

When and Why Ion Nitriding/ Nitrocarburizing Makes Good Sense

E. Roliński and G. Sharp, Advanced Heat Treat Corp., Monroe, Mich.

Ion nitriding can be used in many applications, but some are so unique that they can be called "the best" applications, where the competitive treatments such as salt bath and gas nitriding cannot easily duplicate the unique results of ion nitriding.

Ion, or plasma, nitriding has been studied and used industrially for more than 40 years [1-8], but the technology has not been used to its full potential. Ion nitriding/nitrocarburizing is a low temperature (800-1100°F, or 430-595°C) process, which results in little or no distortion of the treated parts even if significant residual compressive stress is induced to the surface layer of the treated products. The ion nitriding process also has been referred to by other surface treating characteristics including plasma, vacuum, diffusion, low nitriding potential, passive surfaces activating, easy-to-control structure and pollution-free processes. These process characteristics with the exception of the low nitriding potential concept are discussed in the literature.

Low nitriding potential

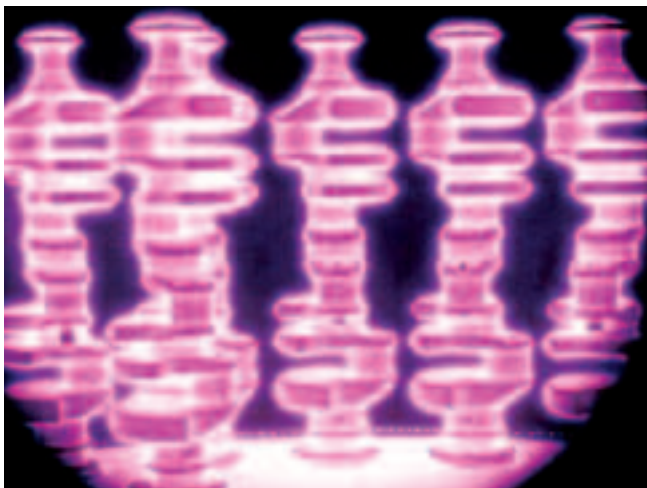
In ion nitriding, as with other diffusional processes, the rate growth of a nitrided layer with time is governed by a parabolic relationship [8]. The growth of the compound zone (white layer) thickness CZ is slower than parabolic [5], and it can be expressed by the simple polynomial equation:

$$CZ = a \cdot b \cdot t + c \cdot \sqrt{t} \quad \text{Eq (1)}$$

where a is the thickness of compound zone produced during ramping, b is the coefficient of sputtering ($b = 0$ in gas nitriding), c is the coefficient of diffusional growth and t is time. The growth of diffusional and sputtering components of Eq (1) developed empirically for 3Cr-Mo-V steel is shown in Fig. 1 [9]. This hypothetical graph shows that even without sputtering, a very long nitriding cycle would not pro-

duce a thick compound zone in this steel. Since ion nitriding is a vacuum process and only a small portion of nitrogen is ionized at this pressure, its effective nitriding potential is low [2]. Without sputtering, the compound zone thickness would not exceed 25 μm after 400 hours of nitriding. When the sputtering component is subtracted from the diffusional component, the real time growth of the compound zone in this steel is even slower (Fig. 2).

Figure 3 shows the growth of the compound zone during the first hours of ion nitriding initially is very rapid (faster than in gas nitriding). However, sputtering becomes a dominant factor in longer processes, limiting final compound zone thickness. As a result, thickness of the compound zone achieved in ion nitriding is similar to the thickness produced by the gas nitriding



Ion nitriding high-performance crankshafts. Masking plates on the tops of the parts protect threaded holes in the nose end of crankshaft from nitriding.

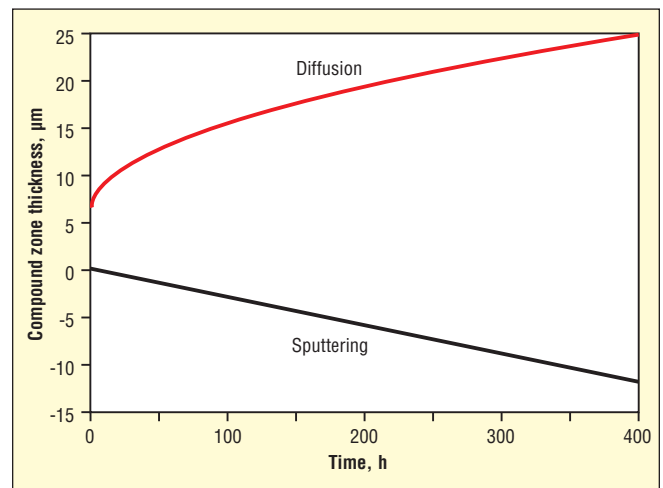


Fig. 1. Diffusional and sputtering components (Eq 1) for 3Cr-Mo-V steel ion nitrided at 1000°F (540°C) in a mixture of 30%N₂ and 70%H₂.

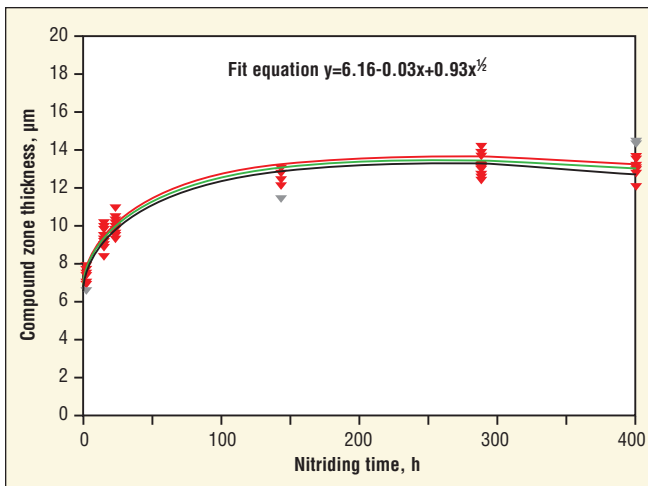


Fig. 2. Compound zone growth in 3%Cr-Mo-V steel during ion nitriding at 1000°F (540°C) in a 30%N₂-70%H₂ mixture (Ref. 9).

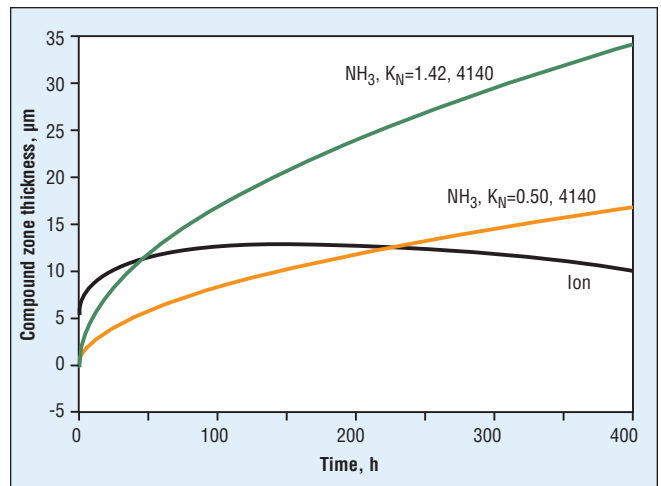


Fig. 3. Compound zone growth kinetic in ion and gas nitriding at 1000°F (540°C); gas nitriding curves are computer simulated for the nitriding potentials K_N indicated (Ref. 9, 10).

process performed at a very low nitriding potential. Also, because of the low nitriding potential, the structure of the long nitrided sample is free of the nitrocarbide network (Fig. 4). Figure 3 shows that a thin compound zone can be produced in gas nitriding, but requires very precise control of the nitriding potential at very low level. The same results (very thin compound zone) are achieved in ion nitriding without special nitriding-potential controlling instruments by the very nature of the process.

When is the Low-Nitriding Potential Process Required?

The unique characteristics of ion nitriding described above have practical consequences. There are several product types

that benefit greatly from ion nitriding treatment including many engineering components made of alloy steels, which must have very good fatigue and wear resistance properties. These parts (e.g., crankshafts) should have a nitrided layer with the limited compound zone thickness. They also often require local masking from nitriding to protect threaded holes from hardening, which can easily be achieved in ion nitriding.

Long parts

Large tools processed in the finished condition benefit from ion nitriding because they require precise control of the nitrided structure and dimensional stability. Cold-wall technology used occasionally for

treating extremely long parts such as rolls, shafts and extruder screws is a very economical way of nitriding. Usually, 90-120 in. (2.3-3 m) long modules are manufactured and assembled as required a few times a year to accommodate long parts. The engraved roll in Fig. 5 is an example of such a part after nitriding. The roll was processed at 840-860°F (450-460°C) in a cold-wall modular chamber and did not require any additional finishing operation after ion nitriding. Very long extruder screws are another example of the “best” application for the ion nitriding process, as they require precise dimensional control (mainly straightness) and a hard surface with a minimized need for polishing after the treatment.

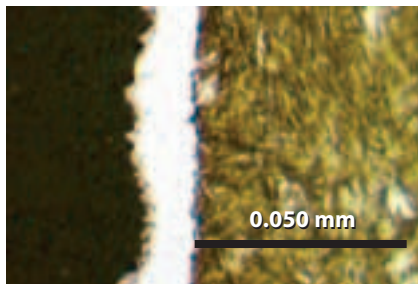


Fig. 4. Photomicrograph of the 3Cr-Mo-V steel sample ion nitrided at 1000°F (540°C) for 400 h in a 30%N₂-70%H₂ mixture shows no nitrocarbide network below the compound zone; nital etch.

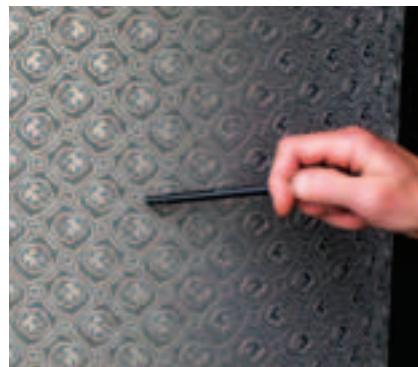


Fig. 5. Surface of embossing roll after ion nitriding



Fig. 6. Mechanical masking of torsion bars during ion nitriding; bars are positioned in the steel bushings, which serve as locators, as well as mechanical masks.

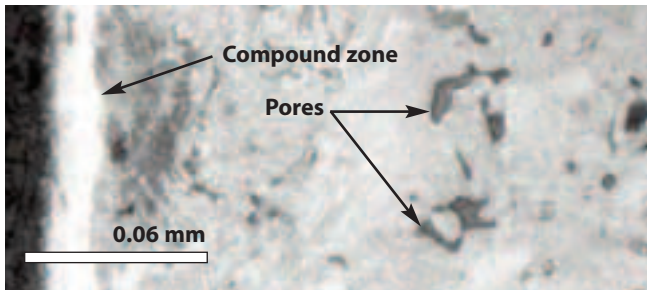


Fig. 7. Photomicrograph of plasma nitrided synchronizer hub showing several dark etched pores; nitral etch.

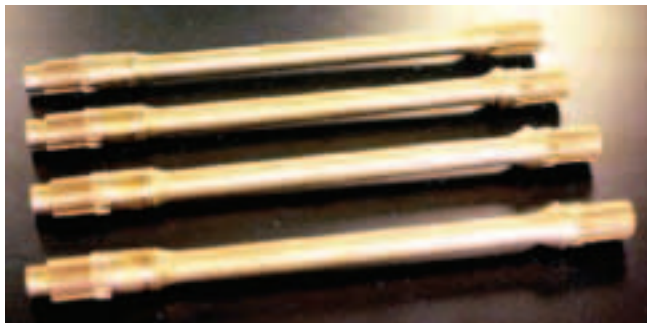


Fig. 9. Ion nitrided (in pure nitrogen) titanium-alloy racing half shafts

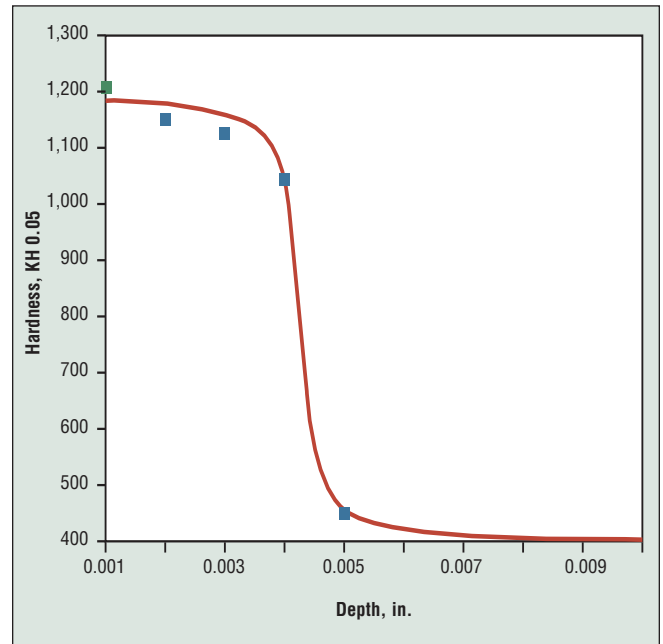


Fig. 8. Hardness profile in ion nitrided AISI Type 304 stainless steel soot blower tube.

Selective hardening

If the direct contact of the glow discharge with a surface is eliminated, the surface will not nitride. This simple principle is used for mechanical masking, effectively protecting a surface from ion nitriding (Fig. 6).

Low-density PM parts

Conventional nitriding of a low-density PM parts using salt or ammonia is difficult because salt or sand may be trapped in the porous structure and must be removed. Also, ammonia penetrates the internal structure of a PM part and causes through hardening, embrittlement and swelling. The practical option is ion nitriding [11].

The most important portion of the nitrided layer in a low-alloy PM part is the compound zone formed by the Fe_4N and Fe_2-3N iron nitrides and $Fe_2-3N(C)$ nitrocarbides. This zone has very good frictional and anticorrosion properties and can be seen easily under the optical microscope (Fig. 7).

Stainless steel products

Since plasma is very effective in removing the passive layer of a CrFe oxide formed

naturally on the surface of stainless steel, ion nitriding is widely used to harden (especially selective hardening) stainless steel parts. Chromium and other metal nitrides formed in stainless steels have good thermal stability and maintain very high hardness at elevated temperatures to 1100°F (595°C). Therefore, nitriding replaces chromium plating in many applications (especially in power-generation industry) where a nitrided layer does not peel off from the surface, as there are no thermal expansion problems. Also, mechanical masking is frequently used in many of these applications. Hardness of the nitrided layer in stainless steels is very high (>70 HRC equivalent), but as in all AISI 300 series stainless steels, there is an abrupt change of hardness between the layer and the substrate (Fig. 8).

Titanium alloys

Titanium and titanium alloys have unique corrosion, nonmagnetic and strength-to-weight ratio properties, but their tribological behavior is considered generally poor [12]. Therefore, machine components made of titanium alloys and subjected to

friction have to be surface treated to improve wear and antigalling resistance. Ion nitriding improves wear resistance and corrosion resistance of titanium alloys in certain environments [12, 13]. It is performed at a temperature higher than 1255°F (680°C) in pure nitrogen. Hydrogen in the atmosphere must be avoided because it forms brittle titanium hydrides in the matrix. The nitrided layer in titanium contains TiN and Ti_2N nitrides, as well as a diffusion zone enriched in nitrogen. The hardness of TiN is very high (2,000 HV), and its characteristic gold/yellow color imparts an appealing appearance to the treated parts (Fig. 9). Ion nitriding applied to one-phase α and β alloys or to lower strength $\alpha + \beta$ alloys improves their frictional properties and increases bending fatigue limit [13].

Large stamping dies

Ion nitriding is used in the auto industry to surface harden large stamping tools (Fig. 10), which usually are made of cast steels or a variety of cast irons. Ion nitriding significantly enhances the endurance of the die by eliminating adhesion of the die to the sheet steel

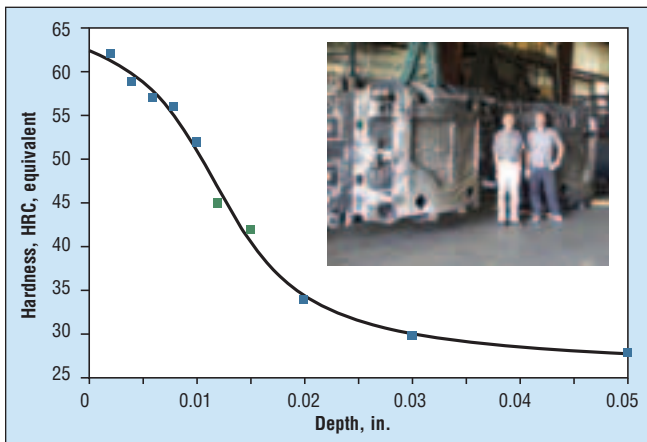


Fig. 10. Ion nitrided SAE 435/0050A cast steel stamping die (inset) and hardness profile after nitriding at 975°F (525°C)

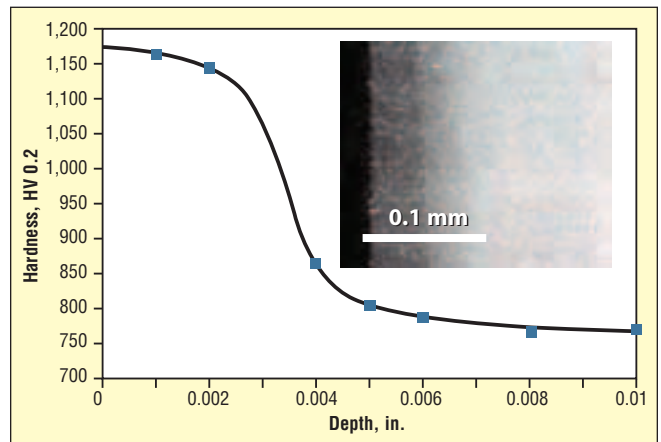


Fig. 11. Photomicrograph of M-2 steel bead insert ion nitrided at 900°F (480°C), inset, and hardness profile; surface hardness is 94.2 HR15N.

product and reducing its scratching and scoring by the significant increase of hardness.

In these applications, forming dies must be made of better materials to withstand more severe working conditions when used for forming high strength steels and dual phase sheet steels [12]. Surface treatment further enhances die performance. The common practice is to ion nitride dies that have high speed steel weld beads on the die in the most critical and stressed areas. High speed steels such as M-2 nitride very well, and if the case structure is controlled with sufficient precision, they offer good performance. Ion nitriding provides good control of the case structure. Typical case structure and hardness profile of M-2 steel insert after ion nitriding is shown in Fig. 11. There is no compound

zone and no evidence of nitrocarbide network at grain boundaries. Hardness of this layer is sufficiently high to withstand almost any stress produced during forming of the HSS sheet auto body parts. **IH**

References

- Knüppel, H. et al., *Stahl Eisen*, V 78, p 1871, 1958
- Seybolt, A.U., *Met. Trans.*, AIME, V 245, April, p 769, 1969
- Keller, K., *Härtereitechnische Mitteilungen*, V 26, p 120, 1971
- Edenhofer, B., *Heat Treatment of Metals*, V 23, p 59, 1974
- Marciniak, A., *Surf. Engng.*, 1986, V 2, p 283, 1986
- Lampe, T. et al., *Surf. Engng.*, V 4(1), p 69, 1993
- Sun, Y. and Bell, T., *Matl. Sci. & Engng.*, 1997, V A 224, p33, 1997

- Roliński, E. et al., *J. Matls. Engng. & Perf.*, V 9(4), p 457, 2000
- Roliński, E. et al., *J. Matls. Engng. & Perf.*, V 10(4), p 444, 2001
- Maldzinski, L. et al., *Proc. 12th Intl. Conf. Surf. Mod. Tech.*, ASM Intl., p 215, 1998
- Roliński, E. and Sharp, G., *Ind. Htg.*, Oct., p33, 2004
- Roliński, E., *Surf. Engng.*, V 2, p 35, 1986
- Shibata, H. et al., *Fatigue*, 1994, Vol. 16, August, p370, 1994
- Konieczny, A., *Proc. 11th Intl. Symp. Proc. & Fab. Adv. Matls*, ASM Intl., p.1, 2002

For more information: Ed Roliński, VP Technology, Advanced Heat Treat Corp., 1625 Rose St., Monroe, MI 48162; tel: 734-243-0063 fax: 734-243-4066; e-mail: doctorglow@ion-nitriding.com; web: www.ahtweb.com.

Visit Advanced Heat Treat Corp. online at: www.ahtweb.com for all your heat treat needs.

