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Enhanced wear resistance by compressive strengthening: A novel combination of laser and ion implantation technology

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In general, neon implantation is not very effective in reducing wear rates. However, neon implantation into laser-melted steel turns out to reduce the wear rate substantially as a result of a conversion of residual tensile stresses into compressive ones. Nitrogen implantation, on the other hand, at a high dose of 3×10^{17} ions/cm² at 90 °C exhibits a deleterious effect on the wear performance. A brittle layer of $\epsilon\text{-Fe}_2(\text{C},\text{N})_{1-x}$ nitrides is formed. At a lower dose of 1×10^{17} /cm² or implantation of nitrogen at 150 °C, the wear rate is also reduced.

Many successful wear-resisting materials consist of particles of a hard phase dispersed in a more ductile matrix. Such dispersion can be prepared by laser melting. The steel under investigation (2.05 wt. % C, 11.05 wt. % Cr, 0.62 wt. % W, and the balance, Fe) turns after laser treatment into austenite dendrites surrounded by segregated M_3C carbides with a hardness of 550–600 HV.^{1,2} However, by melting and resolidifying, surface layer tensile stresses are introduced.³ This tensile stress state detrimentally influences the wear performance by increasing the crack growth velocity. On the other hand, plastic flow is terminated by compressive stresses existing on the shear plane, and crack growth will be impeded. Consequently, ion implantation, introducing compressive stresses, might be an appropriate technique for improving the wear resistance of metals.^{4,5} Commonly used ions are N^+ , B^+ , C^+ , Mo^+ , Ti^+ , and Cr^+ . Responsible mechanisms are compressive strengthening, grain size strengthening, interstitial solid solution hardening, and second-phase strengthening. Furthermore, ion implantation might lower the friction coefficient or initiate a more favorable wear mechanism.⁶ The dominating mechanism is determined by the implanted ions, the surface, implantation conditions, and the wear system. In general, nitrogen implantations improve the wear properties, and neon exhibits either intermediate or negligible effects.⁷

Our basic idea is to convert high tensile stresses in the laser-melted surface into a compressive state after implantation. The effects upon wear can be measured quantitatively. To investigate the importance of compressive strengthening, neon is implanted and compared with nitrogen implantations. Nitrogen forms stable nitrides, and therefore, besides compressive strengthening, hardening mechanisms play a role as well. The wear experiments are designed in such a way that compressive stresses and hardening strongly define the wear process. Adhesive and oxidational wear are excluded.

For the laser treatment, a Spectra Physics 820 1.5 kW cw CO_2 has been used. The beam is focused with a 127 mm ZnSe lens. Melted tracks are made near each other with some overlap. The conditions are 1300 W power on the surface, focus point 15 mm above the surface, and a scanning velocity of 4 cm/s.

Implantations are carried out using an Extrion 200 kV

implanter with doses of 3×10^{16} , 1×10^{17} , and 3×10^{17} ions/cm² at an energy of 50 keV per N^+ or Ne^+ ion. The projected range is 55 nm with a root-mean-square standard deviation of 16 nm in the case of nitrogen, and 42 nm with a deviation of 15 nm of the neon implantation as calculated according to the theory of Lindhard *et al.*⁸ The temperature during implantation is kept at 90 or 150 °C.

Wear experiments are carried out using a pin-on-disk tester.⁹ The pin is a 5 mm ruby crystal ball under a load of 2.3 N. The disk is made of the implanted material under investigation, which is worn at a speed of 5 cm/s under lubrication of pure ethanol. The experiments are done in a dry nitrogen atmosphere to exclude the influence of moisture. Subsequently, the wear volume is determined using an interference microscope. With a strain gauge configuration, the friction force is monitored during the experiment. In all experiments the friction coefficient varies between 0.3 and 0.5.

The Vickers hardness of the implanted surfaces is measured with a load of 25 g. A series of indentations is placed across the laser-melted tracks. The hardness of the only laser-melted material is 550 HV, which does not vary across the mutually heat-affected zones in the laser-melted tracks. A strong increase in hardness up to 1000 HV is observed after nitrogen implantation at 90 °C. Here the hardness varies strongly as a result of the heat-affected zones in the laser-melted tracks. Implanting at 150 °C results in a rather flat hardness profile of 700 HV. Implantation with neon results in an increase of the hardness with increasing dose. The hardness attained using 3×10^{17} ions/cm² varies between 600 and 1000 HV.

The wear performance is depicted in Fig. 1. At the onset of the experiment, there is a running in process. This is expressed in a running in parameter which is determined by the intersection of the extrapolation of the curve with the ordinate. These experimental parameters are listed in Table I. Increasing the dose of neon implantations results in a decreasing wear rate. At a dose of 3×10^{16} ions/cm², the wear rate increases after 6000 turns. The depth of the wear track is then about 160 nm, which is two times the implantation depth. Hereafter, the wear rate increases strongly, which is due to a new running-in process. A transition is also found with the highest dose implantation. Extended measurements

FIG. 1. (a) Measured wear volume of neon implanted laser-melted steel. (+) not implanted; (Δ) 3×10^{16} Ne^+ ions/ cm^2 ; ($\circ$) 1×10^{17} Ne^+ ions/ cm^2 ; ($\square$) 3×10^{17} Ne^+ ions/ cm^2 . (b) Measured wear volume of nitrogen-implanted laser-melted steel. ($\square$) 1×10^{17} N^+ ions/ cm^2 at 90°C ; ($\circ$) 3×10^{17} N^+ ions/ cm^2 at 90°C ; (Δ) 3×10^{17} N^+ ions/ cm^2 at 150°C ; (+) only laser melted.

done at lower loads show, after 15 000 turns, when the depth of the wear track is about the same as compared to the lowest dose implantation, an increase in wear rate. The wear rate increases gradually to a value of the unimplanted laser-melted steel. The absence of a steep increase in the wear rate immediately after penetrating the material implanted with the highest dose is ascribed to a higher wear resistance of the side walls of the track. Scanning electron microscope investi-

gations confirm that all wear tracks are worn in an abrasive manner, and no mutual differences could be found. Consequently, stresses and hardness define the wear process.

Transmission electron microscopic (JEM 200 CX) examination of the 3×10^{17} Ne ions/ cm^2 implanted surface shows the formation of bubbles with an average diameter of about 16 nm (see Fig. 2). Stereomicroscopy reveals bubbles up to a 200 nm depth with a volume concentration of 2.5%. This means an increase in lattice parameter of 0.8%, which is sufficient to convert the surface under tensile stresses into a compressive state. Furthermore, an extra hardening can be expected because of the interaction between dislocations and bubbles.

Independent of dose, neon implantations improve the laser-melted surface. However, nitrogen implantation always shows a worse running-in behavior and, for 3×10^{17} ions/ cm^2 at 90°C , even a slightly worse steady-state wear. The somewhat higher steady-state wear rate, after the nitride layer has been spalled, might be caused by the large amount of hard nitride particles in the wear debris. The composition and phase of the nitrides after implantation strongly influences the final wear behavior. Nitrogen implantation improves the wear resistance mainly by compressive strengthening and hard nitrides. In particular, interstitial nitrogen, precipitation of nitrides, and developing of martensitic phases contribute to the volume expansion. Further aspects determining the wear process are tribo-enhanced diffusion.¹⁰ In Fig. 3 the electron diffraction pattern of the 3×10^{17} N^+ ions/ cm^2 implanted surface is depicted. The diffraction rings originate from $\epsilon\text{-Fe}_2(\text{C},\text{N})_{1-x}$ nitrides, according to calculations of Rauschenbach.¹¹ CrN could not be detected, which is consistent with the convergent electron Mössbauer spectroscopy spectra and in line with the conclusions of Dos Santos *et al.* on the same steel.¹² These ϵ nitrides are known to create a brittle layer which easily spalls during wear.¹³ Inward migration is not to be expected because of the high carbon and chromium concentration.^{14,15}

FIG. 2. TEM picture of coalesced bubbles after implantation of neon with 3×10^{17} ions/ cm^2 .

TABLE I. Measured wear rates after implantation into laser-melted steel.

Implanted ions	Dose ($\times 10^{17}$)	Temperature ($^\circ\text{C}$)	Wear rate ($\times 10^{-15} \text{ m}^3/\text{m}$)	Running in wear ($\times 10^{-12} \text{ m}^3/\text{m}$)
...	0		1.0	4.4
N^+	1	90	0.1	7.0
N^+	3	90	1.8	7.8
N^+	3	150	0.5	11.0
Ne^+	0.3	90	0.7	3.8
Ne^+	1	90	0.7	8.9
Ne^+	3	90	0.2	4.7

FIG. 3. Diffraction ring pattern after implantation of 3×10^{17} 50 keV N^+ ions/cm² at 90 °C. Rings are indexed as 1 = $(11\bar{2}0)$ ϵ -Fe₂(C,N)_{1-x}; 2 = $(0002)_\epsilon$; 3 = $(11\bar{2}1)_\epsilon$; 4 = $(11\bar{2}2)_\epsilon$; 5 = $(10\bar{1}3)_\epsilon$ (superspot) and $(30\bar{3}0)_\epsilon$; 6 = $(11\bar{2}3)_\epsilon$; 7 = $(30\bar{3}2)_\epsilon$; 8 = $(224\bar{1})_\epsilon$.

TEM investigations of the steel implanted at the same dose but at a higher temperature of 150 °C reveal ϵ -Fe₂(C,N)_{1-x} and the nitrogen-poor α'' -Fe₁₆N₂ nitrides. Apparently diffusion of nitrogen occurs to a larger depth. Further, a higher carbon concentration in the carbonitrides is present, by which the hardness is smoothened and no hard brittle layer is present. Implantation of 1×10^{17} N^+ ions/cm² at 90 °C results in a'-martensite diffraction rings. The martensite results in hardening and compression. The wear measurements already showed that these implantation conditions result in a good wear-resistant surface.

In conclusion, neon implantations contribute to compressive strengthening by forming bubbles. This always results in a strong improvement of the wear performance of the laser-melted steel. Nitrogen implantations result in the formation of hard nitrides. Although there will be a volume expansion, the wear performance might be even worse. This is due to a hard brittle spalling layer of ϵ -Fe₂(C,N) carbonitrides. Only for selected dose and temperature is there a significant improvement.

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