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Friction reduction and zero wear for 52100 bearing steel by high-dose implantation of carbon

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Ion implantation of carbon in the AISI 52100 bearing steel yields a distinct reduction in friction and wear. This improvement is strongly dependent on the implanted fluence. The coefficient of friction decreases from 0.6 to 0.2 for doses $> 1 \times 10^{18} \text{ cm}^{-2}$ (energy 100 keV) and a wear reduction to nearly "zero wear" was obtainable even under severe wear conditions. The counterpart (unimplanted AISI 52100 steel ball) shows a similar behavior, which demonstrates that the tribological system is totally changed. Mössbauer spectroscopy and x-ray diffraction revealed that hexagonal ϵ -carbide is formed on implantation. On the other hand, Rutherford backscattering spectrometry shows that for high doses a large fraction of the implanted carbon is not contained in this carbide.

With nitrogen implantation, a distinct reduction in wear occurs especially for high chromium steel, but without any decrease of the coefficient of friction.¹ On the other hand, the wear behavior of 52100 bearing steel (low alloyed steel) could not be influenced by implantation of nitrogen.^{1,2} Singer *et al.*³ reported a 60% reduction of the friction by Ti implantations at high temperature and in a CO atmosphere. Similar improvements are published by Follstaedt *et al.*⁴ They implanted Ti and C into AISI 304 stainless steel, using transmission electron spectroscopy (TEM) for microstructural analysis.

We investigated the tribological behavior of the 52100 bearing steel implanted with carbon. We have applied Mössbauer spectroscopy and Rutherford backscattering spectrometry (RBS) to characterize the implanted layer. The doses were varied from $4 \times 10^{17} \text{ cm}^{-2}$ to $30 \times 10^{17} \text{ cm}^{-2}$ at an energy of 100 keV. The tribological tests were carried out on an oscillating ball-on-disk tester¹ under severe conditions. The frequency of the ball was 7 Hz at a track length of only 2 mm, and the applied load amounted to 2.1 N. The ball, 4.76 mm in diameter, was also made of the 52100 bearing steel.

The implantations were performed on a Varian 350D implanter and the ion current density was $17 \mu\text{A}/\text{cm}^2$. The disks were mounted on a water-cooled sample holder, so the temperature on the surface never exceeded 100 °C.

The ⁵⁷Fe Mössbauer spectra were obtained with a constant accelerator spectrometer measuring the conversion

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electrons with a 96% He-4% CH₄ gas detector at room temperature. The spectra were fitted with a computer simulation, including isomer shift, quadrupole splitting and magnetic hyperfine field. RBS with 2 MeV He⁺ ions and scattering angle 170° was used to measure Fe and C concentration profiles. The carbon concentration profiles have been calculated from the Fe signal using the missing element option of the computer program SQUEAKIE.⁵

The friction behavior of the steel, implanted with a dose

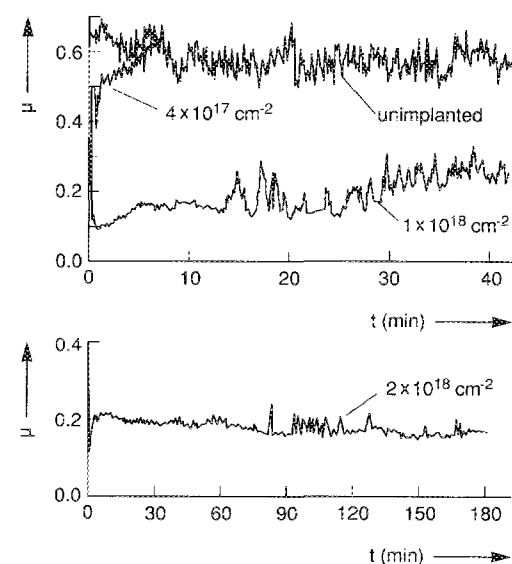


FIG. 1. Friction behavior of the unimplanted and carbon-implanted AISI 52100 steel. Implantation energy 100 keV, fluence $4 \times 10^{17} \text{ cm}^{-2}$, $1 \times 10^{18} \text{ cm}^{-2}$, and $2 \times 10^{18} \text{ cm}^{-2}$.

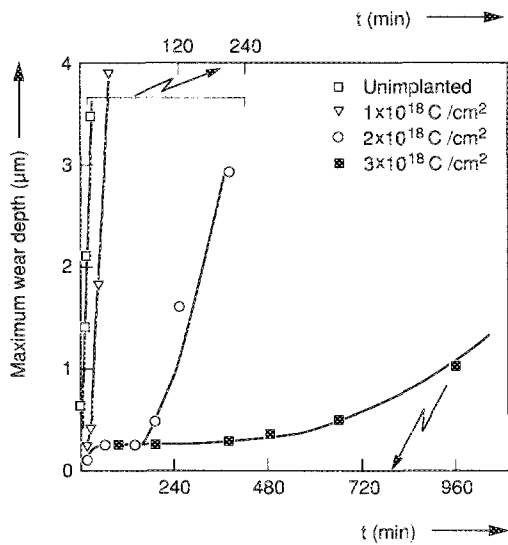


FIG. 2. Wear behavior of the unimplanted and carbon-implanted AISI 52100 steel. Implantation energy 100 keV. For the $3 \times 10^{18} \text{ cm}^{-2}$ disk the lower horizontal axis is used. For the other fluences the upper axis is used.

$4 \times 10^{17} \text{ cm}^{-2}$ shows only a slight effect (Fig. 1). A reduction of the friction coefficient by 0.1 occurs for a very short time (about 4 min). A more marked effect can be seen when the disk is implanted with a dose of $1 \times 10^{18} \text{ cm}^{-2}$ (Fig. 1). Further improvement is obvious for a dose of $2 \times 10^{18} \text{ cm}^{-2}$ (Fig. 1). At a dose of $3 \times 10^{18} \text{ cm}^{-2}$, the reduced friction is observed for more than 6 h (1.5×10^5 cycles).

The corresponding wear results are shown in Fig. 2. The best results are obtained for the highest dose of $3 \times 10^{18} \text{ cm}^{-2}$. Normal wear occurs only for the very first cycles up to a depth of $0.15 \mu\text{m}$ which corresponds to the projected range of the carbon ions measured by secondary-ion mass spectrometry (SIMS) analysis. When this depth is reached, a very low wear rate is obtained with nearly no measurable wear for about 6 h. The ball shows also no wear for this period of time, no facet is formed, only a slight material transfer from the disk to the ball can be seen as black particles.

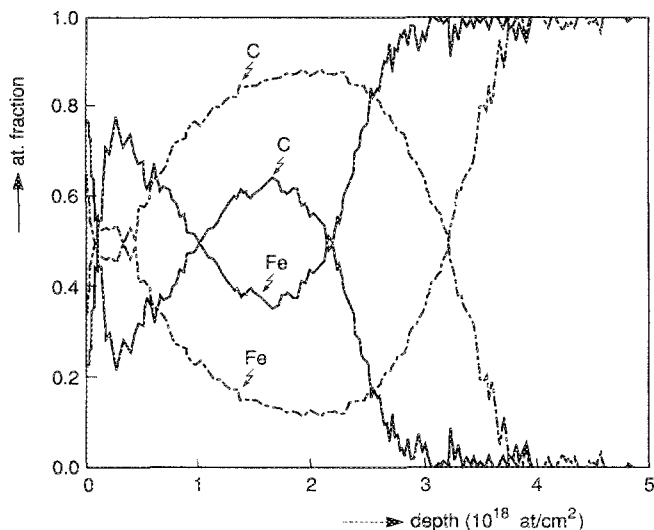


FIG. 3. Fe and C concentration profiles obtained from RBS of carbon-implanted AISI 52100 steel. Implantation energy 100 keV. For the solid curve the implantation influence is $1 \times 10^{18} \text{ cm}^{-2}$. For the dashed-dotted curve it is $2.8 \times 10^{18} \text{ cm}^{-2}$.

Preliminary investigations with 50 keV, $1 \times 10^{18} \text{ cm}^{-2}$ implanted steel revealed that it is possible to get rid of the relatively high wear (normal wear) for the first cycles, due to the fact that the carbon distribution is closer to the surface.

In Fig. 3 the Fe and C concentration profiles resulting from analysis of the RBS spectra are shown for disks implanted with 1×10^{18} and $2.8 \times 10^{18} \text{ C/cm}^2$ at an energy of 100 keV. Since the density of the implanted layer is not known, the depth scale is expressed in at/cm^2 . The high carbon fraction at the surface is an artifact of the computer program. The total amount of carbon found was $(1.1 + 0.1) \times 10^{18}$ and $(2.5 + 0.1) \times 10^{18} \text{ C/cm}^2$, respectively.

In Fig. 4, the Mössbauer spectra of the unimplanted and implanted bearing steel AISI 51200 are shown. For the implanted disks two extra sextets with respect to the unimplanted disk are observed. These subspectra can be assigned to crystalline ϵ -carbide Fe_{3-x}C , with $0 < x < 1$. Similar to ϵ -nitride two iron sites are possible, coordinated with two or three C atoms.⁶ For Fe_3C all Fe atoms are surrounded by two C atoms, while for Fe_2C all the atoms are surrounded by three C atoms. Assuming equal Mössbauer fractions for both sites, the relative intensity of them gives the composition of the carbide. With implantation dose, the relative carbon fraction increases, resulting in $\text{Fe}_{2.2}\text{C}$ for $2 \times 10^{18} \text{ cm}^{-2}$. Assuming equal Mössbauer fractions for Fe (α and γ) and the ϵ -carbide, the relative amount of those phases in the layer observed with Mössbauer spectroscopy can be found from the intensity of the subspectra. The result is shown in Fig. 5.

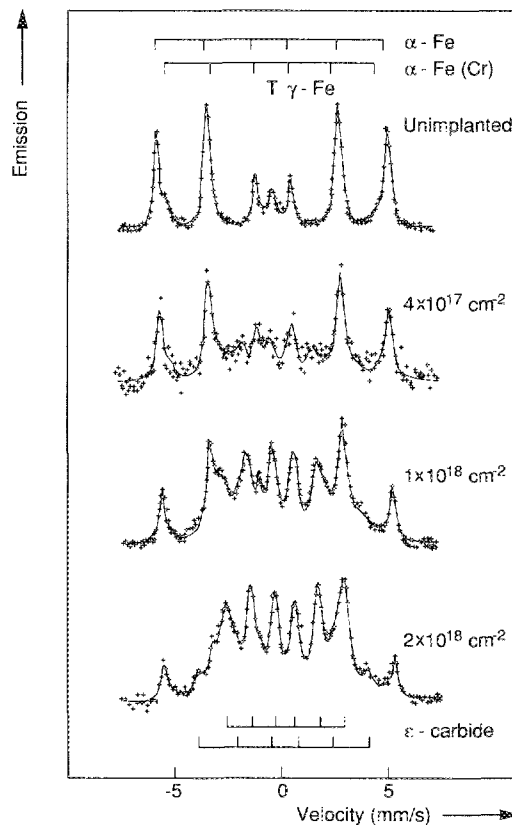


FIG. 4. Mössbauer spectra of the unimplanted and carbon-implanted AISI 52100 steel. Implantation energy 100 keV.

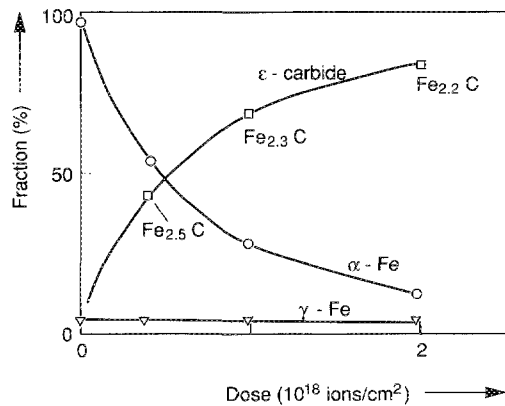


FIG. 5. Relative fractions of the α -Fe, γ -Fe, and ϵ -carbide as a function of the implantation fluence, determined from the area of the Mössbauer sub-spectra.

The relative amount of ϵ -carbide is increasing with implantation dose. Conversion electron Mössbauer spectroscopy (CEMS) probes a depth of 9×10^{17} Fe at/cm² in Fe (for α -Fe about 1000 Å). For the K-conversion electrons of iron in nearly 100% pure carbon the depth is 4.2×10^{18} C at/cm². This means that we probe a depth of 1.1×10^{18} at/cm² for the 1×10^{18} C/cm² implant. For the 2.8×10^{18} C/cm² implant this depth is 2×10^{18} at/cm². Therefore, the thickness of the layer observed with CEMS increases with the implantation dose. In this layer CEMS, able to distinguish the various iron compounds formed upon implantation, shows the presence of a carbide of composition $\text{Fe}_{2.2}\text{C}$. On the other hand, the results in Fig. 3 show an atomic carbon concentration far in excess of the 30% of this carbide. Therefore, it can be concluded that a large fraction of the implanted carbon is present as pure carbon and is not contained in a carbide. This conclusion is confirmed by preliminary results of Auger

electron spectroscopy (AES). The wear reduction, which increases with the dose, is probably a result of the ϵ -carbide phase along with the carbon layer being formed. Our conclusion of finding crystalline ϵ -carbide in a steel with low chromium concentration is in agreement with the results of Föllstaedt *et al.*⁷ Analysis of our samples by x-ray diffraction confirmed the presence of crystalline ϵ -carbide.

After annealing the implanted steel disk (dose 2×10^{18} cm⁻²) at 500 °C the ϵ -carbide is transformed to the Hagg-carbide Fe_7C_5 according to Mössbauer and x-ray diffraction results. This is accompanied by a drastic change of the tribological behavior. A high wear of the ball occurs and the wear of the disk (adjusted to the contact pressure) increased by a factor of 10. This suggests that the ϵ -carbide phase which disappeared on annealing contributes to the impressive reduction in friction and wear. Further investigations by depth selective Mössbauer spectroscopy are in progress.

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