



*This is a preview version of our white paper on surface treatment by ion implantation*

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WHITE PAPER

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## Surface treatment by ion implantation

Case study: Nitrogen implantation in brass as an efficient means to lower the coefficient of friction and improve the wear resistance of miniature parts

## Ion implantation equipment and surface treatment

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Ion implantation can be applied to a wide variety of materials to enhance their surface properties (*e.g.*, stainless steel, titanium, sapphire, silicon, rubber, PEEK).

This white paper showcases some benefits of this technology through a thorough tribological study of machinable brass implanted with nitrogen ions.

The document is divided into two parts that can be read independently:

### I. **Technology** (pp. 2–5)

Introduction to ion implantation technology and equipment used for surface treatment

### II. **Case study** (pp. 6–14)

Multi-charged nitrogen ion implantation in machinable brass

In the case study devoted to brass alloy CuZn39Pb3, we demonstrate the lowering of the coefficient of friction from 0.7 to 0.25 (system: brass vs. quenched steel) as a result of nitrogen ion implantation. Surface wear of brass could be drastically reduced, even with a contact pressure in the range of 300 MPa.



**I**ON IMPLANTATION is the principal technology used to introduce foreign elements into solids in a uniform and controlled way. The technology was initially developed by the semiconductor industry for the doping of materials. It is now being increasingly adopted for the surface treatment of a variety of materials. Before getting into this topic, we shall take a brief look at the history of ion implantation.

### Semiconductor doping and surface engineering

In semiconductor terminology, doping refers to the fabrication process in which impurity atoms are intentionally introduced in the exposed area of a semiconductor for the purpose of modifying its electrical characteristics. Shockley<sup>1</sup> was the first to foresee the potential of ion implantation for the doping of semiconductor wafers, as evidenced by his 1954 patent application entitled “Forming semiconductive devices by ionic bombardment” [1]. However, it was only in the late 1970s that ion implanters entered the industry of semiconductors. Since then, ion implantation has been the principal doping method used for integrated circuits (IC) manufacturing [2].

The technology involves the generation of an ion beam and steering it in a controlled manner. Ionized species are accelerated by electric fields to high energies and shot into a target substrate. They can also be separated by magnetic fields to obtain an ion beam of high purity and a well-defined energy. Research in particle physics shares the same needs in terms of high purity ion beam handling and has played an essential role in the rise of ion implantation. Key technologies have emerged from the efforts made in building efficient ion sources and high-energy particle accelerators, not to mention the hardware required to generate high-power

their researches on semiconductors and their discovery of the transistor effect.”

<sup>1</sup> William Bradford Shockley, John Bardeen and Walter Houser Brattain were jointly awarded the Nobel Prize in Physics 1956 “for

radio-frequency (RF) electromagnetic waves and high-voltage DC (direct current) electric fields.

A major reason in applying ion implantation for doping semiconductors lies in that critical process parameters, such as amount and position of implanted species (concentration and penetration depth), are equipment settings directly controlled by the dose and energy, respectively. To fulfil the many implantation applications found in ICs, the doping requirements span several orders of magnitudes in both dose ( $10^{11}$  to  $10^{17}$  atoms/cm<sup>2</sup>) and energy levels (100 eV to nearly 10 MeV), for a wide range of atomic species (P, B, As, In, Ge, N, H, He, etc.).

Besides technological advances, studies were also undertaken to better understand ion stopping and thus address the needs for accurate doping. Theoreticians have refined models describing collision of ions with matter. On this topic, much is owed to Ziegler *et al.* who have developed SRIM, a Monte Carlo computational algorithm whose acronym stands for "Stopping and Range of Ions in Matter" [3]. SRIM was released as a freeware and is currently the most widely used tool by the scientific community to simulate the interaction of accelerated ions with matter [4]. It is primarily used to determine the penetration depth and distribution of atoms (projected range and straggling) based on the energy of ions and their angle of incidence with respect to a target material.

We now leave aside the field of semiconductors to focus on the needs that are specific to surface treatment. Noticeable changes in material surface properties can be obtained for doses in the range of  $10^{15}$  to  $10^{18}$  atoms/cm<sup>2</sup> and for energies between tens to few hundred keV. Literature survey shows that a number of atomic species have been implanted, often extracted from gaseous elements (N, O, H, He, C, Ar, etc.), but also from solid-state materials, mainly to produce metallic ion beams (Cu, Au, Ag, etc.). Owing to its ease of production and handling – and its many proven effects – nitrogen is the foremost species used for surface treatment by ion implantation.

In light of the requirements mentioned above, the primary specification for a surface treatment implanter is to produce large ion beam currents to ensure cost effectiveness of the process for high-dose applications. Indeed, the greater the beam current, the faster the implantation. However, but not surprisingly, many

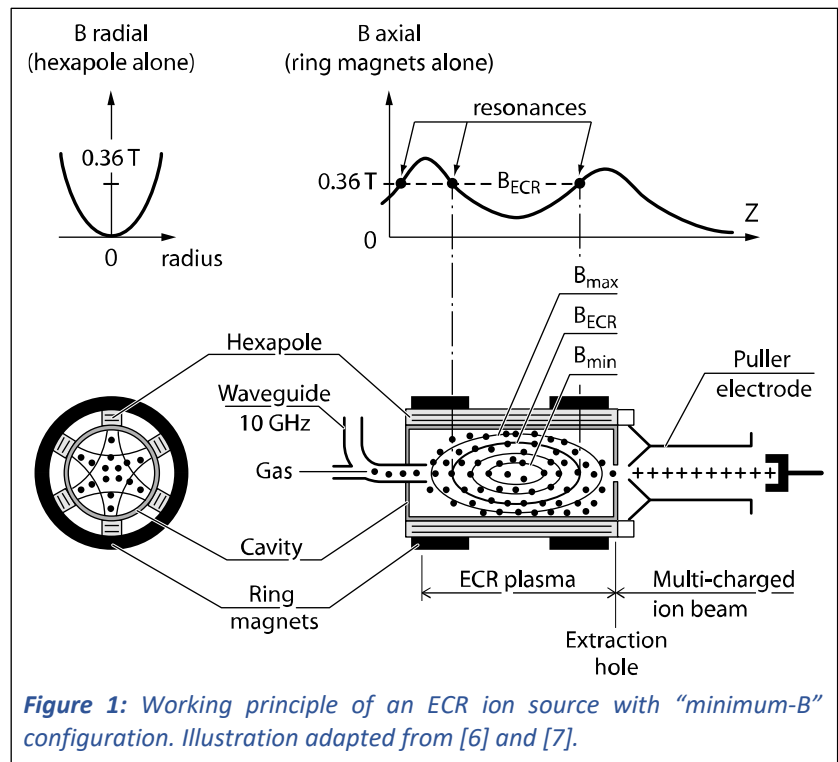


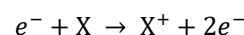
Figure 1: Working principle of an ECR ion source with "minimum-B" configuration. Illustration adapted from [6] and [7].

surface treatment studies have been carried out with implanters dedicated to semiconductor wafers or using in-house modified versions of these facilities. Clearly, the demands for semiconductor implantation are different from those for surface engineering. The same applies to the ion source: While high purity and precise dose control are critical parameters for IC doping, throughput is the primary criterion for surface treatment.

### Electron Cyclotron Resonance (ECR) ion source

Among the wide variety of ion sources that have been invented, the ECR ion source has several great characteristics that makes it particularly suited for surface treatment. It produces positive ions with charge states reaching unparalleled levels [5]. The ECR ion source was pioneered in the 1970s by Richard Geller at the CEA<sup>2</sup> [6, 7]. It is now widely used in accelerator physics and can be found in leading-edge research facilities such as the Linear Accelerator 3 (LINAC 3) at CERN<sup>3</sup>. We shall first present its operating principle (see Figure 1), since it will prove useful for the understanding of the process discussed below.

**Gas discharge** – The ECR ion source uses a gas discharge plasma to create positive ions. A gas discharge plasma is an ionized gas sustained by applying an electric field across it. Positive ions are created by electrons that supply the dissociative ionization energy when they collide against atoms. The basic gas phase reaction is



<sup>2</sup> French Alternative Energies and Atomic Energy Commission or CEA (Commissariat à l'Énergie Atomique).

<sup>3</sup> European Organization for Nuclear Research, known as CERN.





## Case study: Ion implantation of nitrogen in machinable brass

**Abstract** — We investigate the tribological effects of multi-charged ion implantation (MCII) of nitrogen in CuZn39Pb3 alloy, a machinable brass that is widely used in the watchmaking industry. For this study, MCII of nitrogen was performed at 35 kV with a fluence of  $5 \times 10^{17}$  atoms/cm<sup>2</sup> and has resulted in a threefold decrease of the coefficient of friction (COF), from 0.7 to 0.25, as measured with a ball-on-disc tribometer. Wear track profile measurements using Atomic Force Microscopy (AFM) on the tribological traces showed that the wear rate was lowered by two orders of magnitude, from  $10^{-14}$  m<sup>2</sup>/N for the untreated reference to  $2.9 \times 10^{-16}$  m<sup>2</sup>/N for the implanted brass. Implanted samples were observed by transmission electron microscopy (TEM) and the results corroborate well with Monte Carlo simulations. Further analyses with Energy-Dispersive X-Ray Spectroscopy (EDS) and Grazing Incidence X-Ray Diffraction (GIXRD) showed that nitrogen implantation in brass did not result in the formation of a new crystalline phase. Hence, the enhanced tribological properties of brass could be attributed to the modification of surface microstructure of brass following high energy ion implantation.

### Introduction

Ion implantation is well known for significantly modifying the mechanical, electrical, optical, and chemical properties of all categories of solid materials (metals, polymers, ceramics, semiconductors, composites). The primary use of ion implantation is in semiconductor doping, but several other industrial applications have started to emerge [13]. For instance, structural rearrangement in optical materials causes changes in the refractive index. Ion implantation can hence be used as an anti-reflective treatment, or to produce waveguides. Advanced applications based on ion implantation technology are also being developed for catalysis systems, and more generally for chemical surface functionalization, or for biocompatibility enhancement. Finally, the potential for mechanical applications is vast and this field is probably the one that is currently undergoing the strongest developments. In this study, we will discuss the tribological properties of brass that can be improved by ion implantation.

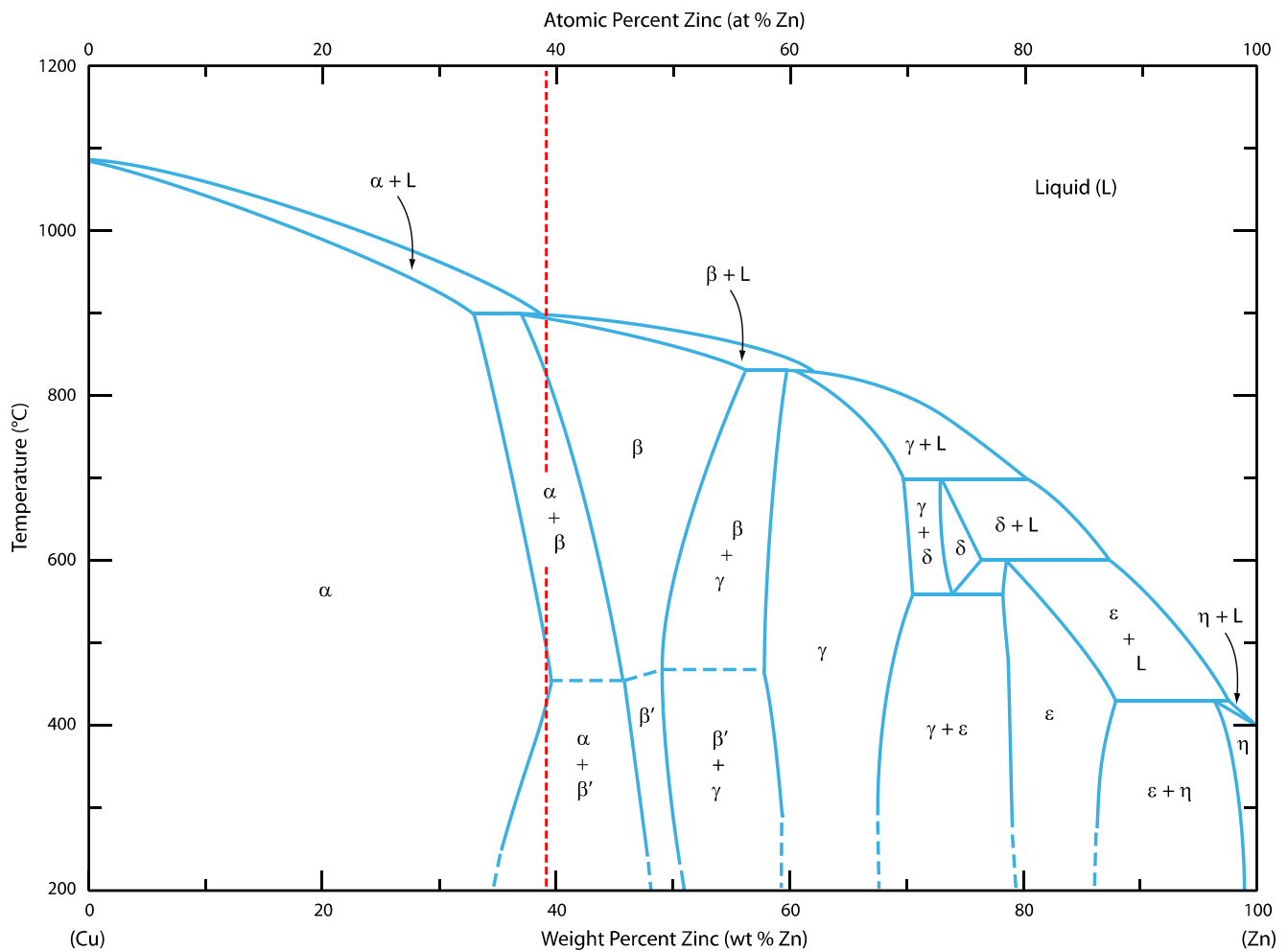
When compared with other surface treatments, ion implantation has several unique advantages:

- Dimensional changes are insignificant, or restricted to the atomic level;
- Solid solubility limit can be exceeded;
- Depth distribution is controllable;
- As its name implies, particles diffuse into the matter, thus eliminating delamination risks;
- The process can be completed at low temperature.

These characteristics are sought after in fine mechanics. Of course, like other processes, the technology also has some limitations. One being that ion beam implantation is a line-of-sight treatment. Re-entrant surfaces cannot be treated effectively. Nevertheless, this issue can be tackled if the functional areas can be scanned under the ion beam using an adapted sample stage. The technology is thus particularly attractive for miniature mechanical parts, like those found in high-end watches.

The tribological effects of ion implantation have been largely studied and the proven improvements include: surface hardening, low friction, increased wear resistance, and improved corrosion reduction. One or several of these effects have been demonstrated in a wide variety of materials: plastics (*e.g.*, PEEK, PMMA, PC) and elastomers (*e.g.*, NBR, EPDM, FKM rubbers), brittle materials (such as silicon, glass, sapphire, ceramics), and of course metals. Ion implantation in metallic alloys has been investigated in detail (especially in steels [14, 15, 16], but also in aluminium alloys [17, 18], and in titanium alloys [19]). Yet, only few studies have been devoted to ion implantation in brass or copper.

In a research report dating from 1985, Shih showed that nitrogen implantation had a positive effect on impact wear for various metals (100 keV, dose of about  $4 \times 10^{17}$  atoms/cm<sup>2</sup>) [20]. For unexplained reasons, it did not prove successful on cartridge brass (70 wt % Cu / 30 wt % Zn), but the results were significant on copper, bronze (90 wt % Cu / 10 wt % Sn), aluminium alloys and 304 stainless steel, among other tested metals. Improvement in impact wear resistance was attributed to the hardening effects of nitrogen implantation. These effects could be explained either in terms of interstitial hardening, or by the formation of very hard nitride phases, or by a combination of both processes. These two strengthening processes act by impeding the movement of dislocations. The first one involves the segregation of interstitial nitrogen atoms at the core of dislocations [21]. More recently, Sari *et al.* showed surface hardening as well as increased corrosion resistance of Cu (99% purity) after nitrogen implantation (50 keV, fluence of  $3 \times 10^{17}$  atoms/cm<sup>2</sup>) [22]. A recent work by Zharkov *et al.* showed wear reduction of copper samples implanted with nitrogen (20 keV, fluence of  $9 \times 10^{17}$  atoms/cm<sup>2</sup>) caused by surface hardening [23]. They attributed hardening in copper to the refinement of the grain structure (nanostructuring) of the main FCC-Cu phase (face-centred cubic structure) occurring at high-



**Figure 4:** Copper-zinc (Cu-Zn) phase diagram. Adapted from Fig. 9.17 in “Binary Alloy Phase diagrams,” 2nd ed., Vol. 2, Massalski (Editor-in-Chief), 1990 [27].

dose implantation [24]. Finally, Cavellier completed a study on pure copper and copper alloys (brass and bronze) implanted with multi-charged nitrogen ions [25]. Several promising tribological results obtained on these materials were highlighted, related to surface hardening, especially in terms of coefficient of friction (COF) and wear rate. However, for confidentiality reasons, the associated process parameters (*i.e.*, implanted doses and energies) were not disclosed. Thus, part of his results can only be considered qualitatively.

### Free machining brass

In a watch mechanism, a variety of parts are made from brass: balance wheel, bridges and bottom plate, gear train (wheels and pinions), barrel (drum and cover). Gears and drum barrel are of interest here since they have mechanical functions that require low friction and good wear resistance. To meet these requirements and ensure reliability and durability of the watch movement,

various types of lubricants are applied on these parts. Aging of lubricants is an issue and watchmakers consider lubricant-free mechanisms as their Holy Grail. Hence, one can clearly see the potential of a machinable brass with much improved tribological characteristics.

CuZn39Pb3 is one of the most commonly used brass alloys in the watchmaking industry. Lead is added to binary brass (Cu-Zn) to obtain an alloy with good machinability, a material said to be “free machining” [26]. The short metal chip breaking and the lubricating effect of lead during machining make leaded brass particularly suited for Swiss-type lathe machining (a barfed automatic turning machine known in French as “*décolleteuse*”).<sup>6</sup> Figure 4 shows the phase diagram of binary brass (adapted from [27]). The vertical dashed line that is superimposed indicates the corresponding nominal concentration of Zn found in CuZn39Pb3.

The choice of CuZn39Pb3 for our scientific study was dictated, on the one hand, by the particularity of this

<sup>6</sup> The European regulation REACH stipulates that Pb must be eliminated from the design of consumer goods. Currently, watchmakers are still allowed to use leaded brass for internal

components of watch timepieces which are inaccessible to consumers [Regulation No. 1907/2006 of the European Parliament and of the Council, Amendment M18].

Year	2010	2011	2012	2013	2014	2015	2016
Net egg production (kg/ha)	100	100	100	100	100	100	100
Net egg production (kg/ha)	100	100	100	100	100	100	100

Table 1. Net egg production (kg/ha) of *Salmonella* in eggs produced in 2010–2016. Net egg production (kg/ha) is the average of 100 eggs.

the egg production from different strains and genotypes. However, in the other study, we used the results of our research conducted in an indoor facility, and in order to determine the impact of different genotypes on the egg production, we used the results of our research in an outdoor facility. In our previous research in a mixed farm, we used the results of our research in an outdoor facility.

The egg production results are presented in Table 1. The net egg production (kg/ha) was calculated as the average of 100 eggs from 100 different genotypes. The net egg production (kg/ha) was calculated as the average of 100 eggs from 100 different genotypes. The net egg production (kg/ha) was calculated as the average of 100 eggs from 100 different genotypes. The net egg production (kg/ha) was calculated as the average of 100 eggs from 100 different genotypes.

#### Discussion of the results

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Figure 2. Micrograph of the egg production. The inset shows a magnified view of the egg's surface, which is likely a bacterial colony.

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#### Conclusion

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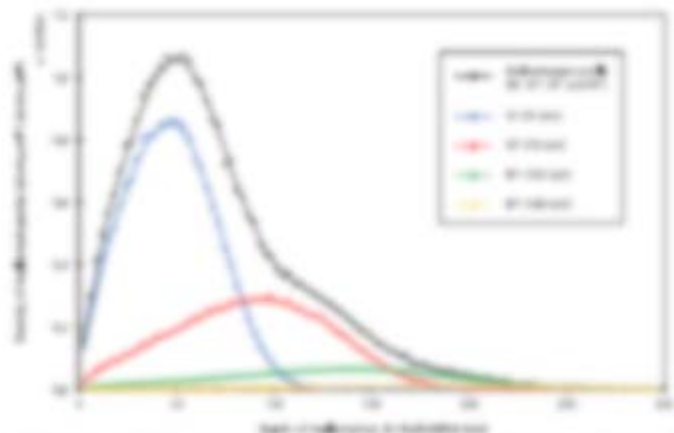


Figure 1. Evolution of the number of particles versus time for each stage of the degradation process. The curves represent the number of particles at each stage: Stage 1 (black), Stage 2 (blue), Stage 3 (red), Stage 4 (green), and Stage 5 (yellow).

Figure 1. It is clear that the number of particles increases over time for each stage of the degradation process. The curves represent the number of particles at each stage: Stage 1 (black), Stage 2 (blue), Stage 3 (red), Stage 4 (green), and Stage 5 (yellow).

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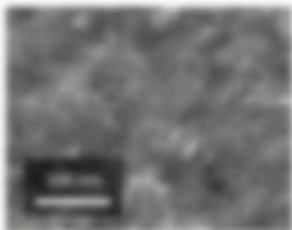
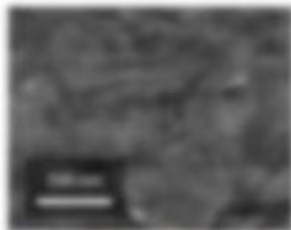


Figure 2. Two images of particles from the degradation process. The left image shows Stage 1 and the right image shows Stage 2.

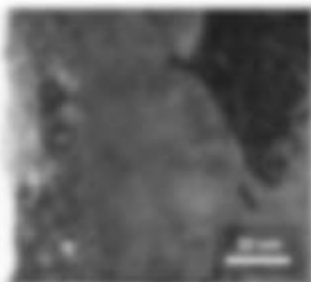
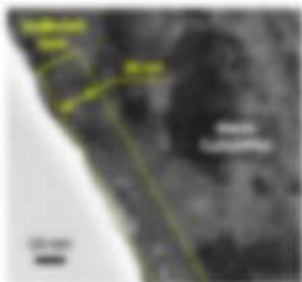


Figure 4. SEM micrographs observation of superabsorbent after 2400 s charge measurement at 20 °C in absence of moist atmosphere. The left image is a cross-sectional view, a representative image of about 20 μm long without any film formation on the electrode surface.

different stage of the sample in the impedance test conducted with the voltage bias in Figure 2 and in the corresponding Nyquist plots. These two measurements are plotted in Figure 3 in the Nyquist plane, as it is well known, the Nyquist plot is composed of a semicircle with a diameter equal to the charge transfer resistance.

SEM analysis shows a porous structure about the electrode surface of 2400 s. For this analysis, we compare the difference between two the structure and impedance spectra with a reference SEM

micrograph. Figure 4 shows that different porous structure are shown in Figure 5. The different porous structure may be due to the different diffusion process after 2400 s corresponding to the time with change of capacitance after 2400 s. Figure 6(a) shows that the porous structure was obtained at 1200 s, and after the discharge time, the porous structure was obtained after 2400 s. Figure 6(b) shows that the porous structure after 2400 s charge measurement is more than 2400 s.

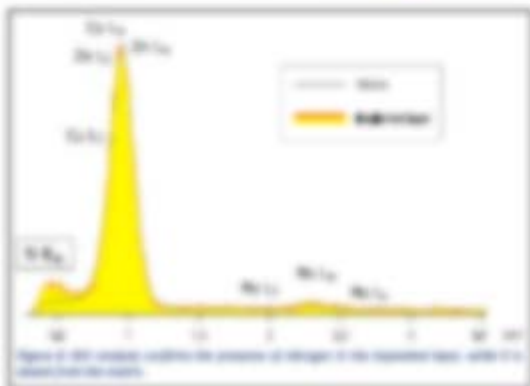


Figure 5. EIS analysis exhibits the presence of charge in the impedance test, with 10 s delay from the start.

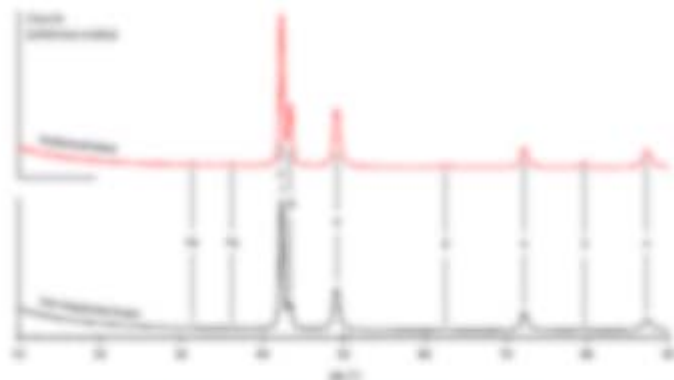


Figure 20. XRD patterns obtained from a polymer sample using an x-ray diffractometer. The top figure shows the experimental data and the bottom figure shows the reference pattern. The x-axis is labeled  $2\theta$  and the y-axis is labeled Intensity.

### Young's modulus

Young's modulus is a measure of the stiffness of a material. It is defined as the ratio of stress to strain in the linear elastic region of the stress-strain curve. It is denoted by  $E$  and is measured in Pascals (Pa). The units of Young's modulus are  $\text{N/m}^2$ .

Young's modulus is a property of a material that depends on its atomic structure. It is a measure of the resistance of a material to deformation under stress. It is a scalar quantity and is denoted by  $E$ . The units of Young's modulus are  $\text{N/m}^2$ . The units of Young's modulus are  $\text{N/m}^2$ .

$$E = \frac{\sigma}{\epsilon} = \frac{F/A}{\Delta L/L} = \frac{FL}{A\Delta L}$$

The units of Young's modulus are  $\text{N/m}^2$ . The units of Young's modulus are  $\text{N/m}^2$ .

The maximum normal stress,  $\sigma_{max}$ , occurs at the ends of the specimen.

$$\sigma_{max} = \frac{F}{A}$$

The angle of inclination,  $\theta$ , is related to the normal stress by

$$\theta = \frac{\sigma}{E}$$

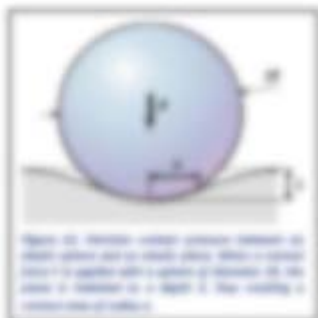


Figure 21. Sphere in contact with a flat surface. The radius of the sphere is  $R$  and the radius of the flat surface is  $r$ . The angle of inclination is  $\theta$ .



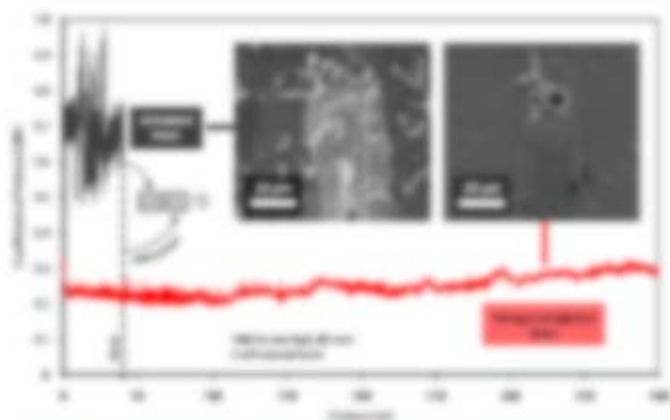


Figure 19. Variation of the average diameter of the particles through the effect of the different flow rates of the compressed air supply flow velocity in the spray chamber of the air sprayer machine and the change in particle size. (Source: *Health Engineering and Manufacturing*, 2019, 1(1), 1-10. <https://doi.org/10.1080/26449300.2019.1611100>)

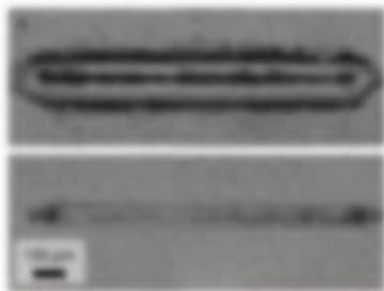
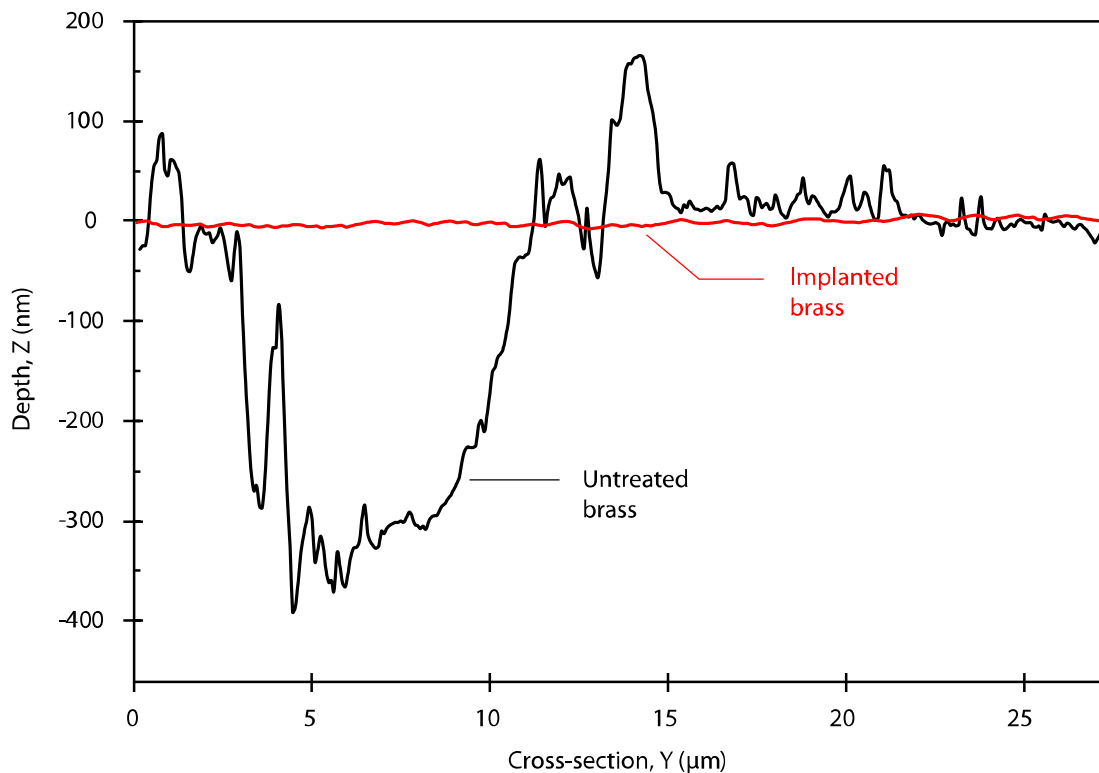


Figure 20. Scanning of the filament made by the ring die extrusion apparatus in making these applications for the use of a spray device of 40 kg. The diameter of the rod made by Figure 20, a single filament made with an extrusion die, showing the effect of a filament that is prepared from water.

#### Extrusion and coating

One of the advantages of this process is that it allows the production of a wide range of products, from simple rods to complex structures, such as tubes or sheets. It is also possible to produce coatings on the surface of the rods. The process involves the extrusion of a polymer melt through a die, which can be modified to produce different shapes and sizes. The extrusion process is controlled by the extrusion rate and the die design. The extrusion rate is the speed at which the polymer melt is pushed through the die, and the die design is the shape and size of the die. The extrusion process is a continuous process, and the extruded material is collected in a container or on a conveyor belt. The extrusion process is a simple and efficient way to produce a wide range of products, and it is widely used in the manufacturing industry.



**Figure 14:** AFM measurements of the traces shown in Figure 13. Note the difference in scales between Y cross-section ( $\mu\text{m}$ ) and Z depth (nm). The wear rate of brass could be reduced by two orders of magnitude thanks to ion implantation.

[21]. These results pave the way for the use of nitrogen-implanted brass parts in applications where lifetime and mechanical load usually proscribe untreated brass.

We conclude this white paper by highlighting that ion implantation is a high potential technology that can be used for many other materials, and for applications that are not limited to tribological enhancement. We are working hand in hand with watchmakers and clients from other industrial sectors to develop the technology and provide dedicated processes tailored to their specific needs. ■

## Acknowledgment

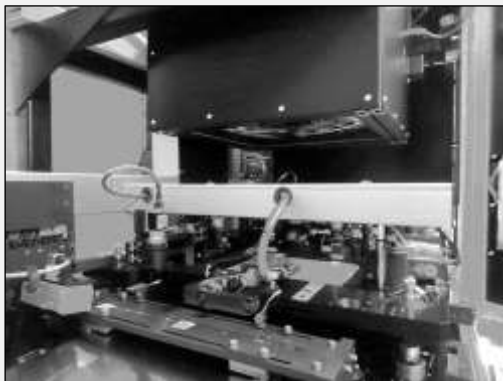
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