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Tribological and Microstructural Characteristics of Ion-Nitrided Steels

(NASA-TM-83368) TRIBOLOGICAL AND
MICROSTRUCTURAL CHARACTERISTICS OF
ION-NITRIDED STEELS (NASA) 12 P
HC A02/MF A01

ABE-24635

CSOI 11F

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Prepared for the
International Conference on Metallurgical Coatings
sponsored by the American Vacuum Society
San Diego, California, April 18-22, 1983

NASA

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TRIBOLOGICAL AND MICROSTRUCTURAL CHARACTERISTICS OF ION-NITRIDED STEELS

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ABSTRACT

Three steels AISI 4140, AISI 4340 and AISI 304 stainless steel were ion-nitrided in a plasma consisting of a 75:25 mixture of $H_2:N_2$, sometimes with a trace of CH_4 . Their surface topography was characterized by SEM and two distinct compound phases were identified: the γ and the ϵ . The core-case hardness profiles were also established. The low Cr alloy steels have an extended diffusion zone in contrast to the 304 stainless steels which have a sharp interface. The depth of ion-nitriding is increased as the Cr-content is decreased. Friction tests reveal that the γ surface phase has a lower coefficient of friction than the ϵ -phase. The lowest coefficient of friction is achieved when both the rider and the specimen surface are ion-nitrided.

INTRODUCTION

Ion-nitriding is a glow discharge surface modification technique primarily used for case-hardening ferrous alloy steels to increase surface hardness and thus provide improved wear resistance. The ion-nitriding process has distinct advantages over the conventional gas-nitriding process: shorter and more effective nitriding times, lower temperatures, practically no structural distortion, uniform case depth and accurate control of the growth rate of the compound layer (refs 1-6).

During ion-nitriding the specimen is the cathode of the dc glow discharge. Impinging ions, usually a mixture of nitrogen and hydrogen, deliver enough energy to the specimen to heat it to a preselected nitriding temperature. Generally, the preferred temperature range is between 490 to 565° C, however, temperatures as low as 325° C have been reported (ref. 7). The best response to nitriding usually occurs when steels contain at least one of the following alloying elements: Al, Cr, Ti, Mo, V, W. Unalloyed carbon steels have a tendency to form an extremely brittle case that readily spalls.

The ion-nitriding mechanism is not completely understood. However it seems to consist of two simultaneous mechanisms: gas absorption and ionic bombardment. Atomic nitrogen diffuses into the steel and forms complex nitrides with the iron and the alloying elements. This generates distinct compound layers with a graded diffusion zone which in turn contributes to improved mechanical and tribological properties. Hard compound layers thus produced are responsible for the significant improvements in resistance to adhesive and scuffing wear. Generally these hard compound layers are considered undesirable because of their brittleness, but when their thickness is controlled as is done in ion-nitriding these surfaces exhibit wear resistance and low friction.

In this study three commercially used steels: AISI 4140, AISI 4340 and AISI 304 stainless steel were ion-nitrided and surface analysed to identify the compound layers formed. Hardness profiles were measured and their frictional and wear characteristics determined.

EXPERIMENTAL CONDITIONS

Ion-Nitriding Chamber and Procedure

The ion-nitriding chamber is shown photographically in Fig 1(a) and schematically in Fig. 1(b). A glass cylinder 45 cm in diam. with a removable aluminum cover rests on the steel feed-through ring. The steel specimen to be nitrided is the cathode and the steel base plate mounted on ceramic insulators is the anode. Temperature of the specimen is measured by a chromel-alumel thermocouple embedded in the specimen. The nitriding gas is admitted to the chamber by a valve which controls the variable leak rate.

The nitriding process parameters were: discharge voltage of 450-600 volts dc, current density 11 to 13 mA/cm², discharge pressure 3.5 to 4 Torr. The nitriding gas was a premixed mixture of 75 percent H₂ - 25 percent N₂. In one experiment the mixture used, also contained a trace CH₄. Ion-nitriding was performed for 6 hours at two temperatures: 350° C and 500° C. Before ion-nitriding, the steel specimens were argon dc sputter-etched for 20 minutes.

Materials Testing

Three steels: AISI 4140, AISI 4340 and AISI 304 stainless steel, were ion-nitrided. The specimens were disks 6.3 cm in diameter by .25 cm thick, with the surfaces progressively ground through 600 grit Si-C paper.

The surface phase structures of the ion-nitrided samples were investigated by X-ray diffractometry. Vickers microhardness measurements using 100 g loads were used to determine the core and case depth. Scanning Electron Microscopy (SEM) was used to determine the morphological growth features. Frictional testing was performed in a vacuum with a pin and disk apparatus using a 440 C stainless steel pin (radius 4.75 mm and 55 HRC) sliding on the ion-nitrided disk surface at a constant speed of 1.52 m per min and a normal load of 2.45 N in a vacuum of 2×10^{-3} Torr.

RESULTS AND DISCUSSION

X-Ray Diffractometry

The X-ray diffraction lines of the AISI 4140 steel specimens are seen in Fig 2(a). Those for ion-nitrided in the 75:25 pure mixture of H₂:N₂ can be attributed to the presence of the γ -Fe₄N single phase Fig. 2(b). However, the samples ion-nitrided in the 75:25 H₂-N₂ gas mixture containing trace CH₄ formed the ϵ -Fe₂₋₃N phase Fig. 2(c). According to the Fe-N phase diagram, the Fe₄N phase is formed between 5.7 - 6.1 wt. percent N with a fcc structure whereas the ϵ -Fe₂₋₃N phase is formed between 11-11.35 wt percent N with an hexagonal structure (Fig. 3).

The nitriding potential of the gas mixture determines the concentration (strictly speaking, the activity) of nitrogen in iron. When the potential, and hence the concentration, exceeds that in equilibrium with the iron nitrides: γ - Fe₄N or ϵ -Fe₂₋₃N, then these phases can form at the specimen surface. The single γ -Fe₄N phase is formed only when the gas mixture ratio resembles that of ammonia, NH₃, and the plasma is free of any carbon con-

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tamination. When the plasma contains traces of carbon, the ϵ -Fe₂₋₃N is formed (this could be possibly Fe₂₋₃N_xC_y). Formation of an homogeneous ϵ -layer is difficult to obtain, since the substrate carbon level continues to change with the gas mixture (ref. 3).

Microscopy

The compound layer is apparent in the metallographic cross section of the 4140 steel, ion-nitrided at 500° C for 6 hours, after etching with 1 percent Nital reagent (Fig. 9). The non-etching white compound layer of γ -Fe₄N appears to be 14 μ m thick and the diffusion zone at least 250 μ m in depth. The hard layer of γ -Fe₄N is of special interest, since the topographical structure of this layer will determine the mode of wear. The surface morphology of the untreated specimen and the compound layer is shown by a Scanning Electron Micrographs in Fig. 5. The basic change in the surface topography after ion-nitriding was the disappearance of sharp grinding marks and surface tearing, and the formation of large conical type of asperities.

Microhardness Measurements

The surface hardness for all three steels 4140, 4340 and 304 stainless steel is about Hv 780 kg/mm² (Fig. 6) the microhardness profiles reveal diffusion zones for the two low-Cr alloy steels extending over a region of 350 μ m, whereas, a very sharp interface forms between the core and the case in the 304 stainless steel.

The ion-nitrided 304 steel surfaces form a very brittle core and exhibit a very pronounced spalling of the compound layer: resulting in a chipped compound layer (Fig. 7) and a distinct boundary of separation between the core and the case (Fig. 8.). This sharp interface for 304 stainless steels has been also observed by several investigators (refs. 8 and 9) who also reported that steels containing more than 5 percent Cr by weight display sharp interfaces and shallow nitrided depth, which is inversely proportional to the Cr-alloy content of the steel.

Frictional Characteristics

Untreated 4140 steel specimens in dry sliding displayed high coefficients of friction: 0.45 (Fig. 9). Ion-nitrided surfaces had much lower friction: 0.13 for the γ -Fe₄N surface and 0.15 for the ϵ -Fe₂₋₃N surface. The average steady-state wear rate after 4000 cycles was 12 x 10⁻¹⁵ m³/m for γ and 15 x 10⁻¹⁵ m³/m for the ϵ phase. A 440 C (HRC 52) rider was used in all the friction tests, except in one experiment where an ion-nitrided 440 C rider was slid against a γ -Fe₄N surface. This combination displayed the lowest coefficient of friction: 0.11.

During sliding friction, the compressive load applied to the contact zone not only modifies the morphology of the surface layer but also affects other layers directly beneath the surface. For untreated surfaces, the surface asperities deform plastically and a rapid expansion of area is observed until a the bulk material supports the applied load. The ion-nitrided surfaces, as shown in Fig. 5, have a topography of conical shaped asperities. As the load is applied the plastic deformation is offset by the hardening effects of the diffusion zone and the compound hardness and the conical asperity shape of the surface compound layer. Thus, the contact area is smaller and the friction coefficient lower.

CONCLUSIONS

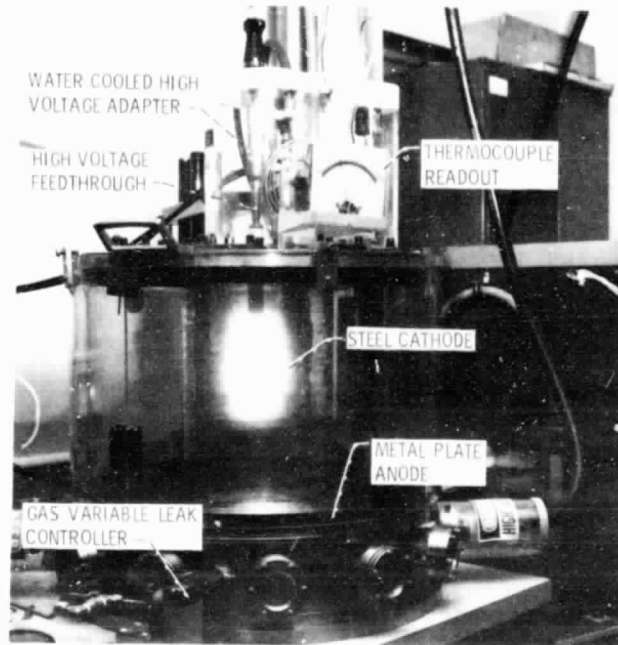
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Evaluation of ion-nitrided steel surfaces was conducted by X-ray diffractometry, optical and SEM microscopy, microhardness measurements and frictional testing. 1) Two distinct surface compound phases were γ and ϵ identified: 2) The surface hardness was measured at Hv 781 Kg/mm² and the hardness depth profiles revealed that low Cr alloy steels formed an extended diffusion zone, where as the 314 stainless steels had a sharp interface. 3) SEM showed that the surface topography of the compound layers had conical type of asperities. 4) Sliding friction tests indicate that the γ surface phase displays a lower coefficient of friction as that of the ϵ phase and that the lowest coefficient of friction was achieved when both the rider and the specimen surface are ion nitrided.

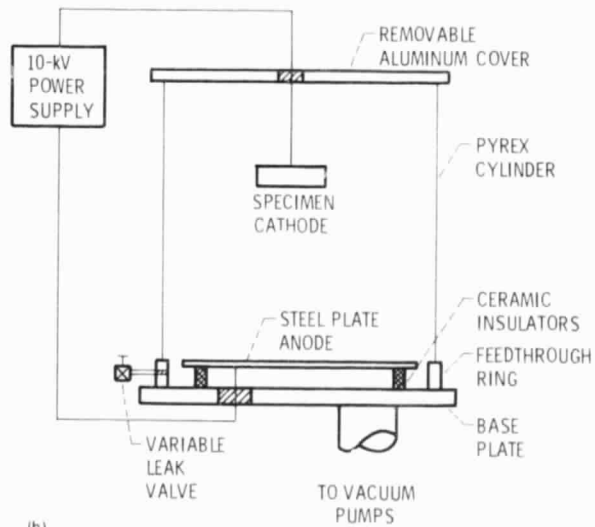
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(a)



(b)

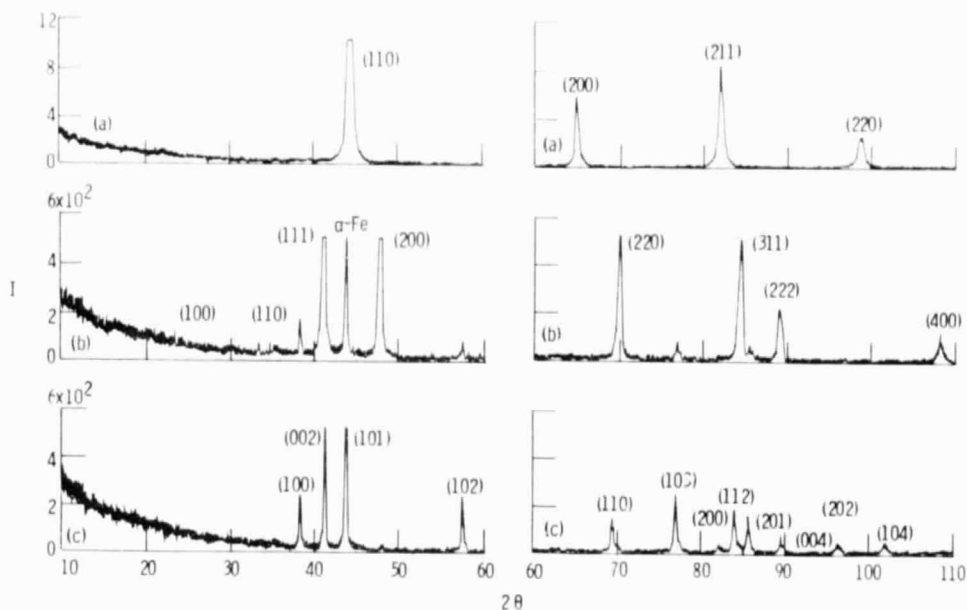
(a) Ion nitriding chamber.

(b) Schematic of ion nitriding apparatus.

Figure 1. - Ion nitriding system.

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(a) Original AISI 4140 steel.

(b) Ion nitrided in 75% H_2 -25% N_2

(c) Ion nitrided in 75% H_2 -25% N_2 +trace CH_4

Figure 2 - X-ray diffractograms of ion nitrided AISI 4140 steel.

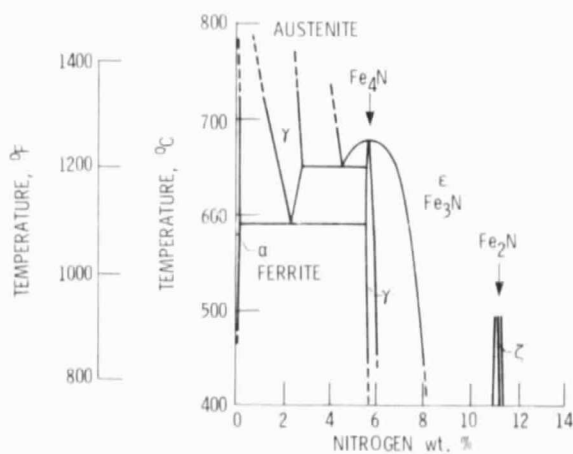


Figure 3 - Iron-nitrogen phase diagram.

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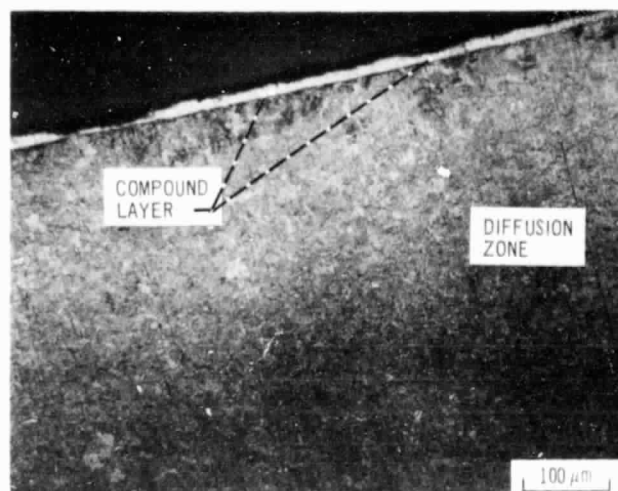
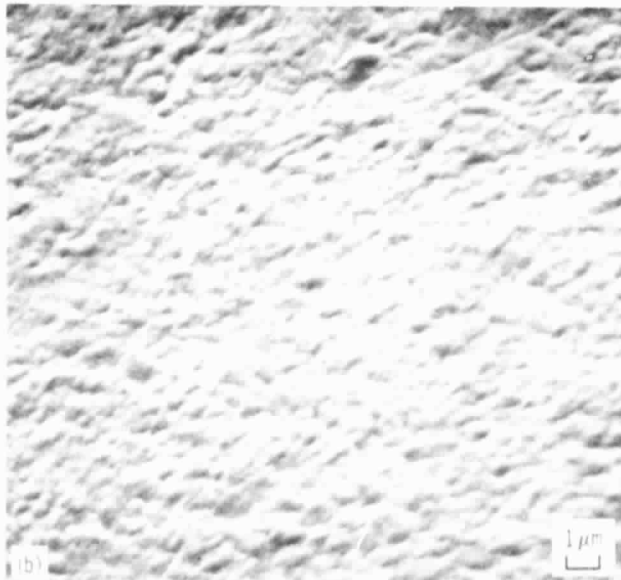
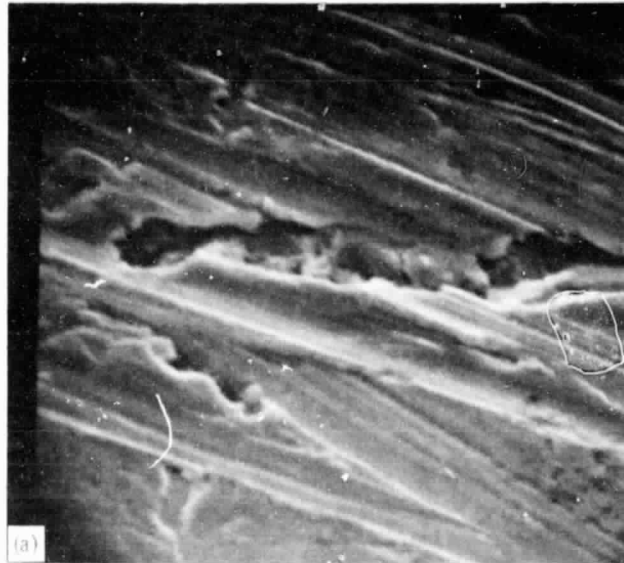


Figure 4. - Metallographic cross section of ion nitrided 4140 at 500° for 6 hours.

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(a) Prior to ion nitriding.

(b) After ion nitriding 304 stainless steel at 500° C for 6 hours.

Figure 5. - SEM micrographs of the topography.

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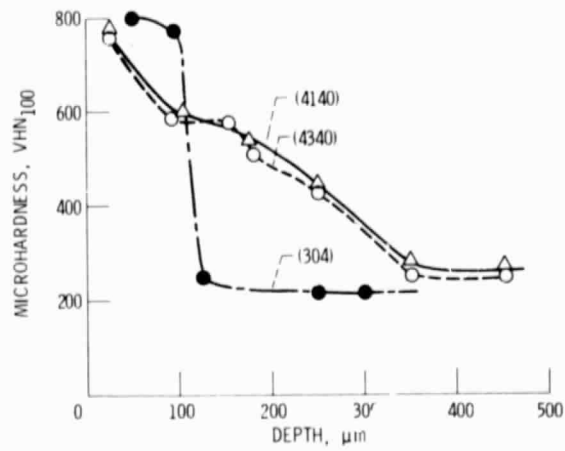


Figure 6. - Microhardness profiles of ion nitrided steels: 4140, 4340 and 304 (plasma nitrided for 6 hours at 500°C, gas 75% H_2 -25% N_2 at 3 torr).



Figure 7. - SEM cross section of ion nitrided 304 stainless steel at 500°C for 6 hours in 75% H_2 -25% N_2 at 3 torr.

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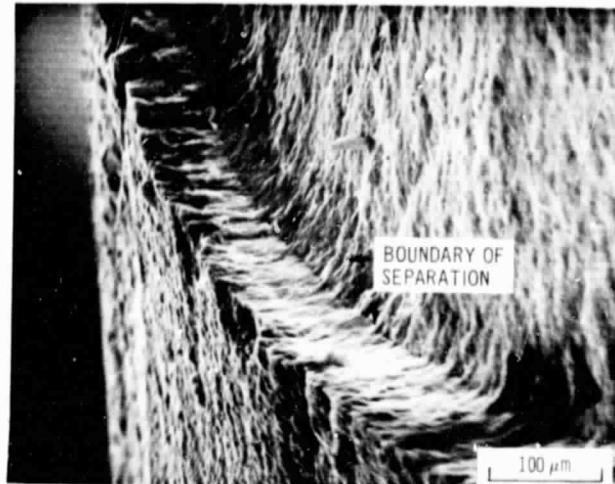


Figure 8. - SEM surface view of ion nitrided 304 stainless steel after spalling of the compound layer.

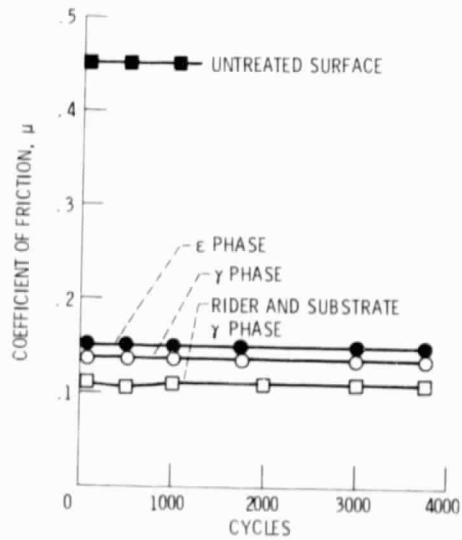


Figure 9. - Comparison of the coefficients of friction of ion nitrided 4140 steel surfaces (440C rider; load, 250 GF; speed, 1.52 Mmin⁻¹; pressure, 2 x 10⁻³ torri).