the order should be drafted by the panel; who should be in charge of the inspection; whether the European Patent Attorney of the patentee could be present during the inspection; whether a bailiff could be appointed; whether police forces could be requested to help during the operation ; how should confidential information be treated)
- the Application for provisional measures and the following procedure (Rules 206, 209, 210 and 211). The Application will follow the result of the inspection and the pending opposition procedure at the EPO will have to be considered.

The mock trial per se will comprise two oral hearings concerning respectively:

- the granting of an order to preserve evidence and to inspect the premises (without hearing the defendant)
- the granting of provisional measures in the form of a preliminary injunction (inter partes hearing)

1) Main facts and venue

The Claimant is a US company named “3 Abrasive (3A), owner of the European patent EP no. 2900000 entitled « *Flexible abrasives* ».

The Patent is valid and in force in the Contracting States designated by the Patent, including France, Germany, The Netherlands, United Kingdom and Sweden. All the Designated Contracting States of the Patent have ratified and implemented the UPC Agreement.

The Claimant exports products made according to the Patent throughout Europe, including France, Germany, The Netherlands and Sweden. Abrasive belts for polishing hard stones and manufactured according to the patented method are being sold all over Europe since more than three years.

The Patent was the subject of an opposition proceedings in the European Patent Office. In a decision dated 3 September 2014, the opposition division rejected the opposition and decided that the Patent be maintained as granted. An Appeal is pending before a Board of Appeal.

The Patent relates to flexible abrasives. It was granted in English and claims a method of forming an abrasive member with a step of electro deposition of abrasive nickel patches, the abrasive patches being maintained laterally by a resin material.

The Defendant is a French company named “La Toilemeri”.

The Defendant has a commercial activity in the field of abrasive products for industrial uses.

2) Infringement action

On 25 August 2014, La Toilemeri published on its Internet website an announcement concerning an allegedly new abrasive product.

On this web site, La Toilemeri posted a photograph of this product as well as marketing statements indicating that the advantages of the product were:

- an excellent lateral bearing of the abrasive patches,
- a reinforced securing of the abrasive patches onto the support.

In addition, the website mentioned that the abrasive patches are embedded in a non woven sheet comprising long fibers of polyamide.

3A considers that the advantages mentioned on this website can only be obtained with a product manufactured according to the method disclosed in EP Patent N° 2 900 000 .

Therefore, 3A decided to engage an infringement action against La Toilemeri, considering that this web site announcement was a clear offer to sale on the territory of the countries designated in its European patent. No Application for opt out having been filed, 3A decided to bring the action before the Paris local division since the defendant is domiciled in France. Another reason for this choice was the hope that an order for inspection at the premises of La Toilemeri would possibly be more easy to obtain from the Paris local division comprising two Judges of French nationality.

The Statement of claim has been filed on October 13, 2014, in the English language, which is one of the official languages of the EPO that the Paris local division accepts (under Art 49(2) UPCA).

The panel comprises three judges: Mr Paul Maier (FR), President, Mrs Sophie Canas (FR) and Mr Colin Birss (UK).

On October 30, 2014, the panel designated Mr Colin Birss as judge rapporteur (Rule 18)

On 18 December 2014, La Toilemeri filed a Defense challenging any evidence of infringement and a Counter claim for revocation of the Patent. Simultaneously, the Defendant filed an Intervention in the Appeal procedure which had been initiated at the EPO on 21 November 2014 by the previous Opponent. The defendant, making use of the provisions of Rule 298, also requested the Court to request that the appeal procedure at the EPO be accelerated and to stay its proceedings pending the outcome of the EPO appeal.

The local division decided, according to Article 33(3)(a) UPCA to keep the complete case (infringement and revocation) for decision and requested the President of the Court of First Instance to allocate from the Pool of Judges, a technically qualified judge with qualifications and experience in the field of materials.

Mr Kim Finnilä (FI) was consequently allocated to the panel as technically qualified judge.

3) Application for preserving evidence and inspection

Following the arguments of La Toilemeri in its Statement of defense, insisting particularly on the absence of evidence of reproduction of the claimed method of manufacturing, 3A decided to file on 29 December 2014 an Application for preserving evidence (Rule 192) with a request of an order for inspection at the premises of La Toilemeri, near Paris.

3A also requests the Court to issue this order without hearing the Defendant so as to avoid any risk of the evidence disappearing.

The oral hearing concerning this request and the Application for preserving evidence took place on 5 January 2015.

By an extraordinary special effect, we are now able to present you today this oral hearing exactly as it happened.

4) Result of the inspection

The inspection ordered by the Court at the end of the oral hearing of 5 January 2015, was performed, as determined by the Court, on 8 January 2015.

The report of the inspection made on the spot by the person nominated by the Court shows how the new product of La Toilemeri is manufactured.

In particular, the inspection revealed that the nickel mixed with diamond powder was effectively electro deposited through a layer of non woven fabric, on copper attached to the support.

The inspection allowed also to find copies of a distribution agreement for the product in Europe, with commercial indications.

Finally, a test report was found, relating to the technical effect of the non woven fabric remaining between the nickel abrasive patches. This test report, although important for the evidence of infringement, was indicated as being confidential by the employees of La Toilemeri. It was consequently sealed in an envelope kept by the person in charge of the inspection and subsequently handed to the judge rapporteur.

After study of the content of the enveloppe, the judge rapporteur ordered that the test report be

be disclosed, only to the Representatives of the parties and to two specially named persons of the Applicant i.e. the chief of the Patent Department Mr. X and a technical manager Mr. Y after those two persons had signed a declaration under oath not to take any copy, and not to disseminate the information contained in the confidential documents for a period of 4 years.

The tests report shows that, on use, in the product manufactured according to the method observed during the inspection and having a non woven material made of long fibers of polyurethane, only about 8% of the metal patches had chipped off.

3A was therefore conforted in their position concerning infringement and intended to file a reply to the statement of defense of La Toilemeri, on the basis of the evidence collected during the inspection.

5) Result of the inspection

However, on 16 January, 2015, 3A detected an announcement on the web site of La Toilemeri indicating that the product of La Toilemeri was about to be launched on a great scale as from next June 8, and presented in an international exhibition in Paris beginning May 28.

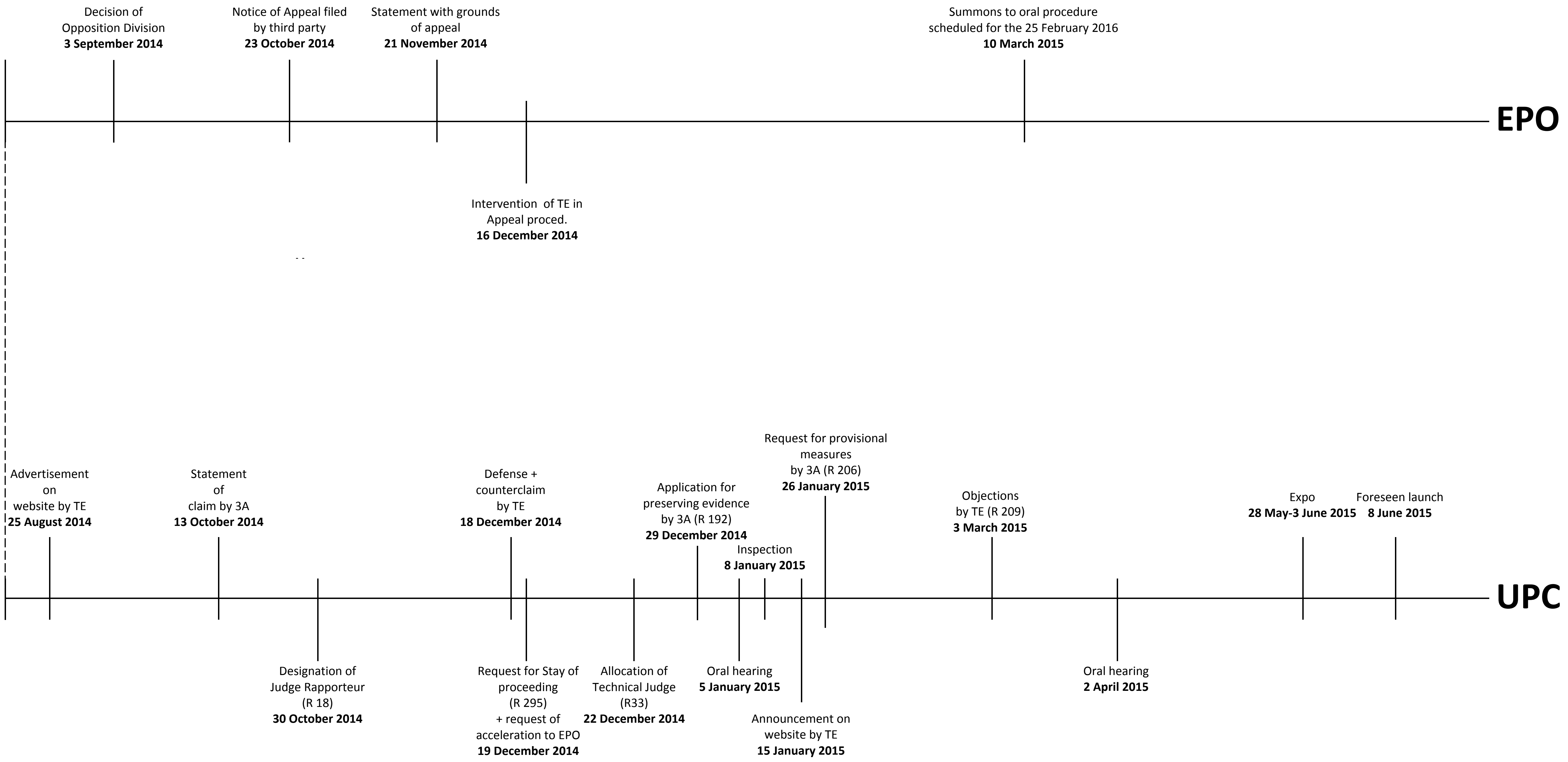
3A decided therefore to file instead an Application for provisional measures (Rule 206) in order to try stopping the acts which 3A considers infringe its European patent. This Application was filed on 26 January 2015.

La Toilemeri filed observations against this Application on 3 March 2015 (Rule 209)

The oral hearing has been scheduled for today.

Mock Trial before the Unified Patent Court - 2 April 2015

Chronology of events



EPO

Decision of Opposition Division
3 September 2014

Notice of Appeal filed by third party
23 October 2014

Statement with grounds of appeal
21 November 2014

Intervention of TE in Appeal proced.
16 December 2014

Summons to oral procedure scheduled for the 25 February 2016
10 March 2015

UPC

Advertisement on website by TE
25 August 2014

Statement of claim by 3A
13 October 2014

Designation of Judge Rapporteur (R 18)
30 October 2014

Defense + counterclaim by TE
18 December 2014

Request for Stay of proceeding (R 295) + request of acceleration to EPO
19 December 2014

Allocation of Technical Judge (R33)
22 December 2014

Application for preserving evidence by 3A (R 192)
29 December 2014

Oral hearing
5 January 2015

Inspection
8 January 2015

Announcement on website by TE
15 January 2015

Request for provisional measures by 3A (R 206)
26 January 2015

Objections by TE (R 209)
3 March 2015

Oral hearing
2 April 2015

Expo
28 May-3 June 2015

Foreseen launch
8 June 2015

The chronology

Mock Trial before the Unified patent Court – 2 april 2015

EPO	
3 September 2014	Decision of Opposition Division
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25 August 2014	Advertisement on website by TE
13 October 2014	Statement of claim by 3A
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22 December 2014	Allocation of Technical Judge (R33)
29 december 2014	Application for preserving evidence by 3A (R192)
5 January 2015	Oral hearing
8 January 2015	Inspection
15 January 2015	Annoucement on website by TE
26 January 2015	Request for provisional measures by 3A (R206)
3 March 2015	Objections by TE (R209)
2 April 2015	Oral hearing
28 May/3 June 2015	Expo
8 June 2015	Foreseen launch

(19) Europâisches Patentamt
European Patent Office
Office européen des brevets

Publication number: **EP 2 900 000 B1**

EUROPEAN PATENT SPECIFICATION

Date of publication of patent specification: **14.11.2010**

Int. Cl. : **B24D 11/00, B24D 18/00, B24D 3/00**

Application number: **22650211.5**

Date of filing: **13.04.2008**

Flexible abrasives.

Priority: **27.05.2007 US 2007 085 240**

Date of publication of application:
13.12.2009 Bulletin 2009/50

Publication of the grant of the patent:
14.11.2010 Bulletin 2010/45

Designated Contracting States:

DE FR GB NL SE

References cited:

US 4,256,467

FR-A- 2 565 870

Proprietor: **3 ABRASIVE (US)**

Inventor: **John SMART**

Representative: **Yves PARENT (CH)**

EP 2 900 000 B1

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid (Art. 99(1) European patent convention).

Rank Xerox (UK) Business Services 13.10/3.6/3.3.1)

Description

This invention relates to flexible abrasive member particularly suitable for abrading, grinding, smoothing, and finishing operations on stone, glass and other materials in heavy-duty applications.

Manufacturing process is known in which a flexible abrasive member comprising a flexible non-conductive mesh carrying a multitude of nickel deposits in which abrasive material, such as diamond grit, is embedded. The flexible abrasive member is manufactured by first laying a sheet of flexible nonconductive mesh material onto a smooth electrically conductive surface, suitably masked to expose only those surface portions where electrodeposition is desired, so that the mesh material is in immovable relationship with the conductive surface. Nickel is then electrodeposited onto the exposed portions of the smooth surface through the mesh material in the presence of abrasive material so that the abrasive material becomes embedded in the metal layer and the mesh becomes embedded in the nickel deposits. Finally, the mesh is stripped from the electrically conductive surface and cut into the desired shape.

The product produced according to this prior art process is structurally weak and only suitable for light-duty operations, such as lens grinding. If the product is used in heavier duty applications, such as abrading belts, the mesh has to be bonded to a suitable substrate. The heat generated during the abrading operation makes it difficult to provide a satisfactory bond, and difficulties have been experienced due to the belts breaking, the nickel deposits chipping off the intrinsically weak mesh, and delamination of the belts.

French patent no. 2,565,870 describes a method of forming an abrasive member wherein a metal layer is fixedly attached to one surface of a thermoplastic sheet, a mask is applied to the exposed surface of the metal layer, the mask having a multitude of discrete openings therein, and metal is electrodeposited through said discrete openings onto the metal layer in the presence of particulate abrasive material so that the particulate abrasive material becomes embedded in the metal deposits.

While such a method represents an improvement over the prior art discussed above, there is a tendency for the metal deposits to chip off the substrate due to the very high shearing forces applied to them.

According to the present invention the voids between the metal deposits are at least partially filled with resin material selected so as to reduce lateral movement of the metal deposits.

The resin material fills the interstices between the deposits, reducing the shearing forces applied to them during the abrading process. As a result, the tendency of the deposits to chip off is dramatically reduced.

The resin material can be chosen from polyurethane resins, polyamide resins, polycarbonate or high density poly ethylene. The resin chosen must be such that less than 10% of metal deposits chip off after five hours grinding of hard stone such as granite.

The resin is preferably filled with a filler, such as silicon carbide.

The flexible substrate is preferably in the form of a woven fabric, but it may be fibre glass epoxy laminate.

The invention will now be described in more details, by way of example, only, with reference to the accompanying drawings, in which:

Figure 1 shows in cross-section a short length of Kevlar fabric carrying diamond-bearing nickel deposits;

Figure 2 shows a laminated substrate bearing a surface mask defining a regular pattern of crescent-shaped holes;

Figure 3a shows a detail of one of the shaped holes; and

Figure 3b shows a detail of a group of holes.

Referring to Figure 1, a length of conductive Kevlar fabric 1 is covered by a mask with a multitude of openings and the laminate placed in an electrolytic deposition bath. Copper is deposited through the openings in the mask. The laminate obtained is then placed in another electrolytic deposition bath. Nickel is deposited onto the copper through the openings in the mask with diamond particles sprinkled into the tank during the electro-deposition. The mask is then removed to leave upstanding diamond-bearing nickel deposits lying on small copper disks 2.

It results from this that the nickel nodules 3 are electrolytically deposited on the copper discs 2 and have diamond particles 4 embedded therein.

The voids between the deposits 3 are filled with polyurethane resin 5. The resin 5 reduces lateral movement of the deposits 3 and has a profound effect on their tendency to chip off during the abrasion process. The resin has a greater effect than would result merely from its adhesive action due to the way in which it stabilizes the nodules in operation.

One of the factors inhibiting widespread use of this type of abrasive product in the past has been the difficulty of retaining the deposits on the substrate in the hostile environment of an industrial abrading machine.

The shoots may be cut into circular disks of different diameters for different grinding machines.

A test made on a grinding machine equipped with an abrasive product made as mentioned above, mounted on a rotating head of the machine, showed a chip off of only about 8% of the nickel nodules after five hours polishing a granite surface.

Alternatively, the shoots may be cut into strips and formed into belts by making a butt joint and applying a tape on the rear side with Bostik 7070$_{TM}$ adhesive. To minimize wear, the rear side should be slightly scuffed in the region where the tape is to be located so as to avoid a noticeable bump when the tape is in place. The edges should desirably be cut in a wavy line to reduce lateral movement.

The laminate 11, shown in figure 2, comprises a conductive Kevlar$_{TM}$ fabric covered with a surface mask 13 of photo-resist material defining crescent-shaped holes 14 through which electro-deposition occurs. The laminate shown in Figure 2 is subsequently placed in electrolytic tanks to permit deposition of copper and then, of nickel in the presence of diamond grit, through the shaped holes 14. This process produces crescent-shaped pellets at the locations of the holes with diamond grit embedded in the nickel.

After removal from the second tank, the mask is removed to leave a sheet consisting of a regular pattern of crescent-shaped pellets firmly attached to the Kevlar$_{TM}$ backing. Each pellet consists of an electrodeposit of nickel bearing the diamond grit carried on a crescent-shaped segment of copper bonded to the underlying fabric.

Figure 3a shows in detail the shape of the holes. The crescent-shapes are defined by overlapping circles of slightly different radii. Figure 3b shows how the holes are arranged in a symmetrical arrangement. The manufactured sheet is subsequently cut into circular disks or elongated strips, which in turn are formed into belts. The crescent-shaped modules make the belts unidirectional, in that the convex edge has to face the direction of movement of the belt. This is generally a significant advantage.

The use of crescent-shapes permits significant savings in diamond grit, since the surface area of the pellets is less than for circular pellets, without deterioration in the abrasive properties, and furthermore the removal of abraded matter is improved.

The holes can have other shapes. For example, honeycomb shapes provide the product with greater rigidity.

The spacing and size of the pellets can be varied to fine tune the properties of the abrasive product according to the intended application. A much greater degree of control can be exercised over the abrasive properties than was previously possible. For rough grinding purposes, the pellets are spaced further apart and larger diamonds employed. For smooth grinding and polishing applications, the pellets are brought closer together and smaller diamonds used.

Claim

1. A method for manufacturing an abrasive member comprising a flexible sheet (1) with a multitude of discrete metal protuberances (2,3)
wherein a multitude of copper protuberances (2) are formed on the flexible sheet (1),
nickel protuberances (3) are electrodeposited over the copper protuberances (2) in the presence of particulate abrasive material (4) so that the particulate abrasive material becomes embedded in the nickel deposits
and wherein the voids between the protuberances (2,3) are at least partially filled with resin material, the resin material being selected so as to reduce lateral movement of the nickel deposits.

Patentanspruch

1. Ein Verfahren zur Herstellung eines Schleifkörpers, der eine flexible Bahn (1) mit einer Vielzahl von metallischen Einzelvorsprüngen (2,3) enthält wobei eine Vielzahl von Kupfervorsrüngen auf der flexiblen Bahn (1) gebildet werden; Nickelvorsprünge (3) galvanisch auf die Kupfervorsprünge (2) in Anwesenheit eines teilchenförmiges Schleifmaterial (4) abgeschieden wird, wodurch letzteres in das abgeschiedenes Nickel (3) eingebettet wird; und wobei die Leerstellen zwischen den Vorsprüngen (2, 3) zumindest teilweise mit Harzmaterial (5) ausgefüllt sind, um die Seitenbewegung der Nickelabscheidungen zu reduzieren.

Revendication

1. Un procédé de fabrication d’un élément abrasif comprenant ’une feuille flexible (1) avec une multitude de protubérances métalliques individuelles dans lequel on forme une multitude de protubérances de cuivre (2) sur la feuille flexible (1) ; des protubérances de nickel sont déposées par électrolyse sur les protubérances de cuivre (2) en présence de particules de matière abrasive (4) de sorte que les particules de matière abrasive (4) se trouvent incrustées dans les dépots de nickel (3; les vides entre les dépôts de nickel (2, 3) étant, du moins en partie, remplis de résine (5), la résine étant choisie afin de réduire le mouvement latéral des dépôts de nickel.

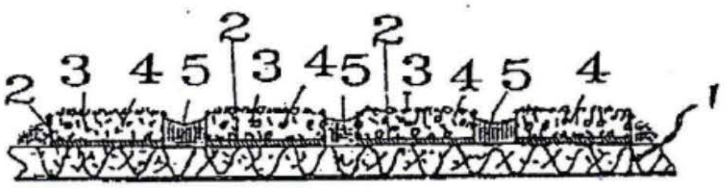

Fig. 1

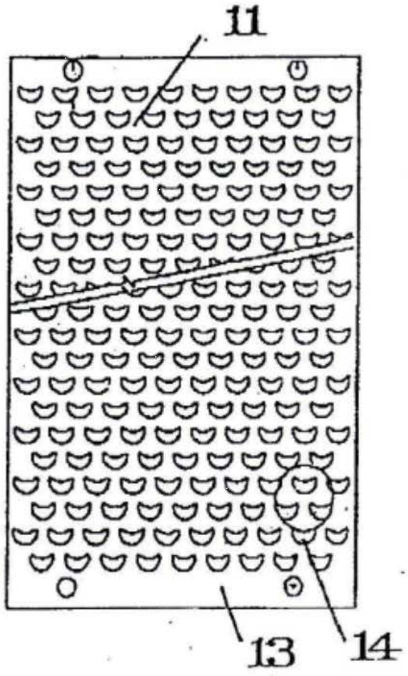

Fig. 2

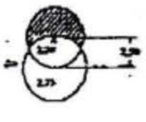

Fig. 3a

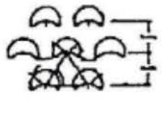

Fig. 3b

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Application No. / Patent No. 22 650 211.5 / 2 900 000	Ref. O04/3808EP	Date 03.09.2014
Proprietor 3 ABRASIVE		

Decision rejecting the opposition (Art. 101(2) EPC)

The Opposition Division - at the oral proceedings dated 11.08.2014 - has decided:

The opposition(s) against the European patent EP-B- 2900000 is/are rejected.
The reasons for the decision are enclosed.

Possibility of appeal
This decision is open to appeal. Attention is drawn to the attached text of Articles 106 to 108 and Rules 97 to 98 EPC.

Registered letter with advice of delivery

FACTS AND SUBMISSIONS

1 European patent application No. 22 650 211.5 filed on 13.04.2008 with US priority 2007 085 240, was published as European patent No. 2 900 000 which and which has the title "Flexible abrasives". Proprietor of the patent is

3 ABRASIVE, Chicago (USA).

2 An opposition was filed on 09.02.2012 by

ABRASIVE SA, Montreux (CH).

The opponent raised an objection under Article 1OO (a) EPC in combination with Article 56 EPC. His argumentation, amongst other, is based on D1 see below, which represents a prior art. Auxiliarily, the opponent requested oral proceedings.

3 With notice of opposition, the following evidence was submitted:

D1 FR 2 565 870

[…]

3. *Inventive step*

3.1 Closest prior art

The only prior art is document D1. Document D1(see Figures 5 and 6) discloses a method of forming an abrasive member, wherein a metal film 3 is fixedly attached to one surface of a non-conductive flexible sheet 2, a mask 4 of plating resistant material is applied to the exposed surface of the metal film 3,said plating resistant material having a multitude of discrete openings 5 therein, and metal 7 is electrodeposited through said discrete openings 5 onto said metal film 3 in the presence of particulate abrasive material 6 so that the particulate abrasive material 6 becomes embedded in the metal deposits 7.

3.2 Problem underlying the invention

The inventors of the patent in suit have found that in the abrasive member obtained by the method of document D1 there is a tendency for the metal deposits to chip off due to the high shearing forces applied to them.

Therefore, the problem to be solved by the present invention is to reduce the tendency of the metal deposits to chip off the substrate due to the high shearing forces applied to them in use (see page 2, lines 26 to 28 of the patent in suit).

3.3 Solution

The above-mentioned problem is solved by the method according to claim 1 of the patent in suit in that the voids between the metal deposits are filled with resin to reduce lateral movement of the metal deposits.

3.4 This solution is not rendered obvious by the document under consideration for the following reasons:

Document D1 seeks to provide a better fixation of the diamonds on the backing member. When looking at the drawings (Figures 5 and 6) of document D1 the skilled person would not recognise any tendency of the metal deposits to chip off the substrate due to the high shearing forces applied to them in use, since the person skilled in the art learns from document D1 that an additional metal layer should be provided over the whole surface of the substrate (see document D1, page 4, line 10 to 13).

Therefore, document D1 teaches that if an additional fixation of the diamonds is needed, then an additional metal layer should be applied to the surface, including the voids between the metal deposits.

Consequently, document D1 teaches away from using a resin as fixing aid by coating and filling with it the voids between the metal deposits.

In view of this teaching of document D1 the person skilled in the art would not be induced by the knowledge of resin-coated sandpapers to replace in the method known from document D1 the metal coating suggested by this document by a resin coating.

Moreover, the person skilled in the art being aware of the general knowledge in the field of size-coated sandpapers would not expect to get any suggestions in such sandpapers for solving the problem of avoiding chipping off of the specifically structured metal deposits according to document D1.

It follows from the above that the product of claim 1 of the patent in suit is not obvious to the skilled person in the light of the combination of the teaching of document D1 with the general technical knowledge in the field of the abrasive members, in particular sandpapers.

3.5 Therefore, the method of claim 1 of the patent in suit involves an inventive step within the meaning of Article 56 EPC.

⑲ RÉPUBLIQUE FRANÇAISE

INSTITUT NATIONAL
DE LA PROPRIÉTÉ INDUSTRIELLE

PARIS

⑪ **N° de publication :** **2 565 870**
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㉑ **N° d'enregistrement national :** **84 09429**

(51) **Int Cl⁴ : B 23 P 5/00; B 23 B 27/20; B 24 D 11/02.**

⑫ DEMANDE DE BREVET D'INVENTION A1

㉒ **Date de dépôt :** 15 juin 1984.

(30) **Priorité :**

(43) **Date de la mise à disposition du public de la demande :** BOPI « Brevets » n° 51 du 20 décembre 1985.

(60) **Références à d'autres documents nationaux apparentés :**

(71) **Demandeur(s) :** *Société anonyme dite : TRIEFUS FRANCE APPLICATIONS INDUSTRIELLES DU DIAMANT - STI.* — FR.

(72) **Inventeur(s) :** Alain Guyomard.

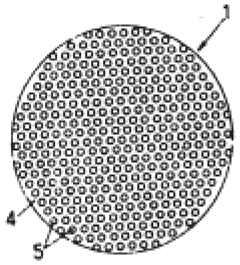
(73) **Titulaire(s) :**

(74) **Mandataire(s) :**

(54) **Procédé de fabrication d'outils diamantés sur support souple et outils en résultant.**

(57) Procédé de fabrication d'outils diamantés caractérisé par le fait que l'on fixe, de préférence par collage ou auto-adhésif, sur un outil non consommable, rigide et précis une feuille consommable diamantée 1 comportant une alternance de zones 5 diamantées séparées par des zones 4 non diamantées.

Application au polissage des métaux.



FR 2 565 870 - A1

D

Vente des fascicules à l'IMPRIMERIE NATIONALE, 27, rue de la Convention — 75732 PARIS CEDEX 15

Procédé de fabrication d'outils diamantés sur support souple et outils en résultant.

La présente invention concerne la fabrication d'outils diamantés pour usages divers, en particulier pour le polissage de laboratoire.

Pour le polissage métallographique par exemple, on utilise déjà des disques ou des plateaux recouverts de poudre de diamant, mais la concentration en diamant étant importante, celle-ci est alors un obstacle à l'évacuation de la matière résultant de l'abrasion, ainsi qu'à l'accès du liquide d'arrosage ou de lubrification s'il y a lieu. De plus la nécessité d'obtenir un échantillon parfaitement plan conduit á un coût élevé du plateau, lequel doit être usiné et rectifié avec précision avant de le recouvrir de la couche diamantée, puis celle-ci rodée après diamantage. En outre, la concentration importante de diamant contribue également au coût élevé du produit.

Le but de l'invention est d'éliminer les inconvénients précédents, c'est-à-dire de réaliser des plateaux de polissage ou autres outils diamantés qui soient beaucoup plus économiques d'emploi, tout en étant aussi précis, et qui soient plus efficaces par une meilleure évacuation de la matière résultant de l'abrasion ainsi que par un meilleur accès du liquide d'arrosage.

Ce résultat est obtenu selon l'invention en limitant la partie consommable de l'outil à une feuille diamantée mince et souple que l'on fixe par tout moyen approprié sur un plateau ou un support approprié, précis mais non consommable. D'autres part, ladite feuille diamantée n'est pas diamantée uniformément sur toute sa surface mais comporte de nombreuses zones diamantées limitées séparées par des zones non diamantées.

Plus particulièrement le procédé de fabrication de la feuille mince diamantée consiste à utiliser une feuille mince et souple d'un produit revêtu sur au moins une face par une mince couche métallique, puis à réaliser sur cette surface métallique une épargne partielle isolante, par un procédé de sérigraphie, par une résine photosensible, ou encore par le dépôt d'un film perforé auto-adhésif, afin d'isoler la surface conductrice excepté dans des zones successives qui doivent être recouvertes de diamant, à réaliser la fixation des diamants dans ces zones laissées à nu par dépôt simultané de diamants et d'un métal, par exemple du nickel, par voie chimique puis à éliminer l'épargne isolante. On réalise de préférence dans une première phase la fixation primaire de diamants par dépôt chimique de nickel, puis dans une deuxième phase on renforce cette fixation des diamants par un dépôt secondaire plus épais de nickel chimique, permettant d'obtenir une meilleure planéité de la-couche diamantée. Selon les cas on peut travailler avec deux bains successifs, par exemple deux bains chimiques, mais on peut également mettre en œuvre le procédé avec un seul bain avec des diamants en suspension, dans une solution pour dépôt chimique. Après les opérations de dépôt, on retire la couche d'épargne pour obtenir le produit final.

D'autres particularité de l'invention apparaîtront dans la description qui va suivre d'un mode de réalisation et de mise en œuvre pris comme exemple et représenté sur la dessin annexé, sur lequel :

- la figure 1 représente un disque de support souple avant traitement;
- la figure 2 ce même disque après application de l'épargne;
- la figure 3 est une coupe fragmentaire ä grande échelle de la figure 1;
- la figure 4 une coupe similaire de la figure 2;
- la figure 5 une coupe correspondante après la première phase de fixation des diamants, et
- la figure 6 une coupe correspondante après la deuxième phase de fixation et avant élimination de l'épargne.

Selon 1'invention on part d'une feuille mince 1 qui peut avoir la forme d'un disque, comme dans l'exemple représenté sur la figure 1, ou toute autre forme rectangulaire ou en bande, selon l'utilisation. Au moins la face de cette feuille destinée ä recevoir les diamants doit être métallique pour l'application du dépôt chimique. Pour cela la feuille mince 1 pourrait être métallique dans son ensemble et isolée sur sa face arrière par une couche de protection, mais pour une raison d'économie, de souplesse et de facilité de collage, il est préférable d'utiliser un support plastique souple, représenté par 2 sur la figure 3, recouvert d'une mince couche de métal 3.

Le support plastique mince 2 peut être une feuille de matière thermo—plastique, ou thermodurcissable, ou encore stratifiée, et être recouverte d'une mince couche métallique, par exemple de cuivre, comme on en utilise pour la fabrication des circuits imprimés.

Conformément à l'invention, après nettoyage, dégraissage et activation de cette surface 3, on réalise sur celle-ci une épargne isolante 4 qui isole cette surface, en laissant toutefois subsister un grand nombre d'orifices 5 dans lesquels le métal, par exemple le cuivre, reste à nu. Le tracé le plus simple est celui représenté sur la figure 2, dans lequel la surface couverte 4 est continue et les orifices 5 sont de nombreux trous circulaires, plus petits et plus nombreux qu'il n'apparaît sur le dessin. Toutefois une infinité d'autres motifs géométriques seraient utilisables, pourvu que conformément á l'invention on ait une certaine alternance de zones à nu, destinées à être recouvertes de diamants, et de zones épargnées 4 qui ne seront pas recouvertes.

Cette épargne peut être réalisée par sérigraphie, avec une peinture ou un vernis isolant. Elle peut aussi être réalisée à l'aide d'une résine photosensible (photorésist), positive ou négative, traitée par insolation à travers un masque représentant le motif voulu, puis dissoute sélectivement. Enfin elle peut également être réalisée à l'aide d'un film isolant perforé auto-adhésif ou thermocollant.

Dans la suite du procédé, on place le disque 1, ainsi recouvert de son épargne, et éventuellement à nouveau activé et désoxydé dans un bain chimique. En outre, conformément à l'invention, ce bain comprend en suspension un grand nombre de diamants microscopiques 6.

On réalise ainsi un dépôt chimique de métal, généralement du nickel, lequel comme illustré sur la figure 5, se dépose essentiellement dans les zones 7 situées entre la couche 3 et les diamants 6 qui se sont déposés dans les orifices 5, assurant ainsi la fixation primaire de ces diamants 6 dans ces zones.

Enfin on opère de préférence dans une deuxième phase une fixation secondaire des diamants 6 en immergeant la feuille précédente dans un bain pour dépôt chimique permettant le dépôt d'une couche régulière de nickel, représentée en 8 sur la figure 6, sur les plots diamantés afin d'assurer un véritable sertissage des diamants 6.

Tant que l'épargne 4 est restée en place ce dépôt de sertissage 8 n'a lieu que sur les plots diamantés. L'épargne est ensuite éliminée pour obtenir le produit final. Il est également possible à titre de variante de retirer l'épargne 4 à l'aide d'un solvant approprié après l'exécution de 1a première phase pour que la seconde phase produise un dépôt de nickel sur toute la surface du support 1.

Naturellement si on le désire on peut réaliser un plus grand nombre de phases de fixation afin de déposer plusieurs couches superposées de diamants. Enfin il est parfaitement possible d'appliquer le procédé à l'aide d'un bain unique dans lequel les deux phases se succèdent. Ce bain unique contient alors du diamant en suspension dans une solution pour dépôt chimique. Dans ce dernier cas on obtient une multicouche de métal et de diamants dont l'épaisseur varie seulement avec le temps d'immersion.

Dans tous les cas on obtient un disque souple diamanté qui peut être fixé sur le support rigide et précis d'entraînement par simple collage, par exemple à l'aide d'un enduit auto-adhésif ou d'un film adhésif double face. De cette manière, le support ainsi fabriqué constitue la seule partie consommable de l'outil qui, après usure de sa couche diamantée, peut être restauré très rapidement et économiquement en décollant la couche usée et en la remplaçant par une couche neuve, sans aucun usinage ni rodage.

Par ailleurs, l'outil ainsi constitué comporte des zones diamantées séparées par des passages libres qui permettent une très bonne évacuation des particules de matière résultant de l'abrasion ainsi que du liquide d'arrosage, ce qui donne une très grande longévité à chaque support consommable, un bon mordant et une excellente planéité qui est celle de l'outil de base grâce à la souplesse du support.

REVENDICATIONS

1. Procédé de fabrication d'outils diamantés caractérisé par le fait que l'on fixe, de préférence par collage ou auto-adhésif, sur un outil non consommable, rigide et précis une feuille consommable diamantée (1) comportant une alternance de zones (5) diamantées séparées par des zones (4) non diamantées.

2. Procédé selon la revendication 1, caractérisé par le fait que l'on réalise la feuille souple diamantée en partant d'une feuille souple (1) ayant au moins une face (3) métallique, que l'on réalise sur cette face une épargne isolante (4) ménageant des zones successives (5) dans lesquelles le métal (3) reste à nu, puis que l'on réalise dans ces zones un dépôt chimique d'un métal, de préférence du nickel, à l'aide d'un bain de dépôt chimique contenant des diamants en suspension.

3. Procédé selon la revendication 2, caractérisé par le fait que le dépôt de métal a lieu au cours de deux phases successives, l'une, au cours de laquelle du métal (7) est déposé principalement entre les diamants (6) et la couche métallique(3), de manière à assurer l'accrochage primaire des diamants (6), et une deuxième phase, réalisée par voie chimique, au cours de laquelle une couche plus importante (8) de métal recouvre au moins partiellement les diamants (6) de manière à assurer leur sertissage.

4. Procédé selon l'une des revendications précédentes, caractérisé par le fait que le support souple 1 utilisé est constitué par un isolant mince (2) recouvert de cuivre (3), du type utilisé pour la fabrication des circuits imprimés.

5. Procédé selon une des revendications 2 à 4, caractérisé par le fait que l'épargne (4) est réalisée pat sérigraphie avec une peinture ou vernis isolant.

6. Procédé selon une des revendications 2 à 4, caractérisé par le fait que l'épargne est réalisée à l'aide d'une résine photosensible positive ou négative traitée par insolation à travers un masque, puis dissoute sélectivement.

7. Procédé selon une des revendications 2 à 4, caractérisé par le fait que l'épargne est réalisée à l'aide d'un film isolant perforé auto-adhésif ou thermocollant.

8. Procédé selon la revendication 3, caractérisé par le fait que les deux phases ont lieu simultanément dans un bain unique, de dépôt chimique.

9. Procédé selon une des revendications 3 à 8, caractérisé par le fait que l'opération de dépôt métallique de sertissage (8) a lieu uniquement sur les surfaces diamantées.

10. Procédé selon l'une des revendications 3 à 7, caractérisé par le fait que l'on retire l'épargne (4) à l'aide d'un solvant approprié entre la première et la deuxième phase, et que le dépôt métallique de sertissage (8) a lieu sur toute la surface du support souple (1).

11. Procédé selon une des revendications précédentes, caractérisé par le fait que l'on réalise successivement plusieurs couches superposées de diamants (6) par répétition ou prolongation du procédé.

12. Support diamanté consommable résultant de la mise en œuvre du procédé selon une des revendications précédentes et caractérisé principalement par la présence de zones diamantées (5) séparées par des zones non diamantées (4) sur un support souple (1).

FIG.1

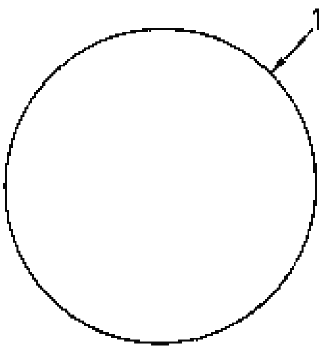


FIG.2

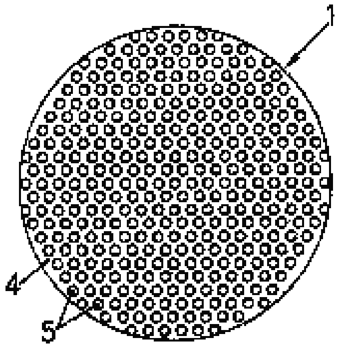


FIG.3

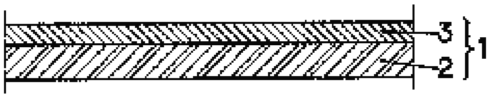


FIG.4

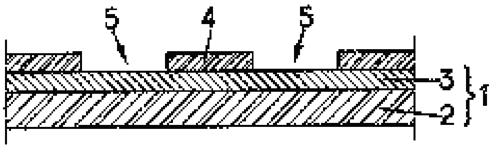


FIG.5

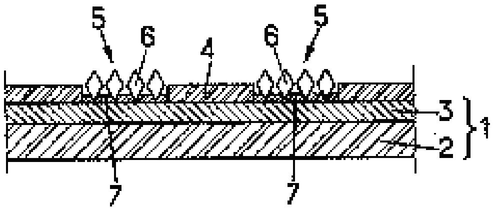
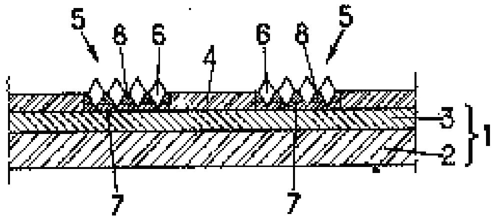


FIG.6



FR2565870

TRANSLATION

Manufacturing process of diamond tools on a flexible support and tools resulting, thereof

The present invention relates to the manufacture of diamond tools for various uses, particularly for laboratory polishing.

For metallographic polishing for example, discs or trays covered with diamond powder are already used, but the diamond concentration being significant, it is then an obstacle to the removal of the material resulting from abrasion, and to the irrigation or lubrication liquid if any. Also the need for a perfectly flat sample leads to a high cost of the plate, which must be machined and precision ground before covering it with the diamond layer, and it must be lapped after diamond polishing. In addition, the high concentration of diamond also contributes to the high cost of the product.

The object of the invention is to eliminate the above drawbacks, that is to say, to make polishing plates or other diamond tools that are much more economical to use while being as accurate, and that are more effective through better evacuation of the material resulting from abrasion as well as better access of coolant.

This is achieved according to the invention by limiting the consumable portion of the tool to a thin and flexible diamond sheet which is fixed by any suitable means on a plate or a suitable support, but not consumable. On the other hand, said diamond sheet is not uniformly covered with diamond over its entire surface but includes many diamond limited areas separated by non-diamond areas.

More particularly the method of manufacturing the diamond thin film is to use a thin and flexible sheet of a product on at least one side by a thin metallic layer, and to perform on this covered metallic surface an insulating partial savings, by a screen printing process, by a photosensitive resin, or by depositing a self-adhesive perforated film, to isolate the conductive surface except in successive areas to be covered with diamond, to realize the attachment of diamonds in these areas left exposed by simultaneous deposition of diamond and a metal, for example nickel, by chemical means and finally to eliminate the insulating savings. The process comprises preferably, a first stage where the primary attachment of diamonds is made by chemical deposition of nickel, then in a second phase the fixation of the diamonds is reinforced with a thicker secondary layer of chemical nickel, to obtain a better flatness of the diamond layer. As the case may be working with two successive baths, for example, two electroplating baths, but it is also possible to implement the process with a single bath with diamonds in suspension in a solution for electroless plating. After the deposit operations, the savings layer is eliminated to arrive at the final product.

Other features of the invention will become apparent from the following description of an embodiment and implementation as an example and shown in the accompanying drawing, in which

Figure 1 shows a flexible support disk before processing;

Figure 2 that same disk after application of savings;

Figure 3 is a fragmentary section on a large scale of Figure 1;

Figure 4 a section similar to Figure 2;

Figure 5 a corresponding section after the first diamond attachment phase, and

Figure 6 a corresponding section after the second fixing stage and before elimination of the savings.

According to the invention one starts with a thin sheet 1 which may have the shape of a disk, as in the example shown in Figure 1 or any other rectangular or strip, depending on use. At least the face of this sheet for receiving the diamonds must be metallic for the application of chemical deposition. For this the thin sheet metal 1 could be metallic as a whole and isolated on its back by a protective layer, but for reasons of economy, flexibility and ease of bonding, it is preferable to use a flexible plastic support represented by 2 in Figure 3, covered by a thin metal layer 3.

The thin plastic support 2 may be a sheet of thermoplastic material, or thermoset, or laminated, and be covered with a thin metallic layer, for example of copper, such as those used for the manufacture of printed circuits.

According to the invention, after cleaning, degreasing and activating this surface 3 an insulating savings 4 is applied which insulates the surface, but leaving a large number of apertures 5 in which the metal, for example copper, remains bare. The simplest design is the one shown in Figure 2, wherein the covered area 4 is continuous and the openings 5 are many circular holes, smaller and more numerous than shown on the drawing. However an infinity of other geometric patterns would be usable, provided that in accordance with the invention there is some alternating exposed areas, to be coated with diamond, and spared areas 4 that are not covered.

These savings can be achieved by screen printing, with paint or varnish insulation. It can also be performed using a photosensitive resin (photoresist), positive or negative, processed by irradiation through a mask representing the desired pattern, and then selectively dissolved. Finally it can also be carried out using a perforated self-adhesive or thermo-adhesive insulating film.

In the remaining part of the method, the disk 1 is placed, covered with its savings, and optionally activated again and deoxidized in a chemical bath. Furthermore, in accordance with the invention, the bath includes in suspension a large number of microscopic diamonds 6.

Metal, usually nickel, is thus deposited chemically as illustrated in Figure 5, and is mainly deposited in the regions 7 situated between the layer 3 and the diamonds 6 which were deposited in the holes 5, thus ensuring the primary fixing of said diamonds 6 in these areas.

Finally, in a second stage, a secondary fixing of the diamonds 6 is performed by immersing the previous sheet in a bath for chemical plating allowing deposition of a uniform layer of nickel, shown at 8 in Figure 6,onto the diamond studs to ensure a real crimp of the diamonds 6.

As long as the savings 4 remain in place, this crimping deposition takes place only on the diamond studs. The savings is then eliminated to arrive at the final product. It is also possible alternatively to withdraw the savings 4 with a suitable solvent after completion of the first phase so that the second phase can produce a nickel plating over the entire surface of the support 1.

Of course if desired one can achieve a greater number of securing steps to deposit several superimposed layers of diamonds. Finally, it is perfectly possible to apply the method using a single bath in which the two phases proceed successively. This unique bath then contains diamond suspended in a solution for chemical plating. In the latter case a multilayer of metal and diamonds is obtained whose thickness varies only with the time of immersion.

In all cases one obtains a flexible diamond disc which can be fixed to the rigid and precise drive bracket by simple bonding, for example by using a self-adhesive coating or a double-sided adhesive film. In this way, the thus produced carrier is the only consumable part of the tool that, after wear of its diamond coating, can be restored very quickly and economically by peeling the waste layer and substituting it with a new layer without any machining or grinding.

Moreover, the thus formed diamond tool comprises zones separated by free passageways which allow a very good discharge of the particulate matter resulting from the abrasion and the coolant, which gives a high durability to each consumable support, good bite and excellent flatness which is that of the basic tool thanks to the flexibility of the support.

CLAIMS

1.

A method of manufacturing diamond tools characterized by the fact that one secures preferably by adhesive or self-adhesive on a non-consumable, rigid and accurate tool diamond consumable sheet (1) having alternating areas (5) of diamond separated by regions (4) without diamond.

2.

A method according to claim 1, characterized by the fact that the flexible diamond sheet is made by starting from a flexible sheet having at least one metallic face (3) that one makes on this face an insulating savings (4) providing successive zones (5) in which the metal (3) remains exposed, and one provides then in these zones a chemical deposition of a metal, preferably nickel by using a chemical bath containing diamonds in suspension.

3.

A method according to claim 2, characterized in that the metal deposition takes place in two successive stages, one in which metal (7) is deposited mainly between the diamonds (6) and the metallic layer (3), so as to ensure the primary attaching of diamonds (6), and a second phase, performed by chemical means, in which a more important layer (8) of metal covers at least partially the diamonds (6) so as to ensure their crimping.

4.

Method according to one of the preceding claims, characterized in that the flexible support 1 used consists of a thin insulating layer (2) covered with copper (3), of the type used for the manufacture of printed circuits.

5.

Method according to one of claims 2 to 4, characterized in that the savings 4 is made by screen printing with an insulating paint or varnish.

6.

Method according to one of claims 2 to 4, characterized by the fact that the savings is performed using a positive or negative photosensitive resin treated by irradiation through a mask, then selectively dissolved.

7.

Method according to one of claims 2 to 4, characterized by the fact that the savings is performed using a perforated self-adhesive or thermo-adhesive insulating film.

8.

A method according to claim 3, characterized in that the two phases occur simultaneously in a single chemical bath.

9.

Method according to one of claims 3 to 8, characterized in that the operation of metallic crimping deposit (8) takes place only on the diamond surfaces.

10.

Method according to one of claims 3 to 7, characterized in that one removes the savings (4) using an appropriate solvent between the first and the second phase, and that the crimping metal deposit (8) takes place over the entire surface of the flexible support (1).

11.

6 Method according to one of the preceding claims, characterized in that one successively performs several layers of diamonds (6) by extension or repetition of the process.

12.

Consumable diamond carrier resulting from the implementation of the method according to one of the preceding claims and characterized mainly by the presence of diamond regions (5) separated by non-diamond areas (4) on a flexible support (1). 12

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La surface abrasive est formée d'une multitude de plots métalliques à surface diamantée. La fixation des plots sur le support de la bande est améliorée par rapport aux bandes abrasives classiques grâce à l'existence d'une couche de cuivre préalablement formée sur le support.
Les plots abrasifs sont noyés dans une couche de non-tissé comprenant des fibres longues de polyamide. L'usure est réduite grâce à l'excellent maintien latéral des plots métalliques abrasifs par la couche de non-tissé.

Prix de lancement envisagé: 5 € le m

Nos produits

Livraison 48h

Notre réseau

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Loose translation of La Toilemeri SA.’s website

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Latest news :

Our laboratories have developed a new revolutionary abrasive tape which abrasive and wear-resistant qualities are outstanding.

The abrasive surface is made of multiple metallic patches with a diamante surface. The fixation of the patches on the tape’s support is improved compared to classic abrasive tapes. Thanks to the existence of a copper layer previously formed on the support.
The abrasive patches are embedded in non woven sheet comprising long fibers of polyamide. Wear is reduced thanks to the excellent lateral bearing of the metallic abrasive patches by the non woven sheet.

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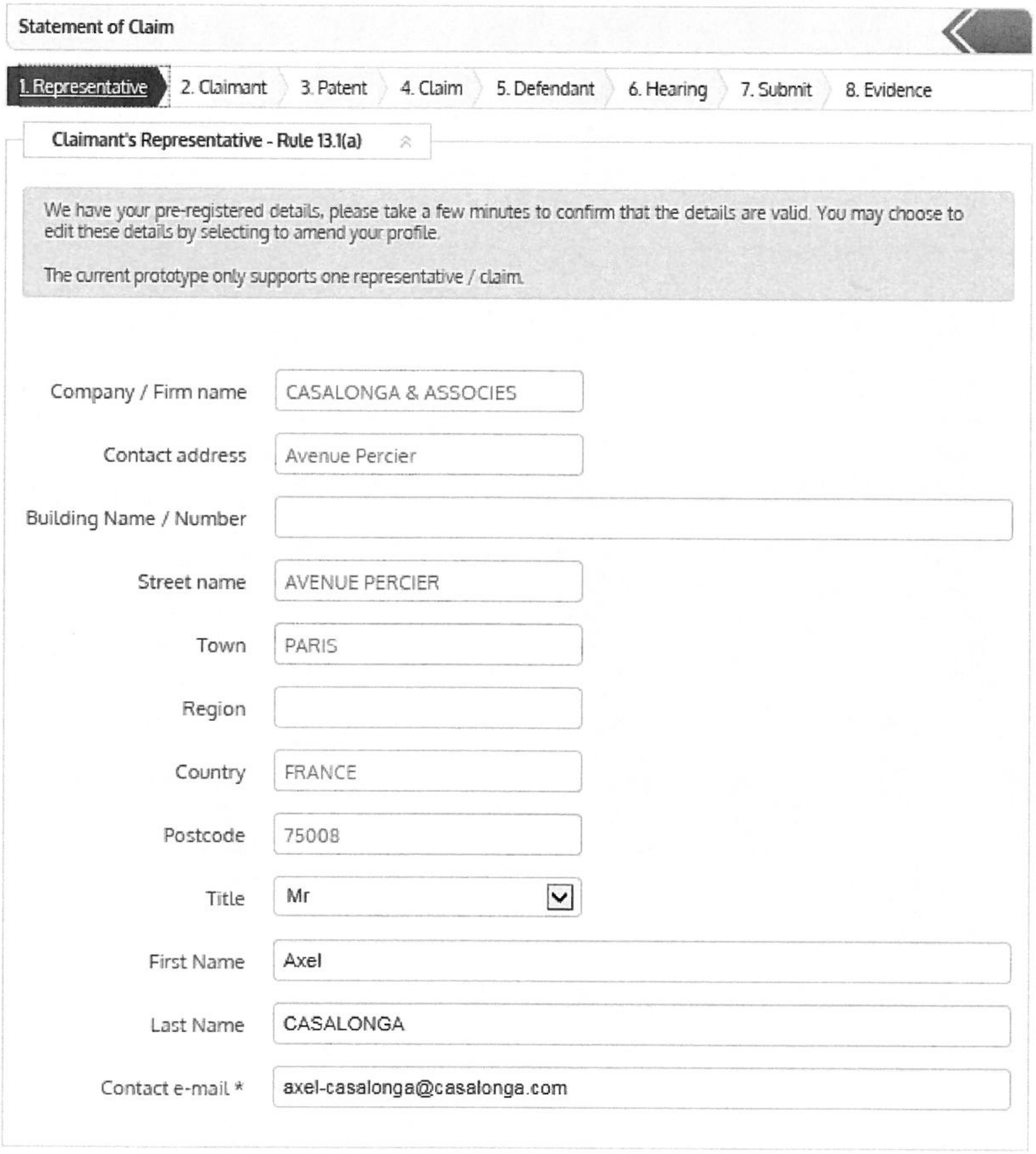
Draft Form of Statement of Claim

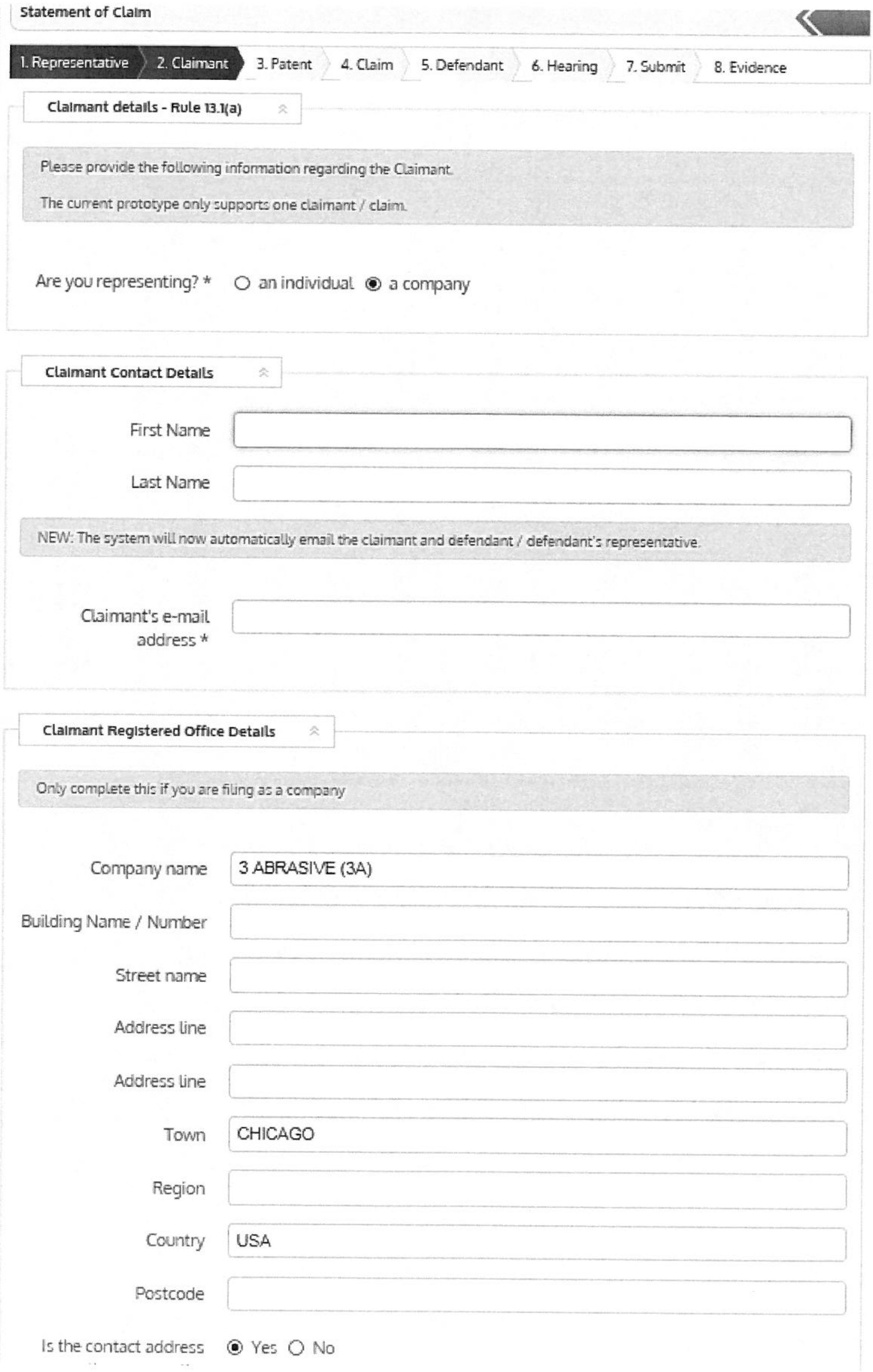


Claimant's Representative - Rule 13.1(a)

We have your pre-registered details, please take a few minutes to confirm that the details are valid. You may choose to edit these details by selecting to amend your profile.

The current prototype only supports one representative / claim.

Company / Firm name	CASALONGA & ASSOCIES
Contact address	Avenue Percier
Building Name / Number	
Street name	AVENUE PERCIER
Town	PARIS
Region	
Country	FRANCE
Postcode	75008
Title	Mr
First Name	Axel
Last Name	CASALONGA
Contact e-mail *	axel-casalonga@casalonga.com



Claimant details - Rule 13.1(a)

Please provide the following information regarding the Claimant.

The current prototype only supports one claimant / claim.

Are you representing? * ○ an individual ● a company

Claimant Contact Details

First Name

Last Name

NEW: The system will now automatically email the claimant and defendant / defendant's representative.

Claimant's e-mail address *

Claimant Registered Office Details

Only complete this if you are filing as a company

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Building Name / Number	
Street name	
Address line	
Address line	
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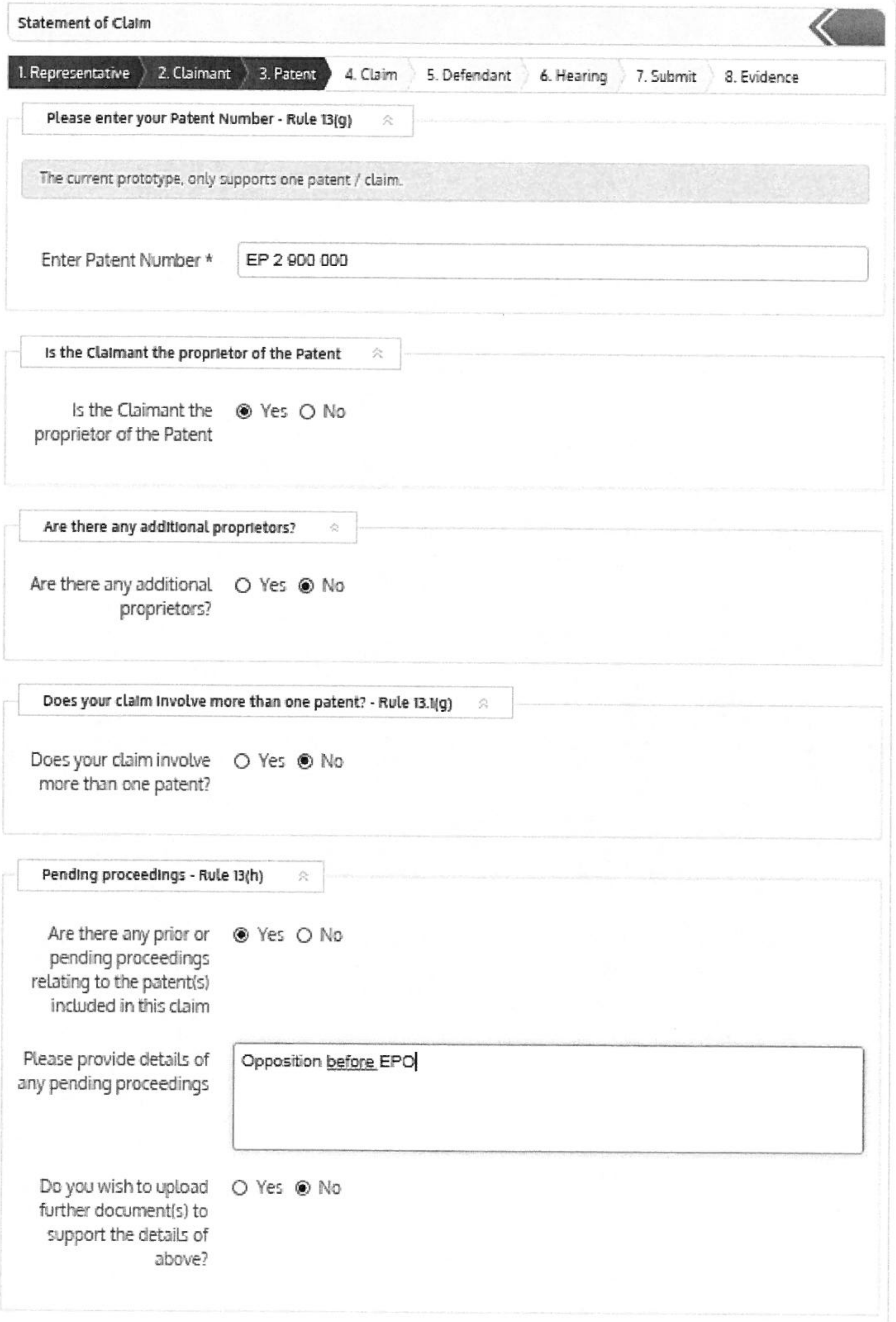
Is the contact address ● Yes ○ No



Statement of Claim

Please enter your Patent Number - Rule 13(g)

The current prototype, only supports one patent / claim.

Enter Patent Number * EP 2 900 000

Is the Claimant the proprietor of the Patent

Is the Claimant the proprietor of the Patent ● Yes ○ No

Are there any additional proprietors?

Are there any additional proprietors? ○ Yes ● No

Does your claim involve more than one patent? - Rule 13.1(g)

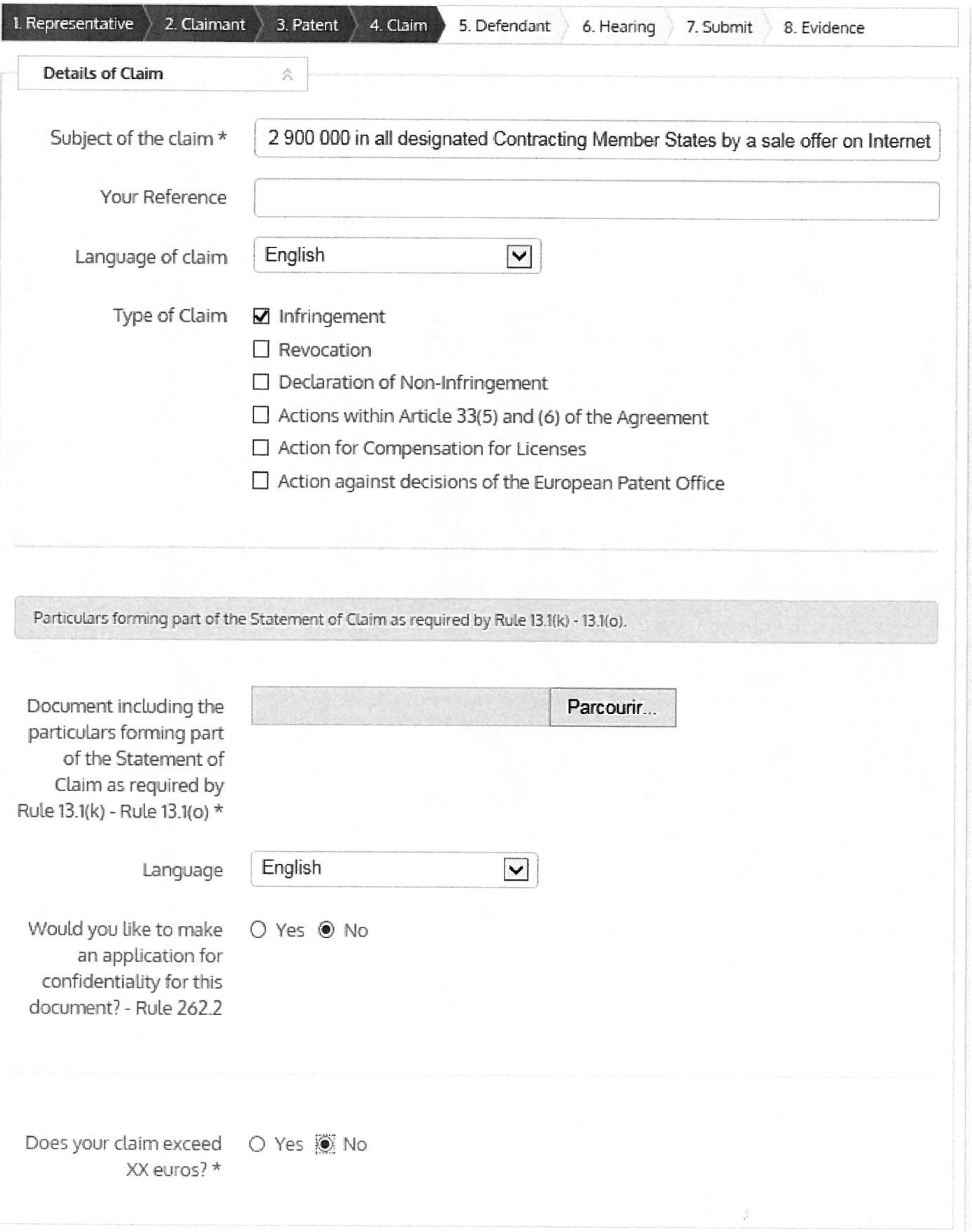
Does your claim involve more than one patent? ○ Yes ● No

Pending proceedings - Rule 13(h)

Are there any prior or pending proceedings relating to the patent(s) included in this claim ● Yes ○ No

Please provide details of any pending proceedings: Opposition before EPO

Do you wish to upload further document(s) to support the details of above? ○ Yes ● No



Details of Claim

Subject of the claim * 2 900 000 in all designated Contracting Member States by a sale offer on Internet

Your Reference

Language of claim English

Type of Claim
- ☑ Infringement
- ☐ Revocation
- ☐ Declaration of Non-Infringement
- ☐ Actions within Article 33(5) and (6) of the Agreement
- ☐ Action for Compensation for Licenses
- ☐ Action against decisions of the European Patent Office

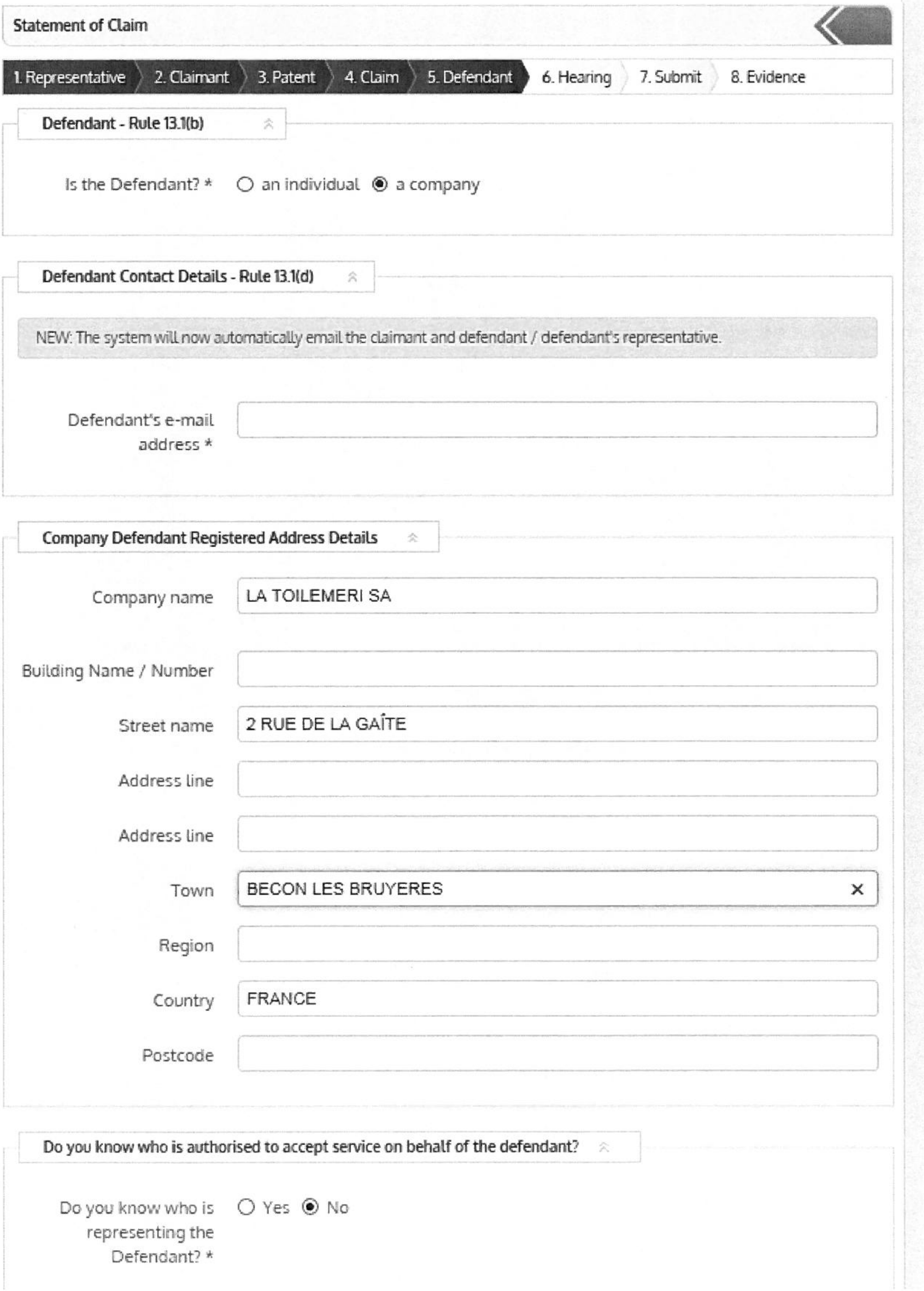
Particulars forming part of the Statement of Claim as required by Rule 13.1(k) - 13.1(o).

Document including the particulars forming part of the Statement of Claim as required by Rule 13.1(k) - Rule 13.1(o) *

Language English

Would you like to make an application for confidentiality for this document? - Rule 262.2 ○ Yes ● No

Does your claim exceed XX euros? * ○ Yes ● No



Statement of Claim

Defendant - Rule 13.1(b)

Is the Defendant? * ○ an individual ● a company

Defendant Contact Details - Rule 13.1(d)

NEW: The system will now automatically email the claimant and defendant / defendant's representative.

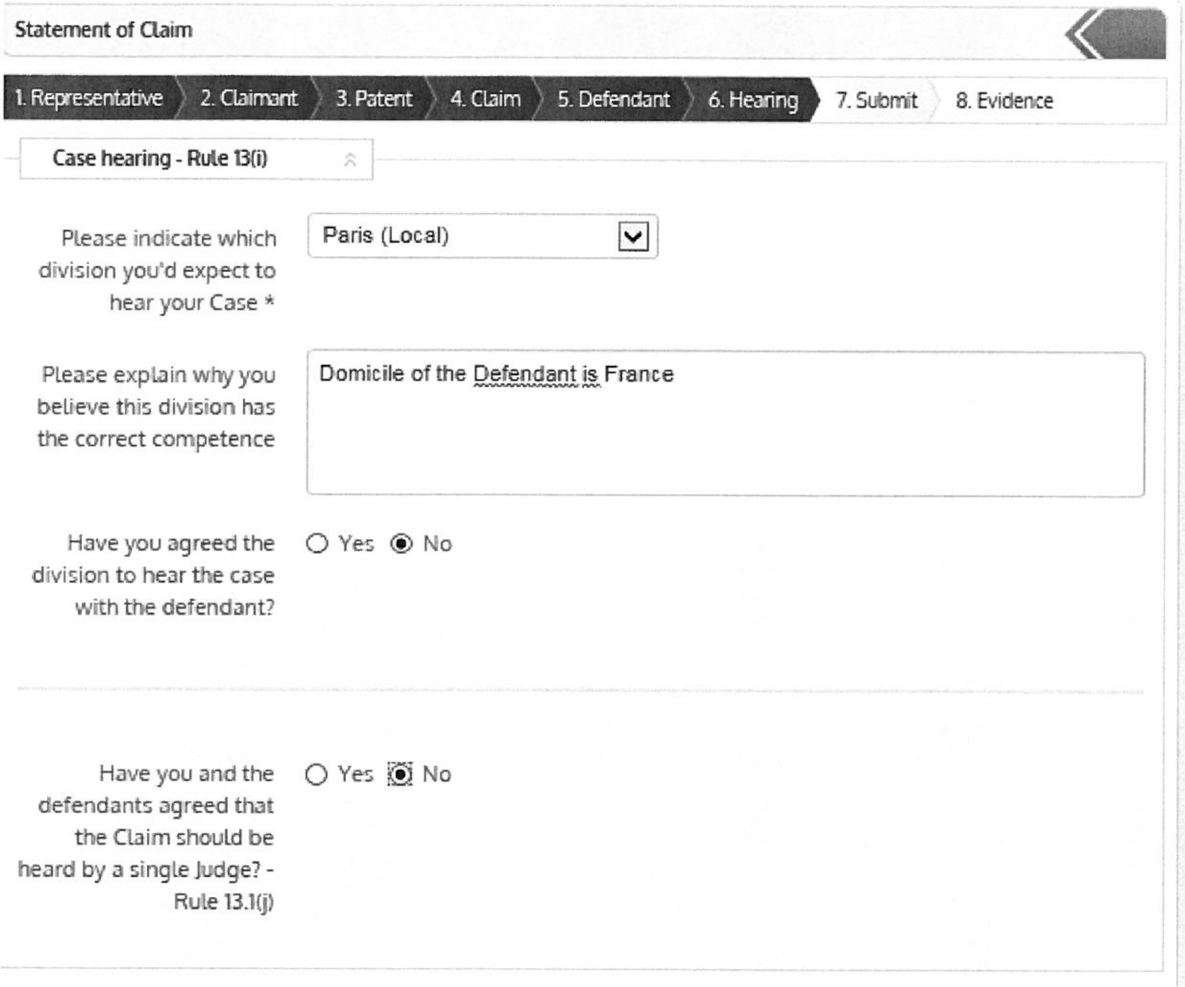
Defendant's e-mail address *

Company Defendant Registered Address Details

Company name	LA TOILEMERI SA
Building Name / Number	
Street name	2 RUE DE LA GAÎTE
Address line	
Address line	
Town	BECON LES BRUYERES
Region	
Country	FRANCE
Postcode	

Do you know who is authorised to accept service on behalf of the defendant?

Do you know who is representing the Defendant? * ○ Yes ● No

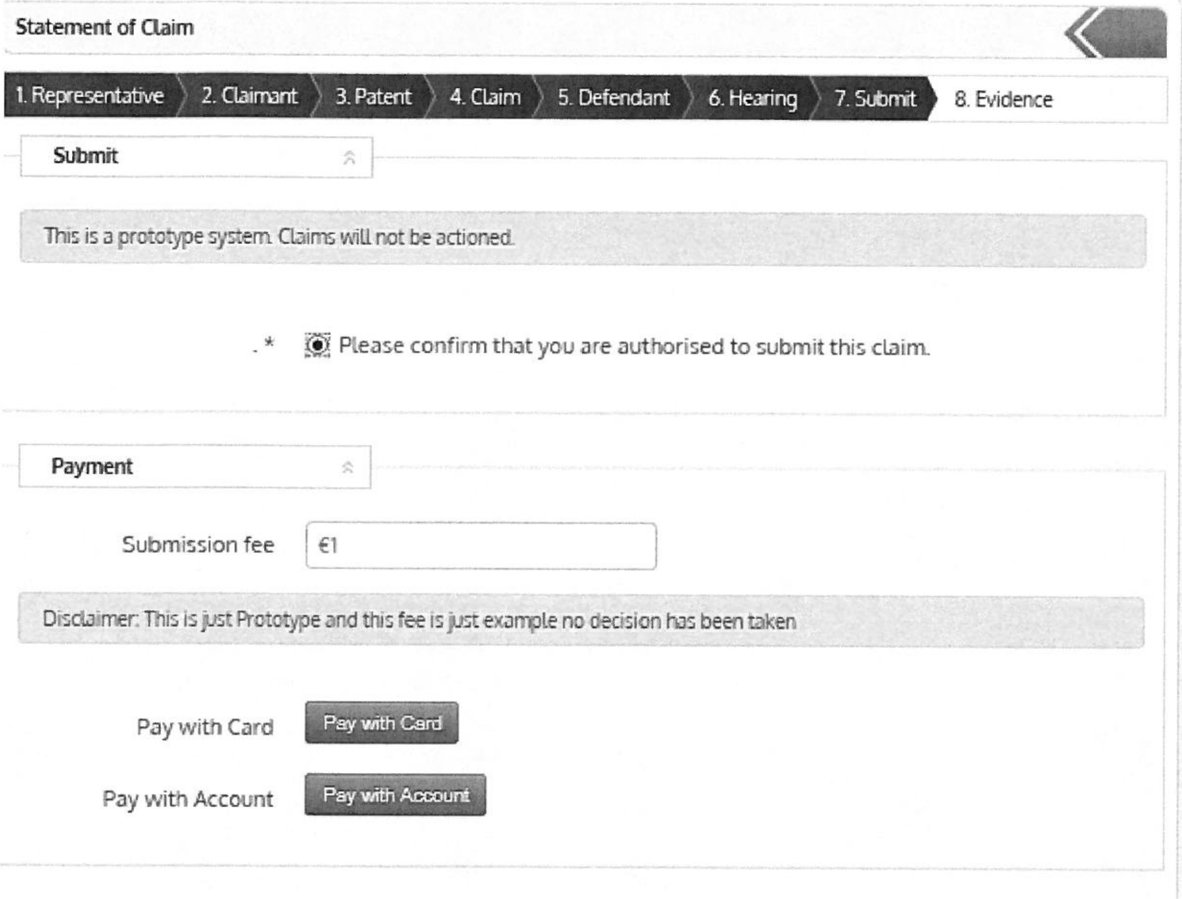


Please indicate which division you'd expect to hear your Case * Paris (Local)

Please explain why you believe this division has the correct competence: Domicile of the Defendant is France

Have you agreed the division to hear the case with the defendant? ○ Yes ● No

Have you and the defendants agreed that the Claim should be heard by a single Judge? - Rule 13.1(j) ○ Yes ● No



Statement of Claim

Submit

This is a prototype system. Claims will not be actioned.

. * ● Please confirm that you are authorised to submit this claim.

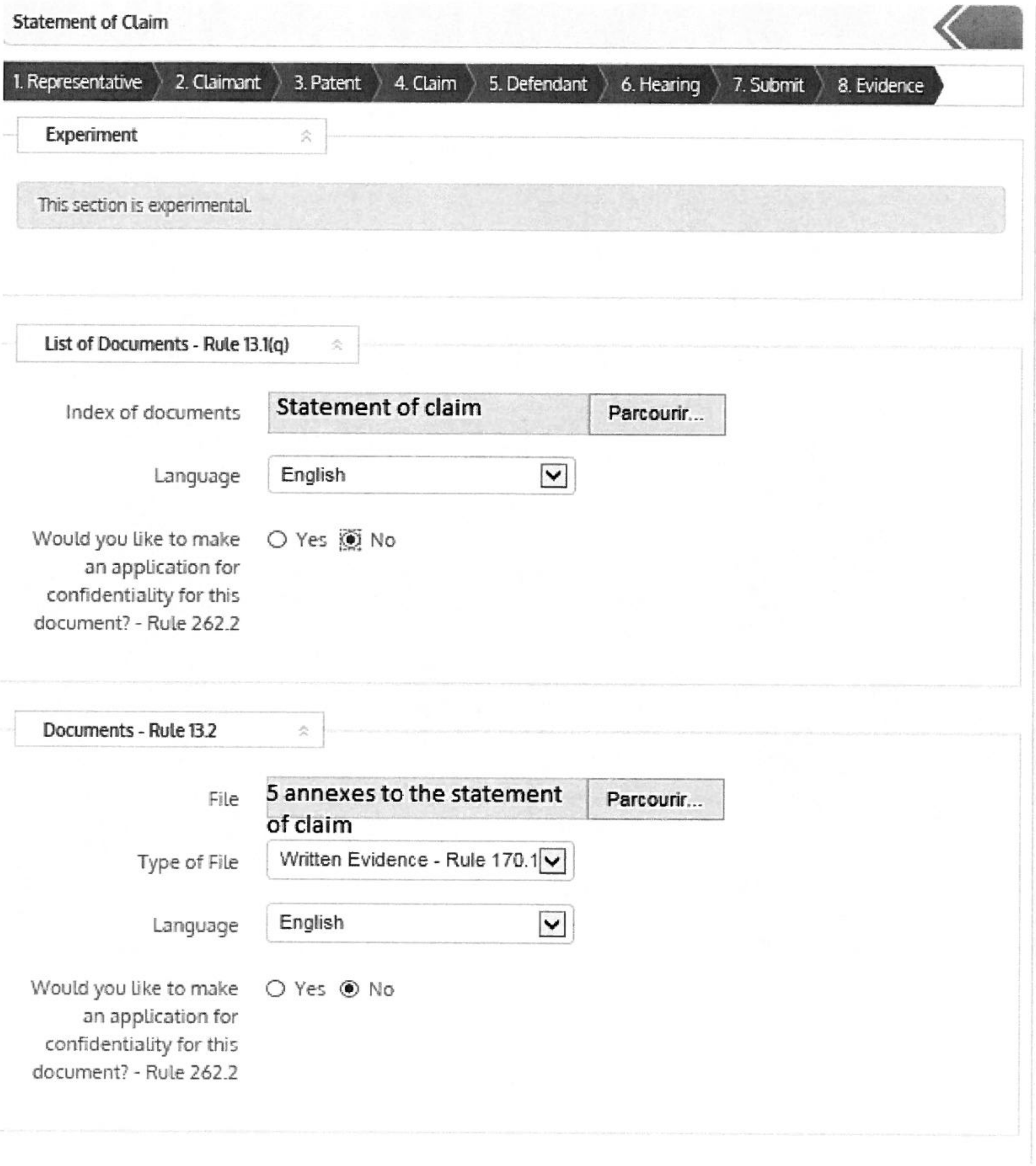
Payment

Submission fee: €1

Disclaimer: This is just Prototype and this fee is just example no decision has been taken

Pay with Card

Pay with Account



This section is experimental.

List of Documents - Rule 13.1(q)

Index of documents: Statement of claim

Language: English

Would you like to make an application for confidentiality for this document? - Rule 262.2 ○ Yes ● No

Documents - Rule 13.2

File: 5 annexes to the statement of claim

Type of File: Written Evidence - Rule 170.1

Language: English

Would you like to make an application for confidentiality for this document? - Rule 262.2 ○ Yes ● No

October 13th, 2014

Docket No 2015/01
UNIFIED PATENT COURT
PARIS LOCAL DIVISION

3 ABRASIVE (3A)

(A company with its registered office located in the USA)

v La Toilemeri SA

(A company with its registered office located in France)

CLAIMANT'S STATEMENT OF CLAIM

FOR INFRINGEMENT OF EP No. 2900000

Claimant's Address for Service:

Mr. Axel Casalonga, Partner of CASALONGA & Associés, is authorized to accept service in relation to these proceedings.

8, avenue Percier
75008 PARIS
France

a.casalonga@casalonga.com

Mr. Martin Köhler is authorized to accept service in relation to these proceedings.

martin.koehler@rokh-ip.com

Defendant's Address for Service:

[Persons authorized to accept service]

[Postal address]

[Email address]

1. Competence of the local division of the Court of First Instance

1- The defendant has his residence and principal place of business in France.

Therefore, article 33.1 b) of the Agreement on a Unified Patent Court allows the claim to be brought before the local division of the Court of Instance in Paris.

2. The Claimant and its Patent

2- The Claimant is a company registered under the laws of the USA with its registered office at Chicago USA.

3- The Claimant is the proprietor of EP no. 2900000 entitled « *Flexible abrasives* » which claims priority from US patent application no. 2007085240 ("the Patent").

(Annex 1)

4- The Patent is valid and in force in the Contracting States designated by the Patent, including France, Germany, The Netherlands, United Kingdom and Sweden. All the Designated Contracting States of the Patent have ratified and implemented the UPC Agreement.

5- The Claimant exports, through a distribution network, products made according to the Patent throughout Europe, including France, Germany, The Netherlands and Sweden. Abrasive belts for polishing hard stones and manufactured according to the patented method are being sold all over Europe since more than three years. The belts are sold at a price of 8 Euros for one meter. Over the last three years the average yearly benefit resulting from this distribution network in the above mentioned European countries was of 100 000 Euros.

(Annex 4)

6- The Patent was the subject of opposition proceedings in the European Patent Office.

However, in its decision dated 3 September 2014, the opposition division rejected the opposition and decided that the Patent be maintained as granted.

(Annex 2)

3. Claims of the Patent

8- The Patent relates to flexible abrasives. It was granted in English and discloses a method of forming an abrasive member with a step of electro deposition on a metal film as well as an abrasive product.

9- The abrasive product has great advantages over the previously known abrasives.

10- The flexible support has namely a plurality of metallic abrasive patches which are particularly strongly secured to the surface of the support thanks to the specific manufacturing process including an electrodeposition step of a second metal on a first metal.

11- In addition, the patches are perfectly maintained against any lateral movement, thanks to a resin layer embedding the metal patches.

12- The independent claim defines the manufacturing method of the abrasive product as follows:

A method for manufacturing an abrasive member comprising a flexible sheet (1) with a multitude of discrete metal protuberances (2,3) wherein :

C1 a multitude of copper protuberances (2) are formed on the flexible sheet (1),

C2 nickel protuberances (3) are electrodeposited over the copper protuberances (2) in the presence of particulate abrasive material (4) so that the particulate abrasive material becomes embedded in the nickel deposits

C3 and wherein the voids between the protuberances (2,3) are at least partially filled with resin material

C4 the resin material being selected so as to reduce lateral movement of the nickel deposits (3).

The resulting abrasive product is illustrated on figure 1 of the Patent:

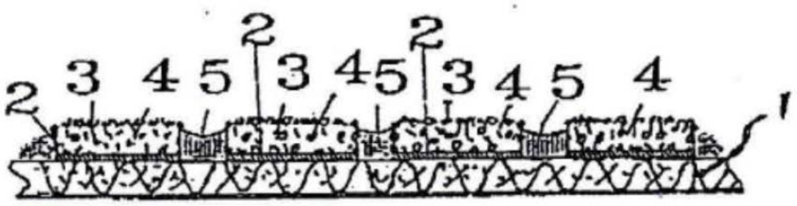


Fig. 1

4. The Defendant's acts of infringement

13- The Defendant has a commercial activity in the field of abrasive products for industrial uses.

14- The Defendant manufactures in France and sells in Europe a variety of abrasive products.

15- On 25 August 2014, the Defendant published on its Internet website an announcement concerning an allegedly new abrasive product.

On this web site, the defendant posted a photograph of this product as well as marketing statements indicating that the advantages of the product were:

- an excellent lateral bearing of the abrasive patches,
- a reinforced securing of the abrasive patches onto the support.

In addition, the website mentioned that the abrasive patches are embedded in a non woven sheet comprising long fibers of polyamide.

The price of product was also indicated at 5 € per meter.

16- The Claimant asked a bailiff to issue a report on this website in order to preserve the facts mentioned above. The report includes a copy of a page of this website showing a photograph of a new product for which a set of advantages are mentioned.

(Annex 3)

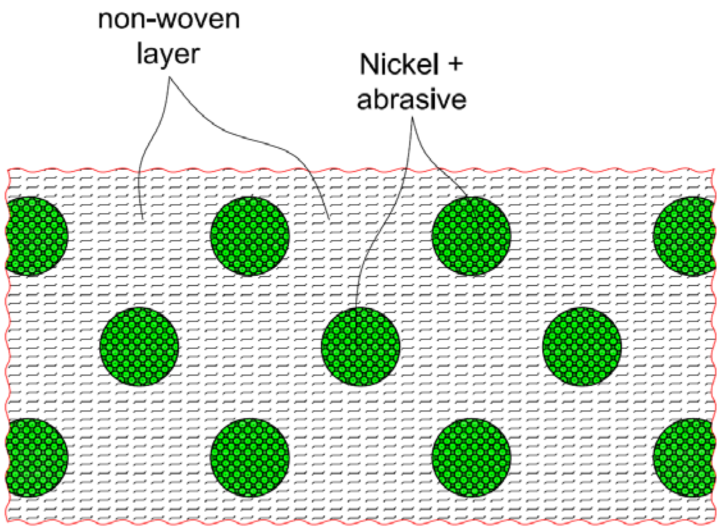
17- The advantages mentioned on this website can only be obtained with a product manufactured according to the method disclosed in EP Patent N° 2 900 000 owned by the Claimant.

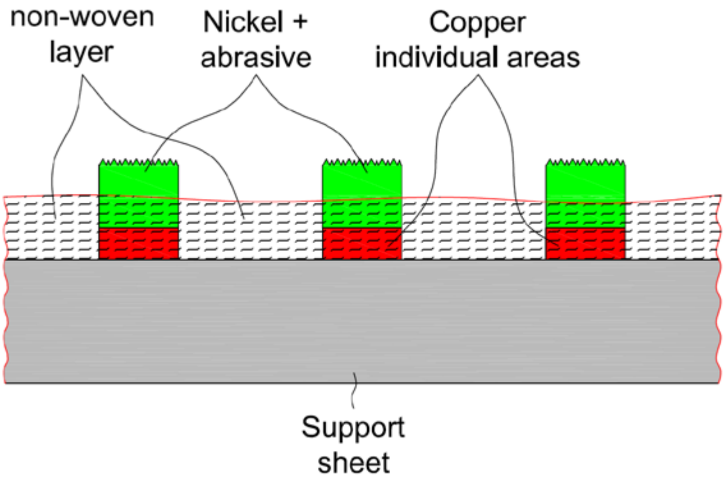
18- Consequently, subsequent to the grant of the Patent and prior to the issue of this Claim, the Defendant has infringed the Patent under Article 25 of the UPC Agreement, by doing, without the consent of the Claimant, in some or all of the Designated Contracting States the acts of offering to sell, a product which is manufactured according to the method which is the subject matter of claim 1 of the Patent.

5. Infringement Analysis

19- The infringing product is an abrasive member comprising:

- a support sheet
- a plurality of metal patches made of nickel having particulate abrasive therein on their upper surfaces,
- said metal patches having been attached to the support via individual areas of metallic copper in direct contact with the support sheet
- a layer of a nonwoven fabric comprising polyamide fibers stuck to the support between the metal patches maintains the metal patches laterally





20- In other words, the infringing product also has the two different metal layers recited in the method claim of EP 2900000.

21- The infringing product has a nonwoven polyamide layer between the independent abrasive metal patches. This layer forms a resin layer partially filling the voids between the abrasive patches and reducing lateral movement of the abrasive patches as required in the claim of EP 2900000.

22- The commercial statements on the website of the Defendant clearly acknowledge the existence of the two essential features of the product manufactured according to the patented method *i.e.* the reinforced securing of the metal abrasive patches onto the flexible support and the reduced lateral movement of the patches.

6. Relief

23- Unless restrained by this Court, the Defendant will continue to infringe the Patent, whereby the Claimant will suffer further loss and damage.

24- The Claimant is at present unable to give more information of the Defendant's infringements of the Patents but will seek relief at the trial of this action in respect of such infringement.

7. Allocation of a Technically qualified Judge

25- According to Rule 33 of the Rules of Procedure of the Unified Patent Court, any party may lodge an application for allocating a technically qualified judge to the panel.

Considering the difficulty of this matter and the high specificity of the Patent, the Claimant requests the designation of a technically qualified judge, the relevant field of technology being the abrasive material.

(Annex 5)

8. Value of the action

26- In results from the web site of La Toilemeri that the selling price which the Defendant intends to determine for the product offered for sale is of 5 Euros per meter.

27- Moreover, the fact that this offer was made through the internet in three languages and was therefore addressed to potential buyers in all the Contracting member States covered by the patent leads to an important prejudice for the Claimant.

28- The approximate market share of La Toilemeri in the abrasive products business in Europe can be estimated at 10%.

29- Since the overall value of the market for those products is about 5 Mi Euros, and the usual beneficial gross margin on that type of product is high and can be estimated, for the case of La Toilemeri at 30%, the unfair benefit which the Defendant may obtain for one year can be assessed at 150 000 Euros.

30- Besides, the Claimant exports in Europe, through a distribution network, abrasive products manufactured according to the method claimed in the Patent, at an average price of 8 Euros for one meter. Over the last three years the average yearly benefit of the Claimant resulting from the selling of those products in the European countries designated in the Patent was of 100 000 Euros.

31- In view of the remaining duration of the Patent (13 years) the Claimant may expect a total benefit of at least 1,3 million Euros over the life of the Patent.

32- On the other hand, the Defendant could produce similar abrasive products with another manufacturing method which would be outside the scope of protection of the Patent, even if the quality of the product would then be less attractive for the consumers.

33- Eventually, the Claimant will suffer a moral prejudice and a loss of market share.

34- Therefore, and taking into consideration this factual situation, the Claimant considers, for the time being that the value of the action should be established at 500 000 Euros.

9. Recoverable costs

35- The Claimant has incurred costs to ensure the protection of its IP right.

36- Hence, the Claimant considers that it justifies the allocation of the sum of 150 000 Euros.

10. Summary

37- The Claimant claims with respect to all Designated Contracting States:

1. The Allocation of a technically qualified judge

2. A declaration that the Patent is valid and has been infringed by the Defendant,

3. An injunction to restrain the Defendant from infringing the Patent, whether by using the method claimed in the Patent or by offering, placing on the market, using, importing or storing a product whichhas been manufactured according to the method claimed in the Patent;

3. That any non-compliance with the aforesaid injunction shall be subject to a recurring penalty payment payable to the Court;

4. An order for the delivery up or destruction upon oath of any product which infringes the Patent, whether by recalling the products from the channels of commerce, removing the products from the channels of commerce, and/or destroying the products concerned;

5. Damages suffered by the Claimant as a result of the Defendant’s acts of infringement;

6. An order for publication, at the Defendant’s expense, of any judgment in which the Defendant is found to have infringed;

7. Legal costs ;

8. Further or other relief.

List of documents referred to in the statement of claim

Annex 1: Patent EP 2900000

Annex 2: Decision of the opposition division of the EPO dated 3 September 2014

Annex 3: Bailiff Report

Annex 4: Accounting Certificate

Annex 5: Application for allocating a technically qualified Judge dated October 13th, 2014

Annex 6: Fixed fee payment

PROCÈS-VERBAL DE CONSTAT INTERNET

L'AN DEUX MILLE QUATORZE ET LE SIX OCTOBRE

À LA REQUETE DE :

La société 3A ABRASIVE, société de droit américain dont le siège social est situé 10008 S. Western Avenue, Chicago, IL 60643, USA.

LAQUELLE M'EXPOSE :

Qu'elle a le plus grand intérêt à faire constater le contenu de pages mises en ligne sur internet.

Qu'elle me requiert, en conséquence, pour assurer la sauvegarde de ses droits, de procéder à toutes constatations utiles et d'en dresser procès-verbal.

POURQUOI DÉFÉRANT À CETTE RÉQUISITION :

Je, Claude Dupuis, Huissier de Justice associés près le Tribunal de Grande Instance de PARIS, demeurant 23, rue des fermiers 75017, soussignée

Ce jour, en mon Etude :

J'AI VU, RECONNU ET CONSTATÉ CE QUI SUIT :

I - ENONCIATIONS PREALABLES

NORMES AFNOR NF Z67-147

1) Les présentes constatations sont effectuées sur le poste informatique décrit ci-après :

Ordinateur de bureau

Modèle : HP ProDesk 400 G1 MT

Carte mère HP18E9

Processeur : Intel(R) Core(TM)2 Duo CPU E8400 @ 3.00GHz, 3000 MHz, 2 cœur(s), 2 processeur(s) logique(s)

Mémoire physique : 4096 Mo

Carte graphique : Intel® HD Graphics 4400

Disque dur : TOSHIBA DT01ACA050 SCSI Disk Device (500Go)

Lecteur DVD-Rom : Hewlett-Packard DVDRAM GT80N

Type de moniteur : ViewSonic VG390m-3-19 pouces

Carte réseau : Realtek Semiconductor RTL8168/8111

Système d’exploitation : Windows 7 Professionnal Professionnel Medis Center 6…

DirectK : Version 11.00

Windows Performance Index : 4.8 sur 7.9

Connecté au serveur informatique de mon étude.

Les copies écran sont effectuées au moyen de la touche « Impr écran » et son directement incorporées au présent procès-verbal de constat.

Les captures sont effectuées au moyen du logiciel Capturino 2.43 et également de l’outil de capture windows Outil Capture et sont directement incorporées au présent procès-verbal de constat.

Les impressions sont réalisées au moyen du matériel d’impression suivant :

KONICA MINOLTA C360

Ce matériel permet d’imprimer les pages consultées. Les impressions réalisées sont annexées au présent procès-verbal.

2) Je synchronise l'horloge interne de mon poste avec le serveur de temps interenet « time.windows.com » en effectuant une mise à jour de « Propriétés de date et heure » « temps internet » de mon logiciel d'exploitation.

L'horloge a été synchronisée avec time.windows.com le 06/10/2014 à 13.01

3) Je mets à jour mon logiciel antivirus

Kaspersky

Endpoint Security 10

For Windows

Puis je lance une analyse antivirale.

4) je mets à jour mon programme de suppression des logiciels espions

Malwarebytes

ANTI-MALWARE

Puis je lance une analyse.

5) je détermine la configuration de ma machine de travail

(configuration reprise ci-dessus) (=description détaillée des éléments de mon poste informatique-matériel et système d'exploitation) à l'aide du logiciel : PC WIZARD 2014 Classic Edition – Version 2.13

6) j'accède au réseau étendu par un routeur connecté au serveur et dont le modèle est

D-LINK ADSL ROUTER modèle n°DSL6502T

Via carté réseau :

Realtek Semiconductor RTL8168/8111 Gigabit Ethernet Adapter

7) mon fournisseur d'accès est WANADOO/ORANGE

Abonnement : Internet pro solo – 8M

8) je détermine l’adresse MAC de la carte réseau active de ma machine de travail en utilisant la commande « ipconfig/all » précédée de cmd+ok dans l’invite de commande de mon logiciel d’exploitation et je relève que celle-ci est identique à celle obtenue à l’aide du logiciel gratuit BeLarc Advisor.

Je note que le DHCP n’est pas activé

Cette adresse est la suivante : 2C:44:F :3A:A8:C2

9) Je procède ensuite à diverses opérations de purge, comme suit :

- je procède à une purge complète et à un vidage intégral de ma corbeille
- je m’assure que les lecteurs CD Rom et de disquettes sont vides ;
- je m’assure que l’ordinateur ne contient aucun support de mémoire amovible externe.
- Purge complète de l’historique de navigation et paramétrage de mon navigateur Firefox 3.5 par le chemin : « Outils, « Options », « Vie privée » « Historique » « Ne jamais conserver l’historique ».
- Paramétrage du cache local et purge complète du cache local par le chemin : « Outils », « Options », « Avancé » « Réseau » « contenu web en cache » bouton « Vider maintenant »

10) Je m’assure que mon navigateur n’est pas paramétré pour utiliser un proxy par le chemin : « Outils », « Avancé », onglet « Réseau », « Connexion », « paramètres » je m’assure que concernant la Configuration du serveur proxy pour accéder à Internet ‘Pas de proxy » et coché comme suit :

Paramètres de connexion

Configuration du serveur proxy pour accéder à Internet

- Pas de proxy

11) Je configure mon navigateur pour accepter les cookies

12) Je paramètre une page vierge comme page de démarrage par le chemin : « Outils », « Options », onglet « général », « Démarrage », Au démarrage de Firefox « Afficher une page vide ».

13) Je récupère mon adresse IP pulique « WAN » - en utilisant la commande « ipconfig/all » dans l'invite de commande de mon système d'exploitation et en me connectant à la page http://www.mon-ip.fr

82.123.21.85

14) Je procède à un ultime nettoyage au moyen du logiciel CCleaner.com

***

II - CONSTATATIONS

Je démarre mes constatations à 14 heures 45

Lancement de mon navigateur internet Firefox 3.5

Dans la barre du navigateur, je saisis l'adresse suivante

http://www.latoilemeri.fr

et frappe sur la touche « Entrée ».

La page d'accueil du site apparaît.

Sur cette page d'accueil je clique sur le lien « NEWS », situé en haut de la page.

Une nouvelle page apparaît laquelle est imprimée (Annexe 1) et dont je fais des copies d'écran :

LA TOILEMERI SA.

LE SPÉCIALISTE FRANÇAIS DE L'ABRASIF INDUSTRIEL

Vous êtes ici / Accueil / News

Dernières nouvelles:

Nos laboratoires ont développé une nouvelle bande abrasive révolutionnaire, dont les qualités de résistance à l'usure et d'abrasion sont particulièrement remarquables.

La surface abrasive est formée d'une multitude de plots métalliques à surface diamantée. La fixation des plots sur le support de la bande est améliorée par rapport aux bandes abrasives classiques grâce à l'existence d'une couche de cuivre préalablement formée sur le support.
Les plots abrasifs sont noyés dans une couche de non-tissé comprenant des fibres longues de polyamide. L'usure est réduite grâce à l'excellent maintien latéral des plots métalliques abrasifs par la couche de non-tissé.

Prix de lancement envisagé: 5 € le m

La Toilemeri © 2014 / mentions légales / Contact

TELLES SONT MES CONSTATATIONS

Et de tout ce que dessus, j'ai fait et rédigé le présent procès-verbal de constat, pour servir et valoir ce que de droit.

SOUS TOUTES RÉSERVES

BAILIFF REPORT ON THE INTERNET

DATE: October 6th, 2014

Acting upon request of the company 3A ABRASIVE, a company with its registered office located in Chicago, USA.

STATING THAT:

It has the greatest interest in establishing a report of websites' pages' content on the internet.

It then requests me, in order to preserve its rights, to establish all necessary facts and to draw up an official report.

I, CLAUDE DUPUIS, COURT BAILIFF, WHOSE ADDRESS IS 23 RUE DES FERMIERS 75017 PARIS, CERTIFIES THAT I REPORTED THE FOLLOWING:

After having done all the necessary technical measures to ensure that the report is valid.

At 2.45 pm, I start my findings.

I start my web browser. Firefox 3.5

I enter the following address in the browser's address field:

http://www. latoilemeri.fr

and press "Enter".

The website’s homepage appears.

On this homepage, I click on the link “NEWS”, located on the top of the page.

A new page appears, which is printed (annex 1), and I make a screenshot of it:

Dernières nouvelles:

Nos laboratoires ont développé une nouvelle bande abrasive révolutionnaire, dont les qualités de résistance à l’usure et d’abrasion sont particulièrement remarquables.

La surface abrasive est formée d’une multitude de plots métalliques à surface diamantée. La fixation des plots sur le support de la bande est améliorée par rapport aux bandes abrasives classiques grâce à l’existence d’une couche de cuivre préalablement formée sur le support.
Les plots abrasifs sont noyés dans une couche de non-tissé comprenant des fibres longues de polyamide. L’usure est réduite grâce à l’excellent maintien latéral des plots métalliques abrasifs par la couche de non-tissé.

Prix de lancement envisagé: 5 € le m

La Toilemeri © 2014 / mentions légales / Contact

<u>THESE ARE MY FINDINGS.</u>

I certify that I have done and wrote this bailiff report.

Made for all legal intents and purposes,

FAJB Chicago

Accounting, Audit and Consulting

3A ABRASIVE

10008 S. Western Avenue
Chicago, IL 60643

Chicago, October 1st, 2014

I, certified public accountant at FAJB Chicago for the 3A ABRASIVE Company, hereby certifies that the average yearly benefit over the last three years (2012, 2013, 2014) in all the European countries, resulting from the patent No. 290000's exploitation by 3A ABRASIVE, was of 100.000 Euros.

Made for all legal intents and purposes,

John GRISHAM

Certified public accountant

j.grisham@fajb.com

13 October 2014
Application for allocating a technically qualified judge

By e-mail

Docket No 2015/01
UNIFIED PATENT COURT
PARIS LOCAL DIVISION

3 ABRASIVE (3A)
(A company with its registered office located in the USA)
v/
La Toilemeri SA
(A company with its registered office located in France)

APPLICATION FOR ALLOCATING A TECHNICALLY QUALIFIED JUDGE

Claimant's Address for Service:

Mr. Axel Casalonga, Partner of CASALONGA & Associés, is authorized to accept service in relation to these proceedings.

8, avenue Percier
75008 PARIS
France
a.casalonga@casalonga.com

2/ Mr. Martin Koehler, Partner of **REIMANN OSTERRIETH KÖHLER HAFT** is authorized to accept service in relation to these proceedings.

Steinstrasse 20
40212 Düsseldorf
Germany
Martin.Koehler@rokh-ip.com

1. Necessity of a technically qualified judge

1- A statement of claim is filed today based on European patent EP 2 900 0000.

2- The Patent relates to flexible abrasives and claims a method of forming an abrasive member with a step of electro deposition on a metal film.

3- The technology involved in the present case is complex.

6- Allocation to the panel of a technically qualified judge is therefore necessary. The field of technology is the field of abrasive materials.

FOR THESE REASONS, MAY IT PLEASE THE COURT:

7- That a request be made by this panel to the President of the Court of First Instance to allocate a technically qualified judge having qualifications and experience in the field of abrasive products

Enclosures:

fee payment for Application to allocate a technically qualified judge

Unified Patent Court
Paris Local Division

Docket number 2015/01

December 18, 2014

STATEMENT OF DEFENCE
COUNTERCLAIM FOR REVOCATION

On behalf of Defendant **La Toilemeri SA**, a company with registered seat in France.

Represented by
Mr. Grégoire Desrousseaux of August & Debouzy, 6-8 avenue de Messine F-75008 Paris,
gdesrousseaux@augdeb.com
Mr. Kay Rupprecht of Meissner Bolte, Widenmayerstr. 47, D-80538 Munich,
ru@mpb.de
both authorised to accept service in relation to the instant proceedings

Against Claimant **3 Abrasive (3A)**, a company with registered offices in the USA

Represented by
Mr Axel Casalonga, Casalonga & Associés, 8 avenue Percier, F-75008 Paris,
a.casalonga@casalonga.com
Mr Martin Köhler, Reimann Osterrieth Köhler Haft, Steinstr. 20, D-40212 Düsseldorf
martin.koehler@rokh-ip.com
both authorized to accept service in relation to the instant proceedings

1. GENERAL STATEMENTS

1 In response to the Statement of Claim of October 13, 2014, Defendant hereby files the Statement of Defence, together with a Counterclaim for Revocation of claim 1 of EP-B-2 900 000 (the Patent).

2 Defendant did not lodge any Preliminary Objection under Rule 19 RoP. The jurisdiction and competence of the Court are accepted. Competence of the Paris Local Division is not challenged.

Defendant does not object to the language of the Statement of Claim.

3 Defendant concurs with the application under Rule 33 RoP for allocating a technically qualified judge to the panel, made in par. 25 of the statement of claim. As regards the definition of the technical field, Defendant submits this should cover not only abrasive materials, but also manufacturing processes and uses of such materials.

While this application is fully unnecessary as regards the infringement claim, due to the lack of any actual evidence of infringement, Defendant hereby files a counterclaim for revocation. Due to this counterclaim, it is appropriate that a technical Judge be appointed to complete the panel.

4 Value of the dispute is discussed below in par. 37. We offer a value of the dispute (including the Counterclaim for Revocation) of 295 000 €.

5 Facts regarding the plaintiff and the history of the Patent, as listed in par. 2, 3, 4 and 6 of the Statement of claim, are admitted. The statement in par. 5 is denied in the absence of any evidence from Claimant.

As regards opposition proceedings in the EPO, an appeal of the decision of the Opposition Division (Annex 2 to the Statement of Claim) was filed on October 23, 2014, after the filing of the Statement of Claim. As evidenced in Exhibit D2, Defendant intervened in the EPO opposition proceedings pending before Board of Appeal 3.4.2, under Article 105 EPC.

6 The claim for infringement is groundless, in the absence of any evidence. All pleas and requests of Claimant should be rejected (see section 2 below).

A counterclaim for revocation is filed. Defendant requests that the sole claim of the patent be revoked (see section 3 below).

2. STATEMENT OF DEFENCE

7 Pleas 2-7 in the Statement of claims should be rejected, in view of the lack of any evidence of infringement.

As regards the plea for a declaration that the Patent is valid (first part of the second plea), the Court has no ability to declare patents to be valid. Besides, as discussed in Section 3 below, the Patent is invalid and revocation should be ordered,

2.1 FACTS AND EVIDENCE

8 Defendant relies on the facts and evidence listed in the Statement of Claim, to the extent they are admitted.

Facts listed in par. 8-12 of Statement of Claim are admitted, except for the discussion of alleged advantages and effects of the claimed subject-matter (e.g. "great advantages" in par. 9 or the "perfect" maintaining in par. 11). There is no demonstration nor any evidence supporting these advantages and effects.

The feature breakdown offered in par. 12 is used below.

9 Defendant further relies on French patent application FR-B-2 565 870, filed under number 84 09429 (thereafter D1). D1 is full prior art against the Patent, under Article 52(2) EPC.

A copy of D1 is attached. This patent is in the French language. We understand no translation of this document is required pursuant to the provisions of Rule 14.2 (c) (ii) RoP. This document is also relied upon below as regards the Counterclaim for revocation (together with additional evidence).

2.2 CLAIM INTERPRETATION

10 The claim of the Patent was not construed in the Statement of Claim. Alleging advantages of the claimed subject-matter is by no means a proper construction.

11 The following terms require construction

- *resin material* (features C3 and C4)
- *to reduce lateral movement of the protuberances* (feature C4).

Furthermore, the claim requires construction as regards the order of steps.

12 The only example of *resin* offered in the Patent stands in p. 2 lines 4-5, which states that

> *The resin material can be chosen from polyurethane resins, polyamide resins, polycarbonate or high density poly ethylene [sic].*

Polyurethane resin is the material used in the sole example of the Patent.

The description further states that the resin may comprise a filler of silicon carbide (p. 2 line 7).

It is also repeatedly stated in the Patent – including in the claims – that the resin material should achieve the effect of reducing lateral movement of the protuberance.

These statements make it clear for the reader of the Patent – the person skilled in the art of abrasives – that the resin material can only be a solid layer of resin. One cannot imagine a filler in anything else than a solid layer of resin.

This is also consistent with the claimed step of *filling the voids between the protuberances*: this suggest to the reader the pouring of a liquid resin into the voids, which will thereafter solidify and provide the claimed result of limiting lateral movement. No other form of *resin material* can be contemplated – and no alternate solution is offered or suggested in the Patent.

13 The Patent states the problem of avoiding the tendency of the metal deposits to chip off during abrasion (p. 1 lines 27-29). The problem is supposed to be solved thank to the resin reducing lateral movement (p. 1 lines 30-32). This is causing a "dramatic reduction" (p. 2 lines 1-3) on the metal deposits tendency to chip off. See also the reference to a "profound effect" at p. 2 line 27.

The terms *resin to reduce lateral movement of the protuberances* should thus be construed (and limited) in view of this technical effect.

This dramatic effect should take place <u>during abrasion</u> as made clear from the test offered in the specification (p. 3 lines 6-8).

14 The claim further requires construction as regards the order of steps.

The only disclosure in the specification is a process whereby the first step is a step of forming copper protuberances, the second step is a step of electrodepositing nickel protuberances and thereafter filling the voids with resin. See p. 2 lines 18-26.

There is no other suggestion or teaching in the description as regards the order of steps.

The wording of the claim further makes it clear that these steps are carried out in the same order. The fact that the last feature of the claim is drafted as a result (*voids (...) are partially filled*) cannot mask that it is actually the third and last step of the process.

While we expect the patentee to argue that the claim should be read as it stands, we submit that this is inconsistent with the Protocol on the Interpretation of Article 69. Taking the claims irrespectively of the (sole) positive and actual teaching of the description is exactly the very first alternative which is excluded in Article 1 of the Protocol.

Thus, the claim should be construed to cover a process in which the claimed step of filling the voids takes place <u>after</u> the two other steps of forming the protuberances.

2.3 NO EVIDENCE OF REPRODUCTION OF CLAIM 1

15 It is admitted that

- the Defendant has a commercial activity in the field of abrasive products for industrial uses (par. 13 of the Statement of claim);
- the Defendant manufactures in France and sells in Europe a variety of abrasive products (par. 14 of the Statement of claim);
- the Defendant published an announcement on its Internet website, as evidenced in Annex 3 of the Statement of claim (par. 15 and 16).

16 It is not admitted that

- the advantages mentioned in Annex 3 can only be obtained with a product manufactured according to the Patent claim (par. 17 of the Statement of claim);
- the Defendant committed any act of infringement (par. 18 of the Statement of claim).

The allegations in par. 19-22 of the Statement of claim are not admitted, to the extent they differ from the contents of Annex 3. This is the case, e.g. as regards *individual areas of metallic copper*, which does not reflect the disclosure of Annex 3.

17 As regards reproduction of claim 1 of the Patent, no evidence is offered by Claimant of the step *nickel protuberances are electrodeposited film*. Par. 19 of the Statement of claim correctly states that metal patches are attached to the support.

However, no evidence is offered that such patches are formed of by electrodepositing.

Alternative technologies to electrodeposition exist in the prior art, e.g. electrolytic or chemical processes (see the discussion of chemical deposition in D1, page 1, line 35 and page 2, lines 10 and 11). It is certainly undisputed that the allegedly infringing product comprises a plurality of metal patches made of **nickel** having particulate abrasive therein on their upper surfaces, said metal patches being attached to the support via individual areas of metallic **copper** in direct contact with the support sheet.

A frivolous claim is by no means a reason for Defendant to provide Claimant with proprietary trade secrets and manufacturing information.

18 No evidence is offered by Claimant of a *resin* being present in the allegedly infringing product. According to Claimant, a layer of nonwoven fabric comprising polyamide fibers extends between metal patches.

This layer is not a resin, as construed above. Such nonwoven layer cannot comprise a filler and is widely different from the generally accepted meaning of a resin material.

19 Even assuming, *arguendo*, that the nonwoven layer were to be construed as a *resin material*, there is no evidence whatsoever that the process steps are carried out in the order imposed by the claim language.

According to Defendant's process, the non-woven layer is formed <u>before</u> the metal chips are formed.

Infringement is excluded, inasmuch the claimed order of steps is not reproduced.

20 Last, no evidence is offered as regards the feature *to reduce lateral movement of the protuberances.*

Par. 19 of the Statement of claim recites that the mere presence of the nonwoven layer between the patches will reduce lateral movement. This is not supported by any form of evidence.

In view of the construction of these terms outlined above, a non-woven layer does not have the ability to provide the "dramatic reduction" against the tendency of the metal patches to chip off.

21 There is no evidence of reproduction of claim 1 for the manufacture of the product discussed in Annex 3 of the Statement of claim. There cannot be any evidence that this product was *obtained directly* as indicated in Article 25, letter (c) of the UPC Agreement.

The relief sought by Claimant has no basis and should be rejected.

2.4 NO ACT OF INFRINGEMENT

22 The only basis for the alleged infringement is the publication of Annex 3 on the website of Defendant.

Claimant argues in par. 18 of the Statement of claim that *offering to sell a product which is manufactured according to the [patented] method* is an act of infringement.

For the reasons discussed above, there is no evidence whatsoever that the product discussed in the website was manufactured according to the patented method. Actually, there is no evidence whatsoever that this product was ever manufactured at all – beyond the manufacture for experimental purposes. Such manufacture for experimental purposes is not an act of infringement pursuant to Article 27, letter (b) of the UPC Agreement.

In addition, the mere publication of Annex 3, which announces a product, without offering any possible way for the public to purchase the product, is not the act of *offering* a product which can be prevented under Article 25 letter (c) of the UPC Agreement. Offering a product can only take place when the product exists and can be purchased. A mere announcement of a future product is not an act that can be prevented under Article 25 UPCA.

In the absence of any act of infringement, Claimant's pleas should be dismissed.

3. COUNTERCLAIM FOR REVOCATION

23 Revocation of EP-B-2 900 000 (the Patent) is requested. Revocation is requested as regard the sole claim and for all designated States, under Article 65(2) of the UPC Agreement.

Claimant is the sole proprietor of the Patent according to the EPO on-line Register and has standing.

Defendant was sued for infringement before the Court and thus, has obvious standing for requesting revocation.

24 Ground for revocation is the lack inventive step of the sole independent claim under Articles 138(1) a) and 56 EPC.

As an auxiliary plea, revocation is request for insufficient disclosure, Article 138(1) b) EPC.

25 Prior art relied upon is D1 (already discussed above).

Defendant further relies on D3. D3 shows the first 25 results of an Espacenet search for applications with a date of publication between 2000 and 2007, the abstract of title of which contains the words *electrodepo** and *abrasive*.

26 The fee for the Counterclaim for Revocation provided in Rule 26 is paid concurrently.

27 As outlined above, Defendant intervened in the EPO opposition proceedings (now pending before the Board of Appeal). Notice of intervention was filed on December 16 at the EPO and is attached as document D2.

3.1 DISCLOSURE OF D1

28 D1 discloses the same type of abrasive as the Patent. D1 is discussed in the Patent at p. 1 lines 20-25.

D1 disclosed a two layered structure of metal patches. See in p. 2 line 30 to 32 the example of a support of supple plastic 2 covered by a thin metal layer 3. Over the thin metal layer, patches 8 are formed by chemical deposition (see p. 4 line 3-7, for instance).

D1 teaches that a resin fills the space between the patches. This resin (p. 3 line 13) forms a layer (*épargne*) which is, according to D1, removed to obtain the final product (p. 9-13).

3.2 LACK OF INVENTIVE STEP

29 Claim 1 of the Patent lacks inventive step over D1, in view of the common general knowledge of the person skilled in the art.

30 For the purposes of this counterclaim, we will consider the broadest possible construction of Claim 1, whereby the order of steps is irrelevant.

While we strongly believe this construction is contrary to the Protocol, it is the only possible construction (as regards the order of steps) under which the Defendant's products might infringe (again, only taking into account the order of steps).

31 There are two possible differences between the claim and D1.

Difference 1 is use of electrodeposition, while D1 teaches chemical deposition.

Difference 2 is the filling of resin, while D1 teaches removing the resin layer. Whether this is actually a difference is discussed below.

32 Differences 1 and 2 are unrelated one to the other.

The way the Ni protuberances are deposited does not affect the filling of voids with resin.

Conversely, the filling of voids with resin can be carried out irrespective of the nature of the deposition step.

Thus, partial technical problems should be defined and inventive steps should be discussed separately for Differences 1 and 2.

33 Difference 1 cannot involve any inventive step.

There is no disclosure of any technical effect in the Patent for the step of electrodepositing. There is no suggestion that this method of forming the Ni protuberances would provide any advantageous effect over the prior art chemical deposition of the same protuberances in D1.

Thus, the partial objective technical problem is at best to provide an alternative deposition method for the Ni protuberances.

The solution is to use electrodeposition rather than chemical deposition.

This solution is one of the alternatives available to the person skilled in the art of manufacturing of abrasives. We offer as evidence thereof the result of an Espacenet search using the key words *electrodepo** and *abrasive*. This provides 141 results for applications with a publication date between 2000 and 2007 – that is earlier than 2008. Hits 6, 7, 9 and 10 are obvious examples of the use of electrodeposition techniques for manufacturing abrasive products.

In the unlikely event that Claimant would challenge that this technique belong to the CGK, we submit that the Patent lacks any proper indication to the skilled reader as to the implementation of the electrodeposition step. Thus, unless it can be assumed that the technology at stake is CGK, the Patent lacks sufficient disclosure.

Thus, there is no inventive step whatsoever in changing the chemical deposition of D1 into the claimed electro-deposition.

Difference 1 cannot involve an inventive step.

34 Difference 2 cannot involve an inventive step either.

First, D1 teaches the sequence of forming the copper layer, forming the protuberances and having the voids partially filled with resin. See description of D1, up to p. 4 line 9.

D1 suggests that the resin filling be removed.

However, it is undebatable that the product of D1, before the resin is removed, is already fully usable as abrasive member. This product has all functionalities for being used as an abrasive.

Thus, we submit D1 teaches manufacturing of an abrasive member, with resin filling the voids. In view of the nature of this resin (especially the photoresist mentioned in p. 4 line 22), it is implicit that there will be an effect limiting lateral movement of the protuberances.

Thus, claim 1 lacks inventive step.

35 In the assumption that the Court considers that no abrasive member is obtained in D1 prior to the step of removing the layer, we offer the following auxiliary plea.

D1 would thus provide a method with all the steps of the claim, except for the additional provision of resin material filling the voids.

First, simplifying the process of D1 simply by deleting the step of removing the layer of photoresist is a measure of routine for the person skilled in the art. Simplifying a process cannot involve an inventive step – all the more as there already existed abrasive products with or without material between the protuberances / abrasive parts.

Second, the objective technical problem at stake would (at best) be to improve resistance to lateral movement of the protuberances. Leaving the photoresist around the protuberances is the very first idea that the person skilled in the art would have when faced with this problem.

Thus, Difference 2 – assuming it is indeed a difference – cannot involve an inventive step.

36 The invention is obvious in view of D1 and of the CGK, for the person skilled in the art.

Claim 1 should be revoked for lack of inventive step.

As an auxiliary plea, should the Claimant deny that electrodeposition techniques belonged to the CGK at the priority date of the Patent (for the manufacture of abrasives), the Patent should be revoked for insufficient disclosure.

4. VALUE OF THE DISPUTE – RELIEF

37 The proposed value of the dispute is 295 000 €.

The value of 500 000 € was offered by the Claimant in par. 31 of the Statement of claim. This is based on the argument that the yearly benefit of the Claimant for the sales of products manufactured according to the Patent amount to 100 000 € in the last three years.

The computation offered by the Claimant is not consistent with the rules set out in Art. 13 of the enforcement Directive 2004/48. As regards patentee, the Directive does not refer to patentee's benefits, but to *negative economic consequences, including lost profits*[1], This refers to consequences of the infringement, but not to the economic activities of the patentee working the patent.

In the unlikely event that the Court were to find the patent valid and infringed, this would be in less than one year after the (possible) launch of the product. Negative economic consequences for the Claimant would be extremely limited.

As to *moral prejudice*, we hardly see any reason for taking into account any moral prejudice in this matter. The mere publication on the Defendant's web page of an announcement, without any reference to the Patent or to the manufacturing process protected by the Patent does not create any moral prejudice. There is no need to remind the Court that results – such as resistance to chipping off of the metal inserts – cannot be protected and may thus be freely used by all economic actors.

Even if the Defendant's product were to be launched, there would not be any *moral prejudice* of any sort for the Claimant, failing any specific reference to the Patent or to Claimant's products.

Last, the Directive offers to take into account *unfair profits made by the infringer*[2]. Assuming the allegedly infringing product were to be launched before a decision of the Court, *unfair profits made by the infringer* would not exist. Developing and launching a product require substantial investments – as the Court knows – and would not generally result in profits in a time period as short as the one until a decision of the Court.

We offer a value of 100 000 € for the value of the infringement claim. The remarks made above should not be construed as any admission of the facts underlying this value.

The value for the counterclaim is assessed at 195 000 €.This value should be assessed according to the patentee's sales figures or license fees from the time of filing the revocation action for the prospective term of the patent.

This assessment is based on the fact that the patentee's market shares for the abrasive

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[1] Article 13.1 (a) of the Directive.

[2] Article 13.1 (a) of the Directive.

products business in Europe can be estimated at 30%. Not all abrasive products sold by Patentee work the Patent. We offer the estimate of 15% of the overall market as representative of the products potentially working the Patent[3].

Patentee's own assessment of the value of the market for such products is 5 Mi Euros; while we believe this value is grossly overestimated, we suggest, for the purposes of simplicity, to take it as a working basis for the assessment of the value of the case.

This means that the annual market share of Patentee for the products that possibly work the Patent amount to 750 k€. Assuming a royalty rate of 2%[4], the annual revenue under the Patent might, at the very best, amount to 15 000 €.

Even taking into account the Patentee's sales figures, the benefits supposedly made with products working the Patent amount to 100 000 € per year. However, benefits under these products cannot be equated to the value of the Patent. As pointed out by the Claimant himself in his statement of claim,

> *The Defendant could produce similar abrasive products with another manufacturing method which would be outside the scope of protection of the Patent.*

Irrespective of the Defendant's manufacturing technologies and know-how, it is fully clear that products manufactured as suggested in the Patent but using chemical deposition would not be comprised in the scope of protection and would still offer the same resistance to chipping off. We again refer to the discussion of partial problems above.

Thus, the benefits of the Claimant have nothing to do with the protection under the Patent. The benefits of the Claimant cannot be equated to the value of the counterclaim for revocation.

A value of the Patent corresponding to one third of the Claimant's benefits (according to the usual rule of thumb) would provide a yearly value of about 30 000 € for the Patent. In view of the fact that alternative manufacturing solutions exits (as acknowledged by the Claimant), this figure should be diminished. This confirms the yearly value of 15 000 € offered by Defendant.

We have no evidence of the Patent being licensed and this is not put forward by the Claimant.

In view of the remaining life time of the Patent (13 years), the value of the case for the Counterclaim is proposed to be 13 * 15 000 €, that is 195 000 €.

Should the facts outlined above be challenged by the Claimant, we offer the testimony of our Director of Marketing and Sales. Again, these remarks are by no means to be construed as an admission of facts put forwards by the Claimant.

Therefore the value of the dispute (including the counterclaim, Rule 31 RoP) is less than 500 000 €. There should not be any value-based fee.

38 The following relief is sought.

Defendant requests that

- Claimants pleas be rejected – apart from the plea for allocation of a technical Judge to complete the panel ;

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[3] This estimate is based on products which cannot possibly work the Patent, e.g. products without the filling of resin. Whether other products work or not the Patent – that is use the process as claimed was not determined by Defendant. Defendant has not carried out full examination of the manufacturing process of Plaintiff for the sole purpose of determination of the value of the case.

[4] We again believe this is over-estimated. This is no admission that this would be a fair royalty value.

- EP-B-2 900 000 be revoked, for all designated States and in its entirery, pursuant to Article 65(2) of the UPC Agreement ;
- Costs and expenses incurred by the Defendant (including the fixed fee for revocation under Rule 26) be reimbursed by the Plaintiff, pursuant to Article 69(1) of the UPC Agreement ;
- The decision of the Court be published in 5 newspapers chosen by the Defendant, at the sole costs of Claimant.

The estimate of legal costs to be recovered shall be provided in advance of the hearing, pursuant to Rule 118.6 RoP, taking into account the ceiling for such costs. We submit that these costs should further include costs incurred for the intervention in the opposition proceedings, which was clearly caused (and made possible) by the instant infringement claim.

5. LIST OF DOCUMENTS

39 Documents relied upon in the Statement of Defence and Counterclaim for Revocation include :

- D1 : FR-B-2 565 870 (application number 84 09429)
- D2 : intervention brief in the opposition proceedings before the EPO (at the appeal stage)
- D3 : Espacenet Search for "electrodepo* " and "abrasive", between 2000 and 2007.

EPO
D-80298 Munich

EP-B-2 900 000 (22650211.5)
Title : Flexible abrasives
Patentee : 3 Abrasives

T 2940/14 – Board 3.4.2

On-line filing
Our ref. LTE 14/123

December 16, 2014

Dear Sirs,

On behalf of

> **La Toilemeri SA**, a company with registered seat 2 rue de la Gaieté, F-92400 Bécon-lès-Bruyères,

We, August & Debouzy, association of representatives n°108, 6-8 avenue de Messine F-75008 Paris, hereby intervene in the opposition proceedings against the above referenced patent EP-B-2 900 000 to 3 Abrasives.

We request that the opposition fee be debited from our deposit account No 2.804.0880.

The opposition is based on the grounds of
- Article 100 a) and 56 EPC,
- Article 100 b) EPC.

Facts and arguments are attached.

Opposition proceedings are pending before the Board of Appeal. Intervention is possible under Article 105 EPC, see decision G 3/04.

Intervention under Article 105 EPC is admissible inasmuch as proceedings for infringement of EP-B-2 900 000 were initiated by the patent proprietor in front of the Unified Patent Court, at the Paris Local Division.

We attach the Statement of claim dated October 13, 2014. The deadline of three months of Rule 89(1) EPC is thus respected.

We request that EP-B-2 900 000 be revoked in its entirety, Article 101(2) EPC.

We further requests oral proceedings (art. 116 EPC), should any decision other than setting aside the decision of the Opposition Division dated September 3, 2014 and revoking the patent in its entirety be contemplated by the Board.

Please note that Mr. Kay Rupprecht of Meissner Bolte, Widenmayerstr. 47, D-80538 Munich is also empowered to represent the Intervener in these proceedings as an additional representative. We understand that no power of attorney is required under the decision of the President of the EPO dated 12.07.2007. Should this not be the case, we will gladly provide such a power of attorney.

Yours sincerely

Grégoire Desrousseaux
Authorized representative

Encls:
- Statement of Claim before the UPC
- Documents D1 and D2
- Facts and arguments

FACTS AND ARGUMENTS

1. DOCUMENTS

1 The opposition is based on

- FR-B-2 565 870 (D1)
- Common general knowledge at the priority date, evidenced by the results of an Espacenet search using the key words *electrodepo** and *abrasive*. (D2)

D1 is prior art under Art. 54(2) EPC.

2. DISCLOSURE OF D1

2 D1 discloses the same type of abrasive as EP-B-2 900 000 (the Patent). D1 is discussed in the Patent at p. 1 lines 20-25.

D1 disclosed a two layered structure of metal patches. See in p. 2 line 30 to 32 the example of a support of supple plastic 2 covered by a thin metal layer 3. Over the thin metal layer, patches 8 are formed by chemical deposition (see p. 4 line 3-7, for instance).

D1 teaches that a resin fills the space between the patches. This resin (p. 3 line 13) forms a layer (*épargne*) which is, according to D1, removed to obtain the final product (p. 9-13).

3. LACK OF INVENTIVE STEP

3 Claim 1 of the Patent lacks inventive step over D1, in view of the common general knowledge of the person skilled in the art.

4 There are two possible differences between the claim of the Patent and D1.

Difference 1 is use of electrodeposition, while D1 teaches chemical deposition.

Difference 2 is the filling of resin, while D1 teaches removing the resin layer. Whether this is actually a difference is discussed below.

5 Differences 1 and 2 are unrelated one to the other.

The way the Ni protuberances are deposited does not affect the filling of voids with resin.

Conversely, the filling of voids with resin can be carried out irrespective of the nature of the deposition step.

Thus, partial technical problems should be defined and inventive steps should be discussed separately for Differences 1 and 2. We refer to the Case law of the Boards of Appeal of the EPO, 7th Edition, I.D.9.2.2.

6 Difference 1 cannot involve any inventive step.

There is no disclosure of any technical effect in the Patent for the step of electrodepositing. There is no suggestion that this method of forming the Ni protuberances would provide any advantageous effect over the prior art chemical deposition of the same protuberances in D1.

Thus, the partial objective technical problem is at best to provide an alternative deposition method for the Ni protuberances.

The solution is to use electrodeposition rather than chemical deposition.

This solution is one of the alternatives available to the person skilled in the art of manufacturing of abrasives. We offer as evidence thereof D2. D2 provides 141 results for applications with a publication date between 2000 and 2007 – that is earlier than 2008. Hits 6, 7, 9 and 10 are obvious examples of the use of electrodeposition techniques for manufacturing abrasive products.

In the unlikely event that the patent proprietor would challenge that this technique belongs to the CGK, we submit that the Patent lacks any proper indication to the skilled reader as to the implementation of the electrodeposition step. Thus, unless it can be assumed that the technology at stake is CGK, the Patent lacks sufficient disclosure.

Thus, there is no inventive step whatsoever in changing the chemical deposition of D1 into the claimed electro-deposition.

Difference 1 cannot involve an inventive step.

7 Difference 2 cannot involve an inventive step either.

First, D1 teaches the sequence of forming the copper layer, forming the protuberances and having the voids partially filled with resin. See description of D1, up to p. 4 line 9.

D1 suggests that the resin filling be removed.

However, it is undebatable that the product of D1, before the resin is removed, is already fully usable as abrasive member. This product has all functionalities for being used as an abrasive.

Thus, we submit D1 teaches manufacturing of an abrasive member, with resin filling the voids. In view of the nature of this resin (especially the photoresist mentioned in p. 4 line 22), it is implicit that there will be an effect limiting lateral movement of the protuberances.

Thus, claim 1 lacks inventive step.

8 In the assumption that the Board considers that no abrasive member is obtained in D1 prior to the step of removing the layer, we offer the following auxiliary plea.

D1 would thus provide a method with all the steps of the claim, except for the additional provision of resin material filling the voids.

First, simplifying the process of D1 simply by deleting the step of removing the layer of photoresist is a measure of routine for the person skilled in the art. Simplifying a process cannot involve an inventive step – all the more as there already existed abrasive products with or without material between the protuberances / abrasive parts.

Second, the objective technical problem at stake would (at best) be to improve resistance to lateral movement of the protuberances. Leaving the photoresist around the protuberances is the very first idea that the person skilled in the art would have when faced with this problem.

Thus, Difference 2 – assuming it is indeed a difference – cannot involve an inventive step.

9 The invention is obvious in view of D1 and of the CGK, for the person skilled in the art.

Claim 1 should be revoked for lack of inventive step.

As an auxiliary plea, should the patent proprietor deny that electrodeposition techniques belonged to the CGK at the priority date of the Patent (for the manufacture of abrasives), the Patent should be revoked for insufficient disclosure.

4. REQUESTS

10 Requests are indicated above. We request that the decision of the opposition division be set aside and that the patent be revoked in its entirety.

We also request oral proceedings, should any other decision be contemplated by the Board.

Europäisches Patentamt
European Patent Office
Office européen des brevets

Espacenet

Result list

141 results found in the Worldwide database for:
electrodepo* AND abrasive in the title or abstract AND **2000:2007** as the publication date

1. **PROTECTIVE COATING FOR COMPONENTS OF A WEAPON OR THE LIKE**

Inventor:	**Applicant:**	**CPC:**	**IPC:**	**Publication info:**	**Priority date:**
BILGER GERHARD [DE] TRIPP MARKUS [DE]	RHEINMETALL WAFFE MUNITION [DE] BILGER GERHARD [DE] (+1)	C23C28/023 C23C30/00 F41A27/08 (+2)	F41A27/18	WO2007137716 (A1) 2007-12-06	2006-05-29

2. **WIRE SAW BEAD**

Inventor:	**Applicant:**	**CPC:**	**IPC:**	**Publication info:**	**Priority date:**
CHO SUNG HO [KR] SONG MIN SEOK [KR] (+4)	SHINHANDIAMONDINDUSTRIAL CO LT [KR]		B23D61/18	KR20070102016 (A) 2007-10-18	2006-04-13

3. **SAW WIRE AND ITS MANUFACTURING METHOD**

Inventor:	**Applicant:**	**CPC:**	**IPC:**	**Publication info:**	**Priority date:**
NAKAGAWA HEIZABURO KOBAYASHI MAKOTO	NIPPON SEISEN CO LTD		B24D11/00 B24D3/00 B24D3/06 (+1)	JP2007203393 (A) 2007-08-16 JP4829626 (B2) 2011-12-07	2006-01-31

4. **MANUFACTURING METHOD OF SAW WIRE**

Inventor:	**Applicant:**	**CPC:**	**IPC:**	**Publication info:**	**Priority date:**
OGAMI HIROYUKI UNO KOJI	KANAI HIROAKI		B24D11/00 B24D3/00 B24D3/06	JP2007152486 (A) 2007-06-21	2005-12-05

5. **MANUFACTURING METHOD OF SAW WIRE**

Inventor:	**Applicant:**	**CPC:**	**IPC:**	**Publication info:**	**Priority date:**
UNO KOJI OGAMI HIROYUKI	KANAI HIROAKI		B24D11/00 B24B27/06	JP2007152485 (A) 2007-06-21	2005-12-05

6. **ELECTROPLATED ABRASIVE TOOLS, METHODS, AND MOLDS**

Inventor:	**Applicant:**	**CPC:**	**IPC:**	**Publication info:**	**Priority date:**
SUNG CHIEN-MIN	SUNG CHIEN-MIN	B24D18/0009 B24D18/0018	B24B7/16	WO2007120224 (A2) 2007-10-25 WO2007120224 (A3) 2007-12-27	2005-12-02

7. **ELECTRODEPOSITION GRINDSTONE AND GRINDING METHOD USING IT**

Inventor:	**Applicant:**	**CPC:**	**IPC:**	**Publication info:**	**Priority date:**
AKIBA KENJI SUWA KENICHIRO	TDK CORP		B24D3/06 B24D5/10 B24B55/02	JP2007144597 (A) 2007-06-14	2005-11-30

8. **TRUING MEMBER FOR ABRASIVE PAD AND TRUING METHOD OF ABRASIVE PAD**

Inventor: KUBO TAKASHI [JP] KIMURA HIROSHI [JP]	**Applicant:** ASAHI GLASS CO LTD [JP] KUBO TAKASHI [JP] (+1)	**CPC:** B24B37/042 B24B53/017 B24B53/12	**IPC:** B24B37/04 B24B53/12 B24D7/06 (+1)	**Publication info:** WO2007043263 (A1) 2007-04-19	**Priority date:** 2005-10-14
9. MANUFACTURING METHOD OF ELECTRODEPOSITION GRINDING WHEEL					
Inventor: ITO TATSUYA	**Applicant:** HONDA MOTOR CO LTD	**CPC:**	**IPC:** B24D3/00 B24D3/06	**Publication info:** JP2007083368 (A) 2007-04-05 JP4450781 (B2) 2010-04-14	**Priority date:** 2005-09-26
10. Production of wear-, heat-, corrosion- and oxidation resistant abrasive protective coating on component, e.g. turbine, especially gas turbine, or aircraft engine by deposition of base alloy coating with embedded hard particles					
Inventor: COSACK THOMAS [DE] LINSKA JOSEF [DE] (+2)	**Applicant:** MTU AERO ENGINES GMBH [DE]	**CPC:** C23C10/02 C23C10/60 C23C26/00 (+14)	**IPC:** C23F17/00 F01D25/00	**Publication info:** DE102005038374 (A1) 2007-02-15	**Priority date:** 2005-08-13
11. ELECTRODEPOSITION WIRE TOOL					
Inventor: MURAKAMI TSUTOMU MANITA YOSHIHISA (+1)	**Applicant:** ASAHI DIAMOND IND	**CPC:**	**IPC:** B24B27/06 B24D11/00 B24D3/00 (+3)	**Publication info:** JP2007021677 (A) 2007-02-01	**Priority date:** 2005-07-19
12. ELECTRODEPOSITED ABRASIVE TOOL AND METHOD OF PRODUCING ELECTRODEPOSITED ABRASIVE TOOL					
Inventor: TANIGUCHI KAZUAKI	**Applicant:** ASAHI DIAMOND IND	**CPC:**	**IPC:** B24D3/00 B24D3/06	**Publication info:** JP2007021668 (A) 2007-02-01	**Priority date:** 2005-07-19
13. ELECTRODEPOSITED ABRASIVE TOOL AND METHOD OF PRODUCING ELECTRODEPOSITED ABRASIVE TOOL					
Inventor: TANIGUCHI KAZUAKI	**Applicant:** ASAHI DIAMOND IND	**CPC:**	**IPC:** B24D3/00 B24D3/02 B24D3/06 (+2)	**Publication info:** JP2007021667 (A) 2007-02-01	**Priority date:** 2005-07-19
14. SPROCKET					
Inventor: SAKAGUCHI JUN	**Applicant:** BORG WARNER MORSE TEC JAPAN KK	**CPC:** F16H55/30	**IPC:** F01L1/02 F16H55/30	**Publication info:** JP2007010075 (A) 2007-01-18	**Priority date:** 2005-07-01
15. ULTRASONIC CUTTER AND ITS CUTTING METHOD					
Inventor: PARK BYUNG SUN [KR]	**Applicant:** HAVIT INFORMATION CO [KR]	**CPC:**	**IPC:** B26D7/08 B26F3/00	**Publication info:** KR100647199 (B1) 2006-11-10	**Priority date:** 2005-06-03
16. CORRECTING METHOD FOR FORMING TOOL					
Inventor: TOMITA ATSUSHI	**Applicant:** CANON KK	**CPC:**	**IPC:** B24B13/01 B24B53/07	**Publication info:** JP2006218586 (A) 2006-08-24	**Priority date:** 2005-02-14
17. ELECTRODEPOSITION GRINDING WHEEL AND METHOD OF MANUFACTURING ELECTRODEPOSITION GRINDING WHEEL					

Inventor:	Applicant:	CPC:	IPC:	Publication info:	Priority date:
YAMAGUCHI TAKASHI	DISCO ABRASIVE SYSTEMS LTD		B24D3/00 B24D3/02 B24D3/06	JP2006212763 (A) 2006-08-17 JP4767548 (B2) 2011-09-07	2005-02-07

18. ROTARY GRINDING WHEEL

Inventor:	Applicant:	CPC:	IPC:	Publication info:	Priority date:
KOYANAGI SUSUMU TEJIMA MASATOMO	NORITAKE SUPER ABRASIVE KK NORITAKE CO LTD		B24D7/00	JP2006205314 (A) 2006-08-10	2005-01-28

19. GRINDING WHEEL ROLLER FOR FORMING ROUGHENED SURFACE

Inventor:	Applicant:	CPC:	IPC:	Publication info:	Priority date:
HAMADA KAYOKO MIYAKE EISHIN	TOPPAN PRINTING CO LTD		B24D3/00 B24D3/06 B24D5/00 (+2)	JP2006205288 (A) 2006-08-10	2005-01-27

20. CMP PAD CONDITIONER

Inventor:	Applicant:	CPC:	IPC:	Publication info:	Priority date:
IMAI NORIO	NORITAKE SUPER ABRASIVE KK NORITAKE CO LTD		B24B53/02 B24B53/12 H01L21/304	JP2006190899 (A) 2006-07-20 JP4194563 (B2) 2008-12-10	2005-01-07

21. ELECTRODEPOSITION WIRE TOOL

Inventor:	Applicant:	CPC:	IPC:	Publication info:	Priority date:
TANIGUCHI KAZUAKI [JP] NAKANO MASAHIRO [JP] (+1)	ASAHI DIAMOND IND [JP]	B23D61/185 B24B27/0633 B24D3/06	B24B27/06 B24D11/00 B24D3/00 (+1)	KR20070090074 (A) 2007-09-05 KR101147519 (B1) 2012-05-21	2004-12-28

22. METHOD FOR MANUFACTURING HOURGLASS WORM GEAR

Inventor:	Applicant:	CPC:	IPC:	Publication info:	Priority date:
HORIUCHI AKIYO	SHIN EI TECH KK		B21H5/00 B23F13/08	JP2006198759 (A) 2006-08-03	2004-12-21

23. POLISHING CLOTH, WAFER POLISHING DEVICE, AND METHOD OF MANUFACTURING WAFER

Inventor:	Applicant:	CPC:	IPC:	Publication info:	Priority date:
KAWAHITO SHINICHI MARUOKA DAISUKE (+2)	KOMATSU DENSHI KINZOKU KK		B24B37/20 B24B53/017 B24D3/00 (+2)	JP2006147731 (A) 2006-06-08 JP4749700 (B2) 2011-08-17	2004-11-17

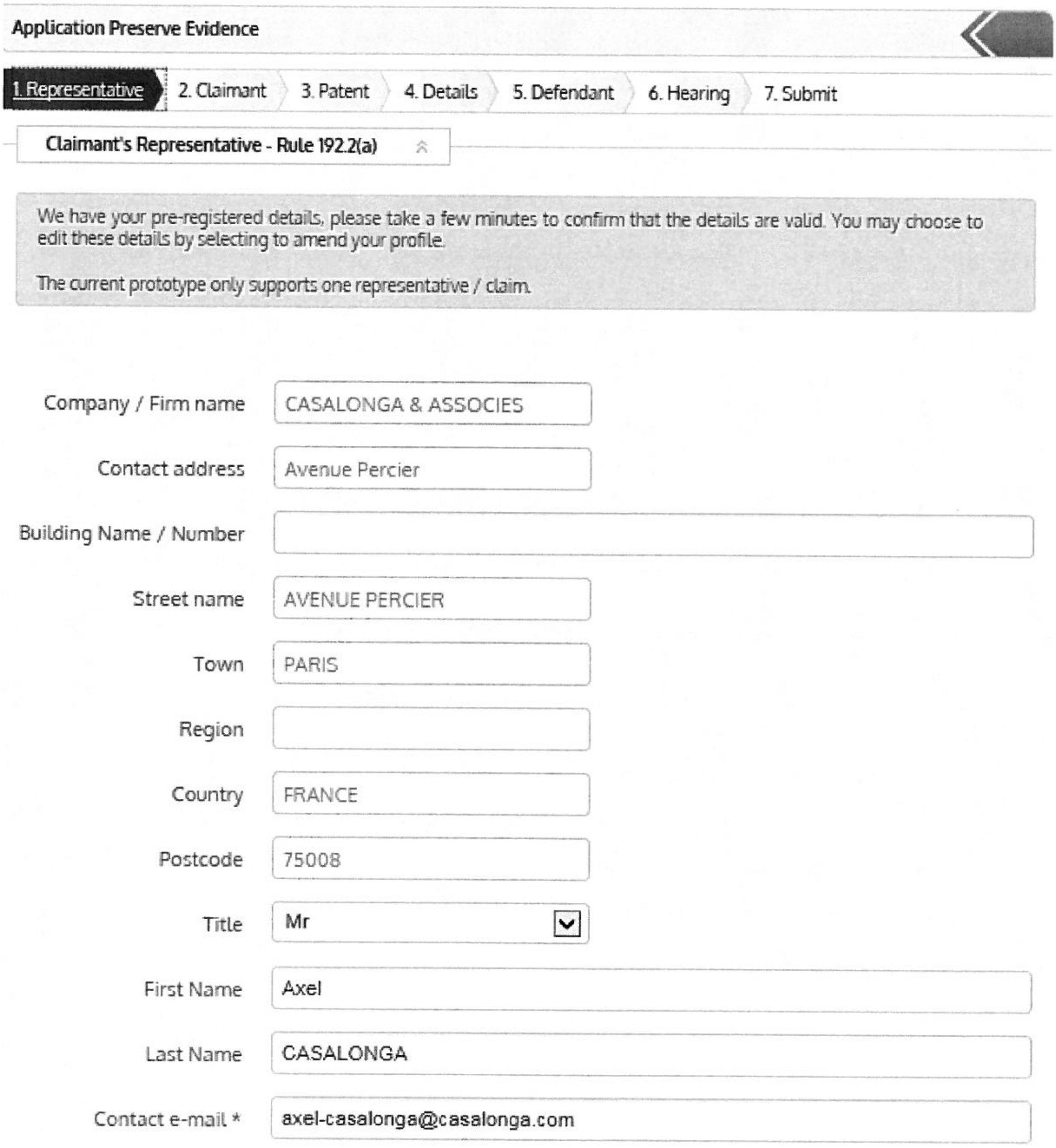
24. MANUFACTURING METHOD FOR WORM WHEEL AND WORM SPEED REDUCER

Inventor:	Applicant:	CPC:	IPC:	Publication info:	Priority date:
MAEDA ATSUSHI CHIKARAISHI KAZUO	NSK LTD	B23F11/00 B23F21/026 F16H55/22 (+1)	B23F11/00 B62D5/04 F16H1/16 (+3)	JP2006142400 (A) 2006-06-08 JP4834978 (B2) 2011-12-14	2004-11-16

25. FIXED ABRASIVE GRAIN TYPE WIRE SAW AND ITS MANUFACTURING METHOD

Inventor:	Applicant:	CPC:	IPC:	Publication info:	Priority date:
MURATA YASUNORI INOUE MAKOTO	NAKAMURA CHOKO KK		B23D61/18 B24B27/06 B24D11/00 (+3)	JP2006123024 (A) 2006-05-18	2004-10-26

Draft Form of Application for Preserving Evidence



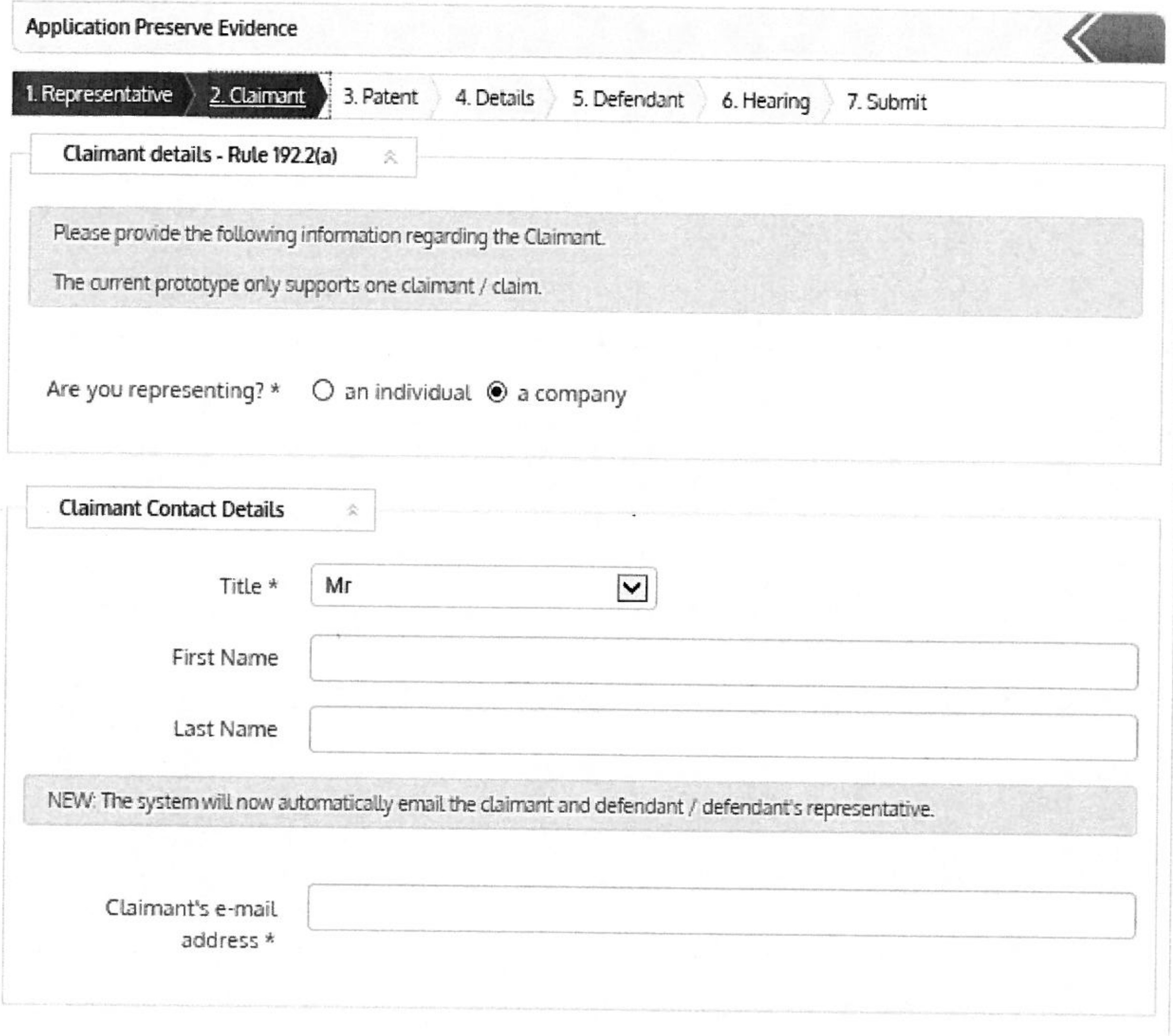
Application Preserve Evidence

Claimant's Representative - Rule 192.2(a)

We have your pre-registered details, please take a few minutes to confirm that the details are valid. You may choose to edit these details by selecting to amend your profile.

The current prototype only supports one representative / claim.

Company / Firm name: CASALONGA & ASSOCIES
Contact address: Avenue Percier
Building Name / Number:
Street name: AVENUE PERCIER
Town: PARIS
Region:
Country: FRANCE
Postcode: 75008
Title: Mr
First Name: Axel
Last Name: CASALONGA
Contact e-mail *: axel-casalonga@casalonga.com



Application Preserve Evidence

1. Representative | 2. Claimant | 3. Patent | 4. Details | 5. Defendant | 6. Hearing | 7. Submit

Claimant details - Rule 192.2(a)

Please provide the following information regarding the Claimant.

The current prototype only supports one claimant / claim.

Are you representing? * ○ an individual ● a company

Claimant Contact Details

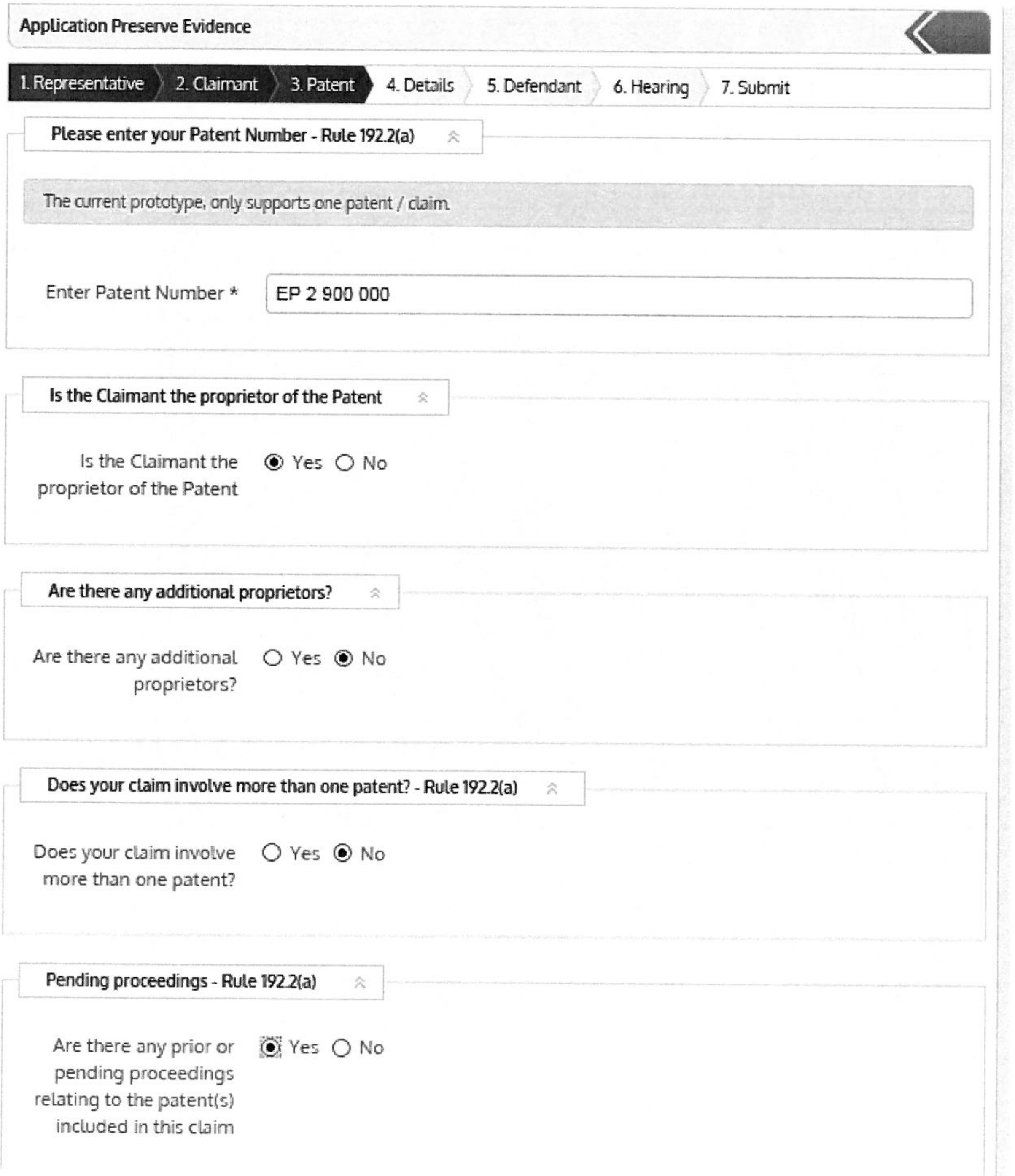
Title * Mr

First Name

Last Name

NEW: The system will now automatically email the claimant and defendant / defendant's representative.

Claimant's e-mail address *



Please enter your Patent Number - Rule 192.2(a)

The current prototype, only supports one patent / claim.

Enter Patent Number * EP 2 900 000

Is the Claimant the proprietor of the Patent

Is the Claimant the proprietor of the Patent ● Yes ○ No

Are there any additional proprietors?

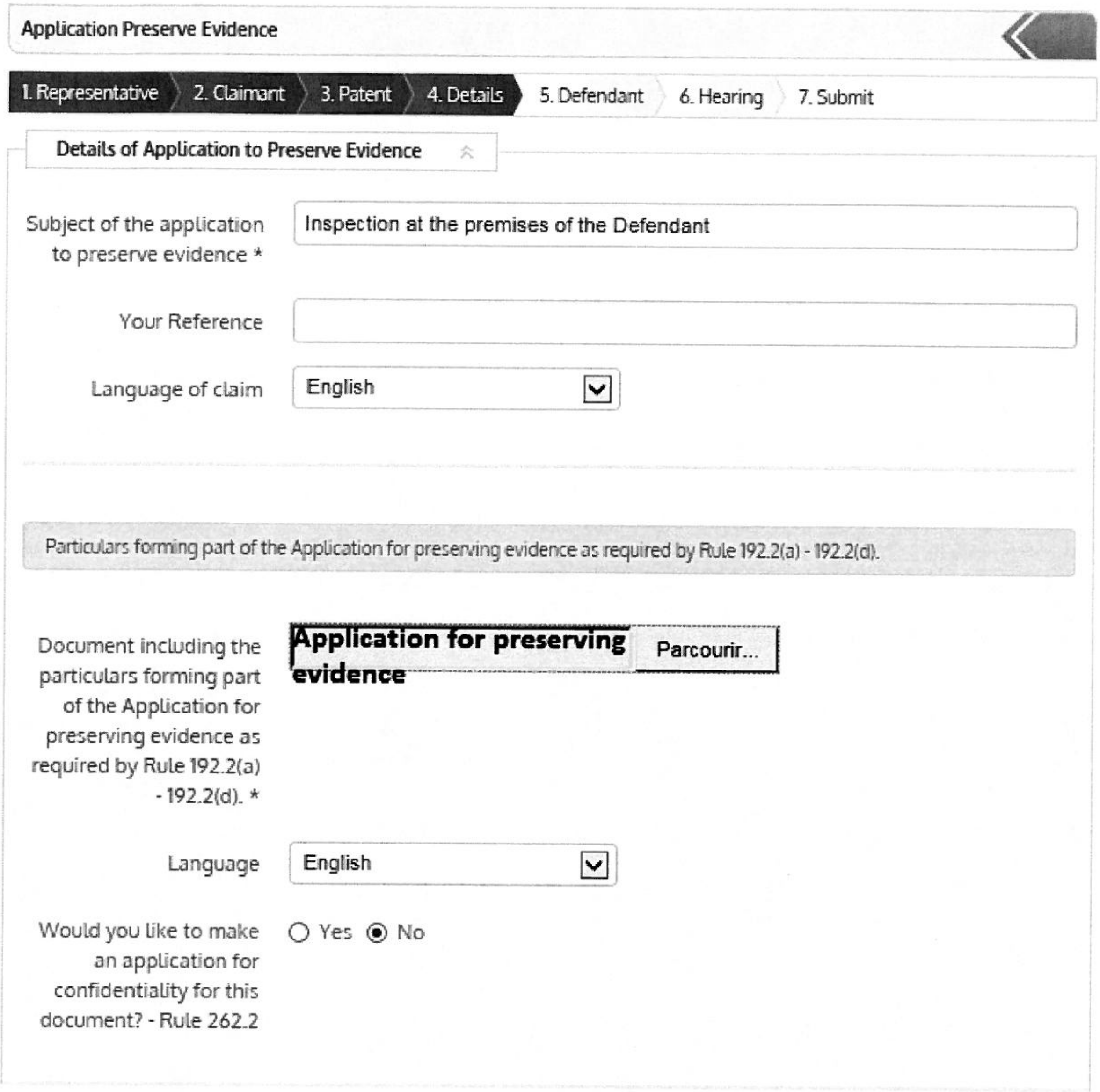
Are there any additional proprietors? ○ Yes ● No

Does your claim involve more than one patent? - Rule 192.2(a)

Does your claim involve more than one patent? ○ Yes ● No

Pending proceedings - Rule 192.2(a)

Are there any prior or pending proceedings relating to the patent(s) included in this claim ● Yes ○ No



Details of Application to Preserve Evidence

Subject of the application to preserve evidence *: Inspection at the premises of the Defendant

Your Reference:

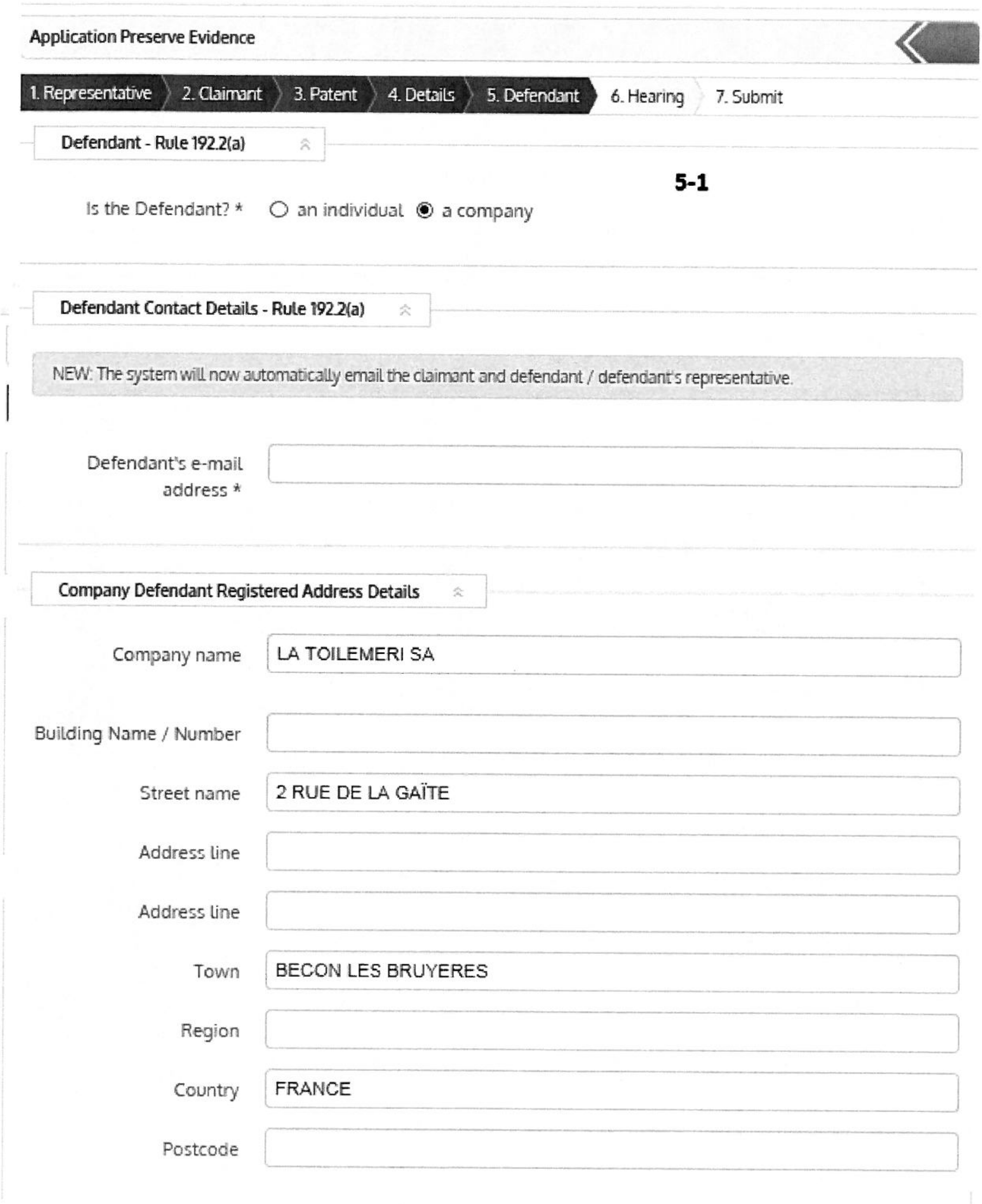
Language of claim: English

Particulars forming part of the Application for preserving evidence as required by Rule 192.2(a) - 192.2(d).

Document including the particulars forming part of the Application for preserving evidence as required by Rule 192.2(a) - 192.2(d). *: **Application for preserving evidence**

Language: English

Would you like to make an application for confidentiality for this document? - Rule 262.2: ○ Yes ● No



5-1

Defendant - Rule 192.2(a)

Is the Defendant? * ○ an individual ● a company

Defendant Contact Details - Rule 192.2(a)

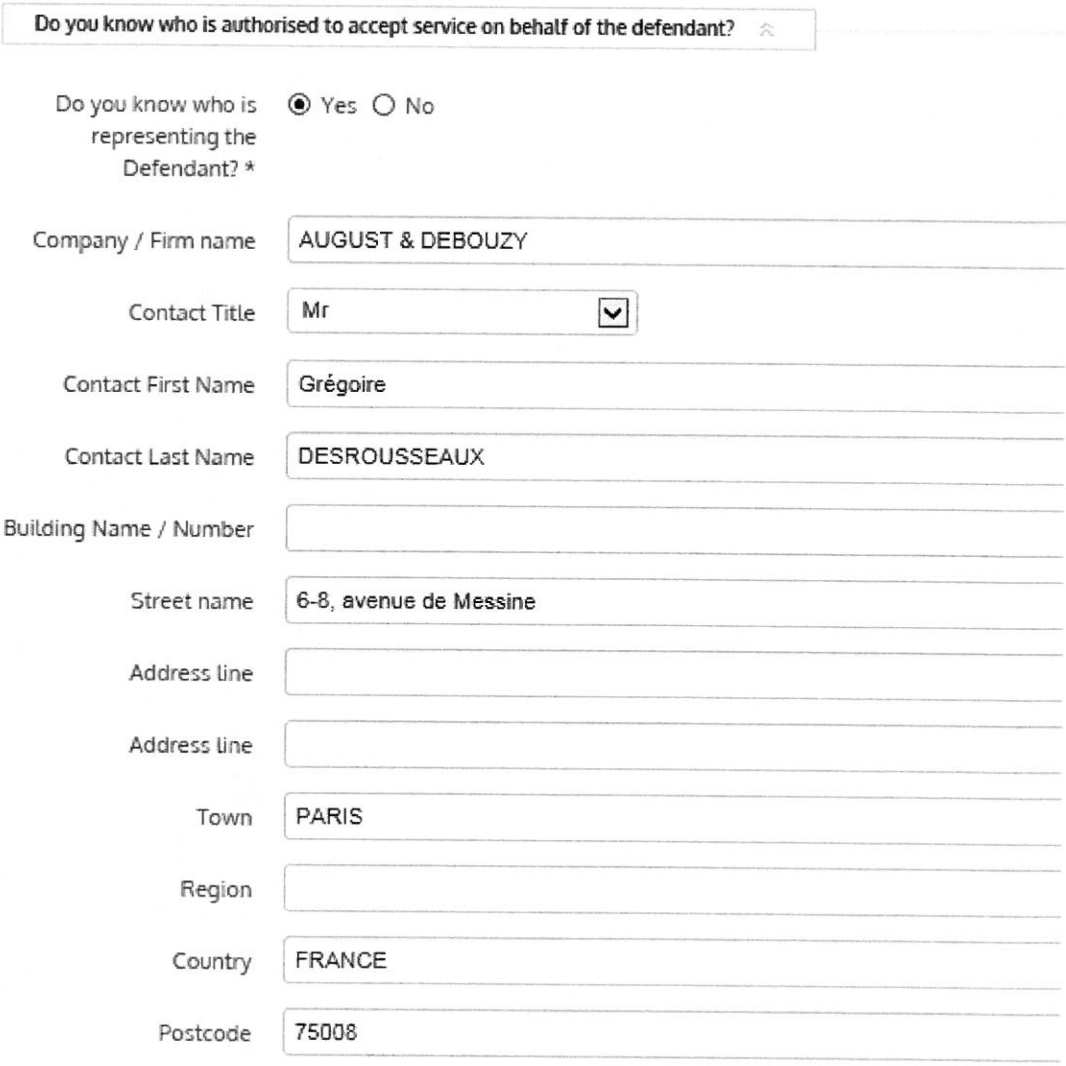
NEW: The system will now automatically email the claimant and defendant / defendant's representative.

Defendant's e-mail address *

Company Defendant Registered Address Details

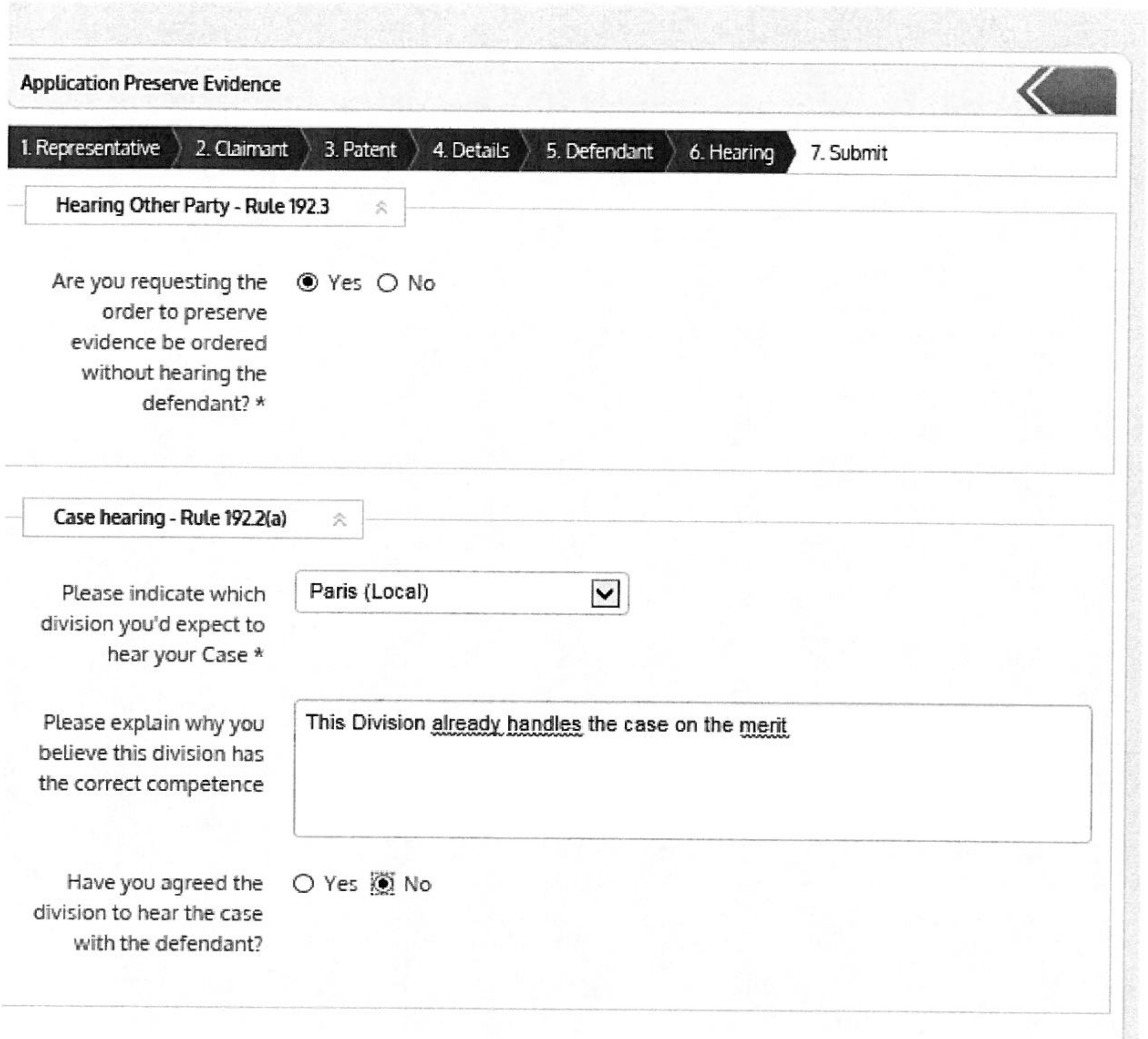
Company name	LA TOILEMERI SA
Building Name / Number	
Street name	2 RUE DE LA GAÏTE
Address line	
Address line	
Town	BECON LES BRUYERES
Region	
Country	FRANCE
Postcode	

5-2



Do you know who is authorised to accept service on behalf of the defendant?

Do you know who is representing the Defendant? *	● Yes ○ No
Company / Firm name	AUGUST & DEBOUZY
Contact Title	Mr
Contact First Name	Grégoire
Contact Last Name	DESROUSSEAUX
Building Name / Number	
Street name	6-8, avenue de Messine
Address line	
Address line	
Town	PARIS
Region	
Country	FRANCE
Postcode	75008



Hearing Other Party - Rule 192.3

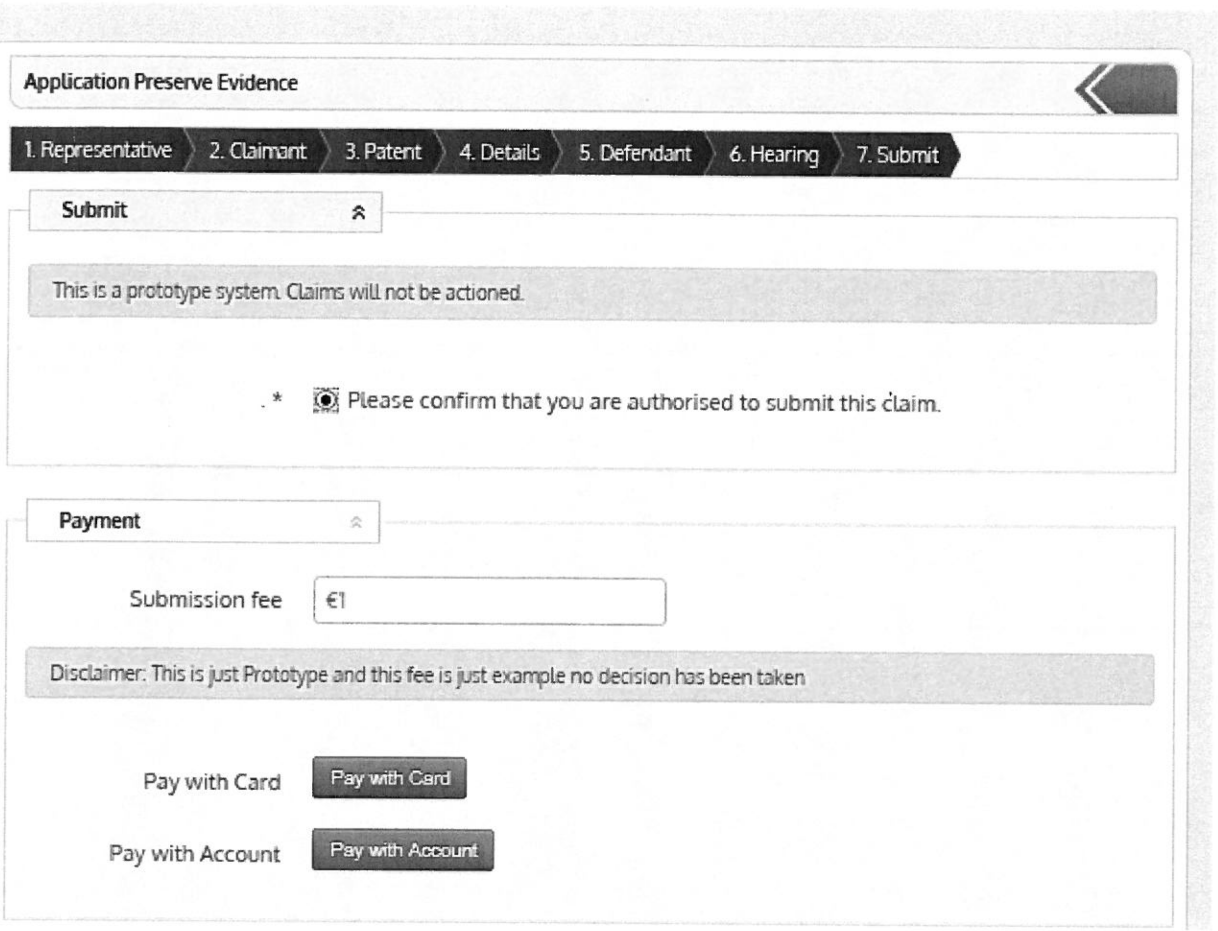
Are you requesting the order to preserve evidence be ordered without hearing the defendant? * ● Yes ○ No

Case hearing - Rule 192.2(a)

Please indicate which division you'd expect to hear your Case * Paris (Local)

Please explain why you believe this division has the correct competence: This Division already handles the case on the merit

Have you agreed the division to hear the case with the defendant? ○ Yes ● No



This is a prototype system. Claims will not be actioned.

.* ● Please confirm that you are authorised to submit this claim.

Submission fee: €1

Disclaimer: This is just Prototype and this fee is just example no decision has been taken

29 December 2014
Application for preserving evidence

By e-mail

Docket No 2015/01
UNIFIED PATENT COURT
PARIS LOCAL DIVISION

3 ABRASIVE (3A)
(A company with its registered office located in the USA)
v/
La Toilemeri SA
(A company with its registered office located in France)

APPLICATION FOR PRESERVING **EVIDENCE AND REQUEST FOR AN ORDER TO INSPECT PREMISES WITHOUT HEARING THE OTHER PARTY**

FOR INFRINGEMENT OF EP No. 2900000

Claimant's Address for Service:

Mr. Axel Casalonga, Partner of CASALONGA & Associés, is authorized to accept service in relation to these proceedings.

8, avenue Percier
75008 PARIS
France
a.casalonga@casalonga.com

2/ Mr. Martin Koehler, Partner of **REIMANN OSTERRIETH KÖHLER HAFT** is authorized to accept service in relation to these proceedings.

Steinstrasse 20
40212 Düsseldorf
Germany
Martin.Koehler@rokh-ip.com

Defendant's Address for Service:

1/ Mr Grégoire Desrousseaux of Auguste & Debouzy is authorized to accept service in relation to these proceedings.

6-8 avenue de Messine – 75008 Paris France

gdesrousseaux@augdeb.com

2/ Mr Kay Rupprecht of   Meissner Bolte & Partner GbR
is authorized to accept service in relation to these proceedings.

Widenmayerstrasse 47, 80538 Munich Germany

ru@mbp.de

1. Competence of the Paris local division of the Court of First Instance and the Full panel

1- As the Applicant has commenced infringement proceedings on the merits against the Defendant before the local division of the Court of First Instance in Paris, this application for preserving evidence and to inspect premises shall be brought in the same jurisdiction.

2- Besides, the Applicant respectfully requests that the Application be transmitted to the full Panel for examination notwithstanding the fact that, in view of the extreme urgency, the application should have been handled by the standing judge according to Rule 194-4.

3- The reason is that it is the first time in the history of the Unified Patent Court that such an extremely urgent request for an order of preservation of evidence and inspection of premises without hearing the other party is filed.

It is therefore appropriate that the full panel hears this urgent request.

2. Summary of facts

4- In the Statement of claim filed by the Applicant on 13 October 2014, it has been indicated that the Defendant had posted on its website, a photograph of a product which is considered by the Applicant as manufactured according to the method claimed in its European patent N° 2900000.

5- The website of the Defendant also contained marketing statements indicating that the advantages of the product were:

- an excellent lateral bearing of the abrasive patches,
- a reinforced securing of the abrasive patches onto the support.

In addition, the website included a page offering the sale of all products mentioned on the site including the previously mentioned product.

6- The Statement of claim was filed on the basis of that evidence.

7- In the Statement of defense the Defendant has challenged the infringement stating that the product could be manufactured by another method.

8- It is therefore of utmost importance that evidence relating to the effective manufacturing process be preserved.

9- The Applicant knows that the Defendant manufactures its products in its factory at Bécon les Bruyères 2 rue de la Gaieté near Paris.

10- For all those reasons it is highly important that complete evidence on the alleged infringement be obtained urgently.

3. Infringement Analysis (Statement of claim)

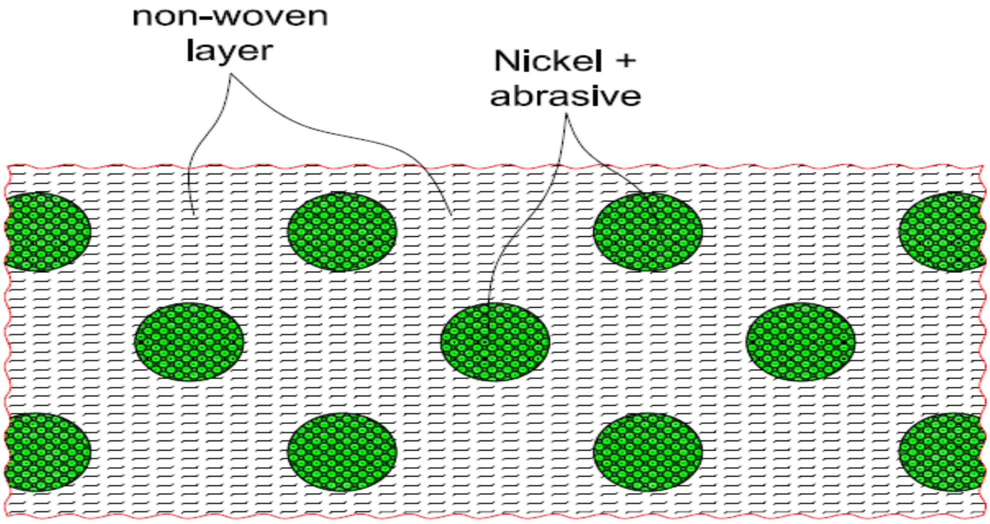
11- The manufactured product is an abrasive member comprising:

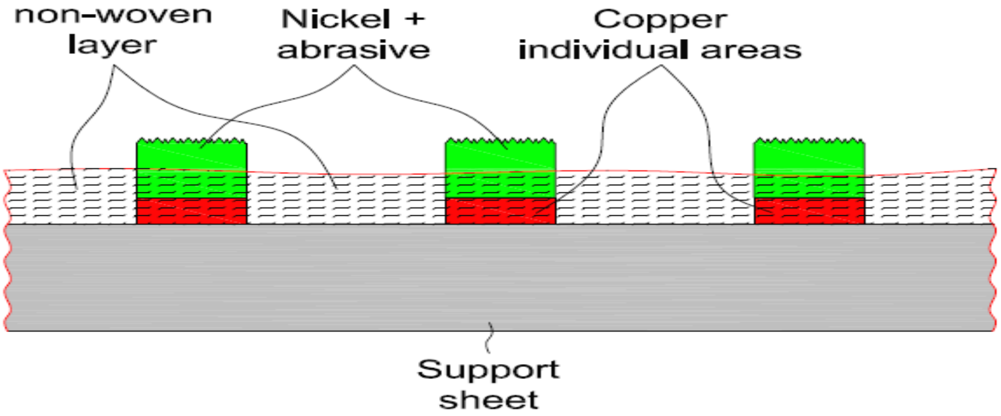
- a support sheet

- a plurality of metal patches made of nickel having particulate abrasive therein on their upper surfaces,

- said metal patches being attached to the support via individual areas of metallic copper in direct contact with the support sheet

- a layer of a non-woven fabric comprising polyamide fibers being stuck to the support between the metal patches.





In other words, the infringing product also has the two different metal layers recited in the method claim of EP 2900000.

12- In its defence, the Defendant has argued that there was no evidence that its product had been manufactured by a method which would reproduce the essential features of claim 1 of the patent of the Applicant.
In particular, the Defendant has argued that the abrasive patches of its product could have been attached to the support by a process of chemical deposition.

13- The Applicant maintains that the product of the Defendant has abrasive patches where the diamond abrasive particles are embedded in Nickel as an external metal layer which is secured to another metal layer made of copper electrodeposited on the support during the manufacturing process.

14- The Applicant has already explained to this court that the provision of two layers of different metals, particularly with patches of Nickel electro-deposited on internal individual areas of copper deposited on the support, had the advantage of a very strong binding of the abrasive patches. A mere chemical deposition of nickel layer would not permit to obtain such a result.

15- The only way to prove that the products of the Defendant are effectively manufactured according to a method reproducing the features of the claim of the Patent is:

- to inspect the Defendant's place of manufacture at 2 rue de la Gaieté Bécon les Bruyères noting that it is specified on the Defendant's website that the products at issue are manufactured in such place;
- and to describe the manufacturing process of the Defendant product.

16- Indeed, once the product is manufactured, it is almost impossible to determine whether or not the Nickel patches have been electro-deposited.

4. The measures requested

17- It is requested that an order be issued authorizing preservation of evidence and inspection at the premises of the Defendant, in accordance with French national law, at Bécon les Bruyères 2 rue de la Gaieté near Paris where there is a strong suspicion that the product is manufactured..

18- The preservation of evidence should include a description of the alleged infringing manufacturing process. It should be particularly described how the metal patches are attached to the support and particularly evidenced that two different metals are provided, the Nickel being deposited by electro-deposition on individual copper areas attached to the support.

All technical and commercial documents relating to the alleged infringing manufacturing process should be photocopied.

19- The physical seizure of three samples of the product manufactured according to the alleged infringing method should be authorized with payment of their price, one sample to be handed to the Court and the two other to be handed to the Representatives of the Applicant.

20- Preservation and disclosure of any password for accessing to any such documents stored in digital medias and computer systems should be ordered.

5. The carrying out of the requested measures

21- The person who will carry out the requested measures should be independent from the parties and absolutely impartial and neutral.

In addition this person should be able to understand the technical aspects of the measures to be carried out.

22- The Applicant presents to the court three names of European Patent Attorneys, Mrs EPI-1, EPI-2 and EPI-3.

23- Each of these persons has made a declaration as to its technical capacities, independence and impartiality.

None of them has had knowledge of the patent involved, be it by filing or granting operations, which means they are completely independent.

24- This independence is also confirmed by the fact that they are all bound by the Code of Conduct of the European Patent Institute (EPI) an official organisation created by the European Patent Convention (EPC).

None of them has any connection with both the Applicant and the Defendant which means they are all completely impartial and neutral in the present action.

25- The Applicant respectfully requests the court to appoint one of those three persons to carry out the requested measures and to draft a written report of the entire operation.

In addition, the Applicant respectfully requests that a bailiff, chosen by the Applicant and having jurisdiction at the place of the requested inspection, could accompany the person nominated by the court to carry out the measures so as to certify the exactness of the report, according to French national law.

26- The bailiff should also be authorized to ask police forces to assist him in case of any difficulty according to French national law.

27- No employee or director of the Applicant should be allowed to be present at the execution of the measures.

28- However, in order to facilitate the execution of the measures it is respectfully requested that one of the Representatives of the Applicant, Mr Axel Casalonga as European Patent Attorney, be present to assist the bailiff and the person in charge of execution of the measures.

Indeed the Representative of the Applicant is fully instructed of the details of the patent and of the alleged infringement and can best help the person in charge to find out the necessary evidence. As European Patent Attorney, the Representative of the Applicant is also bound by the Code of Conduct of the European Patent Institute (EPI) an official organisation created by the European Patent Convention (EPC), and shall refrain to communicate to the Applicant any information he could obtain during the execution of the measures that would go beyond the report drafted by the person in charge of the measures.

The Representative of the Applicant however should not by himself proceed to any investigation or interrogation, this being reserved strictly to the person in charge of the measures assisted if necessary by the bailiff.

29- As a further request, an IT specialist should be authorized to be present during the operations so as to facilitate investigations of the person in charge with the execution of the measures in case information and/or documents would be on memories of computers or of the IT system of the Defendant and difficulties of access for example by passwords would occur.

30- The Applicant proposes in this regard Mr Pomme who has signed a declaration certifying that he is not an employee of the Applicant and has no connection with the Applicant. His declaration also contains an engagement not to disclose any information he could have obtained during the execution of the measures.

31- Before the execution of the measures at the premises of the Defendant, notice of the order will be given to the Defendant by the person in charge with the execution of the measures.

6. Confidential information

32- If the person in charge of the execution of the measures considers that certain information obtained, while being useful to prove infringement, are of confidential nature, or if the Defendant or its employees declare during the execution of the measures that certain information is confidential, all said confidential information shall be kept in a sealed envelope by the person in charge of the execution of the measures and handed to the Court who shall determine that confidential information can be transmitted, only to the Representatives of the parties and to two specially named persons of the Applicant i.e. the chief of the Patent Department Mr. X and a technical manager Mr. Y after those two persons will have signed a declaration under oath not to take any copy, and not to disseminate the information contained in the confidential documents for a period of 4 years.

7. Order without hearing the Defendant

33- The Applicant requests that the order to preserve evidence and to inspect premises be granted without hearing the Defendant in accordance with Rule 197.

34- As explained above, an essential aim of the requested measures is to demonstrate that nickel patches are electro-deposited on copper areas attached to the support when manufacturing the product.

This step of the manufacturing process is made in a batch process at the premises of the Defendant.

35- If the Defendant would be made aware of the inspection, it would be extremely easy for the Defendant simply to disconnect the reactor where the support is immerged during electro-deposition or even to stop operating the process. This would not allow the Defendant to obtain the necessary evidence.

36- In other words, if the Defendant is informed in advance of the execution of the measures, the chances that no evidence could be found would be extremely high.

37- Besides, the Applicant has recently discovered in the press that the Defendant is being accused of destruction of evidence in an ongoing case (two articles from a French newspaper are annexed to this Application). Hence, the Defendant may act in the same way in this case and try to resist to any attempt to obtain evidence of the effective manufacturing method.

38- Therefore, hearing the Defendant would cause *“a demonstrable risk of evidence being destroyed”* as provided in Rule 197.

39- The applicant considers that no security for any compensation is necessary. As a matter of fact the requested measures will cause no injury to the defendant. The requested measures will not necessitate to stop or to slow down the manufacturing process. It will be sufficient to observe how the process is carried out and to make a description thereof.

40- Under the special circumstances of the presently requested measures, no security will therefore be ordered.

8. Oral hearing

41- The Applicant respectfully requests that the order be given orally at the end of an oral hearing held without the presence of the Defendant.

42- The order will be given in writing shortly after the oral hearing.

9. Payment of the fee for the Application for preserving evidence

43- Proof of payment of the fee for the Application for preserving evidence is enclosed.
The Applicant assumes that no additional fee for the request for the order of inspection is required since the preservation of evidence is made precisely by the inspection.

FOR ALL THESE REASONS, MAY IT PLEASE THE COURT TO:

43- authorize preservation of evidence and inspection at the premises of the Defendant at Bécon les Bruyères 2 rue de la Gaieté;

44- authorize this preservation of evidence and inspection at the premises of the Defendant to be carry out by Mr , European Patent attorney, assisted by a bailiff and may request assistance of the police forces if necessary (hereafter “the person in charge of the measures”);

45- authorize the person in charge of the measures to carry out in the Defendant’s premises, a detailed description of the alleged infringing manufacturing process and in particular how the metal patches are attached to the support and evidence that two different metals are provided, nickel patches being deposited by electro-deposition on individual areas of copper attached to the support;

46- authorize the person in charge of the measures to seize physically three samples of the product manufactured according to the alleged infringing method, one sample to be handed to the Court and the two other to be handed to the representatives of the Applicant, after having paid the price asked;

47- authorize the person in charge of the measures to carry out any useful searches to establish the materiality, the nature, the scope, the origin and the destination of the infringement, and to record in his report not only the respondents' statements, but also every statements made in the course of the operation;

48- authorize the person in charge of the measures to obtain and consult promotional material, papers, books, catalogs, leaflets, price lists, vouchers, order books, invoices, drawings, negatives, correspondence, accounting documents and any other documents or computer files that he may find during the operation which will establish the evidence, the materiality, the nature, the scope, the origin and the destination of the infringement;

49- order the preservation and disclosure of any password for accessing to any such documents stored in digital medias and computer systems;

50- authorize the person in charge of the measures to submit and sign *ne varietur* every accounting documents, books and registers and generally, any peculiar documents to establish the materiality, the nature, the scope, the origin and the destination of the infringement;

51- authorize the person in charge of the measures to seize by way of description, photocopies or photographs, every documents consulted during his mission, such as promotional material, papers, books, catalogs, leaflets, price lists, vouchers, order books, invoices, drawings, negatives, correspondence, accounting documents, computer files that determine the materiality, the nature, the scope, the origin and the destination of the infringement. Two copies of each shall be transmitted to the Court and to the Defendant in order to allow it to assert its rights;

52- authorize the person in charge of the measures to carry out a physical seizure of every documents consulted during its mission such as promotional material, order books and accounting documents that determine the materiality, the nature, the scope, the origin and the destination of the infringement. Two copies of each shall be transmitted to the full panel and to the Defendant in order to allow it to assert its rights;

53- authorize that the proofs of photocopies or photographs that might be taken during the operation be transmitted to the Defendant only after the establishment of the report by the person in charge of the execution of the measures;

54- order that confidential information obtained during the operation shall not be transmitted to the Applicant but handed to the Court who shall determine how and to whom said information may be transmitted;

55- authorize the presence of Mr. Axel Casalonga, Representative of the Applicant, noting that :

- Mr. Axel Casalonga shall refrain to communicate to the Applicant any information he could obtain during the execution of the measures that would go beyond the report drafted by the person in charge of the measures;

- Mr. Axel Casalonga shall not by himself proceed to any investigation or interrogation, this being reserved strictly to the person in charge of the measures assisted if necessary by the bailiff;

56- authorize the presence of Mr Pomme, IT specialist, during the operations so as to facilitate investigations of the person in charge with the execution of the measures;

57- authorize the person in charge of the measures to be assisted by a photographer in order to take any photographs necessary for the accomplishment of its mission;

58- order that before the execution of the measures at the premises of the Defendant, notice of the order shall be given to the Defendant by the person in charge of the measures.

59- order that the measures ordered will be carried out within two months of the decision;

60- order that the Court will be referred to in case of difficulties, but only after the measures being carried out and stamped.

Enclosures:

Annex 1: Declarations under oath of Mr EPI 1
Annex 2: Declarations under oath of Mr EPI 2
Annex 3: Declarations under oath of Mr EPI 3
Annex 4: Declarations under oath of Mr Pomme (IT specialist)
Annex 5: Article dated October 22, 2014 from a French newspaper “Le Canard Déchainé » 1
Annex 6: Article dated December 3, 2014 from a French newspaper “Le Canard Déchainé »

DECISION OF THE COURT ON APPLICANT'S APPLICATION FOR PRESERVING EVIDENCE AND REQUEST FOR AN ORDER TO INSPEC PREMISES WITHOUT HEARING THE OTHER PARTY

Having heard the applicant's explanations and in view of the facts and evidence relied on by the Applicant,

the Court:

- authorizes preservation of evidence and inspection at the premises of the Defendant at Bécon les Bruyères (address);

- authorizes this preservation of evidence and inspection at the premises of the Defendant to be carry out by Mr EPI-1, European Patent attorney, assisted by a bailiff and the police forces if necessary (hereafter "the person in charge of the measures");

- authorizes the person in charge of the measures to carry out in the Defendant's premises, a detailed description of the manufacturing process of the alleged infringing product and in particular how the metal patches are attached to the support and evidence that two different metals are provided, nickel patches being deposited by electro-deposition on individual areas of copper attached to the support;

- authorizes the person in charge of the measures to seize physically three samples of the alleged infringing product, one sample to be handed to the Court and the two other to be handed to the representatives of the Applicant, after having paid the price asked;

- authorizes the person in charge of the measures to submit at any time, including at the beginning of the measures, a copy of the bailiff report, annexed to this Request, in order to provoke a reaction of the person being questioned;

- authorizes the person in charge of the measures to carry out any useful searches to establish the materiality, the nature, the scope, the origin and the destination of the infringement, and to record in his report not only the respondents' statements, but also every statements made in the course of the operation;

- authorizes the person in charge of the measures to obtain and consult promotional material, papers, books, catalogs, leaflets, price lists, vouchers, order books, invoices, drawings, negatives, correspondence, accounting documents and any other documents or computer files that he may find during the operation which will establish the evidence, the materiality, the nature, the scope, the origin and the destination of the infringement;

- orders the preservation and disclosure of any password for accessing to any such documents stored in digital medias and computer systems;

- authorizes the person in charge of the measures to submit and sign *ne varietur* every accounting documents, books and registers and generally, any peculiar documents to establish the materiality, the nature, the scope, the origin and the destination of the infringement;

- authorizes the person in charge of the measures to seize by way of description, photocopies or photographs, every documents consulted during his mission, such as promotional material, papers, books, catalogs, leaflets, price lists, vouchers, order books, invoices, drawings, negatives, correspondence, accounting documents, computer files that determine the materiality, the nature, the scope, the origin and the destination of the infringement. Two copies of each shall be transmitted to the Court and to the Defendant in order to allow it to assert its rights;

- authorizes the person in charge of the measures to carry out a physical seizure of every documents consulted during its mission such as promotional material, order books and accounting documents that determine the materiality, the nature, the scope, the origin and the destination of the infringement. Two copies of each shall be transmitted to the full panel and to the Defendant in order to allow it to assert its rights;

- authorizes that the proofs of photocopies or photographs that might be taken during the operation be transmitted to the Defendant only after the establishment of the person in charge of the execution of the measures' report;

- orders that confidential information obtained during the operation shall not be transmitted to the Applicant but only to the Representatives of the parties and exclusively to the chief of the Patent Department of the Applicant, Mr. X and the technical manager of the Applicant, Mr. Y, after those two persons have signed a declaration under oath not to take any copy, and not to disseminate the information contained in the confidential documents for a period of 4 years;

- authorizes the presence of Mr. Axel Casalonga, Representative of the Applicant, noting that :

    - Mr. Axel Casalonga shall refrain to communicate to the Applicant any information he could obtain during the execution of the measures that would go beyond the report drafted by the person in charge of the measures;

    - Mr. Axel Casalonga shall not by himself proceed to any investigation or interrogation, this being reserved strictly to the person in charge of the measures assisted if necessary by the bailiff;

- authorizes the presence of Mr Pomme, IT specialist, during the operations so as to facilitate investigations of the person in charge with the execution of the measures;

- authorizes the person in charge of the measures to be assisted by a photographer in order to take any photographs necessary for the accomplishment of its mission;

- orders that before the execution of the measures at the premises of the Defendant, notice of the order shall be given to the Defendant by the person in charge of the measures.

- orders that the measures ordered will be carried out within two months of the present decision;

- orders that the Court will be referred to in case of difficulties, but only after the measures being carried out and stamped.

Le Canard déchaîné

La Toilemeri ne rit plus : Deux salariés portent plainte après avoir été menacés de licenciement

Le Canard Déchainé.fr | 22.10.2014 à 9h29
Par Camille Lemonnier

Une source judiciaire affirme mercredi 22 octobre que deux salariés de l'entreprise La Toilemeri, spécialisée dans les techniques d'abrasif industriels, ont déposé plainte pour «harcèlement moral » .

Les dirigeants de la société auraient fait pression sur ces employés pour qu'ils détruisent divers documents susceptibles d'apporter la preuve de la responsabilité de l'entreprise dans le cadre de procès en cours.

Devant leurs refus, les dirigeants les auraient menacés de licenciement.

La plainte a été déposée au parquet de Nanterre qui a ouvert une enquête préliminaire en raison « d'indices graves et concordants ». L'enquête a été confiée à l'équipe de l'inspecteur Colombo en charge du SRPJ des Hauts de Seine. Une source interne à l'entreprise parle quant à elle « d'éléments de preuve accablants ».

Bouchée à l'émeri, l'entreprise n'a pas souhaité réagir à ces accusations mais gageons que sa défense sera abrasive …

Le Canard
déchaîné

La Toilemeri does not laugh anymore: two employees lodge a complaint after threats of dismissal

Le Canard Déchainé.fr | 10.22.2014 at 9.29am

By Camille Lemonnier

An unofficial source reveals today that two employees of the company La Toilemeri, specialized in industrial abrasives, have filed a complaint for “moral harassment”.

The company’s managerial staff would have put pressure on the employees in order that they destroy multiple documents that would prove the company’s liability in an ongoing case.

Facing their refusal, the managerial staff would have threatened them of dismissal.

The complaint has been lodged with the public prosecutor of Nanterre, who opened an investigation because of “serious and major clues”. Detective Columbo’s team from the Hauts de Seine’s SRPJ has been put in charge of the investigation. As for a source from the company, it tells that there are “overwhelming evidences”.

For now, the company did not respond to these allegations but we can hope that its defense will be abrasive …

Le Canard déchaîné

Harcèlement moral chez La Toilemeri : tel est pris qui croyait prendre.

La Canard Déchainé.fr | 03.12.2014 à 10h41
Par Camille Lemonnier

Décidément, il ne fait pas bon se frotter à l’équipe dirigeante chez La Toilemeri !

Il y a quelques semaines nous vous parlions du dépôt de plainte de deux salariés ayant subi des menaces de licenciement suite à leur refus de faire disparaître des documents internes susceptibles de compromettre gravement la défense de l’entreprise dans une enquête en cours la visant.

Et hier, rebelote, un troisième salarié aurait également déposé plainte, cette fois ci pour « tentative d’intimidation ».

Spécialiste des techniques d’abrasifs industriels, une chose est sûre, la société La Toilemeri est engagée dans une guerre d’usure …

Le Canard
déchaîné

Moral harassment at La Toilemeri: it’s the biter bit

La Canard Déchainé.fr | 12.03.2014 à 10.41am
By Camille Lemonnier

It is clearly not a good idea to face La Toilemeri’s managerial staff!

A few weeks ago, we told you about two complaints that have been lodged by two employees who have been threatened of dismissal because they have refused to eliminate company’s documents that would be highly compromising for its defense in an ongoing investigation.

And, yesterday, here we go again, a third employee filed a complaint, this time for “intimidation attempts”.

Specialized in industrial abrasives, one thing we know for sure is that La Toilemeri is probably worn out …

Declaration under oath

The undersigned Albert EPI 1, noting that a false declartion is punihable by law, declare the following

I graduated from the technical University of Munich (Germany) in 1980
My major was chemical engineering and material science.
In 1990 I passed successfully the European Qualification Examination (EQE) and became European Patent Attorney.

I am presently chief of the Patent Departement of Berg AG, a company located in Munich and specialized in manufacturing metallic pieces for the automotive industry.
In this capacity I have frequently to do with inventions dealing with electro-deposition of metallic layers on various supports.

I have no connection with 3 A, even if I know this company.
I did not study EP N° 2 900 000 before being contacted by Mr Martin Köhler, representative of 3 A in an infringement action engaged before the UPC.

I also have no connection with La Toilemeri which I do not know.

My company does not work with either one of those companies and has no commercial connection with them.

I am ready to accept the task of executing measures of preservation of evidence in the action engaged by 3 A against La Toilemeri before the UPC.

I shall refrain from disclosing any information I would obtain during the execution of these measures or otherwise relating to this court action, in accordance with the provisions of confidentiality and independance of the Code of Conduct of the EPI

Done in Munich

On 19 December 2014

Declaration under oath

The undersigned Jean EPI 2, noting that a false declartion is punihable by law, declare the following

I graduated from Ecole de Physique et Chimie de Paris in 1975
My major was physics and material science.
In 1980 I passed successfully the European Qualification Examination (EQE) and became European Patent Attorney.

I am presently senior partner of PI & associés, a patent and trademark law firm located in Lyon (France) In this capacity I have from time to time to do with inventions dealing with electro-deposition of metallic layers on various supports.

I have no connection with 3 A, even if I know this company. In particular my firm does not have 3 A among its clients and I do not know any case where my firm would have had a case against 3 A, either before the EPO or before a french court.
I did not study EP N° 2 900 000 before being contacted by Mr Axel Casalonga, representative of 3 A in an infringement action engaged before the UPC.

I also have no connection with La Toilemeri which I do not know.

I am ready to accept the task of executing measures of preservation of evidence in the action engaged by 3 A against La Toilemeri before the UPC.

I shall refrain from disclosing any information I would obtain during the execution of these measures or otherwise relating to this court action, in accordance with the provisions of confidentiality and independance of the Code of Conduct of the EPI

Done in Lyon

On 20 December 2014

Jepi

Declaration under oath

The undersigned Philippe EPI 3, noting that a false declaration is punishable by law, declare the following

I graduated from the University of Strasbourg (France) in 1985
My major was non organic chemistry and material science.
In 1999 I passed successfully the European Qualification Examination (EQE) and became European Patent Attorney.

I am presently employed as Patent Attorney in the firm Brevets et PI, a patent and trademark law firm located in Paris (France) In this capacity I have from time to time to do with inventions dealing with electro-deposition of metallic layers on various supports.

I have no connection with 3 A, even if I know this company. In particular my firm does not have 3 A among its clients and I do not know any case where my firm would have had a case against 3 A, either before the EPO or before a french court.
I did not study EP N° 2 900 000 before being contacted by Mr Axel Casalonga, representative of 3 A in an infringement action engaged before the UPC.

I also have no connection with La Toilemeri which I do not know.

I am ready to accept the task of executing measures of preservation of evidence in the action engaged by 3 A against La Toilemeri before the UPC.

I shall refrain from disclosing any information I would obtain during the execution of these measures or otherwise relating to this court action, in accordance with the provisions of confidentiality and independance of the Code of Conduct of the EPI

Done in Paris

On 22 December 2014

ATTESTATION

Je soussigné, Jules POMME, conscient du fait qu'une fausse déclaration constitue un délit puni par la loi, atteste ce qui suit :

- Je suis domicilié 2 rue de Montrouge, 92220 BAGNEUX.
- J'ai étudié les technologies informatiques à l'Université de Nanterre où j'ai obtenu un master en informatique.
- J'exerce en tant qu'expert indépendant dans le domaine de l'informatique et j'assiste différentes sociétés dans ce domaine.
- Je n'ai jamais travaillé avec la société 3A et je ne connais pas la société LA TOILEMERI.
- J'ai été contacté par Axel CASALONGA, Conseil en Propriété Industrielle et Mandataire en Brevets Européens pour assister à une opération d'inspection dans les locaux de la société LA TOILEMERI ordonnée par la Juridiction Unifiée du Brevet.
- Je m'engage formellement à ne divulguer aucune information que je pourrais obtenir pendant l'exécution de ces mesures et à détruire tout document ou support informatique au cas où je serais amené à les copier au cours de ces opérations.

Fait à Bagneux,

Le 22 décembre 2014

Report on proceedings for preserving evidence and inspection at the premises

Date: On [TBC]
At 10.05 pm

Acting by virtue of an order to preserve evidence granted by the local division of the Court of First Instance in Paris (herein referred to as “the Court”) on [date TBC]

Upon request of 3 ABRASIVE (3A), a company with its registered office located in the USA, holding a European Patent EP 2900000 B1,

I, Albert EP-1, European Patent Attorney, duly appointed by the Court, certifies that I operated as follows:

I came to the premises of Toile Emeri, located [TBC] at Bécon les Bruyères.

I was accompanied by Mr Jacques Battot, a bailiff having jurisdiction at the place of the premises, Mr Axel Casalonga, Representative of 3A, Mr Pomme, IT specialist, and Mr René Spaur, Police Officer.

I knocked the door, a lady opened, I asked her to meet a director. Mr Collé, CEO of Toile Emeri, came in. I stated my identity to him.

I explained him my mission and gave him a copy of the Order for preserving evidence and inspection of the premises. I offered him to take time to read it and contact his attorney, if needed.

Fifteen minutes later, he invited us to come into the factory.

I asked to be provided with the abrasive products announced on the website of the company.
Mr Collé brought me three different products.

Upon indication of Mr Casalonga, I looked more carefully at one of them.

This product presents a support. Said support bears some hard metallic patches. A layer of nonwoven fabric is disposed between the metallic patches, partially filling the voids between the patches. I put my finger on the upper surfaces of the metallic patches, moved it along these surfaces, and was able to note that these upper surfaces of the metallic patches are abrasive.

I requested to see the manufacturing process of this product. Mr Collé firstly offered some resistance, and then conducted us to the manufacturing area.

On a workstation, I could see a piece of support sheet of about 1 m^2. One main surface was sprayed with a thin layer of glue, upon which a nonwoven fabric was stuck.

I asked to examine the nonwoven fabric used to cover the upper surface of the support. I could note that it was slightly porous.

A mask was then applied on the two faces of the support sheet, allowing only certain areas of the fabric to be apparent.

I then moved on another place of the manufacturing area where many tanks were located.

With the help of Mr Casalonga, I was able to find out a tank were several pieces of similar support sheets were treated. The treatment was in process at that time.

I could observe the following steps.

The support sheets covered by the nonwoven fabric and the mask that I previously observed are immersed into a first tank containing a solution. A small label at the bottom of the tank mentions : “Cu electrodeposition”.

Mr Robert, apparently in charge of the operation of the tanks, explained to me that the sheets stayed in the tank for a determined duration and were then removed from these first tanks and immersed in other tanks for another treatment. Each tank is equipped with a timer controlling the duration of the switching on and the intensity of the electric current feeding the bath.

I could examine a support sheet which was just extracted from the first tank. I could see the copper deposited in the areas left open by the mask.

For the following stage of the treatment, support sheet is immersed into a second tank containing a solution. A small label at the bottom of this second tank mentions : “Ni electrodeposition”.

Mr Robert explained to me that Nickel is thus electrodeposited on the cupper areas in order to form nickel patches. The support sheets remain in these second tanks for a determined duration. Each tank is equipped with a timer controlling the duration of the switching on and the intensity of the electric current feeding the bath.

Assisted by Mr Casalonga, I could observe that, at the end of the duration programmed on the timer of one tank, the electric current is switched off. A powder which Mr Robert identified for me as being industrial diamond particles is poured manually into the same electrolytic bath. The electric current is then again switched on.

This leads to Nickel with diamond particles embedded herein being deposited upon the nickel patches. Mr Robert explained to me that this is done until the desired thickness of the patches is reached. The duration of the second nickel-electrodeposition step was much shorter than the duration of the first nickel-electrodeposition step.

I was told by Mr Collé, that the durations of the copper deposition as well as the first nickel deposition and the second nickel deposition were part of the know how of the Company and should be treated as highly confidential information. This information is part of annex 1 of my report.

Once the support sheets are finally removed from the second tanks, the mask is removed by stripping off the mask.

I also asked to examine one of the support sheets extracted from a first tank, before its immersion in a second tank and could notice that the copper was attached to the support through the nonwoven fabric.

I asked Mr Robert what are the compositions of the copper-based electrolytic bath and of the nickel-based electrolytic bath. Mr Coppé intervened to say that they are confidential.

I asked to be given a notice describing the process, as well as commercial documents. Mr Collé answered me that this information is confidential and that the files are on a computer.

I could also observe that the sheets after treatment were cut into round pieces having a diameter of 20 cm.

I then carried on the inspection of the premises accompanied by Mr Coppé. I inspected two rooms. The first room was the commercial department. Three computers were in that room.

I tried to have access to the files relating to the manufacturing process in one of the computers. But each time a password was asked. I asked it to the persons present in the room. They did not answer anything. I then asked to Mr Pomme to get through the system to have access to the documents on the computers. He managed to do it after 10 minutes.

I found a file called “Contrat de distribution nouvel abrasif”. I printed it. It was indicated that a company named Leichtschleif (LS) had been selected to distribute the product in Germany, France, Great Britain and in the Netherlands. A price of 50 Euros was fixed for each product of 20 cm diameter, with a royalty of 5% of the selling price for Toile Emeri. A minimum amount of 10 million of products to be sold over 5 years was fixed.

On this computer I was also able to find out two test reports concerning these products. I printed them.

In the conclusion of the first test report it is stated that the presence of the nonwoven fabric increases the resistance to lateral movement of the metal abrasive patches.

In the conclusion of the second test report it is stated that adhesion of the metal abrasive patches on the support is improved when two metals are used (nickel and copper) instead of one (nickel alone).

I also found out a document named “ET-500-02 Procédé de fabrication”. I printed it.

The inspection of the premises was finished at 2.15 pm.

I gave a copy of my report to Mr Collé. My report has been signed and authentified by myself, by Me Battot, bailiff and by Mr Casalonga, European Patent Attorney

Albert EPI 1 Jacques Battot Axel Casalonga

Encl: Extracts of a Distribution contract
Extracts of twoTest reports
Durations of the two process steps
Document named “ET-500-02 Procédé de fabrication”

EXCLUSIVE DISTRIBUTION AGREEMENT

THIS AGREEMENT dated the 13th of November, 2014 (the "Effective Date") is made between:

(1) **La Toilemeri SA,** a French company whose administrative offices are 2 rue de la Gaieté 92400 Bécon les Bruyères ("the **Manufacturer**");

AND

(2) **Leichtschleif** (LS), a company registered in Germany whose address is Petuelring 130 80788 Munich ("the **Distributor**").

Each being a "Party" and together the "Parties".

Whereas, Manufacturer manufactures an abrasive product ("the **Product**") according to the method as listed in Schedule 1.

Whereas, Distributor wishes to obtain an exclusive right to distribute the Product and Manufacturer wishes to grant Distributor such a right, all in accordance with the terms and conditions of this Agreement;

Now, therefore, the Parties hereto, intending to be legally bound, hereby agree as follows:

1. EXCLUSIVE DISTRIBUTION RIGHT GRANTS

The Manufacturer grants to the Distributor and its Group Companies an exclusive right to distribute the Product in Germany, France, Great Britain and the Netherlands.

2. COMMERCIALIZATION

The Distributor shall take its best efforts to sell a minimum amount of 10 million of Products over 5 years.

If this milestone is not achieved within the deadline, Manufacturer shall have the right to convert this Agreement into a non exclusive distribution agreement with a prior notice of 3 months after the expiry of the term.

3. CONSIDERATION FOR GRANT OF EXCLUSIVE DISTRIBUTION RIGHT

In consideration for the grant of the exclusive distribution right:

3.1 Distributor shall pay a 5% royalty on all Net Sales Prices received on a country by country basis.

3.2 Royalties shall be calculated by Distributor, annually in arrears on the Net Sales Price invoiced by the Distributor, such royalties to be payable in Euros on each anniversary of the Effective Date of this agreement ("Year") and shall be paid by Distributor to Manufacturer within a 30 (thirty) day period thereafter. The Parties shall co-operate and take all steps reasonably and lawfully available to them to avoid deducting any withholding or similar tax from any royalty payment payable under this clause 3 and to obtain double taxation relief. If the Distributor or its SubDistributors are required to make any such deduction they shall do so in accordance with applicable law, and it shall provide the Manufacturer with such certificates or other documents as it can reasonably obtain to enable the Manufacturer to obtain appropriate relief from double taxation of the payment in question.

3.3 VAT shall, if applicable, be added to any royalty payment calculated as due under this clause 5.

3.4 Each royalty payment shall be accompanied by a statement setting out the Distributor's calculation of the amount properly due for that Year, and certified by the Distributor to be correct. If no royalty payment is payable a statement to that effect shall be provided within the time by which any such payment would have been due. In the case of monies received by the Distributor from sales in a currency other than Euros, be calculated in the other currency and then converted into Euros at the buying rate of such other currency as quoted by Barclays Bank plc in London as at the close of business on the last business day of the payment period with respect to which the payment is made.

3.5 Distributor shall maintain properly such records and books of accounts as are necessary to verify the calculation of any royalties on reasonable written request with notice shall make available such books and records for inspection and audit by an independent accountant appointed by Manufacturer during normal working hours.

4. TERM AND TERMINATION

4.1 Term

The term of this Agreement shall commence on the Effective Date and, unless earlier terminated as provided in this Article 4, shall continue in full force and effect for the time the Manufacturer manufactures the Product.

4.2 Termination

4.2.1 Termination without cause

The Distributor may terminate this Agreement by not less than three (3) months' prior written notice at any time, provided that the Distributor indemnifies the Manufacturer in respect of any and all claims arising or threatened as a consequence of failing to do so.

4.2.2 Termination for Default

In the event that either party commits a material breach of its obligation under this Agreement and fails to cure that breach within thirty (30) days after receiving written notice thereof, the other party may terminate this Agreement immediately upon written notice to the party in breach.

4.3 Effect of Termination

Upon any expiration or termination of this Agreement the right granted to Distributor under Article 1 shall terminate and neither Distributor nor its Affiliates may make any further use or exploitation of the Product.

Done in two (2) originals, one for each of the Parties

At PARIS
On 13.11. 2014

LA TOILEMERI SA

At Paris
On 13 11. 2014.

LEICHTSCHLEIF

Tests report – July 2014
Confidential – to internal use only

Comparison of the performances of our new abrasive product with the performances of an abrasive product which do not contain nonwoven fabric in the voids between the metallic patches:

Our new product:

- A conductive Kevlar support sheet is covered by fibers of nonwoven polyurethane 4 mm thick.
- Metal patches are attached on the Kevlar sheet through the layer of nonwoven polyurethane.
- Each patch is made of copper fixed on the support by electrodeposition, and of nickel electrodeposited on copper.
- Diamond particles are embedded in the nickel.

Comparative product:

- A conductive Kevlar support sheet.
- Metal patches are attached on the Kevlar sheet through the layer of nonwoven polyurethane.
- Each patch is made of copper fixed on the support by electrodeposition, and of nickel electrodeposited on copper.
- Diamond particles are embedded in the nickel.

Evaluation of resistance to lateral movement:

The samples tested were 15 cm by 10 cm sample size strips formed into belts of abrasive product. They were mounted on a testing machine and rubbed on a granite surface for 5 hours.

After 5 hours, the testing machine was stopped, the belt removed.

Results:

Our product: about 8% of the metal patches have chipped off.
Comparative product: about 25% of the metal patches have chipped off.

Process description-Electrodeposition durations
Confidential – to internal use only

June 2014

In manufacturing our new product, 3 steps of electrodeposition are to be implemented.

The first electrodeposition step is to deposit copper on specific areas of the surface.
The second electrodeposition step is to deposit nickel on the copper.
The third electrodeposition step is to deposit nickel with diamond embedded herein on the nickel deposited in the second step.

Optimum duration of electrodeposition for 6 mm high patches:

Cu electrodeposition: 5-8 minutes
1st Ni electrodeposition: 12-15 minutes
2nd Ni electrodeposition: 12-15 minutes

via electronic filing

UNIFIED PATENT COURT
PARIS LOCAL DIVISION

Related Docket No 2015/01

26 January, 2015

In the matter

3 ABRASIVE (3A)
(A company with its registered office located in the USA)
v/
La Toilemeri SA
(A company with its registered office located in France)

APPLICATION FOR PROVISIONAL MEASURES

Applicant's Address for Service:

1/ Mr. Axel Casalonga of CASALONGA & Associés
8, avenue Percier
F-75008 PARIS
a.casalonga@casalonga.com

2/ Mr. Martin Köhler of REIMANN OSTERRIETH KÖHLER HAFT
Steinstrasse 20
D-40212 Düsseldorf
Martin.Koehler@rokh-ip.com

Defendant’s Address for Service:

1/ Mr. Grégoire Desrousseaux of Auguste et Debouzy
6-8 avenue de Messine
F-75008 Paris,
gdesrousseaux@augdeb.com

2/ Mr. Kay Rupprecht of Meissner Bolte
Widenmayerstr.47
D-80538 Munich
ru@mpb.de

we kindly request the Court to summon the parties to an oral hearing as early as possible due to the urgency of this matter and decide by way of provisional measures:

A. that the defendant be ordered

I.

1.

upon pain of an administrative fine to be determined by the Court of EUR 5,000 for each case of non-compliance to refrain from using

in the territory of the following countries: France, Germany, Sweden, The Netherlands, United Kingdom,

a method for manufacturing an abrasive member comprising flexible sheet (1) with a multitude of discrete metal protuberances (2,3)

if the method comprises the following steps:
a multitude of copper protuberances (2) are formed on the flexible sheet (1);
nickel protuberances (3) are electrodeposited over the copper protuberances (2) in the presence of 5 particulate abrasive material (4) so that the particulate abrasive material becomes embedded in the nickel deposits;
the voids between the protuberances (2,3) are at least partially filled with resin material, the resin material being selected so as to reduce lateral movement of the nickel deposits;

2.

upon pain of an administrative fine to be determined by the Court of EUR 5,000 for each case of non-compliance to refrain from offering, placing on the market using or importing or storing for those purposes

in the territory of the following countries: France, Germany, Sweden, The Netherlands, United Kingdom, an abrasive member obtained directly by the process according to A.I.1;

II. deliver up products according to A.I.2 which are in defendant's possession to a bailiff commissioned by applicant;

B. that the judgment be provisionally enforceable against provision of a security which may also be provided in the form of a bank or savings bank guarantee.

Grounds

1 Applicant has lodged an infringement action against defendant by statement of claim filed on 13 October 2014 followed by an application for an order to preserve evidence and for inspection filed on 29 December 2014.

2 The information contained in the written report on the measures to preserve evidence has clearly confirmed the literal infringement of claim 1 of the patent in suit by the attacked product. The validity of the patent in suit is beyond doubt because of the decision of the EPO Opposition Division. The applicant has just become aware by information contained on the website of defendant that a market launch is imminent. This will create significant and irreparable damage to the applicant. The only way to protect applicant's legitimate rights in enforcing the patent in suit is by ordering the requested provisional measures.

In detail:

I.

Formalities

3 This Court has jurisdiction to decide on the request for provisional measures according to R208 (3) RoP. Proceedings on the merits of the case have already been started before this Court under the Docket No 2015/01. We kindly request that the existing Court file No 2015/01 shall be made part of the current proceedings on the application for provisional measures.

4 The Applicant is the registered owner of the patent in suit, EP 2 900 000. The patent in suit is in force. We refer to the copy of the patent specification and the register which we submit as

Exhibit A 1.

II.

Infringement of the patent in suit

II.1

The patent in suit

5 With regard to the teaching of the patent in suit we refer to the statement of claim and the request for an order to preserve evidence.

6 The patent in suit relates to a process for manufacturing an abrasive member according to claim 1 which may be presented in form of a feature analysis as follows:

[1] A method for manufacturing an abrasive member
  [1a] comprising flexible sheet (1)
  [1b] with a multitude of discrete metal protuberances (2,3)
[2] whereby the method comprising the steps of
  [2a] forming a multitude of copper protuberances (2) on the flexible sheet (1),
  [2b] electrodepositing nickel protuberances (3) over the copper protuberances (2) in the presence of particulate abrasive material (4) so that the particulate abrasive material becomes embedded in the nickel deposits
[3] wherein the voids between the protuberances (2, 3) are at least partially filled with resin material,
  [3a] the resin material being selected so as to reduce lateral movement of the nickel deposits (2, 3).

Four copies of this feature analysis are presented as

Exhibit A 2.

7 The abrasive product obtained with the claimed process has great advantages over the previously known abrasives. The step of electrodepositing a second metal on a first metal leads to a particularly strong fixation of the plurality of metallic abrasive protuberances on the surface of the flexible support.

8 In addition, the protuberances are secured against lateral movement, thanks to a resin layer embedding the metal patches.

9 A preferred embodiment of a product directly obtained by the claimed process is disclosed in Fig 1 of the patent in suit, represented below (colors added). Fig. 1 shows a flexible support (1 – grey), upon which copper protuberances are positioned (2 – red). Nickel protuberances (3 – green) containing particulate abrasive material (4) are positioned on top of the copper protuberances. The voids between the protuberances (2,3) are filled with resin:

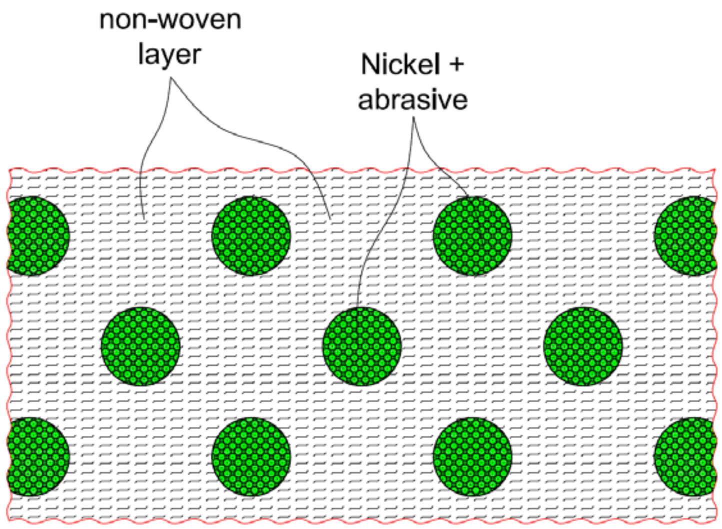
II.2
Literal infringement

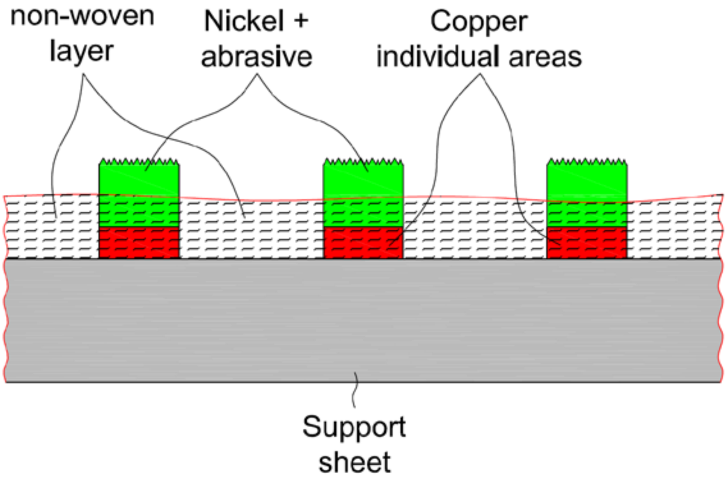
10 The attacked product is directly obtained by a manufacturing process making literal use of all features of claim 1 of the patent in suit. This is confirmed by the report on the measures to preserve evidence which we present as

Exhibit A 3.

11 The use of this manufacturing process violates applicant's rights according to Art. 25 (b) UPCA and the distribution of the attacked product obtained by this manufacturing process violates applicant's rights under Art. 25 (c) UPCA.

12 The attacked product is an abrasive member comprising a flexible sheet and a multitude of discrete metal protuberances (features [1]-[1b]. This can be seen in the below drawing which is an undisputed representation of the attacked product:





13 As has been confirmed by the report on the measures to preserve evidence (Exhibit A 3), the process used by defendant furthermore makes use of all further features [2a]-[3] of claim 1.

14 The attacked product consists of a support sheet covered by a nonwoven fabric and a mask. The support sheet with the nonwoven fabric and the mask is immersed into a first tank where copper protuberances are applied by way of electrodeposition (p. 2 of exhibit A 3) (step [2a]). The copper protuberances cover the areas of the support sheet left open by the mask.

15 Subsequently nickel is deposed on the copper areas in order to form nickel patches. This deposition is effected by electrodeposition in a nickel bath. Industrial diamond

particles are poured into the same electrolytic bath in order to form abrasive material embedded in the nickel patches (p. 2 of exhibit A 3) (step [2b]).

16 Subsequently the mask is removed. As can be seen from the picture above, the voids between the copper-nickel patches are filled with the non-woven fabric made of polyamide fibers. Polyamid is expressly mentioned on p.2 of the patent in suit as a material qualifying as a resin in the meaning of claim 1 of the patent in suit:

> "The resin material can be chosen from polyurethane resins, polyamide resins, polycarbonate or high density poly ethylene. The resin chosen must be such that less than 10% of metal deposits chip off after five hours grinding of hard stone such as granite."

17 According to a test report made by defendant and described in exhibit A3, the non-woven fabric filling the voids between the patches increase the resistance to lateral movement of the patches so that the stability test disclosed in the above cited passage of the patent in suit is matched (p. 3 of exhibit A 3) (step [3], [3a]).

III.

Validity of the patent in suit confirmed by EPO

18 The validity of the patent in suit has been confirmed by the EPO OD with a decision dated 3 September 2014 rejecting the defendant's opposition. The appeal currently pending at the EPO BoA is without merits and will not lead to a revocation of the patent in suit. The same applies to the counterclaim for revocation filed by defendant on 18 December 2014.

19 Both the appeal to the EPO BoA as well as the counterclaim for revocation refer as sole prior art to FR-B-2 565 870 (D1) which we present as

Exhibit A 4.

20 The EPO OD has already dealt in detail with prior art D1 and confirmed the validity of the patent in suit in the light of this prior art with a decision dated 3 September 2014 which we present as

Exhibit A 5.

21 The OD decision comes to the conclusion that D1 lacks in particular features [2b] and [3] of claim 1. Therefore, patent in suit is undoubtedly new over D1.

22 Furthermore, the patent in suit is clearly inventive over D1. There is no motivation for a person skilled in the art to modify the process step of chemical deposition as disclosed in D1 for forming copper protuberances by the different process step of electrodepositing as claimed in feature [2b].

23 Nor is there any motivation for the person skilled in the art starting from D1 to fill the voids between the protuberances with resin in order to prevent lateral movement of the protuberances. Rather, D1 teaches in a different direction in order to prevent lateral movement of the protuberances.

24 Against this background there can be no reasonable doubt that the patent in suit is valid in the light of the prior art presented by defendant.

V.

Urgency

25 Applicant has knowledge of the process used by defendant only since receiving the report on measures to preserve evidence after the inspection of 8 January 2015. Thus, applicant has acted without any unreasonable delay in seeking provisional measures to protect its interests (R211 (4) RoP).

VI.
Balance of interest in support of provisional measures

26 The weighing of interests between the interests of both parties has to be decided in favor of the applicant's application, because of

- the clear infringement of the patent in suit
- the confirmed validity of the patent in suit
- the imminent introduction of the infringing product during the 2015 International Abrasive Exhibition and via the simultaneous start of distribution in Europe
- the substantial damage and loss of market share that applicant will suffer in case of a launch of the infringing product
- the irreparable harm that the launch of the infringing product will cause to applicant because of irreversible price erosion.

27 With regard to the imminent launch of the attacked product in Europe, we refer to the findings on p. 3 of exhibit A 3. According to the Distribution agreement between defendant and its distributor Leichtschleif (LS) the product launch is to start in Germany, France, Great Britain and in the Netherlands immediately after the 2015 International Abrasive Exhibition (28 Mai - 3 June 2015). According to information currently posted on the website of defendant, the European product launch is actually announced for 8 June 2015. We submit a copy of the website as

Exhibit A 6.

28 The sales price fixed in the distribution agreement is of €5 per 1m of abrasive product. This is almost 40% less than the sales price of €8 per 1m applied to the abrasive products manufactured by applicant on the basis of the patented process. The current prices for the applicant's products result from invoices and a current price list which we present as

Exhibit A 7.

29 Due to the highly competitive market there is a very high risk for the applicant to suffer irreversible price erosion should the defendant be able to launch the product. Even if the defendant would be prevented from continuing sales after a later decision of the Court in the main action, there is a high risk that the customers would not accept going back to the current price level for abrasive products sold by applicant. The market for abrasives is dominated by a restricted number of big customers. Because of the strong market power of these customers, the current price level could not be sustained if competing products would be available for 50% less of the price even for a short period of time. The damage caused to applicant due to price erosion only is estimated at € 2 million for the lifetime of the patent.

30 We offer evidence for the above market facts and economic risk by written witness statement by Mr. Peter Smith, in charge of sales and marketing for the applicant as

Exhibit A 8.

31 As a consequence, applicant would suffer substantial and irreversible harm if the defendant should be permitted to launch the product in Europe as planned.

32 On the other hand, defendant's interests are merely of monetary nature. These interests may be fully secured by ordering the applicant to provide an adequate security (R211 (5) RoP).

33 While the applicant thus may only enforce its legitimate rights by way of provisional measures in the form of injunction and delivery up of the infringing products in order to avoid the significant and irreparable harm which would follow from the imminent distribution of the attacked product, defendant has no legitimate interest beyond securing claims for potential subsequent compensation. The balance of interests thus has to be decided in favor of applicant (R211 (3) RoP).

VII.
Legal Consequences

34 Applicant is entitled to request by way of provisional measures an injunction against the use of the infringing process and against the distribution of the infringing products directly obtained by this process (R. 211 (1a)).

35 Applicant furthermore is entitled to request by way of provisional measures the delivery up of the infringing products in order to prevent their entry into the market (R. 211 (1b)).

36 The value of the action is estimated at € 330.000,- which corresponds to 2/3 of the value of the complaint. Because the provisional measures only relate to a provisional injunction, a reduction of 1/3 compared to the value of the complaint appears appropriate form the defendant's point of view.

(Axel Casalonga) (Martin Köhler)

Claim 1 of EP 2 900 000

[1] A method for manufacturing an abrasive member

- [1a] comprising flexible sheet (1)
- [1b] with a multitude of discrete metal protuberances (2,3)

[2] whereby the method comprising the steps of

- [2a] forming a multitude of copper protuberances (2) on the flexible sheet (1),
- [2b] electrodepositing nickel protuberances (3) over the copper protuberances (2) in the presence of particulate abrasive material (4) so that the particulate abrasive material becomes embedded in the nickel deposits

[3] wherein the voids between the protuberances (2, 3) are at least partially filled with resin material,

- [3a] the resin material being selected so as to reduce lateral movement of the nickel deposits (2, 3).

PROCÈS-VERBAL DE CONSTAT INTERNET

L'AN DEUX MILLE QUINZE ET LE VINGT JANVIER

À LA REQUETE DE :

La société 3A ABRASIVE, société de droit américain dont le siège social est situé 10008 S. Western Avenue, Chicago, IL 60643, USA.

LAQUELLE M'EXPOSE :

Qu'elle a le plus grand intérêt à faire constater le contenu de pages mises en ligne sur internet.

Qu'elle me requiert, en conséquence, pour assurer la sauvegarde de ses droits, de procéder à toutes constatations utiles et d'en dresser procès-verbal.

POURQUOI DÉFÉRANT À CETTE RÉQUISITION :

Je, Claude Dupuis, Huissier de Justice associés près le Tribunal de Grande Instance de PARIS, demeurant 23, rue des fermiers 75017, soussignée

Ce jour, en mon Etude :

J'AI VU, RECONNU ET CONSTATÉ CE QUI SUIT :

I - ENONCIATIONS PREALABLES

NORMES AFNOR NF Z67-147

1) Les présentes constatations sont effectuées sur le poste informatique décrit ci-après :

Ordinateur de bureau

Modèle : HP ProDesk 400 G1 MT

Carte mère HP18E9

Processeur : Intel(R) Core(TM)2 Duo CPU E8400 @ 3.00GHz, 3000 MHz, 2 cœur(s), 2 processeur(s) logique(s)

Mémoire physique : 4096 Mo

Carte graphique : Intel® HD Graphics 4400

Disque dur : TOSHIBA DT01ACA050 SCSI Disk Device (500Go)

Lecteur DVD-Rom : Hewlett-Packard DVDRAM GT80N

Type de moniteur : ViewSonic VG390m-3-19 pouces

Carte réseau : Realtek Semiconductor RTL8168/8111

Système d’exploitation : Windows 7 Professionnal Professionnel Medis Center 6…

DirectK : Version 11.00

Windows Performance Index : 4.8 sur 7.9

Connecté au serveur informatique de mon étude.

Les copies écran sont effectuées au moyen de la touche « Impr écran » et son directement incorporées au présent procès-verbal de constat.

Les captures sont effectuées au moyen du logiciel Capturino 2.43 et également de l’outil de capture windows Outil Capture et sont directement incorporées au présent procès-verbal de constat.

Les impressions sont réalisées au moyen du matériel d’impression suivant :

KONICA MINOLTA C360

Ce matériel permet d’imprimer les pages consultées. Les impressions réalisées sont annexées au présent procès-verbal.

2) Je synchronise l'horloge interne de mon poste avec le serveur de temps interenet « time.windows.com » en effectuant une mise à jour de « Propriétés de date et heure » « temps internet » de mon logiciel d'exploitation.

L'horloge a été synchronisée avec time.windows.com le 06/10/2014 à 13.01

3) Je mets à jour mon logiciel antivirus

Kaspersky

Endpoint Security 10

For Windows

Puis je lance une analyse antivirale.

4) je mets à jour mon programme de suppression des logiciels espions

Malwarebytes

ANTI-MALWARE

Puis je lance une analyse.

5) je détermine la configuration de ma machine de travail

(configuration reprise ci-dessus) (=description détaillée des éléments de mon poste informatique-matériel et système d'exploitation) à l'aide du logiciel : PC WIZARD 2014 Classic Edition – Version 2.13

6) j'accède au réseau étendu par un routeur connecté au serveur et dont le modèle est

D-LINK ADSL ROUTER modèle n°DSL6502T

Via carté réseau :

Realtek Semiconductor RTL8168/8111 Gigabit Ethernet Adapter

7) mon fournisseur d'accès est WANADOO/ORANGE

Abonnement : Internet pro solo – 8M

8) je détermine l’adresse MAC de la carte réseau active de ma machine de travail en utilisant la commande « ipconfig/all » précédée de cmd+ok dans l’invite de commande de mon logiciel d’exploitation et je relève que celle-ci est identique à celle obtenue à l’aide du logiciel gratuit BeLarc Advisor.

Je note que le DHCP n’est pas activé

Cette adresse est la suivante : 2C:44:F :3A:A8:C2

9) Je procède ensuite à diverses opérations de purge, comme suit :

- je procède à une purge complète et à un vidage intégral de ma corbeille
- je m’assure que les lecteurs CD Rom et de disquettes sont vides ;
- je m’assure que l’ordinateur ne contient aucun support de mémoire amovible externe.
- Purge complète de l’historique de navigation et paramétrage de mon navigateur Firefox 3.5 par le chemin : « Outils, « Options », « Vie privée » « Historique » « Ne jamais conserver l’historique ».
- Paramétrage du cache local et purge complète du cache local par le chemin : « Outils », « Options », « Avancé » « Réseau » « contenu web en cache » bouton « Vider maintenant »

10) Je m’assure que mon navigateur n’est pas paramétré pour utiliser un proxy par le chemin : « Outils », « Avancé », onglet « Réseau », « Connexion », « paramètres » je m’assure que concernant la Configuration du serveur proxy pour accéder à Internet ‘Pas de proxy » et coché comme suit :

Paramètres de connexion

Configuration du serveur proxy pour accéder à Internet

- Pas de proxy

11) Je configure mon navigateur pour accepter les cookies

12) Je paramètre une page vierge comme page de démarrage par le chemin : « Outils », « Options », onglet « général », « Démarrage », Au démarrage de Firefox « Afficher une page vide ».

13) Je récupère mon adresse IP pulique « WAN » - en utilisant la commande « ipconfig/all » dans l'invite de commande de mon système d'exploitation et en me connectant à la page http://www.mon-ip.fr

82.123.21.85

14) Je procède à un ultime nettoyage au moyen du logiciel CCleaner.com

***

II - CONSTATATIONS

Je démarre mes constatations à 10 heures 30

Lancement de mon navigateur internet Firefox 3.5

Dans la barre du navigateur, je saisis l'adresse suivante

http://www.latoilemeri.fr

et frappe sur la touche « Entrée ».

La page d'accueil du site apparaît.

Sur cette page d'accueil je clique sur le lien « EVENT », situé en haut de la page.

Une nouvelle page apparaît laquelle est imprimée (Annexe 1) et dont je fais des copies d'écran :

Prochain évènement:

La Toilemeri participera comme chaque année au salon "International Abrasive" qui aura lieu à Villepinte du 28 mai au 3 juin 2015

Notre nouveau produit abrasif "Supergrind" sera présenté en avant première

Venez nous voir sur notre stand E24C3

Le lancement de ce produit révolutionnaire est prévu immédiatement après le salon, dans toute l'Europe, avec un pré lancement le 8 juin

Soyez les premiers à bénéficier des multiples avantages de ce nouveau produit

La Toilemeri © 2014 / mentions légales / Contact

<u>TELLES SONT MES CONSTATATIONS</u>

Et de tout ce que dessus, j'ai fait et rédigé le présent procès-verbal de constat, pour servir et valoir ce que de droit.

<u>SOUS TOUTES RÉSERVES</u>

BAILIFF REPORT ON THE INTERNET

DATE: January 20th, 2015

Acting upon request of the company 3A ABRASIVE, a company with its registered office located in Chicago, USA.

STATING THAT:

It has the greatest interest in establishing a report of websites' pages' content on the internet.

It then requests me, in order to preserve its rights, to establish all necessary facts and to draw up an official report.

I, CLAUDE DUPUIS, COURT BAILIFF, WHOSE ADDRESS IS 23 RUE DES FERMIERS 75017 PARIS, CERTIFIES THAT I REPORTED THE FOLLOWING:

After having done all the necessary technical measures to ensure that the report is valid.

At 10.30 pm, I start my findings.

I start my web browser.

I enter the following address in the browser's address field:

http://www. latoilemeri.fr

and press "Enter".

The website's homepage appears.

On this homepage, I click on the link "EVENT", located on the top of the page.

A new page appears, which is printed (annex 1), and I make a screenshot of it:

Vous êtes ici / Accueil / Event

Prochain évènement:

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Soyez les premiers à bénéficier des multiples avantages de ce nouveau produit

La Toilemeri © 2014 / mentions légales / Contact

<u>THESE ARE MY FINDINGS.</u>

I certify that I have done and wrote this bailiff report.

Made for all legal intents and purposes,

Abrasive 1
Rue Principale 5

Paris
France

15 July 2014

Invoice: 10013700/14

Client number 35273

For the delivery of abrasive products in June 2014 we charge:

date	product amount	price
03.6.2014	100 m abrasive product	€ 1.000,-
24.6.2014	900 m abrasive product	€ 9.000,-
Total:		€ 10.000,-

(Peter F. Smith)

Payable within 14 days. Please quote the invoice number.

Written witness statement by Mr. Peter Smith according to R. 175 RoP

I, Peter F. Smith, born 3 July 1959 in Chicago, am aware of the obligation to tell the truth and that any breach of such obligation may engage my liability under French law. I provide the following statement to be presented as written witness evidence in front of the Unified Patent Court, Paris Local Division:

- I am in charge sales and marketing (Vice President Sales and Marketing) at 3 Abrasive since January 2000.
- The market for abrasive products is dominated by only 3 number major customers Abrasive 1, Abrasive 2, and Abrasive 3. These customers cover about 90% of the market share for abrasive products. These customers have a strong market power impacting on the price level at which abrasive products may be sold. In case that a competitor should enter the market with products technically comparable to our abrasive product line at a 37,5% lower sales price compared to our current price, we would expect that we would not be able to sustain our current price level. Even if such competitor would not be able to continue sales at a later point, we would expect that we would not be in a position to get back to our current price level due to the market power of the 3 relevant customers. Therefore, even a short presence of products with a significant lower price would very likely lead to a significant permanent price erosion.
- Based on our current sales price of €8 per 1m and a sales price of € 5 per 1m for the competing product we expect damages caused by price erosion in the range of € 2 million for the next 13 years.

Chicago, 24 January 2015

(Peter F. Smith)

Unified Patent Court
Paris Local Division

Docket number 2015/01

OBJECTION TO THE APPLICATION FOR PROVISIONAL MEASURES

On behalf of Defendant **La Toilemeri SA**, a company with registered seat in France.

Represented by
Mr. Grégoire Desrousseaux of August & Debouzy, 6-8 avenue de Messine F-75008 Paris,
gdesrousseaux@augded.com
Mr. Kay Rupprecht of Meissner Bolte, Widenmayerstr. 47, D-80538 Munich,
ru@mpb.de
both authorized to accept service in relation to the instant proceedings

Against Claimant **3 Abrasive (3A)**, a company with registered offices in the USA

Represented by
Mr Axel Casalonga, Casalonga & Associés, 8 avenue Percier, F-75008 Paris,
a.casalonga@casalonga.com
Mr Martin Köhler, Reimann Osterrieth Köhler Haft, Steinstr. 20, D-40212 Düsseldorf
martin.koehler@rokh-ip.com
both authorized to accept service in relation to the instant proceedings

1. GENERAL STATEMENTS

1 Defendant hereby files, on invitation by the Court according to Rule 209 (1) (a) of the Rules of Procedure (in the following “RoP”), an Objection to the Application for Provisional Measures including some reasons considered as very relevant why the Application shall fail.

2 The objection will focus on

(i) challenging some facts and the evidence relied on and declared most relevant by the Applicant;

(ii) challenging the necessity of the already conducted Order to Preserve Evidence (“Saisie”); and

(iii) requesting, according to Rule 197 (3) RoP, a review of said Order to Preserve Evidence with the aim that it shall be revoked, with the consequence that the infringement has not yet been proven.

2. REASONS WHY THE APPLICATION SHALL FAIL

3 According to Art. 62 (2) UPCA, the Court shall have discretion to weigh-up the interests of the parties and in particular to take into account the potential harm for either of the parties resulting from the granting or the refusal of the injunction. In this context, the Court's discretion shall be footed on the visible application of the principles of proportionality, fairness and equity as laid down in Art. 42 (1) UPCA and in the preamble of the Rules of Procedure.

4 In the following, it will be shown that the sought order for provisional measures would not sufficiently take into account the Defendant's potential harm, and it will further be shown that the Court exerted its earlier discretion in the course of the Order for Preserving Evidence on the basis of incomplete facts presented by the Claimant/Applicant.

5 A further reason to reject the Claimant's Application for Provisional Measures is that the patent in suit is under Appeal by a third party, the outcome of said Appeal still being unknown. However, as the Decision of the Opposition Division (in the following referred to as "OD") seems to be weak, it should be taken into consideration that the Boards of Appeal might well overturn the first instance decision and revoke the patent in suit, for the reasons set out in the Defendant's Statement of Defence and Counterclaim for Revocation, and in the Defendant's Intervention to the EPO.

6 Last, but not least, it will in the following be briefly and in addition to the Defendant's already submitted Counterclaim for Revocation shown that the Defendant's attack does seem promising, as the OD took quite a vulnerable approach to argue in favor of an inventive step.

2.1 NO NECESSITY OF THE ALREADY CONDUCTED ORDER TO PRESERVE EVIDENCE ("SAISIE")

7 As will be shown hereinafter, the relatively drastic action of the inspection of the Defendant's premises and manufacturing process would not have been necessary at all. The Claimant/Applicant would have had other commercial and legal instruments at hand to substantiate its claim and to obtain the missing clarity about the layers of the abrasive member, their manufacturing method and about the nature of the abrasive elements.

2.1.1 EASY ACCESS TO THE PRODUCT

8 In its Application for Preserving Evidence and Request for an Order to Inspect Premises without Hearing the other Party (in the following also referred to as "Ex-Parte Inspection Request"), the Claimant/Applicant admitted in its summary of facts under point 5 that the Defendant's webpage included a page offering the sale of all products mentioned on the site including the allegedly infringing product.

9 Hence, instead of obtaining the Court's permission to enter the Defendant's premises, the Claimant/Applicant could have simply purchased one or two sample products. If one applied Union Law, which should in the absence of a broad basis of antecedent Union jurisprudence, be interpreted as the most possible harmonized law of the Member States, the public availability of an alleged infringing product rules out any order to inspect premises which would in such cases be stigmatized as "fishing expedition".

10 Even if, for whatever reasons, an attempt to purchase the alleged infringing product would have failed, the Claimant/Applicant could have requested the Court to issue an order to produce evidence in accordance with Art. 59 UPCA and Rule 190 RoP. As is obvious from the Claimant's/Applicant's Ex-Parte Inspection Request, page 8, para. 36, the only missing information was the nature of the method of attaching the two metal layers to each other. This aim, however, could have been achieved by smaller "legal calibers", as explained above and in more detail hereinafter.

2.1.2 POSSIBLE EXAMINATION OF THE PRODUCT

11 Alternative technologies to electrodeposition exist in the prior art, e.g. electrolytic or autocatalytic plating, i.e. chemical processes (see the discussion of chemical deposition in D1, page 1, line 35 and page 2, lines 10 and 11). It is certainly undisputed that the infringing product comprises a plurality of metal patches made of **nickel** having particulate abrasive therein on their upper surfaces, said metal patches being attached to the support via individual areas of metallic **copper** in direct contact with the support sheet.

12 As opposed to the Claimant's/Applicant's allegation in its Ex-Parte Inspection Request, para. 14, last sentence on top of page 5, nickel coatings or layers which have been derived from electroless nickel baths (this is called "electroless plating") has excellent properties. The coatings are characterized by excellent corrosion resistance, good wear and abrasion resistance and high hardness. Electroless deposited nickel also forms very uniform layers and results in accurately defined contours, even on complex materials and interior surfaces, see e.g. the article "Oliveira et. al.: The effect of the hypophosphite ion oxidation on the Ni surface electrode – an XPS study", attached as **Annex DEF 3**.

13 Hence, the Claimant/Applicant provided the Court with incomplete if not wrong information. There clearly existed an alternative to electrodeposition before the inspection order was issued. And the question which deposition technology the Defendant is using for its accused products could have been answered without entering its premises, as will be briefly explained in the following:

14 If an alternative deposition technology to electrodeposition has been used, namely autocatalytic deposition which as a rule uses the chemical solution sodium-hypophosphite, then the combined **nickel-copper** composite would show a significant phosphor content within the upper nickel film, see e.g. the European Standard EN ISO 4527, attached as **Annex DEF 2**. The two short paragraphs of the introduction on page 4 of said European Standard briefly explain how this works and the Annex D on page 22 of the European Standard describe how the analysis of the product would have worked. Namely, said phosphor content – or its absence – could have easily been determined by using EDX (Energy Dispersive X-ray spectroscopy), an examination method which is commonly being used in all Materials Testing Institutes. An example of such an institute may be retrieved at: http://www.mpa.uni-stuttgart.de/index.en.html. Namely, according to said European Standard for materials testing and examination, any phosphor content within a nickel film would demonstrate that the nickel film was coated by an electroless method, or, applying an argumentum e contrario (converse argument), if no phosphor would be traceable within the product's nickel film, the composite structure would have been created by electrodeposition.

15 Consequently, this means that if the upper nickel film in the accused product was coated by using an electrodeposition method, the phosphor content within the nickel film is zero.

16 Therefore, and contrary to the Claimant's/Applicant's allegation, it is and would have been very easy to examine, if the nickel film was coated by electrodeposition or not.

2.1.3 NO CAUSE OF IRREPARABLE HARM TO THE APPLICANT

17 According to Rule 197 RoP, the Court may issue an order to preserve evidence without the Defendant having been heard where any delay is likely to cause irreparable harm to the Applicant or where there is a demonstrable risk of evidence being destroyed or otherwise ceases to be available.

18 Despite the Defendant’s simple allegation in its Application for Provisional Measures, page 4, para. 2, last sentence, none of these preconditions seem to exist. The harm to the Applicant, if any, could have been avoided by just procuring the product and examining it. Neither has a risk of evidence being destroyed or otherwise ceasing to be available been demonstrated. To the contrary, the Defendant’s company is well-established and, as the Applicant itself has conceded, recently started the manufacturing process which certainly could not have suddenly been spirited away.

2.1.4 VIOLATION OF THE PRINCIPLE OF PROPORTIONALITY

19 According to the Defendant’s conviction, there was no real urgency or other reason to use the “steam hammer method” of conducting an inspection at the Defendant’s premises and to provide the Claimant with proprietary trade secrets and manufacturing information. Namely, the alleged infringing product could have been obtained either by a simple purchase order through the Defendant’s internet platform or by applying for a Court Order to Produce Evidence in accordance with Rule 190 RoP. Subsequently, the product could have been examined in order to find out whether the first and the second metal layer were brought together by electrodeposition.

20 The Claimant/Applicant has also not provided any information that unsuccessful attempts were made to obtain the advertised product from the Defendant. E.g., according to the Rules of Court in England, a party alleged to infringe a patent can avoid giving disclosure on the issue of infringement if it elects to serve a product or process description with full particulars instead. Whereas the Defendant acknowledges that there is no such provision in the UPCA or in the Rules of Procedure, this procedural handling in England might give an example of how to avoid excessive use of inspection orders.

21 With all due respect, the Defendant cannot see the proportionality and fairness as provided for in Art. 42 UPCA and in the preamble of the Rules of Procedure.

2.1.5 INTERIM RESULT

22 According to the above arguments, the Defendant’s request to review the issued Order to Preserve Evidence according to Rule 197 (3) RoP, in particular without hearing the Defendant, seems to be justified, with the consequence that it shall be revoked and the result of the inspection shall be kept confidential and not used for the infringement proceedings.

2.2 INVALIDITY OF THE PATENT

23 Further to the Defendant’s Statement of Defence and Counterclaim for Revocation, the contents of which is expressly incorporated into the present Objection to the Application for Provisional Measures, it is respectfully and additionally submitted that the OD erred by taking the position that D1 teaches the person skilled in the art away from the patent in suit, as will be shown hereinafter.

24 The OD fails to recognize that D1 discloses the sandwich construction of an abrasive member including the very same general construction as claimed in the patent in suit, namely

(1) a flexible basic support;
(2) a first metal layer (of copper);
(3) a second metal layer (of nickel) including abrasive elements therein;
(4) an intermittent cover sheet of raisin;
(5) the first and the second layers being laminated together by suitable means.

25 The only difference between the claimed method of making the accused product and the product disclosed in D1 is that in the product of D1, the resin is applied first and then the second metal layer is deposited onto the first metal layer where the intermittent raisin cover has left notches, whereas the claimed method to produce the allegedly infringing product, the second layer is first deposited in an intermittent manner onto the first layer and then the resin cover is filled into the notches left between the intermittent patches of the second layer.

26 This sole difference is the same as if one compares a negative with the corresponding positive. The person skilled in the art in the field of making sandwich layer constructions is well aware of the possibilities of carrying out the lamination either the one way or the other. Hence, it would have been obvious to the person skilled in the art to start with the technical teaching of D1 in order to arrive at the claimed product without being inventive.

27 As a conclusion the patent in suit should be declared invalid.

2.3 NO DELIVER-UP OF THE PRODUCTS

28 The Claimant/Applicant requested to deliver-up the allegedly infringing products which are in the Defendant’s possession.

29 In fact, according to Art. 62 (3) UPCA, the Court may order the seizure or delivery up of the products suspected of infringing a patent so as to prevent their entry into, or movement, within the channels of commerce.

30 But given the circumstances, namely that the infringement is neither proven nor the patent’s validity ensured, the Defendant would suffer significant financial harm resulting from the granting of the injunction. Namely, the Defendant only recently established its production line for the accused product with high corresponding costs. The Defendant’s financial situation is currently under extreme pressure because of the initial investments into said production line facilities. In this context, the Defendant herewith provides a Witness Statement of its CFO, Mr. Donald Duck, resident in F-74999 Canardville, which is attached as **Annex DEF 1**.

3. SUMMARY OF THE ARGUMENTS

31 The Court Order for Preserving Evidence without hearing the Defendant was based on incomplete facts presented by the Applicant and should thus be revoked and hence, the evidence gathered from the corresponding inspection at the premises of the Defendant was unlawful and should not be taken into account due to a violence of the principle of proportionality.

32 Consequently, the alleged infringement must be deemed not yet been proven.

33 The patent in suit must be declared invalid and cannot form the basis for the sought Application for Provisional Measures.

34 The request to deliver-up the allegedly infringing products must fail.

35 As a consequence, the Application for Provisional Measures has to be rejected.

4. RELIEF

36 Hence, the following relief is sought:

(1) The order for preserving evidence be revoked and its result be set aside;

(2) The application for provisional measures be rejected;

(3) The Claimant/Applicant shall bear the costs of the proceedings in accordance with Art. 69 UPCA; and

(4) The Court shall order an interim award of costs to the benefit of the Defendant in accordance with Rule 211(1) (d) RoP, because the Claimant/Applicant has no legal seat within the European Union.

Respectfully submitted on March 3, 2015,

Kay Rupprecht Grégoire Desrousseaux

Attachments:

Annex DEF 1
Annex DEF 2
Annex DEF 3

Annex DEF 1

Witness Statement

I, the undersigned Donald Duck, residing in F-74999 Canardville, knowing that I have to tell the truth and that a false declaration is punishable by national French law, herewith declare the following

I graduated from the Ecole d' Economie de Paris in the year 1987 with focus on finance management for small and medium sized companies.

I am since 5 years CFO of the French company La Toilemeri SA located at Bécon les Bruyères, France.

My company is since a number of years active in the field of manufacturing and distributing abrasive products for industrial applications. Most recently, we established a production line for manufacturing an abrasive member which has been developed by our engineers.

Currently, our company's financial situation is under some considerable pressure, as we had to finance the production line for our new abrasive member which did cost the company EUR 2.579.000. The intention is to bring said new abrasive member on the market and to gain the financial break even within 3 years.

Done in Paris on the date of February 18, 2015

D. Duck

D. Duck

October 2003

DEUTSCHE NORM

Metallic coatings

Autocatalytic (electroless) nickel-phosphorus alloy coatings

Specification and test methods
(ISO 4527 : 2003)
English version of DIN EN ISO 4527

DIN
EN ISO 4527

ICS 25.220.40

Supersedes DIN 50966,
May 1988 edition.

Metallische Überzüge – Autokatalytisch (außenstromlos) abgeschiedene Nickel-Phosphor-Legierungs-Überzüge – Spezifikationen und Prüfverfahren (ISO 4527 : 2003)

European Standard EN ISO 4527 : 2003 has the status of a DIN Standard.

A comma is used as the decimal marker.

National foreword

This standard has been published in accordance with a decision taken by CEN/TC 262 to adopt, without alteration, International Standard ISO 4527 as a European Standard.

The responsible German body involved in its preparation was the *Normenausschuss Materialprüfung* (Materials Testing Standards Committee), Technical Committee *Galvanische Überzüge*.

The DIN Standards corresponding to the International Standards referred to in clause 2 of the EN are as follows:

ISO Standard	DIN Standard
ISO 1463	DIN EN ISO 1463
ISO 2064	DIN EN ISO 2064
ISO 2177	DIN EN ISO 2177
ISO 2178	DIN EN ISO 2178
ISO 2819	DIN EN ISO 2819
ISO 2859-1	DIN ISO 2859-1
ISO 2859-2	DIN ISO 2859-2
ISO 2859-3	DIN ISO 2859-3
ISO 3497	DIN EN ISO 3497
ISO 3543	DIN EN ISO 3543
ISO 3882	DIN EN ISO 3882
ISO 4516	DIN EN ISO 4516
ISO 6158	DIN EN ISO 6158
ISO 9220	DIN EN ISO 9220
ISO 10289	DIN EN ISO 10289

Continued overleaf.

EN comprises 26 pages.

Ref. No. DIN EN ISO 4527 : 2003-10
English price group 14 *Sales No. 1114*
02.04

Amendments

DIN 50966, May 1988 edition, has been superseded by the specifications of EN ISO 4527, which is identical to ISO 4527.

Previous edition

DIN 50966: 1988-05.

National Annex NA

Standards referred to

(and not included in **Normative references, Bibliography** and **Annex ZA**)

DIN EN ISO 1463	Metallic and oxide coatings – Measurement of coating thickness by the microscopical method (ISO 1463 : 1982)
DIN EN ISO 2064	Metallic and other inorganic coatings – Definitions and conventions concerning the measurement of thickness (ISO 2064 : 1996)
DIN EN ISO 2177	Metallic coatings – Measurement of coating thickness with the coulometric method by anodic dissolution (ISO 2177 : 1985)
DIN EN ISO 2178	Non-magnetic coatings on magnetic substrates – Measurement of coating thickness by the magnetic method (ISO 2178 : 1982)
DIN EN ISO 2819	Electrodeposited and chemically deposited coatings on metallic substrates – Review of methods available for testing adhesion (ISO 2819 : 1980)
DIN EN ISO 3497	Metallic coatings – Measurement of coating thickness – X-ray spectrometric methods (ISO 3497 : 2000)
DIN EN ISO 3543	Metallic and non-metallic coatings – Measurement of thickness – Beta backscatter method (ISO 3543 : 2000)
DIN EN ISO 3882	Metallic and other inorganic coatings – Review of methods of measurement of thickness (ISO 3882 : 2003)
DIN EN ISO 4516	Metallic and other inorganic coatings – Vickers and Knoop microhardness tests (ISO 4516 : 2002)
DIN EN ISO 6158	Metallic coatings – Electroplated coatings of chromium for engineering purposes (ISO/DIS 6158 : 2002)
DIN EN ISO 9220	Metallic coatings – Measurement of coating thickness with the scanning electron microscope method (ISO 9220 : 1988)
DIN EN ISO 10289	Methods for corrosion testing of metallic and other inorganic coatings on metallic substrates – Rating of test specimens and manufactured articles subjected to corrosion tests (ISO 10289 : 1999)

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 4527

May 2003

ICS 25.220.40

English version

Metallic coatings
Autocatalytic (electroless) nickel-phosphorus alloy coatings
Specification and test methods
(ISO 4527 : 2003)

Revêtements métalliques – Dépôts autocatalytiques (sans courant) d'alliages de nickel-phosphore – Spécifications et méthodes d'essai (ISO 4527 : 2003)

Metallische Überzüge – Autokatalytisch (außenstromlos) abgeschiedene Nickel-Phosphor-Legierungs-Überzüge – Spezifikationen und Prüfverfahren (ISO 4527 : 2003)

This European Standard was approved by CEN on 2003-04-25.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, the Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland, and the United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Management Centre: rue de Stassart 36, B-1050 Brussels

Ref. No. EN ISO 4527 : 2003 E

Foreword

International Standard

ISO 4527 : 2003 Metallic coatings – Autocatalytic (electroless) nickel-phosphorus alloy coatings – Specification and test methods,

which was prepared by ISO/TC 107 'Metallic and other inorganic coatings' of the International Organization for Standardization, has been adopted by Technical Committee CEN/TC 262 'Metallic and other inorganic coatings', the Secretariat of which is held by BSI, as a European Standard.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, and conflicting national standards withdrawn, by November 2003 at the latest.

In accordance with the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard:

Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, the Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland, and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 4527 : 2003 was approved by CEN as a European Standard without any modification.

NOTE: Normative references to international publications are listed in Annex ZA (normative).

Contents

Introduction

Autocatalytic nickel-phosphorus alloy coatings are produced by the catalytic reduction of nickel ions in hot, usually mildly acidic solutions at atmospheric pressure using hypophosphite ion as the reducing agent. Because the deposited nickel alloy is a catalyst for the reaction, the process is self-sustaining. The coatings produced are uniform in thickness on irregularly shaped parts if the processing solution circulates freely over their surfaces.

The as-deposited coating is a thermodynamically metastable, supersaturated solid solution of phosphorus in nickel containing up to 14 % mass fraction phosphorus. The physical and chemical properties and the structure of autocatalytic nickel-phosphorus coatings are dependent on the composition of the coating, the chemical make-up of the plating solution, the pre-treatment and quality of the substrate, and heat treatment after deposition.

Autocatalytic nickel-phosphorus coatings are applied in order to improve corrosion protection and to provide wear resistance. In general, corrosion performance is significantly improved as the phosphorus content of the deposit is increased to 8 % mass fraction or higher, whereas wear resistance is improved as the phosphorus content of the coating is decreased below that level. With suitable heat treatment however, coatings with high phosphorus contents display greatly improved microhardness and hence, wear resistance.

1 Scope

This International Standard specifies the requirements and test methods for autocatalytic nickel-phosphorus alloy coatings applied from aqueous solutions on to metallic substrates.

This International Standard does not apply to autocatalytic nickel-boron alloy coatings, nickel-phosphorus composites and ternary alloys.

WARNING — The use of this International Standard may involve hazardous materials, operation and equipment. This International Standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices, and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1463, *Metal and oxide coatings — Measurement of coating thickness — Microscopical method*

ISO 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*

ISO 2079, *Surface treatment and metallic coatings — General classification of terms*

ISO 2080[1], *Surface treatment, metallic and other inorganic coatings — Vocabulary*

ISO 2177, *Metallic coatings — Measurement of coating thickness — Coulometric measurement by anodic dissolution*

ISO 2178, *Non-magnetic coatings on magnetic substrates — Measurement of coating thickness — Magnetic method*

ISO 2819, *Metallic coatings on metallic substrates — Electrodeposited and chemically deposited coatings — Review of methods available for testing adhesion*

ISO 2859-1, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

ISO 2859-2, *Sampling procedures for inspection by attributes — Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection*

ISO 2859-3, *Sampling procedures for inspection by attributes — Part 3: Skip-lot sampling procedures*

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[1] To be published. (Revision of ISO 2080:1981)

ISO 2859-4, *Sampling procedures for inspection by attributes — Part 4: Procedures for assessment of declared quality levels*

ISO 3497, *Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods*

ISO 3543, *Metallic and non-metallic coatings — Measurement of coating thickness — Beta-backscatter method*

ISO 3882, *Metallic and other inorganic coatings — Review of methods of measurement of thickness*

ISO 4288, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Rules and procedures for the assessment of surface texture*

ISO 4516, *Metallic and other inorganic coatings — Vickers and Knoop microhardness tests*

ISO 4519, *Electrodeposited metallic coatings and related finishes — Sampling procedures for inspection by attributes*

ISO 4526[2], *Metallic coatings — Electroplated coatings of nickel and nickel alloys for engineering purposes*

ISO 6158, *Metallic coatings — Electrodeposited coatings of chromium for engineering purposes*

ISO 9220, *Metallic Coatings — Measurement of coating thickness — Scanning electron microscope method*

ISO 9227, *Corrosion tests in artificial atmospheres — Salt spray tests*

ISO 9587, *Metallic and other inorganic coatings — Pretreatments of iron or steel to reduce the risk of hydrogen embrittlement*

ISO 9588, *Metallic and other inorganic coatings — Post-coating treatments of iron or steel to reduce the risk of hydrogen embrittlement*

ISO 10289, *Methods for corrosion testing of metallic and other inorganic coatings on metallic substrates — Rating of test specimens and manufactured articles subjected to corrosion tests*

ISO 10587, *Metallic and other inorganic coatings — Test for residual embrittlement in both metallic-coated and uncoated externally-threaded articles and rods — Inclined wedge method*

ISO 12686, *Metallic and other inorganic coatings — Automated controlled shot-peening of articles prior to nickel, autocatalytic nickel or chromium plating, or as a final finish*

ISO 15724, *Metallic and other inorganic coatings — Electrochemical measurement of diffusible hydrogen in steels — Barnacle electrode method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064, ISO 2079, ISO 2080, ISO 9587 and ISO 9588 apply. See [1] for European terminology.

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[2] To be published. (Revision of ISO 4526:1984)

4 Information to be supplied to the producer of the coating

4.1 Essential information

When ordering articles to be coated in accordance with this International Standard, the purchaser shall provide the following information for all essential items, in writing, as part of the contract, the purchase order, the detailed product specification and/or on engineering drawings:

a) the coating designation (see clause 5);

b) the tensile strength of the part and the requirements for any heat treatment before and after coating deposition (see 6.2, 6.9, 6.10, 6.11, 6.12 and Annex A);

c) the significant surface, indicated by drawings of the articles or by suitably marked samples;

d) the nature, condition and finish of the basis metal, if any of these can affect the serviceability and/or the appearance of the coating (see 6.2);

e) the location, type and dimensions where defects, such as rack marks, may be tolerated (see 6.2);

f) the finish required, e.g., bright, dull, satin or other finish and, if applicable, a sample of the desired finish keeping in mind that approved samples may deteriorate over time and may need to be replaced at regular intervals;

g) any requirements for undercoats (see 6.17);

h) sampling methods, acceptance levels, or other inspection requirements, if different from those given in ISO 4519 (see Clause 7);

i) standard methods for thickness, hardness, adhesion, porosity, corrosion resistance, wear or solderablility testing (see 6.4, 6.5, 6.6, 6.7, 6.8, 6.13, 6.14, and Annex B), and the requirements for special test specimens (see 6.1);

j) requirement for treatments to induce compressive surface stresses, e.g., shot-peening before coating (see 6.16);

k) special requirements for, or restrictions on, pretreatment;

l) special requirements for, or restrictions on, post-treatment;

m) special requirements for maximum coating thickness, especially for the build-up of worn or over-machined parts. It should also be specified whether these thicknesses shall be measured before or after machining of the coating;

n) special requirements for a coating over the autocatalytic nickel coating (see 6.17).

4.2 Additional information

The following additional information may be supplied by the purchaser, as appropriate:

a) the necessity for degaussing (demagnetizing) steel parts before coating to minimize the inclusion of magnetic particles in the coating;

b) the final surface roughness of the coating (see 6.3);

c) any special requirements for the chemical composition of the coating (see 6.15);

d) any special requirements for recovering rejected articles;

e) any other special requirements.

5 Designation of basis metal, metal layers and heat treatment requirements

5.1 General

The designation shall appear on engineering drawings, in the purchase order, the contract or in the detailed product specification. The designation specifies, in the following order, the basis metal, the specific alloy (optional), stress relief requirements, the type and thickness of undercoats, the nominal phosphorus content and thickness of the autocatalytic nickel coating, type and thickness of coatings applied over the autocatalytic layer and post-treatments including heat treatment. Double separators (//) shall be used to indicate that a step or operation has either not been specified or has been omitted.

The designation shall comprise the following:

a) the term "Autocatalytic nickel coating";

b) the number of this International Standard, i.e., ISO 4527;

c) a hyphen;

d) the chemical symbol of the basis metal (see 5.2);

e) a solidus (/);

f) symbols for the autocatalytic nickel coating (see 5.4), as well as coatings applied prior to and after autocatalytic deposition (see 5.4), separated by solidi for each stage in the coating sequence in the order of application. The coating designation shall include the thicknesses of the coatings in µm, as well as heat treatment requirements (see 5.3).

5.2 Designating the basis metal

The basis metal shall be designated by its chemical symbol or, if an alloy, by its principal constituent.

It is recommended that the specific alloy be designated by its standard designation, e.g., its UNS number or the local national equivalent placed between the symbols, < >; e.g., Fe<G43400> is the UNS designation for a high-strength steel. See [2] to [6].

NOTE To ensure proper surface preparation and hence adherence of the coating to the substrate, it is important to identify the specific alloy and its metallurgical condition (tempered, nitrided, etc.).

5.3 Designation of heat treatment requirements

The heat treatment requirements shall be in brackets and designated as follows:

a) the letters SR, for heat treatment for stress relief purposes; the letters HT, for heat treatment to increase the hardness of the coating or the adhesion of the coating to the basis metal; and the letters ER, for hydrogen embrittlement relief;

b) in parentheses, the minimum temperature, in degrees centigrade;

c) the duration of the heat treatment, in hours.

EXAMPLE

A stress relief treatment at 210 °C for 1 h is designated as follows:

[SR(210)1]

5.4 Designating the type and thickness of metal layers

The autocatalytic nickel coating shall be designated by the symbol, NiP, followed by a whole number in parentheses giving the nominal phosphorus content of the coating, followed by a number giving the specified minimum local thickness of the autocatalytic nickel coating, in µm.

Metallic undercoats shall be designated by the chemical symbol(s) for the deposited metal(s) followed by a number specifying the minimum local thickness of the layer, in µm (see 6.17). The symbol Ni is the designation for an electrodeposited nickel undercoat.

Subsequent coatings that are to be deposited upon the autocatalytic nickel layer, e.g., chromium, shall be designated by the chemical symbol of the electrodeposited coating followed by a number specifying the minimum local thickness of the coating, in µm (see 6.17).

5.5 Examples of designations

The following are examples of designations:

a) An autocatalytic nickel-phosphorus coating having a nominal phosphorus content of 10 % mass fraction and a thickness of 15 µm applied to G43400 steel, requiring stress relief prior to coating at 210 °C for 22 h, and subsequently electroplated with chromium, 0,5 µm thick, requiring heat treatment for hydrogen embrittlement relief at 210 °C for 22 h is designated as follows:

   Autocatalytic nickel coating ISO 4527–Fe<G43400>[SR(210)22]/NiP(10)15/Cr0,5[ER(210)22]

b) The same coating on an aluminium alloy, for which there are no heat treatment requirements, is designated as follows:

   Autocatalytic nickel coating ISO 4527–Al<A96061-T6>//NiP(10)15/Cr0,5//

c) The same coating on a copper-base alloy, for which there are no heat treatment requirements, is designated as follows:

   Autocatalytic nickel coating ISO 4527–Cu<C10800>//NiP(10)15/Cr0,5//

For ordering purposes, the detailed product specification shall not only comprise the designation, but shall also contain clear statements of the other essential requirements listed in Clause 4.

6 Requirements

6.1 Special test specimens

Special test specimens may be used to measure adhesion, thickness, porosity, corrosion resistance, hardness and other properties when the coated articles are of a size, shape or material that is not suitable for the test, or if it is not practical to submit the coated articles to destructive tests because the parts are few in number or too expensive. Special test specimens shall be of the same material, shall be in the same metallurgical condition, shall have the same surface condition as the coated articles and shall be processed along with the coated articles that they represent.

The use of special or representative test specimens to determine that the requirements of this International Standard have been met, the number of test specimens to be used, the material from which they shall be made and their shape and size shall be specified by the purchaser.

6.2 Appearance

The autocatalytic nickel coating on the significant surface shall be bright, semi-bright or dull, as specified by the purchaser and, when visually inspected, shall be free from pits, blisters, exfoliation, nodular growths, cracks and other defects detrimental to the final finish, unless otherwise specified. Approved samples with the specified appearance shall be used for comparison purposes [see 4.1 f)].

Imperfections and variations that arise from surface conditions of the basis metal (scratches, pores, roll marks, inclusions), and that persist in the finish despite the observance of good metal finishing practices, shall not be cause for rejection. The purchaser shall specify limits for the tolerable defects on the finished and unfinished product. Damaged basis metals shall not be coated.

Blisters or cracks that are visible to the naked eye and that result from heat treatment performed by the producer of the coating shall be cause for rejection.

NOTE Defects that are present in the basis metal before coating, including hidden defects, may be reproduced by the coating. In addition, stains and the formation of coloured oxides may result from post-coating heat treatment. The latter shall not be cause for rejection unless a special heat treatment atmosphere is specified. It is advisable for the interested parties to agree on the acceptability of such defects.

6.3 Surface finish

If a specified final surface roughness is required, the method of measurement shall be that specified in ISO 4288.

NOTE The surface finish of autocatalytic nickel coatings is not usually superior to that of the substrate before coating, except when the surface of the substrate is extremely smooth and micro-levelling occurs.

6.4 Thickness

The thickness of the coating specified in the designation shall be the minimum local thickness. The minimum local thickness of the coating shall be measured at any point on the significant surface that can be touched by a ball 20 mm in diameter, unless otherwise specified by the purchaser.

Annex C provides guidance on the thickness requirements for corrosion protection under various conditions of service. The thickness shall be measured by one of the methods given in Annex B.

6.5 Hardness

When hardness is specified, it shall be measured by the method given in ISO 4516. The measured hardness of the coating shall be within ± 10 % of that specified by the purchaser.

6.6 Adhesion

The autocatalytic nickel coating shall be adherent to the substrate, and any metallic undercoats. Coatings shall be capable of passing one or more of the adhesion tests given in ISO 2819, as specified by the purchaser.

6.7 Porosity

If required, a maximum degree of porosity of the autocatalytic nickel-phosphorus alloy coating shall be specified by the purchaser together with method of testing for porosity.

6.8 Corrosion resistance

If required, the corrosion resistance and the corrosion test method shall be specified by the purchaser who shall specify acceptance criteria in accordance with ISO 10289. The test methods included in ISO 9227, acetic acid salt spray and copper accelerated salt spray, may be specified for evaluating the pitting corrosion resistance of the coatings.

NOTE Corrosion testing in artificial atmospheres does not necessarily relate to the service life or performance of the finished article.

6.9 Stress relief heat treatment prior to coating

When specified by the purchaser [see 4.1 b)], steel parts that have an ultimate tensile strength equal to or greater than 1 000 MPa and that contain tensile stresses caused by machining, grinding, straightening or cold-forming operations shall be given a stress relief treatment prior to cleaning and metal deposition. The procedures and classes for stress relief treatment shall be as specified by the purchaser or the purchaser may specify appropriate procedures and classes in accordance with ISO 9587. Stress relief shall be carried out before any acidic or cathodic electrolytic treatments are applied.

NOTE Steels with oxide or scale should be cleaned before application of the coatings. For high strength steels, non-electrolytic alkaline and anodic alkaline cleaners, as well as mechanical cleaning procedures, are preferred in order to avoid the risk of producing hydrogen embrittlement during cleaning operations.

6.10 Hydrogen embrittlement relief heat treatment after coating

Steel parts having an ultimate tensile strength equal to or greater than 1 000 MPa, as well as surface hardened parts, shall receive hydrogen embrittlement relief heat treatment after coating according to the procedures and classes given in ISO 9588 or as specified by the purchaser [see 4.1 b)].

Any heat treatment for the relief of hydrogen embrittlement after coating shall be carried out as soon as possible, preferably within 1 h but not later than 3 h after surface finishing and before commencement of grinding or other mechanical operation.

The effectiveness of the embrittlement relief treatment may be determined by a test method specified by the purchaser or by test methods described in ISO Standards; e.g., ISO 10587 describes a method of testing threaded articles for residual hydrogen embrittlement and ISO 15724, one for measuring the diffusible hydrogen concentration in steels.

NOTE Heat treatment in accordance with the procedures and classes given in ISO 9588 does not guarantee complete freedom from hydrogen embrittlement, and tests for residual hydrogen embrittlement should be specified, whenever possible. Freedom from failure of test samples can demonstrate the effectiveness of the hydrogen embrittlement heat treatment procedure, depending on the number of samples that are tested.

6.11 Heat treatment to harden the coating

Table A.1 provides guidance on heat treatment to increase the hardness of autocatalytic nickel-phosphorus alloy coatings so as to improve wear resistance (see 6.13).

Heat treatment to increase the hardness of autocatalytic nickel-phosphorus alloy coatings so as to improve wear resistance shall be performed within 1 h of coating, when required. Heat treatment shall be carried out before mechanical finishing. The duration of the heat treatment shall be 1 h minimum after the part has reached the specified heat treatment temperature.

If heat treatment to harden the coating is carried out, separate hydrogen embrittlement relief heat treatment may not be necessary, provided that the requirements of ISO 9588 have been met (see 6.10).

6.12 Heat treatment to improve adhesion

Heat treatment to improve the adhesion of autocatalytic nickel coatings on certain basis metals shall be carried out in accordance with Table A.1, unless the purchaser specifies other procedures.

6.13 Wear resistance

If required, the wear resistance of the coating shall be specified by the purchaser who shall also specify the wear resistance test method to be used to ensure that the requirement has been met.

NOTE Wear resistance can be affected by heat treating autocatalytic nickel coatings (see 6.11 and Annex A).

6.14 Solderability

If required, the solderability of the coating shall be specified by the purchaser who shall also specify the solderability test method to be used to ensure that the requirement has been met (see C.5).

NOTE Coatings containing greater than 10 % mass fraction phosphorus are sometimes used for soldering in order to minimize the possibility of corrosion during the soldering process, especially in electronic applications. More often, coatings containing low levels of phosphorus (1 % to 3 % mass fraction) are specified for soldering purposes.

6.15 Chemical composition

The phosphorus content of the nickel-phosphorus alloy coating shall be that specified in the designation (see 5.4 and Table C.2). When measured by the method given in Annex D, the mass fraction of phosphorus shall be within ± 0,5 % of that required. If the phosphorus content is not specified, it shall be within the range 1 % mass fraction to 14 % mass fraction, unless otherwise specified [see 4.2 c)].

6.16 Peening of metal parts

If peening prior to coating is specified by the purchaser, it shall be performed in accordance with ISO 12686 which also describes the method for measuring peening intensity, and shall be carried out before any acidic or cathodic electrolytic treatments.

NOTE Shot-peening prior to coating can minimize the reduction in fatigue strength and adhesion that occurs when high-strength steels are coated with autocatalytic nickel coatings and is recommended for parts subjected to repeated applications of complex load patterns in service. Other factors that affect fatigue strength include thickness, which should be kept as thin as is compatible with the expected service condition. The compressive stresses resulting from controlled shot-peening increase corrosion resistance and resistance to stress corrosion cracking, and can have a beneficial effect on coating adhesion.

6.17 Undercoats and overcoats

Electrolytic nickel undercoats [see 4.1 g)] shall comply with ISO 4526. Chromium coatings applied on top of autocatalytic nickel-phosphorus alloy coatings shall comply with ISO 6158.

NOTE Electrodeposited nickel undercoats 2 µm to 5 µm thick may be applied to basis metals (except brass and bronze) that contain more than trace quantities of antimony, arsenic, bismuth, copper, lead or tin. Autocatalytic nickel, electrodeposited nickel or electrodeposited copper undercoats 2 µm to 5 µm thick may be applied to basis metals that contain more than trace quantities of magnesium and zinc. An electrolytic nickel strike between the copper undercoat and the autocatalytic nickel coating may be applied. Electrodeposited nickel undercoats 1 µm to 2 µm thick may be applied to basis metals containing more than trace amounts of chromium, lead, molybdenum, nickel, tin, titanium or tungsten. The purpose of undercoats is to reduce the risk of contaminating the processing solution with elements that may lower the rate of deposition. In addition, electroplated metallic undercoats help prevent the diffusion of impurities from the basis metal into the autocatalytic coating, and help improve adhesion.

7 Sampling

The method of sampling shall be selected from the procedures specified in ISO 2859-1, ISO 2859-2, ISO 2859-3, ISO 2859-4 or ISO 4519, or the purchaser shall specify an alternate plan. The purchaser shall specify the acceptance levels.

Annex A
(normative)

Heat treatment to improve adhesion and increase hardness

A.1 Heat treatment to improve adhesion

The times and temperatures given in Table A.1 shall be used to improve the adhesion of autocatalytic nickel-phosphorus alloy coatings deposited directly on various alloys, unless otherwise specified by the purchaser. For coatings thicker than 50 µm, the heat treatment times shall be increased.

NOTE The tensile strength of heat treatable aluminium and various alloys can be reduced by heating above 130 °C. It is recommended that the effect on the mechanical properties of the substrate be considered and, if necessary, verified when the purchaser specifies heat treatment after coating in order to improve adhesion.

A.2 Heat treatment to improve hardness and wear resistance

Autocatalytic nickel-phosphorus alloy coatings are frequently precipitation hardened by heat treatment in order to improve wear resistance. The times and temperatures given in Table A.1 shall be used.

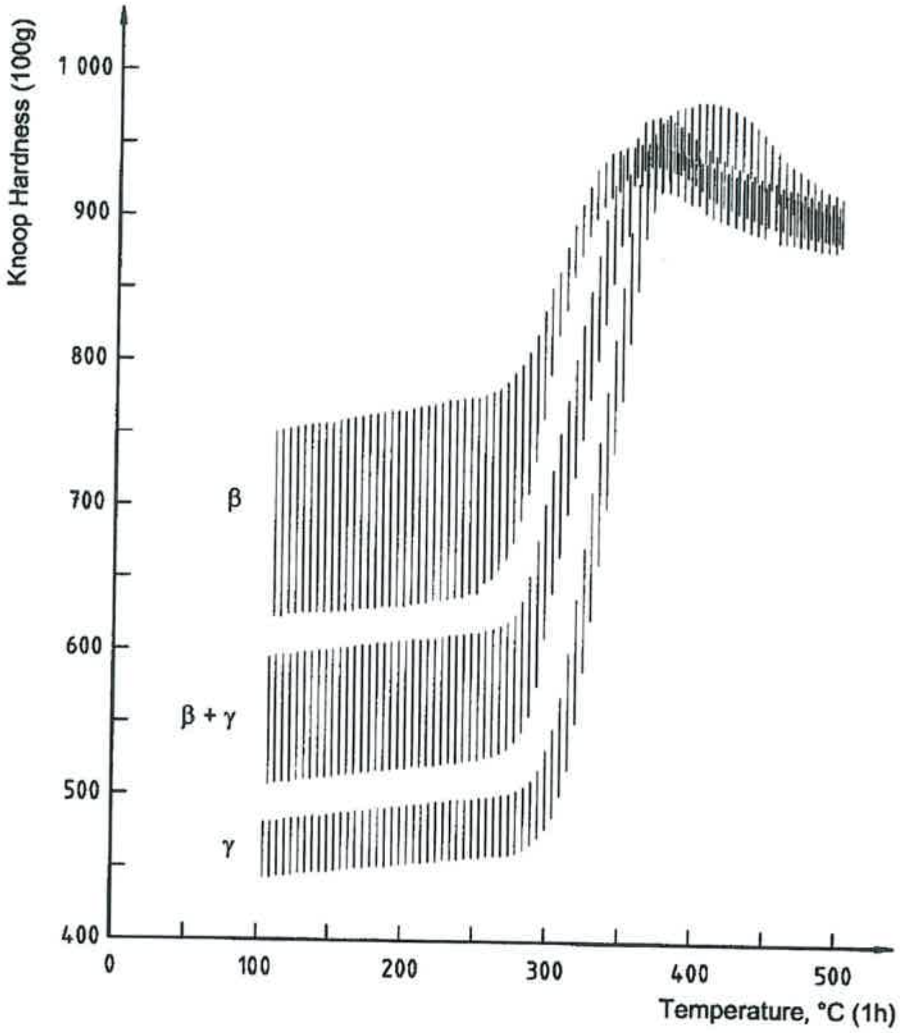
Table A.1 — Recommended heat treatments to improve hardness and adhesion

Class	Description	Temperature °C	Time h
1	No heat treatment; as-deposited		
2	Heat treatment for maximum hardness, by type (see Table C.2):		
	1	260	20
		285	16
		320	8
		400	1
	2	350 to 380	1
	3	360 to 390	1
	4	365 to 400	1
	5	375 to 400	1
3	For adhesion on to steel	180 to 200	2 to 4
4	For adhesion on to carburized steel and age-hardened aluminium	120 to 130	1 to 6
5	For adhesion on to beryllium and non-aged hardened aluminium	140 to 150	1 to 2
6	For adhesion on to titanium and titanium alloys	300 to 320	1 to 4
7	For adhesion on to magnesium and its alloys; copper and its alloys	180 to 200	2 to 2,5
8	For adhesion on to nickel and its alloys	220 to 240	1 to 1,5
9	For adhesion on to molybdenum and its alloys	190 to 210	2 to 2,5

In general, the hardness after heat treatment increases as the phosphorus content is decreased (Figure A.1). The hardness can be increased further by heat treating between 250 °C and 400 °C for longer than 1 h. Heat treatment above 220 °C that causes the hardness to exceed 850 KHN100 can reduce the corrosion resistance of the coatings. Heat treatment at temperatures below 200 °C to improve adhesion or to minimize the risk of hydrogen embrittlement do not impair corrosion resistance or substantially increase the hardness or improve the wear resistance of the coating. When necessary, the heat treatment should be performed in an inert or reducing atmosphere, or in vacuum to prevent coloured oxides from forming on the surface. Heat treatment above 260 °C will cause type 5 coatings to become magnetic.

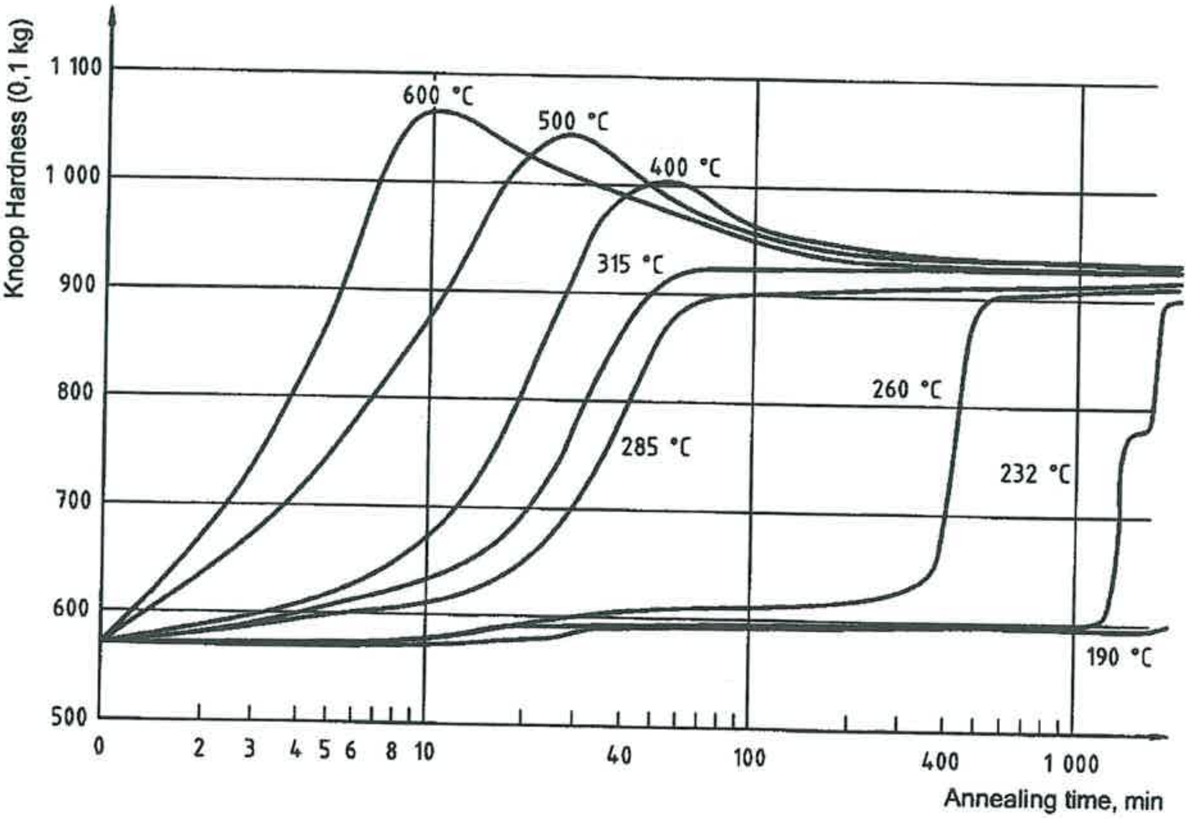
The relationship between heat treatment temperature and hardness after 1 h of heating is shown in Figure A.1 for different types of autocatalytic nickel-phosphorus coatings. The relationship between hardness and annealing time is shown in Figure A.2. The data in Figure A.2 indicate that equivalent coating hardness can be obtained by lowering the temperature and extending the annealing time.

NOTE The Knoop indenter is generally preferred for measuring the hardness of metallic coatings because the uncertainty in the measurement is reportedly less with Knoop than with Vickers. The greater variability may be due to a tendency for thin, brittle coatings to crack during Vickers hardness testing. If measured properly, the numerical values are approximately the same. However, to make proper measurements would involve using different loads to obtain the same depth of indentation. To establish a valid correlation, side-by-side measurements would have to be made on identical coatings using different loads. As far as is known, that type of study has not been performed with autocatalytic nickel-phosphorus alloy coatings.



Phase	Alloy type	Phosphorus % by mass
β	2, 3	1 to 4
β + γ	4	5 to 9
γ	5	> 10

Figure A.1 — Relationship between heat treatment temperature and hardness after 1 h's heating for different types of autocatalytic nikel deposits



NOTE By changing the annealing time and lowering the temperature, equivalent deposit hardness can be achieved. The curves are based averages and the results may vary with deviations from standard practice.

Figure A.2 — Relationship between hardness and annealing time

Annex B
(informative)

Thickness test methods

B.1 General

ISO 3882 reviews methods of measuring thickness of metallic and other inorganic coatings, including methods that are not cited below.

B.2 Destructive

B.2.1 Microscopical method

Use the method specified in ISO 1463.

B.2.2 Coulometric method

The coulometric method specified in ISO 2177 may be used to measure the total thickness of the autocatalytic nickel thickness and the thickness of copper and nickel underlayers, when present, at any point on the significant surface than can be touched by a ball 20 mm in diameter.

B.2.3 Scanning electron microscope method

The scanning electron microscope method described in ISO 9220 may be used to measure the thickness of the autocatalytic nickel and the thickness of underlayers.

NOTE In cases of dispute, the coulometric method should be used for measuring the thickness of coatings less than 10 µm, and the microscopical method for measuring the thickness of nickel coatings and undercoats 10 µm and above.

B.3 Non-destructive

B.3.1 Beta backscatter method (applicable only in the absence of copper undercoats)

Use the method specified in ISO 3543. This method is suitable for measuring coatings on aluminium substrates and determines the total coating thickness.

B.3.2 X-Ray spectrometry

Use the method specified in ISO 3497. X-ray apparatus should be calibrated with thickness standards whose coatings contain known quantities of phosphorus that are similar to or encompass the phosphorus content of the coatings being tested.

NOTE Due to possible local variation in the phosphorus content of the coating, integral methods for phosphorus determination covering a sector of the referenced area are recommended and are frequently utilized.

B.3.3 Weigh-plate-weigh method

Using a part of known surface area (or a special test specimen with a similar substrate material of known surface area), weigh the part or test coupon to the nearest milligram before and after coating. Ensure that the part or coupon is dry and at room temperature for each measurement. Calculate the thickness from the increase in weight, the coating density, and area as follows:

$$T = \frac{10W}{(A \times D)}$$

where

T is the coating thickness, in micrometres;

W is the weight gain, in milligrams;

A is the total surface area, in square centimetres;

D is the density, in grams per cubic centimetre.

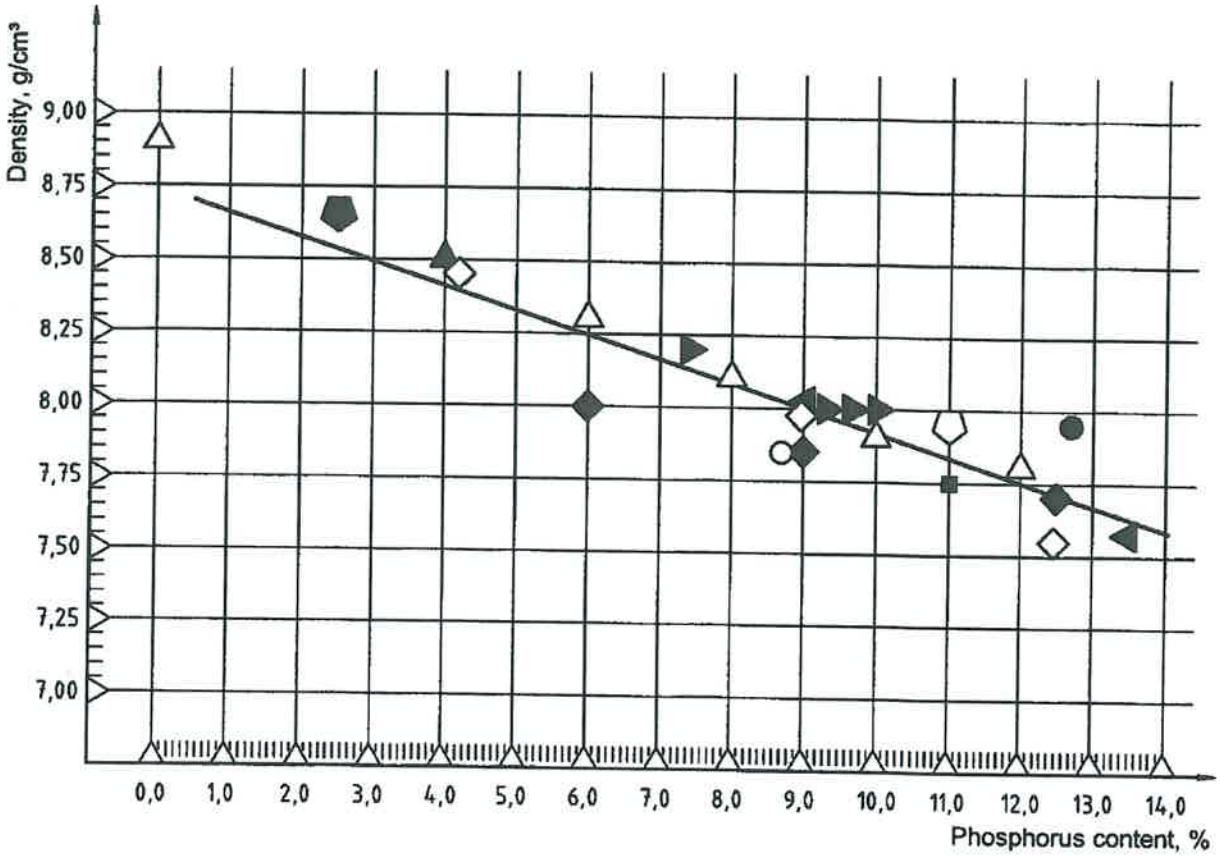
The density of the coating depends on the phosphorus content of the deposit. The density of autocatalytic nickel coatings is given in Figure B.1, based on values reported in the technical literature.

EXAMPLE A coupon of mild steel has a weight of 3 198 mg with an area of 19,736 cm^2 before plating. After plating, the coupon weighs 3 583 mg. The phosphorus content of the coating is 9 % and the density is 8,01 g/cm^3 (from Figure B.1). The thickness is calculated as follows:

$$T = \frac{10(3583-3198)}{19,763 \times 8,01} = 24,3\,\mu\text{m}$$

B.3.4 Magnetic method

Use the method described in ISO 2178 to measure the thickness of autocatalytic nickel-phosphorus alloy coatings containing greater than 8 % mass fraction phosphorus that are sufficiently non-magnetic to be measured by this method.



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◆ From Annex A

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Figure B.1 — Density of autocatalytic (electroless) nickel-phosphorus alloy coatings

Annex C
(informative)

Guidance on thickness, composition and use of autocatalytic nickel-phosphorus coatings

C.1 General

The properties of autocatalytic nickel-phosphorus alloy coatings depend primarily on the composition and structure of the coating. The composition and structure of the coating are in turn determined by the composition of the plating solution, the conditions of deposition and subsequent heat treatment that alters the structure of the deposit. The nature of the substrate, such as surface roughness, may also affect the properties of the coating, including its corrosion resistance.

C.2 Corrosion resistance, coating thickness and service conditions

The minimum coating thickness required for adequate corrosion resistance in different service conditions is given in Table C.1. Coatings should be thicker on rough or porous surfaces in order to minimize the influence of the substrate on the deposit's properties. To obtain optimum corrosion resistance with the minimum thickness of coating, the surface of the substrate should be smooth and pore-free. A substrate surface roughness of approximately $Ra < 0{,}2$ µm may be used as a guide.

Table C.1 — Minimum coating thickness required for corrosion resistance in service

Service condition number	Description	Minimum coating thickness on ferrous base µm	Minimum coating thickness on aluminium base µm
5 (Exceptionally severe)	Service outdoors where frequent wetting and abrasion are factors; e.g., oil field applications.	125	–
4 (Very severe)	Outdoor marine and other aggressive environments, severe abrasion, exposure to acid solutions, elevated temperature and pressure.	75	–
3 (Severe)	Non-marine service outdoors where frequent wetting due to rain and dew, moderate abrasion, exposure to alkali salts at elevated temperatures are factors.	25	60
2 (Moderate)	Service indoors where condensation can occur; indoor industrial exposures in dry or oiled environments.	13	25
1 (Mild)	Service indoors in warm dry atmosphere; for soldering and mild abrasive wear.	5	13
0 (Very mild)	Highly specialized electronic and semiconductor applications, thin film resistors, capacitors, inductors and diffusion welding.	0,1	0,1

In addition to thickness, the phosphorus content of the coating and other factors influence the corrosion performance of autocatalytic nickel-phosphorus coatings. In general, the corrosion resistance of the coatings in acidic environments improves as the phosphorus content is increased. The excellent corrosion resistance of these coatings is due to a phosphorus-rich, passive oxide film usually present on the surface. Impurities that are co-deposited with the alloy, however, can undermine the passive oxide film and, consequently, reduce corrosion resistance.

C.3 Type and phosphorus content of coatings for different applications

The autocatalytic nickel deposition process can be controlled and adapted to yield coatings with properties that meet the requirements of different applications, thus affording engineers the opportunity to specify the properties required for a specific end-use. Table C.2 describes the types and phosphorus contents of coatings that are commonly specified for various applications.

Table C.2 — Types of autocatalytic nickel coatings recommended for different applications

Type	Phosphorus, mass fraction %	Applications
1	No specified requirement for phosphorus content	General purpose coating
2 (low-phosphorus)	1 to 3	Electrical conductivity, soldering, wire bonding
3 (low phosphorus)	2 to 4	Adhesive and abrasive wear requiring high as-deposited hardness
4 (medium phosphorus)	5 to 9	General purpose wear and corrosion resistance
5 (high phosphorus)	> 10	High as-deposited corrosion resistance, non-magnetic, diffusion weldable, flexible with high elongation; e.g., deposits containing 12,5 % mass fraction phosphorus as underlayers on hard discs.

C.4 Reclaiming worn or over-machined articles

Autocatalytic nickel coatings equal to or greater than 125 µm can be deposited to repair worn articles or to salvage ones that have been over-machined. The likelihood of nodule formation, staining, pitting and surface roughness increases with thickness, and the level of acceptance relative to those defects shall be agreed between purchaser and supplier. Because of lower internal stress, higher ductility and enhanced corrosion resistance, a coating with 10 % mass fraction or more phosphorus may be more suitable for reclaiming worn or over-machined articles than a coating with low or medium amounts. Heat treatment to improve adhesion may be performed (see Annex A).

Electrodeposited nickel undercoats are sometimes applied prior to autocatalytic deposition when coating thickness exceeds 125 µm. The electrodeposited nickel may have to be machined to size before applying the autocatalytic nickel deposit.

C.5 Imparting solderability to metals that are difficult to solder

Autocatalytic nickel coatings are used to improve the solderability of aluminium and other alloys that are difficult to solder, the coating thickness being greater than 2,5 µm. A mildly active rosin flux is normally required for soldering.

C.6 Additional information for different applications

Autocatalytic nickel coatings of similar properties are not suitable for applications where adhesive wear is likely to occur, unless the coated surfaces are lubricated.

Medium- and low-phosphorus coatings are not recommended in applications where flexing or resistance to shock is required. Special care should be taken when welding items that have been coated. Welds made on coated areas may be embrittled by the diffusion of phosphorous from the coating. The coatings have a low hot hardness and are not suitable for use in cases where both wear and elevated temperatures are involved.

Some steels containing chromium and molybdenum may become passivated by anodic cleaning if a periodic reverse-current technique is used. For steels with a tensile strength below 1 000 MPa, cathodic cleaning in place of periodic reverse-current cleaning may be used.

Most metals have oxides on their surfaces that may affect the adhesion of the coating to the base metal. Special cleaning and activation procedures exist for many metals including stainless steels and aluminium. The presence of oxide films can cause adhesion failure, therefore surfaces will require removal of oxide films and any micro-constituents that may interfere with the formation of a continuous coating on the surface.

For cast iron and aluminium alloys, the presence of pores on the surfaces may cause corrosion problems due to entrapment of solutions in the pores and/or absence of continuity of the coating. The coating of cast surfaces having excessive porosity, therefore, requires special treatments for achieving the desired service life of the article.

In leaded copper alloys, lead present on the surface can contaminate the coating solution and can cause adhesion failures and porosity in the coating. Hence, special treatments should be used to cover or remove lead, prior to autocatalytic nickel deposition.

Annex D
(normative)

Methods for chemical analysis of autocatalytic nickel coatings for phosphorus content

D.1 Inductively coupled plasma (ICP) method

D.1.1 General

Method of analysis by either emission or absorption spectra produced by inductively coupled plasma (ICP).

D.1.2 Reagents

Analytical grade chemicals and distilled or deionized water shall be used for preparing the following test solutions:

a) nitric acid (HNO_3), 40 % volume fraction;

b) sodium nitrite ($NaNO_2$), 20 g/l solution;

c) potassium permanganate ($KMnO_4$), 7,6 g/l solution.

The nitric acid, 40 % volume fraction test solution is prepared by mixing 2 parts by volume of nitric acid having a specific gravity of approximately 1,42 g/ml with 3 parts by volume of water.

D.1.3 Procedure

Carefully weigh approximately 0,2 g of the test specimen and dissolve in 50 ml of the nitric acid solution in a glass beaker. Heat gently in a fume cupboard until the specimen material is dissolved, then boil until emission of brown fumes ceases. Dilute the solution to approximately 100 ml, bring to the boil and add 25 ml of the potassium permanganate solution. Boil for 5 min and then add the sodium nitrite solution drop by drop until the precipitated manganese dioxide is dissolved. Boil the solution for 5 min and allow it to cool to ambient temperature. Transfer to a 250 ml standard flask and dilute to the mark with distilled or deionized water. Stopper the flask and shake well.

D.1.4 Blank determination

Carry out a blank determination by preparing a blank solution as above, omitting only the test material.

D.1.5 Spectral analysis

Carry out the determination in accordance with the operating instructions for the ICP equipment. The following spectral lines have been found to have low interference when using argon ICP techniques:

Ni 216,10 nm	Cd 214,44 nm	Fe 238,20 nm
P 215,40 nm	Co 238,34 nm	Pb 283,30 nm
P 213,62 nm	Cr 284,32 nm	Sn 198,84 nm
Al 202,55 nm	Cu 324,75 nm	Zn 206,20 nm

NOTE The inductively coupled plasma method is capable of determining the phosphorus content of the autocatalytic nickel coating to within 0,5 %.

D.2 Molecular absorption spectrometric method

D.2.1 General principle

This annex specifies a molecular absorption spectrometric method for the determination of the phosphorus content of autocatalytic nickel-phosphorus coatings. A portion of the sample is dissolved in nitric acid. The solution is treated with potassium permanganate to precipitate manganese dioxide which is dissolved by adding sodium nitrite. Ammonium molybdate and ammonium vanadate are added and the absorbance of the solution is measured spectrometrically at a wavelength of 420 nm.

D.2.2 Reagents

D.2.2.1 General

Analytical-grade reagents and distilled water or water of equivalent grade shall be used throughout.

D.2.2.2 Reagents for dissolution and oxidation

a) Nitric acid, 40 % volume fraction solution, prepared by mixing 2 parts by volume of nitric acid having a specific gravity of approximately 1,42 g/ml with 3 parts by volume of water;

b) Sodium nitrite, 20 g/l;

c) Potassium permanganate, 7,6 g/l.

D.2.2.3 Molybdate–vanadate solution

Dissolve separately in hot water 20 g of ammonium molybdate and 1 g of ammonium vanadate. Mix the two solutions, add 200 ml of nitric acid (specific gravity approximately 1,42 g/ml), and dilute to 1 l with water. Mix well.

D.2.2.4 Phosphorus standard solution (100 mg/l of P)

Weigh 0,439 2 g of potassium dihydrogen orthophosphate $(KH_2)PO_4$, dissolve it in water and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix well.

1 ml of this solution contains 0,1 mg of P.

D.2.3 Apparatus

D.2.3.1 Ordinary laboratory apparatus

D.2.3.2 Spectrophotometer or photoelectric absorptiometer, fitted with a filter providing maximum transmission at a wavelength of 420 nm, equipped with optical cells of path length 10 mm.

D.2.4 Procedure

D.2.4.1 Preparation of the test solution

a) Weigh, to the nearest 0,1 mg, 0,19 g to 0,21 g of the test specimen. Transfer it to a beaker and dissolve it in 50 ml of nitric acid solution [D.2.2.2 a)].

b) Heat gently until the test specimen is completely dissolved. Then boil to remove brown fumes.

c) Dilute the solution to approximately 100 ml, bring to the boil and add 25 ml of the potassium permanganate solution [D.2.2.2 c)].

d) Boil the solution for 5 min.

e) Add the sodium nitrite solution [D.2.2.2 b)] drop by drop until the precipitated manganese dioxide is dissolved.

f) Boil the solution for 5 min and then allow it to cool to ambient temperature.

g) Transfer the solution to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix well.

D.2.4.2 Blank test

Carry out a blank test in parallel with the determination, by the same procedure, using the same quantities of all reagents as in the determination, but omitting the test specimen.

D.2.4.3 Preparation of the calibration graph

Into a series of 100 ml one-mark volumetric flasks, introduce the volumes of the phosphorus standard solution (D.2.2.4) shown in Table D.1.

Table D.1 — Preparation of phosphorus calibration solutions

Volume of phosphorus standard solution ml	Corresponding mass of phosphorus (P) mg
0	0
2	0,2
4	0,4
6	0,6
8	0,8
10	1,0

Treat the contents of each flask as follows:

a) Add 25 ml of the molybdate-vanadate reagent (D.2.2.3), add water to the mark and let the solution stand for 5 min. Fill one of the optical cells with the solution.

b) Carry out the spectrometric measurement using either the spectrophotometer at the wavelength of maximum absorption (about 420 nm) or the photoelectric absorptiometer fitted with suitable filters after having, in each case, adjusted the apparatus to zero absorption against water. Deduct the absorption of the calibration compensation solution from those of the other calibration solutions.

c) Plot a graph with the phosphorus mass, in milligrams, contained in the calibration solution on the abscissa versus the corresponding value of the absorbance on the ordinate.

D.2.5 Dose

a) Transfer 10 ml of the test solution to a 100 ml one-mark volumetric flask, add 50 ml of water, 25 ml of the molybdate-vanadate solution, add water to the mark, and mix well. Allow the solution to stand for 5 min. Fill one of the optical cells with the solution. For purposes of comparison, also test a control sample that does not contain the molybdate-vanadate solution.

b) Carry out the spectrometric measurement as described in D.2.4.3 b).

D.2.6 Expression of results

By means of the calibration graph, determine the mass of phosphorus corresponding to the spectrometric measurement.

The phosphorus content, as a percentage by mass, is given by the following formula:

$$\text{Phosphorus content} = \frac{2{,}5(m_3 - m_4)}{m}$$

where

m_3 is the mass of phosphorus, in milligrams, found in the aliquot portion of the test solution used for the determination;

m_4 is the mass of phosphorus, in milligrams, found in the corresponding aliquot portion of the blank test solution;

m is the mass, in grams, of the test portion.

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[6] EN 10088-1:1995, *Stainless steels — Part 1: List of stainless steels*

NOTE European Standards (EN) and CEN Reports (CR) are available from the European Committee for Standardization (CEN), rue de Stassart 36, B-1050, Brussels, Belgium. ASTM Standards are available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA, 19428-2959, USA.

Annex ZA
(normative)

Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

NOTE Where an International Publication has been modified by common modifications, indicated by (mod.), the relevant EN/HD applies.

Publication	Year	Title	EN	Year
ISO 1463	1982	Metallic and oxide coatings - Measurement of coating thickness - Microscopical method	EN ISO 1463	1994
ISO 2064	1996	Metallic and other inorganic coatings - Definitions and conventions concerning the measurement of thickness	EN ISO 2064	2000
ISO 2177	1985	Metallic coatings - Measurement of coating thickness - Coulometric method by anodic dissolution	EN ISO 2177	1994
ISO 2178	1982	Non-magnetic coatings on magnetic substrates - Measurement of coating thickness - Magnetic method	EN ISO 2178	1995
ISO 2819	1980	Metallic coatings on metallic substrates - Electrodeposited and chemically deposited coatings - Review of methods available for testing adhesion	EN ISO 2819	1994
ISO 3497	2000	Metallic coatings - Measurement of coating thickness - X-ray spectrometric methods	EN ISO 3497	2000
ISO 3882	1986	Metallic and other non-organic coatings - Review of methods of measurement of thickness	EN ISO 3882	1994
ISO 4516	2002	Metallic and other inorganic coatings - Vickers and Knoop microhardness tests	EN ISO 4516	2002
ISO 9220	1988	Metallic coatings - Measurement of coating thickness - Scanning electron microscope method	EN ISO 9220	1994
ISO 10289	1999	Methods for corrosion testing of metallic and other inorganic coatings on metallic substrates - Rating of test specimens and manufactured articles subjected to corrosion tests	EN ISO 10289	2001

Annex DEF 3

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The effect of the hypophosphite ion oxidation on the Ni surface electrode—an XPS study

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Abstract

The effect of the hypophosphite electrooxidation on the Ni surface state was analysed by XPS on electrodes previously submitted to constant-potential polarization in an alkaline solution containing the hypophosphite ion. It was found that Ni surface exhibits the same electronic states than in a hypophosphite-free solution. The XPS spectra of P 2p and P 2s revealed the formation of alloyed phosphorus on the Ni surface. Its dependence on the electrode potential allows concluding that hypophosphite ion participates in competitive surface reactions in the same potential range. The overall results are discussed in the light of the mechanisms proposed in the literature for the phosphorus incorporation process during Ni–P alloy electrodeposition.

Keywords: Electrode materials; Metals and alloys; Electrochemical reactions; Photoelectron spectroscopies

1. Introduction

The hypophosphite ion is a well-known reducing agent for electroless deposition process [1] and electrodeposition of alloys [2] and it is also a promising hydrogen donor in catalytic hydrogenation of organic molecules [3]. The electrooxidation mechanism of the hypophosphite ion on nickel has been the subject of several studies in last 10 years [4–8]. The use of in situ spectroelectrochemical techniques has allowed to characterize adsorbed and solution species involved in this electrode process: (a) hypophosphite ions are adsorbed via its two hydrogen atoms on the nickel surface electrode, from open-circuit potential until the onset of the oxidation potential; (b) at rather negative potentials the adsorption of hypophosphite leads to the cleavage of the P–H bond with the formation of a phosphorus-centred radical, which is subsequently oxidized to the final product, phosphite.

According to the literature, no studies have been undertaken so far with the purpose of evaluating the effect of the hypophosphite oxidation on the nickel surface electrode. The detection and identification of surface species would be important not only to provide more information about hypophosphite oxidation mechanism, but also to better understand the mechanism of phosphorus incorporation during electroless deposition and electrodeposition of Ni–P alloys [9] and to evaluate whether surface species could be responsible for unusual selective organic reactions on Ni in hypophosphite containing solutions [10].

The main purpose of this study is to characterize, by X-ray photoelectron spectroscopy (XPS) technique, Ni surface electrodes previously submitted to constant potential electrolysis in alkaline solutions containing hypophosphite ion. A comparative study was also performed on electrodes prepared in a hypophosphite-free solution.

2. Experimental

2.1. Sample preparation

In the preparation of Ni electrodes for the XPS analysis a 1.5 cm × 1.5 cm Ni foil substrate (99.9%) previously polished with 0.3 μm alumina and electropolished in 57% H_2SO_4 was used. After the electrode surface was rinsed several times with distilled deionised water it was submitted to a constant electrode potential, for 3600 s, in a 0.375 M $H_2PO_2^- + 0.10$ M NaOH solution. Afterwards it was rinsed again with distilled deionised water and dried on a N_2 flow. Details of the cell set up and polishing procedure were described elsewere [10].

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The electrode potential was controlled by an Autolab potentiostat model 100 and is referred to a saturated calomel electrode (SCE).

2.2. XPS analysis

The spectrometer used was a XSAM800 (KRATOS) operated in the fixed analyser transmission (FAT) mode, with a pass energy of 10 eV, a power of 130 W (10 mA and 13 kV) and the non-monochromatised MgK^* X-radiation (h^* = 1253.6 eV). Samples were analysed in a UHV chamber ($\sim10^{-7}$ Pa) at room temperature, using 0° and 60° analysis angles relative to the normal to the surface. Samples were transferred to the fast introduction chamber under ambient atmosphere. Spectra were recorded by a Sun SPARC Station 4 with Vision software (Kratos) using a step of 0.1 eV. A Shirley background was subtracted and curve fitting for component peaks was carried out with a non-linear least-squares algorithm using pseudo-Voigt profiles. No charge compensation (flood-gun) was used. Binding energies were corrected by using contamination carbon (binding energy = 285 eV) as a reference. X-ray source satellites were also subtracted. For quantification purposes, sensitivity factors were 0.66 for O 1s, 0.25 for C 1s, 4.55 for Ni 2p and 0.26 for P 2s. These factors are included in the Vision software and were checked using several calibrating salts as $NiBr_2$, for instance.

3. Results and discussion

On the preparation of the sample electrodes, the negative potential that was applied (−1.1 V) was selected on account of previous work [3,4,11]. It is known that at this potential the electrode exhibits the highest activity to oxidize the hypophosphite ion. Despite the rather long electrode polarization time, 3600 s, the current evolution recorded during the electrode polarization, Fig. 1, reveals that the oxidation of hypophosphite persists all along. In the present experimental conditions no gas evolution was detected, contrasting to what was observed in other experimental conditions [1,5]. Surface characterization of the Ni electrodes prepared at −1.1 V for 3600 s was performed by XPS by analysing the electronic states of nickel and phosphorus. For comparison, the spectrum obtained in the Ni 2p region for the Ni electrode prepared in a hypopohosphite-free solution was also examined.

In Fig. 2, the XPS Ni 2p region is shown for electrodes submitted to polarization in 0.375 M $H_2PO_2^-$ + 0.10 M NaOH and 0.10 M NaOH solutions. For both samples, the Ni 2p exhibits its doublet—Ni $2p_{1/2}$ and Ni $2p_{3/2}$ with a distance around 17.6 eV. Analysis of the Ni $2p_{3/2}$ region in both electrodes show three main components centred at 852.2 ± 0.1 eV, 855.7 ± 0.1 eV and 861.5 ± 0.1 eV. The first one is assignable to metallic nickel and has a full width at medium height (fwmh) of 1.37 eV. Its relative importance is larger in the electrode submitted to potentiostatic polarization in the hypophosphite solution showing that it is the less oxidized sample. In the literature (see Table 1) a large dispersion of values for Ni $2p_{3/2}$ biding energy (BE) is found ranging from 852.6 to 858.3 eV. The source of the lowest value dates from 1983 [13] whereas the source of the highest one dates from 1972 [14a] when XPS spectrometers were far from reliable. Recent data from a French group gives a value of 852.6 eV [14b]. Concerning the second component, the assignment is much more difficult: this value may be assigned to NiO, Ni_2O_3 or $Ni(OH)_2$ (see Table 1). Given its width (fwmh ∼3 eV) it may be a mixture of them. Since its position and shape are the same for both electrodes and taking into account the fact that the modified electrodes were prepared at rather negative potentials and in a solution containing a reducing agent (hypophosphite), Ni_2O_3 is

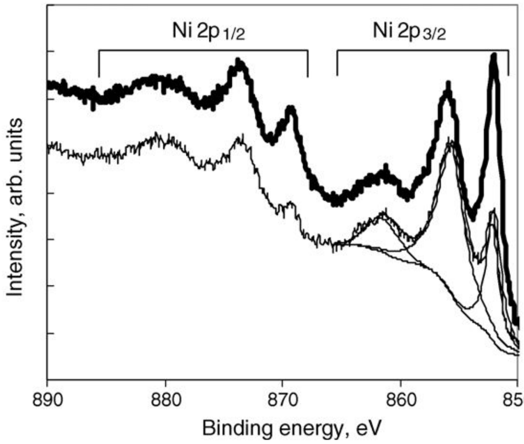

Fig. 2. XPS Ni 2p for nickel electrodes submitted to constant potential polarization in NaOH (bottom line) and $H_2PO_2^-$ + NaOH (top line) solutions by constant potential electrolysis at −1.1 V. Curve fitting with three peaks for the Ni $2p_{3/2}$ regions is shown in the lower spectrum. For the sake of clarity, just the curve fitting for the NaOH treated electrode is shown.

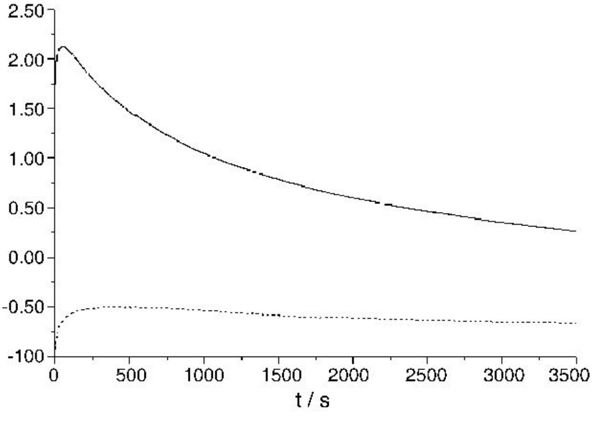

Fig. 1. Current–time profile for the Ni electrode at −1.1 V in 0.375 M $H_2PO_2^-$ + 0.10 M NaOH solution (solid line) and in 0.10 M NaOH solution (dashed line).

Table 1
Binding energies of P_{2p}, P_{2s} and $Ni_{2p3/2}$ core-level lines of standard substances

Standard compounds	BE (eV)			Ref.
	P 2p	P 2s	Ni $2p_{3/2}$	
Ni			852.6–853.8	[12–14]
NiO			853.6–857.2	[12,15,16]
Ni_2O_3			855.8–856.0	[12]
$Ni(OH)_2$			855.1	[12]
NiOOH			856.1–861.7	[12,14]
NaH_2PO_2	132.5	190.1		[17,19]
Na_3PO_3	134.1			[18]
Na_2HPO_3	132.9			[12]
Na_2HPO_4	133.0	190.8		[17–19]
P	130.0	187.7–188.05		[19,20]
PH_3	129.8			[20,21]

not expectable. The distance between peaks is less sensitive to spectrometer energy calibration and, in reference 13 differences around 3.5 eV between metallic and Ni^{2+} can be found confirming that the oxidation state must be the 2+. For very well defined NiO (1 0 0) surfaces, cleaved under vacuum, two components are detected at 854.1 and 855.6 eV [15,16], which is not the case of our sample, pointing to an amorphous oxide or a mixture of oxides and/or hydroxides. The third component, centred around 861.7 ± 0.1 eV and approximately 6 eV wide, is assigned to the complex multiplet structure existing in nickel [15]. In fact, both Ni and Ni^{2+} have unpaired electrons in 3d orbitals which may couple differently with the unpaired electron left in the 2p orbital after the photoelectron ejection giving rise to a complex series of multiplet peaks spreading over 10 eV [15].

The comparative analysis of these spectra lead us to conclude that despite the extent of Ni oxidation is higher in the hypophosphite-free solution, the nickel exhibits the same oxidation states in an solution containing the hypophosphite ion.

Although some authors endorse the formation of nickel hydride upon the hypophosphite oxidation [22], evidence for its formation was not given yet. Hydrogen-metal bonds can be indirectly detected by XPS, via a chemical shift in the photoelectron energies of the metal atom and via line shape changes, but it requires very clean sample surfaces and a rather high amount of hydrogen on the surface. Surface contamination by elements like oxygen, carbon or chloride would have to be avoided, which was not feasible in the present experimental conditions.

Concerning phosphorus, two XPS regions were studied: P 2p and P 2s. The most commonly studied region is the P 2p one and extensive data are found in the literature [17,20,23]. However, in this system, P 2p is not an adequate region to properly study the amount and the nature of phosphorus at the surface because the P 2p overlaps with a very broad shake-up (most probably, a plasmon) of the Ni 3s peak. Although it was also found that the P 2s peak overlaps with Cl 2p satellites (centred around 190 eV), it was possible to subtract the satellites and to obtain the neat P 2s peak from it. Quantification was thus performed with the P 2s region and the binding energies were evaluated either from P 2s or the P 2p region. No attempt was made to eliminate contaminating chloride ions from the surface because we wanted to maintain the same experimental methodology that was previously used [10].

It was found that the P 2s region could be fitted with two components: one centred at 190.3 eV and another one (absent in the −1.10 V treated electrode) centred at 188.4 eV, Fig. 3. The first component is assigned to hypophosphite (Table 1). Albeit the same binding energy is also characteristic of phosphate, previous studies have revealed that only phosphite is formed upon the oxidation of hypophosphite and that it does not remain adsorbed on the electrode surface. The detection of hypophosphite on the surface is consistent with data obtained by SNIFTIRS that revealed that hypophosphite adsorbs on the nickel surface, even at open circuit potential [3,4].

On account of Table 1 data, the second component could be assigned to elemental phosphorus; however this species is not expected to be detected because it is very unstable in aqueous solutions. Two other hypotheses are proposed for its assignment: alloyed phosphorus (normally represented as Ni–P, independently of its stoichiometry) and phosphine. The formation of phosphorus bound to nickel is the most plausible hypothesis to be considered. Literature data on the characterization of Ni–P alloys by XPS reveal the formation of a reduced form of phosphorus which is bound to nickel [17,20,24–27], however such analysis concerns exclusively the P 2p region. According to literature data, the binding energy of alloyed phosphorus lies within 129.0–129.7 eV in the P 2p region, depending on the phosphorus content and sample preparation method. The qualitative analysis of the present electrodes in the P 2p region also reveals a peak in that energy range, which is indicative of alloyed phosphorus formation. In order to confirm this assignment the P 2s and 2p regions of a Ni–P sample prepared by electroless deposition on a Ni foil were also analysed. The obtained values, 190.4 and 187.0 eV for the P 2s region, 132.7 and 129.2 eV for the P 2p region, are consistent with the binding energies reported on the sample electrodes, which supports our hypothesis that alloyed phosphorus is formed upon polarization of the Ni electrode in a solution containing hypophosphite. Data obtained on the analysis of the P 2s region in a $Fe_{40}Ni_{40}P_{14}B_8$ glass sample, further corroborates the assignment of 187.95 eV peak to phosphorus bound to nickel [24]. Phosphine presents a binding energy at 129.9 eV (there are no published results on the P 2s region) [20] but its detection by an ex situ technique (XPS) is unlikely because phosphine, like phosphorus, is very unstable in aqueous solution.

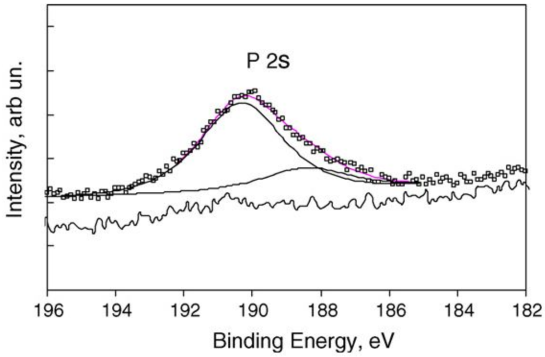

Fig. 3. XPS P 2s region for the Ni electrode after electropolishing (bottom line) and potentiostatic polarization at −1.2 V in 0.375 M $H_2PO_2^-$ + 0.10 M NaOH solution (top dashed line). P 2s was fitted with two peaks (top continuous line).

The amount of each phosphorus component on the surface (measured as P/Ni atomic ratio) as a function of the applied potential is shown in Fig. 4. The fact that the atomic ratio P/Ni increases for an analysis angle of 60° is a clear evidence that phosphorus, in both forms, is on the top layers of the surface electrode, as expected. From the figure it is also evident that the dependence of each phosphorus component on the applied potential follows opposite routes. The amount of hypophosphite on the surface electrode, highly dependent on the electrode potential, reaches its maximum value at −1.10 V. This result is consistent with the potential dependence of the electrode activity to oxidize the hypophosphite ion [3,6,11]. In contrast, the amount of alloyed phosphorus is approximately constant in the −1.00 to −1.30 V potential range, except at −1.10 V where it

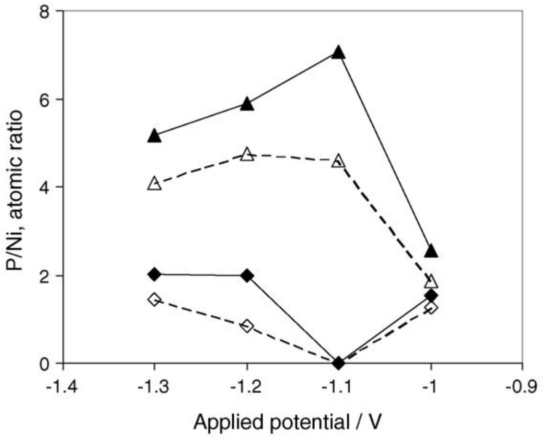

Fig. 4. Diamonds correspond to the lower binding energy P 2s component (188.4 eV) and triangles to the higher binding energy P 2s component (190.3 eV). Results for 0° (relative to the normal to the surface) are represented by empty symbols and for 60° by full symbols.

is not formed. These observations suggest that hypophosphite ion participates in two competitive surface reactions in the same potential range: its reduction to alloyed P and its oxidation to phosphite. The latter is known to occur through an intermediate, HPO_2^- [7,11]:

$$H_2PO_{2(ads)}^- \rightarrow H_{(ads)} + HPO_{2(ads)}^-$$

$$H_2O + HPO_{2(ads)}^- \rightarrow HPO_3^{2-} + e + 2H^+$$

$$H_{(ads)} \rightarrow H^+ + e.$$

At this stage it seems pertinent to analyse these results in the light of the mechanisms that are proposed in the literature for the incorporation of phosphorus during the Ni–P electrodeposition. According to the literature, two different mechanisms are proposed: the direct and the indirect mechanism. The former supports that phosphorous acid, or phosphite (depending on the pH), is electrochemically reduced to elemental phosphorus, which would react with Ni giving rise to Ni–P alloy [25]:

$$H_3PO_3 + 3H^+ + 3e \rightarrow P + 3H_2O$$

$$n\mathrm{Ni} + \mathrm{P} \rightarrow \mathrm{Ni}_n\mathrm{P}.$$

The latter is based on the assumption that phosphine (PH_3), itself formed by hypophosphite or phosphite ion reduction (or their respective acids), reacts with Ni^{2+} ions present in the solution to produce the Ni–P alloy [7,9,26–28]:

$$H_3PO_3 + 6H^+ + 6e \rightarrow PH_3 + 3H_2O$$

$$\mathrm{w}PH_3 + \mathrm{y}Ni^{2+} \rightarrow Ni_yP_w + \mathrm{z}H^+.$$

This mechanism has been more widely accepted than the direct mechanism.

Based on the above results it can be concluded that, independently of the type of mechanism (direct or indirect), phosphite ion does not seem to be the species that is reduced to alloyed P, otherwise, it would have been expected that alloyed P was formed at the potential corresponding to the highest electrode activity to oxidize hypophosphite to phosphite (−1.1 V). This conclusion is in agreement with the Zeller and Landau [27] and Harris and Dang [9] results in Ni–P electrodeposition experiments: the phosphorus content of the alloy is null or very low when H_3PO_3 is used instead of H_3PO_2.

It is also important to note that the detection of alloyed phosphorus, on sample electrodes that were prepared in the absence of Ni^{2+}, reveals that the presence of this metallic cation is not a constraint for the incorporation of phosphorus (in very small amounts) on nickel. This result is indicative that probably different reaction pathways (supported by both direct and indirect mechanisms) will be responsible for the Ni–P alloy formation. To our knowledge this is the first time that alloyed phosphorus is detected on a nickel electrode in a Ni^{2+}-free solution in an alkaline medium. Although Kurowski et al. have reported its detection by XPS in an acidic solution [28], in such medium the dissolution of the Ni may occur, contributing to the presence of the Ni^{2+} ions in the solution. Zeng and Zhou have shown by in situ surface enhanced Ramam spectroscopy, that a Ni–phosphine compound is formed in a solution without Ni^{2+} in a neutral medium [8], but no evidence for the formation of alloyed P was given.

Although it is not the aim of this work to study the phosphorus incorporation mechanism during Ni–P electrodeposition, these peculiar results reveal that this matter should be subjected to further investigation in the future.

4. Conclusions

In this work it was concluded that the Ni surface electrode is modified upon polarization in a hypophosphite containing solution. It was revealed by XPS that alloyed phosphorus is the responsible species for the surface electrode modification. Its dependence on the electrode potential reveals that hypophosphite ion participates in two surface competitive reactions in the same potential range: its oxidation to phosphite and its reduction to alloyed phosphorus.

The analysis of these results suggest that the mechanism for the incorporation of phosphorus in the Ni–P electrodeposition process involves the hypophosphite ion, and not phosphite, and that alloyed P can be formed even in the absence of Ni^{2+} ions.

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