

# ANALYSIS OF PROCESSES AND PROPERTIES OF PLASMA NITRIDING

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## Abstract

The steady increase worldwide of requirements on the quality and performance of ferrous materials as well as ever stricter environmental regulations make new developments necessary to constantly improve the wear and corrosion protection of components and tools. Furthermore, because the treatment of steel surfaces can cut the need for valuable alloying elements in the base materials, the development of environmentally friendly and industrially applicable methods for the modification and coating of ferrous materials is one of the most important challenges in surface technology. This work is focused on the general characteristics of plasma nitriding, its technical and structural properties, and applications.

**Keywords:** surface, diffusion layer, nitriding, plasma,

## 1 Introduction

Plasma technology is used in the field of heat treatment and it is not a new technology. The use of plasma nitriding as a process system has received a great deal of success. Its acceptance by engineers and metallurgists has grown. It is now seen not so much as a new process, but as an accepted process that has a great deal to offer in terms of repeatable and consistent metallurgy. It is known as ion nitriding, glow discharge nitriding, plasma nitriding, and plasma ion nitriding [1].

The phenomena of plasma are well-known natural phenomena as have been previously described. Two German physicists named Drs. Wehnheldt and Berghaus first developed the use of the plasma phenomena for metallurgical processing in Germany early 1930s. Plasma technology is used on a daily basis for (a) pre cleaning for surgical instruments, (b) plasma coating technology, and in plasma-assisted surface treatments, such as nitriding, FNC, carbonitriding, and carburizing. Plasma is a technology that can be used for many process applications, which include the treatments of a metal, plastic, or other surface treatment methods. The use of plasma assistance for thin-film hard coatings has grown considerably. The company is still employing plasma processing as a means of nitriding for surface treatment. It is quite evident that the company believes in the technology of plasma nitriding as a method of precisely producing a desired, repeatable, and consistent metallurgically formed case [2].

## 2 The nitriding process

The generic term nitriding refers to a thermochemical treatment with which the surface zone of ferrous materials is enriched with nitrogen (see DIN 17014). When nitrogen diffuses into the surface zone, it is at first dissolved interstitially in the iron matrix. If the nitrogen concentration exceeds the solubility limit of 2.5 weight percent, a single- or multi-phase nitride layer is formed. This treatment is preferably carried out in the temperature range between 400 and 600°C. The familiar nitriding processes provide not only excellent corrosion protection but also outstanding protection against wear whilst also allowing the dynamic characteristics of components made of ferrous materials to be improved [3].

The suggested mechanism of plasma nitriding is the formation of uniform layer of iron nitride on the surface through combination of sputtered iron atoms from cathode surface and nitrogen ions from plasma. Plasma process allows both bulk and grain boundary diffusion compared to only grain-boundary diffusion in gas nitriding. In plasma nitriding, it is possible to form single phase  $\gamma$  or  $\epsilon$ - nitride in contrast to mixed ( $\gamma + \epsilon$ ) phase in conventional nitriding. White layer formed due to single phase  $\gamma$  is comparatively more tough with minimum residual stress [5].

### 2.1 Typical plasma nitriding process

A typical plasma nitriding process is shown in Figure 1 and the processing parameters are detailed in Table 1 [7].

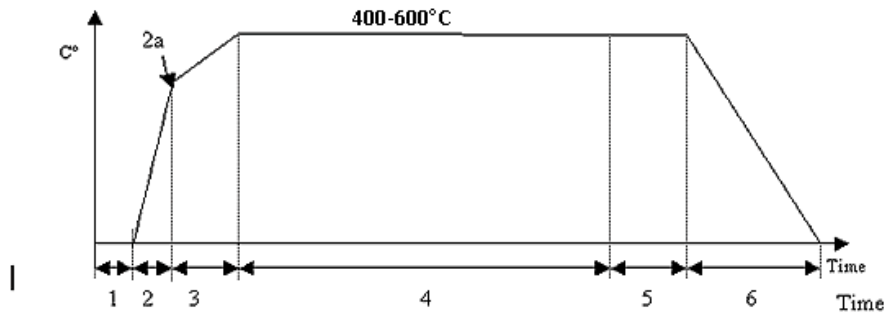


Fig. 1 General treatment cycle of plasma nitriding [7]

Table 1 Description of treatment cycle [7]

Phase	Description
1- Pumping	The vessel is purged and then filled up with gas at a chosen pressure.
2- Heating	Divided into several phases, depending on the temperature and physical or chemical reaction between the furnace atmosphere and the part.
2a- Pre oxidation (Usually not done)	A thin oxide film is produced in an atmosphere of water vapor to accelerate nitro-cementation (i.e. to accelerate the nitriding process).
3- Sputtering	Positive ions impacting on the surface heats the job to slightly below nitriding temperature and ejects surface atoms.
4- Nitriding	Nitrogen ions absorbed into the surface to form finely dispersed nitrides.
5- Oxidation (Upon customer request)	A thin oxide film is produced in an atmosphere of water vapor to prevent corrosion of part after treatment.
6- Cooling	Done as fast as possible without causing part distortion.

### 2.2 Typical process parameters

The environmentally friendly plasma nitriding process offers some key advantages over traditional nitriding in the salt bath or with gas. In particular the layer structure, the depth of the hardness gradient and the homogeneity of the surface layers produced can be selectively controlled in a manner largely independent of each other through control of the discharge parameters (power, voltage, pulse frequencies and mark-to-space ratio), the process gas conditions (gas composition, pressure and flow rate) and the component batch parameters (temperature, time, heating and cooling rate) [4].

Typical process parameters:

- ✓ primary gases used in nitriding: nitrogen, hydrogen
- ✓ primary gases used in nitrocarburizing: nitrogen, hydrogen, carbon dioxide or methane
- ✓ additive gas: argon
- ✓ temperature: 350 to 600°C
- ✓ gas pressure: 50 to 500 Pa
- ✓ gas consumption: from 20 l/h (lab scale) to 500 l/h (industrial plant)
- ✓ treatment time: 0.5 to 60 hours
- ✓ plasma power: 500 A at 0 to 800 V [8].

Compared to conventional hardening processes, plasma nitriding treatments are distinguished by the following manufacturing characteristics:

- ✓ high dimensional accuracy
- ✓ little increase in surface roughness
- ✓ low, predictable increase in volume
- ✓ repair welding is possible
- ✓ simple partial treatment [3].

### 2.3 Improvement of mechanical properties

The diffusion zone determines the fatigue properties of the nitrided components. Workpieces with nitrided cases prove effective under dynamic stresses. In a ferrous material, Nitrogen will exist within the lattice structure as individual, separate atoms up to the limit of solubility of Nitrogen in Iron. Precipitates of the nitrides are formed as the concentration of Nitrogen approaches the limit of solubility. The mechanism by which the hardness is increased is believed to be due to the distortion of the lattice structure when Nitrogen penetrates it [5].

Plasma nitrided surfaces have less porosity in contrast to conventionally nitrided surfaces, which have a highly porous structure. In certain wear applications in which a fretting wear is encountered, it is sometimes desirable to impart some porosity to the surface, as porous layers are known to retain lubricants in the contact zones of opposing wear surfaces. Porosity can be imparted through the addition of Ammonia to the gas composition. Nitriding of high-speed tools is done to increase the wear resistance of the cutting edge and reduce the tendency of the work to weld to the cutting edge. These factors depend on whether the workpiece is soft and sticky, such as mild steel, or hard and abrasive, such as tool steel. Also, mechanical strain due to tensile, compressive, bending, shear, and torsion act on the part. Typically, nitriding is done to enhance sliding wear resistance and is usually a poor choice for heavy-impact wear [6].

Nitriding is considered a low or no-growth treatment, ideal for most production applications like casting, forging and machining. Prior mechanical working and heat treatment are the sources of distortion in a workpiece. If they have been properly annealed for stress relieving at a temperature of at least 50°C above the nitriding temperature, the treatment should involve no distortion. Long thin parts can be hung in fixtures to relieve the stress due to self-weight at the treatment temperatures. Under these conditions, distortion can be held to a minimum [5].

Sputtering can cause an increase in the roughness of a surface, though to a lesser extent than in most conventional case hardening processes. A typical range in increase in surface roughness is 0.2-0.5 microns. Moreover, Simple polishing can restore the original finish [6].

### 3 Overview of applications plasma nitriding

The first applications of the plasma nitriding process were limited to tools and devices such as extrusion screws. The objective was to make use of the improved wear resistance resulting from the greater surface hardness and of the reduced tendency to cold weld due to the ceramic character of the compound layer. The lower process temperatures, compared with other surface hardening and nitriding processes, also permit low-distortion nitriding of tool steels and steels with low tempering temperatures. Variation of the process gas mixture even offers the possibility of reducing the passive layer on stainless steels, thus making these nitridable for the first time. This opens up completely new fields of application, among others in the valve industry [3].

### 4 Conclusion

In the past 30 years, the plasma nitriding of iron-based alloys has become a recognized technique for industrial production along with salt bath nitriding and gas nitriding. The established advantages of the plasma process cause it to be used with increasing frequency. In the future, conventional nitriding processes such as salt bath nitriding will be subject to increasing regulation by environmental authorities. However, plasma technology is available as an alternative which with respect to emission values, can be classified as environmentally friendly.

### References

- [1] Totten, G.E., Howes, G.A.H.: The Steel Heat Treatment Handbook, Marcel Dekker, New York, 1997.
- [2] Totten, G.E.: Steel Heat Treatment: metallurgy and technologies. 2006. CRC Press Taylor & Francis Group, USA, 2006, ISBN 0-8493-8455-9.
- [3] Rainer, H., Werner, O.: Plasma-Assisted Surface Treatment. 2006. Sellier Druck GmbH, D-85354 Freising, Germany, 2006, ISBN 3-937889-39-6.
- [4] Kakař, D., řkorić, B., Novakov, T., Kovačević, L., Miletić, A., Laboratory for heat treatment and surface engineering, In.: Serbian society for microscopy. Academy of medical sciences, SMS, p. 112-115.
- [5] [15.11.2016],[http://books.google.sk/books?id=5T9YZvB8JKcC&pg=PA90&dq=plasma+nitriding&hl=sk&ei=E51nTeP6CozKswa8h7TaDA&sa=X&oi=book\\_result&ct=result&redir\\_esc=y#v=onepage&q=plasma%20nitriding&f=false](http://books.google.sk/books?id=5T9YZvB8JKcC&pg=PA90&dq=plasma+nitriding&hl=sk&ei=E51nTeP6CozKswa8h7TaDA&sa=X&oi=book_result&ct=result&redir_esc=y#v=onepage&q=plasma%20nitriding&f=false)
- [6] [2.7.2016],[http://books.google.sk/books?id=hTlwyGQ4puIC&pg=PA127&dq=plasma+nitriding&hl=sk&ei=E51nTeP6CozKswa8h7TaDA&sa=X&oi=book\\_result&ct=result&redir\\_esc=y#v=onepage&q=plasma%20nitriding&f=false](http://books.google.sk/books?id=hTlwyGQ4puIC&pg=PA127&dq=plasma+nitriding&hl=sk&ei=E51nTeP6CozKswa8h7TaDA&sa=X&oi=book_result&ct=result&redir_esc=y#v=onepage&q=plasma%20nitriding&f=false)
- [7] [8.11.2016],[http://www.tppinfo.com/heat\\_treatment/plasma\\_nitriding\\_coating/plasma\\_nitriding.html](http://www.tppinfo.com/heat_treatment/plasma_nitriding_coating/plasma_nitriding.html)
- [8] [12.11.2016],[http://books.google.sk/books?id=A2QG3UssqKUC&pg=PA163&dq=plasma+nitriding&hl=sk&ei=E51nTeP6CozKswa8h7TaDA&sa=X&oi=book\\_result&ct=result&redir\\_esc=y#v=onepage&q=plasma%20nitriding&f=false](http://books.google.sk/books?id=A2QG3UssqKUC&pg=PA163&dq=plasma+nitriding&hl=sk&ei=E51nTeP6CozKswa8h7TaDA&sa=X&oi=book_result&ct=result&redir_esc=y#v=onepage&q=plasma%20nitriding&f=false)