CONF-960401--46

LA-UR-96- 1321

Title: Tribological Properties of Nitrogen Implanted and Boron Implanted Steels

RECEIVED
MAY 31 1996
OSTI

Author(s): K. T. Kern, Norfolk State University
K. C. Walter, MST-4
S. Fayeulle, Ecole Centrale De Lyon
A. Griffin, Jr., MST-4
M. Nastasi, MST-4
J. Tesmer, CMS
H. Kung, CMS
Y-C. Lu, CMS

Submitted to: Proceedings of Materials Research Society
1996 Spring Meeting
San Francisco, CA
April 8-12, 1996

Presented April 10, 1996

Los Alamos
NATIONAL LABORATORY

TRIBOLOGICAL PROPERTIES OF NITROGEN IMPLANTED AND BORON IMPLANTED STEELS

K.T. Kern[1], K.C. Walter[2], S. Fayeulle[3], A.J. Griffin[2], Jr., H. Kung[2], Y. Lu[2], M. Nastasi[2], and J.R. Tesmer[2].

[1]Center for Materials Research, Norfolk State University, Norfolk, VA 23504, K_Kern@lanl.gov
[2]Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM 87545
[3]Laboratore MMP_URACNRS 447 Ecole Centrale De Lyon, Ecully, France.

ABSTRACT

Samples of a steel with high chrome content was implanted separately with 75 keV nitrogen ions and with 75 keV boron ions. Implanted doses of each ion species were 2-,4-, and 8 x 10^{17} /cm^2. Retained doses were measured using resonant non-Rutherford Backscattering Spectrometry. Tribological properties were determined using a pin-on-disk test with a 6-mm diameter ruby pin with a velocity of 0.94 m/min. Testing was done at 10% humidity with a load of 377g. Wear rate and coefficient of friction were determined from these tests. While reduction in the wear rate for nitrogen implanted materials was observed, greater reduction (more than an order of magnitude) was observed for boron implanted materials. In addition, reduction in the coefficient of friction for high-dose boron implanted materials was observed. Nano-indentation revealed a hardened layer near the surface of the material. Results from grazing incidence x-ray diffraction suggest the formation of Fe_2N and Fe_3N in the nitrogen implanted materials and Fe_3B in the boron implanted materials. Results from transmission electron microscopy will be presented.

INTRODUCTION

Ion implantation is one method for modifying the near-surface region of materials, in which the implanted species forms a layer that may be different from the substrate in chemical composition and in physical properties. Implantation may result in changes in the surface properties of a material, including hardness, wear, coefficient of friction and other properties Characterization of the implanted layer will allow for the use of this surface modification for control of surface properties, including tribological properties. Wear of mechanical systems is a $200 billion per year problem, and therefore control of tribological properties is important in the production of moving parts.

Previous studies of ion implanted steels have given evidence of the mechanisms for property changes due to ion implantation. In particular, nitrogen implanted 304 stainless steel has been extensively studied. The wear mechanism for 304 SS involves the conversion of austenitic (fcc) phase material by plastic deformation into a hard, brittle martensitic (bct) surface layer, which fractures under the load of the wearing body [1].

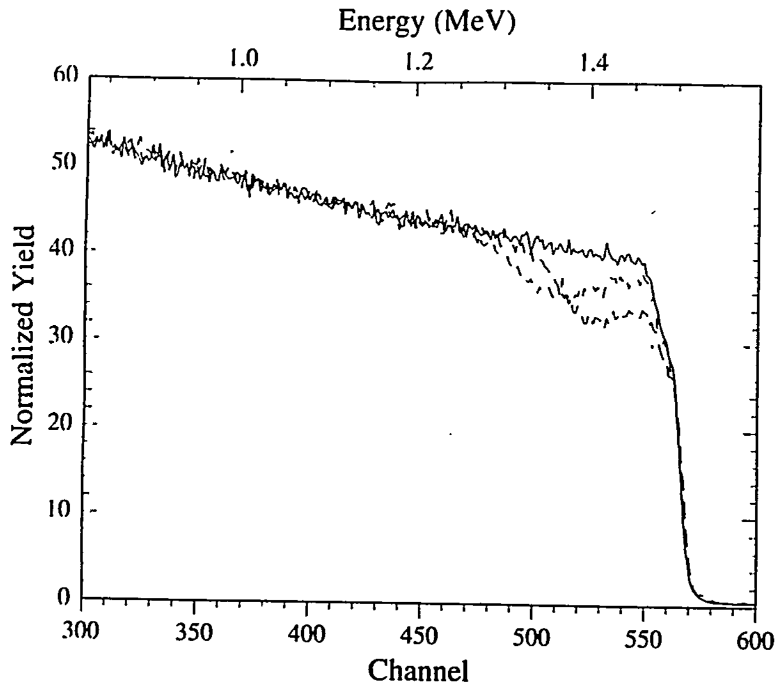

Fig. 1. RBS (2 MeV alpha) on white iron and white iron implanted to $4x10^{17}$ N^+/cm^2 and $4x10^{17}$ B^+/cm^2.

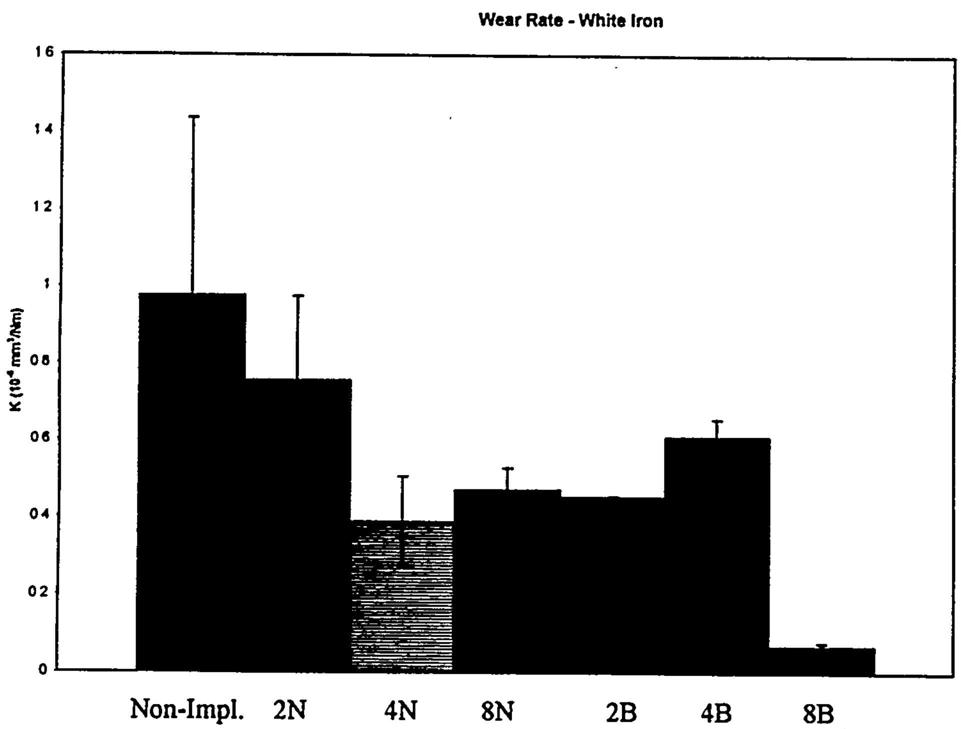

Fig. 2. Wear rates of non-implanted and implanted white iron.

Implantation of nitrogen into 304 SS stabilizes the austenitic phase and prevents transformation to the martensitic phase [2]. Evidence that boron implantation may modify the wear properties of 304 SS via the same mechanism is mixed [3, 4]. The modification of wear properties is sensitive to the phases existing in the material prior to implantation, as implantation of boron into steels with higher bcc content shows a greater hardening effect than in pure fcc materials [5]. In other steels, wear improvement of nitrogen implanted steels has been linked to the chromium content of the steel, implantation of N^+ into higher Cr content steels resulting in greater improvement of wear properties than low Cr content steels. It is expected that this observation is a result of chrome forming nitrides. No similar compositional effect has been reported for boron implantation.

White iron is a high chromium (about 30% Cr) steel with a small percentage of carbon. In this study, this high chrome steel was modified through nitrogen implantation and boron implantation. Surface characterization included wear testing, coefficient of friction determination, and hardness testing. The nature of changes in these surface properties will be correlated with changes in the microstructure of the material resulting from the implantation.

EXPERIMENT

Samples of white iron were prepared by mechanical polishing coupons (0.5 in x 0.5 in) of material to surface roughness of less than 20 nm. Samples were implanted with N_2 ions at 150 keV to incident fluxes of $2x10^{17}$ /cm^2, $4x10^{17}$ /cm^2, and $8x10^{17}$ /cm^2, and implanted with boron ions at 75 keV to incident fluxes of $2x10^{17}$ /cm^2, $4x10^{17}$ /cm^2, and $8x10^{17}$ /cm^2. Samples were cooled during implantation. Retained ion doses were determined using Rutherford Backscattering Spectrometry (RBS), including analysis at a nitrogen resonance (8.9 MeV) and at a boron resonance (6.6 MeV) [6]. The analysis software RUMP was used to determine implant species quantity and distribution.

Surface properties were determined by tribology and nano-indentation. Tribological properties were studied using a pin-on-disk tribotester, in which the pin was a 6-mm diameter ruby ball and the disk was the sample being tested. In these tests, the pin rests on the sample to be tested which is turned at a constant rate. The pin thus traces a circular path on the disk. In this study, the disk spun at 100 RPM and the pin traced a track 3-mm in diameter, for a relative velocity of 0.94 m/min. Weight was added to the pin for a total load of 4.5 Newtons and a maximum contact stress of 1.4 G Pa. All tribotests were conducted in a 10% humidity atmosphere. Tests were halted so that the resulting wear was within the implanted region, for most samples less than 6000 cycles. During tests, the coefficient of friction was calculated and recorded using an automated computer system. Following tests, the wear track was cleaned of wear debris and the wear track depth and cross-sectional area were measured using a profilometer. Wear rates were calculated as K = wear volume/(load x distance). Nano-indentation was used to measure the hardness of the implanted layers. Twelve indents were made at each of four depths, nominally 25, 100, 200, and 300 nm. The 12 hardness values at each depth were then averaged.

Grazing incidence x-ray diffraction (GXRD) was used to determine micro-structure of the implanted materials. The grazing incidence was used to probe only the near-surface. Resulting diffraction peaks were compared to a standard data base for identification of compounds. In addition, transmission electron microscopy (TEM) was used for micro-structural analysis.

RESULTS

RBS of non-implanted white iron indicated an atomic Cr to Fe ratio of 28 to 72. In addition, the non-implanted material contains a small percentage of carbon, which could not be accurately determined. RBS results for implanted materials are summarized in Table 1. Boron implanted materials retained the entire implant dose, while the nitrogen implanted material became sputter limited at the higher doses and thus did not retain the full doses. The implant distributions could be modeled using gaussian distributions, with the exception of the $8x10^{17}$ /cm^2 nitrogen implant, which was skewed toward the surface due to sputtering. In addition, the nitrogen implant distribution was shallower than the boron distribution. These effects can be seen in the $4x10^{17}$ /cm^2 dose results shown in figure 1, in which the decrease in the Fe-Cr edge is an indication of implanted atoms being substituted for the Fe and Cr atoms.

Implanted Species	Implant Dose (x 10^{17} ions/cm^2)	Retained Dose (x 10^{17} ions/cm^2)	Peak Depth (nm)	Peak FWHM (nm)
N	2.0	1.9	79	100
N	4.0	3.9	87	108
N	8.0	5.5	73	146
B	2.0	2.0	125	120
B	4.0	4.0	136	133
B	8.0	8.0	129	126

Table 1. Results of RBS analysis on implanted White Iron.

Wear rates for non-implanted and implanted white iron are shown in figure 2. Some reduction in wear rate is evident for samples implanted to $2x10^{17}$ N^+/cm^2 while samples implanted with 4- and $8x10^{17}$ N^+/cm^2 have wear rates that are less than half that of the non-implanted material. In contrast, all materials implanted with boron show significant reduction in wear rate, the rates for samples implanted to 2- and $4x10^{17}$ B^+/cm^2 being similar to the rates for the materials implanted to 4- and $8x10^{17}$ N^+/cm^2. The wear rate for white iron implanted to $8x10^{17}$ B^+/cm^2, however, showed the largest improvement and was only 3% of the wear rate for the non-implanted material. This sample showed this wear resistance even when tested to 12,000 cycles.

The coefficient of friction for a ruby pin sliding on a white iron disk was recorded for each implant condition. Coefficient of friction for selected samples are shown in figure 3. No significant change in coefficient of friction was observed for nitrogen implanted white iron, although for the material implanted to $4x10^{17}$ N^+/cm^2 the coefficient of friction was 10% higher than the coefficient for the non-implanted material. This difference is not significant and is not interpreted as being a result of the implantation. For the boron implanted materials, the materials implanted to $2x10^{17}$ B^+/cm^2 and $4x10^{17}$ B^+/cm^2 showed a slightly higher coefficient of friction than the non-implanted material, but again this difference is not considered significant. For the $8x10^{17}$ B^+/cm^2 implanted material, however, the coefficient of friction was 20% of that for the non-implanted material. This low coefficient of friction, while corresponding to the low wear rate, requires further investigation as the RBS data indicated a thin layer of carbon on this sample which may be acting as a lubricant during the wear test. It is suspected that the carbon layer resulted from backstreaming during the ion implantation of boron.

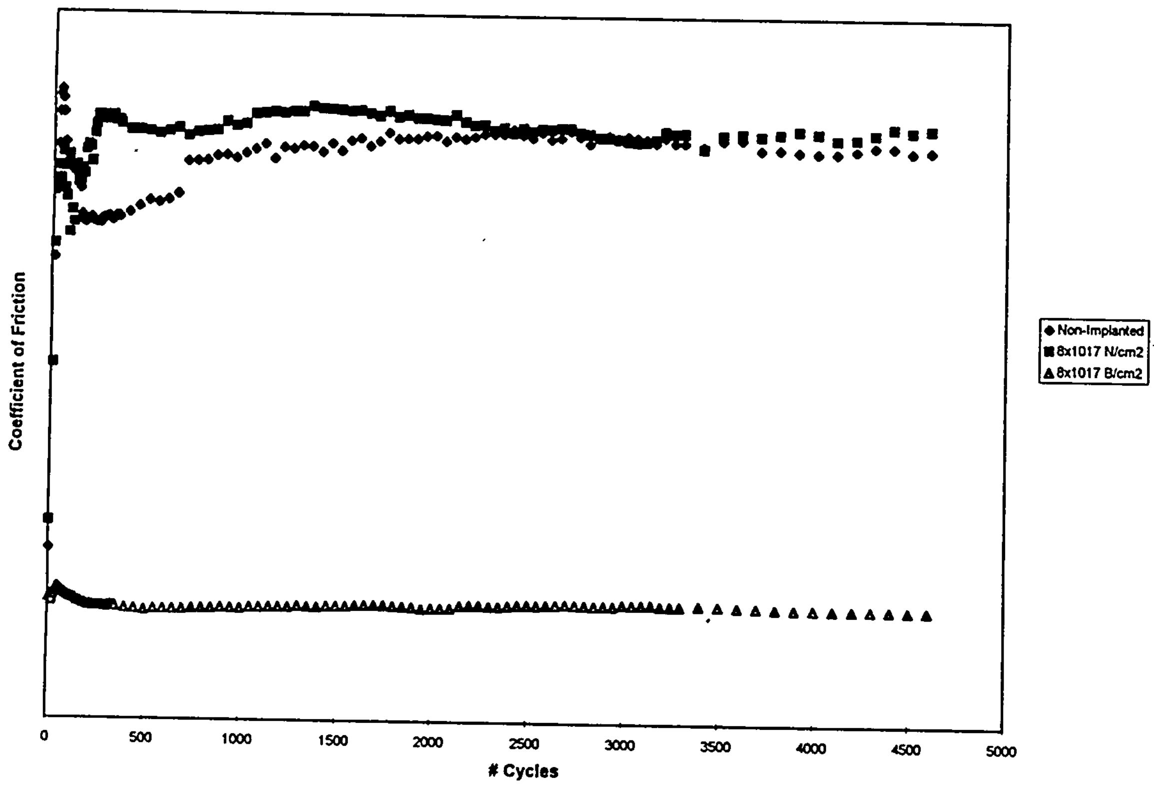

Figure 3. Coefficient of friction for non-implanted white iron and white iron implanted to $8x10^{17}$ N^+/cm^2 and $8x10^{17}$ B^+/cm^2.

Nano-indentation showed that the hardness of the implanted materials varied through the implanted region. Implanted materials, regardless of species implanted or dose, had a hardness at a depth of 23 nm that was 20% to 40% greater than the hardness of the non-implanted materials. Due to surface roughness, the hardness of white iron implanted to $8x10^{17}$ N^+/cm^2 could not be determined at this depth. At 102 nm, near the peak of the implanted species distribution, the hardness was not significantly different from that of the non-implanted material, while indents to depths greater than the depth of the implants showed that the implanted materials were slightly less hard than the non-implanted material.

GXRD and TEM were used to determine micro-structural information for the implanted materials. TEM and XRD of non-implanted materials showed a bcc structure. For the nitrogen implanted white iron, evidence of Fe_2N and Fe_3N was seen in these samples as has been reported in previous studies [7]. GXRD of boron implanted materials showed a peak corresponding to Fe_3B, which was particularly prominent in the $8x10^{17}$ B^+/cm^2 spectrum, although TEM results indicated an amorphous implanted region.

CONCLUSIONS

Implantation of nitrogen into white iron results in hardening of the surface layer and reduced wear for implanted doses greater than $4x10^{17}$ N^+/cm^2. Micro-structural analysis show the formation of Fe_2N and Fe_3N in the implanted materials. For boron implanted white iron, hardening of the surface layer was also observed, however improvements in wear resistance occurred for doses as low as $2x10^{17}$ B^+/cm^2. Additionally, boron implantation resulted in a low coefficient of friction for a dose of $8x10^{17}$ B^+/cm^2, although this result may be influenced by a layer of carbon on the surface of the material. The micro-structure of boron implanted white iron indicates Fe_3B is forming in the material, although TEM indicates a largely amorphous surface layer. Curiously, no evidence of hard compound formation with Cr has been found. The increased wear resistance of N and B implanted white iron is due to the confirmed presence of hard compounds (Fe_2N, Fe_3N, Fe_3B) in the implanted surface.

ACKNOWLEDGMENTS

This work was supported by DOE grant DE-FG01-94EW11493.

REFERENCES

1. K.L. Hsu, T.M. Ahn, and D.A. Rigney in K.C. Ludema, W.A. Glaeser, and S.K. Rhee (eds.) *Wear of Materials*. ASME. New York, 1979.
2. R.N. Bolster and I.L. Singer, *Appl. Phys. Lett.*, **36** (1980) 208.
3. S. Raud, H. Garem, A. Naudori, J.P. Villain, and P. Moine, *Mater. Sci. Eng.*, **A115** (1989) 245.
4. S. Shrivastava, A. Jain, and C. Singh, *Acta Metall. Mater.*, **43** (1995) 59.
5. S. Shrivastava, A. Jain, R.D. Tarey, D.K. Avasthi, D. Kabiraj, L. Senapati, and G.K. Mehta, *Vacuum.*, **47** 3 (1996) 247.
6. Handbook of Modern Ion Beam Analysis, J.R. Tesmer & M. Nastasi, editors, Materials Research Society, Pittsburgh, PA, 1995, pgs. 497, 499.
7. S. Fayuelle, *Appl. Surface Sci.* **25** (1986) 288.