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Enhanced wear resistance of production tools and steel samples by implantation of nitrogen and carbon ions

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Abstract

In recent years ion implantation has become a feasible technique for obtaining improved wear resistance of production tools. However, basic knowledge of how and in which cases ion implantation is working at its best is still needed. The present paper discusses structural and tribological investigations of carbon and nitrogen implanted steels. The nitrogen data were obtained mainly from field tests and the investigation of carbon implantations took place mainly in the laboratory. A study was made of how the tribological behaviour of implanted steels changes with different implantation parameters. The tribological laboratory investigations were carried out using pin-on-disc equipment under controlled test conditions, and deal with high dose carbon implantation (approximately $(1-2)\times10^{18}$ ions cm^{-2}). The wear resistance of steels was enhanced dramatically, by up to several orders of magnitude. The field test results cover a broad range of ion implanted production tools, which showed a marked improvement in wear resistance. Nitrogen implanted tools are also compared with carbon and titanium implanted tools.

1. Introduction

The use of ion implantation to improve tool performance in real production type environments has increased considerably in recent years. A major proportion of this type of implantation is still performed with nitrogen ions, but with a tendency towards more diversified implantations. The development of new very versatile high current ion implanters has made it commercially viable to implant ions other than nitrogen in production tools and it has thereby become possible to a greater degree to tailor the implantation to a specific steel and wear situation. Research and development work on nitrogen implantation has been performed by many investigators over many years. Although more scientific investigations of nitrogen implantation are needed to understand it fully, there is also a growing demand for research and development work on ion implantation of other ions with high potential commercial uses.

This presentation is divided into two parts. The first is mainly a basic laboratory study of the effects of high dose carbon implantations in steel samples that have shown great potential, and the second mainly deals with case studies of nitrogen (but also carbon and titanium) implantation of production tools.

2. Experimental details (carbon)

Both unhardened and hardened (approximately 59 HRC) Sverker 21 (werkst. nr. 1.2379, AISI D2) cold working tool steel samples (discs) were implanted with carbon ions. The discs were mechanically polished with a 1 μm grade diamond paste and had a surface roughness of about 5 nm before implantation. After polishing, the samples were degreased and cleaned in ultrasonic baths with trichloride, acetone and ethanol.

The discs were implanted with C^+ in a Danfysik 1090-200 high current accelerator [1] especially designed for the treatment of tools. The C^+ implantation energies were 75 and 150 keV and the doses were 0.5×10^{18} C^+ cm^{-2}, 1.0×10^{18} C^+ cm^{-2}, and 2.0×10^{18} C^+ cm^{-2}. In all cases the sample temperature during implantation was less than 200 °C and the beam current density was about 6–9 μA cm^{-2}. The mass separated beam was magnetically scanned over the samples and the chamber pressure was kept below about 2.5×10^{-3} Pa.

2.1. *Tribological test conditions*

The discs were tested in a unidirectional ball-on-disc tribometer under unlubricated conditions. During the tests a steel ball (SKF 3 approximately equivalent to AISI 52100, 5 mm diameter) was made to slide (0.1 m s^{-1}) with a specified load (2.4 N) against the rotating discs (wear track diameter 20 mm), and the coefficient of friction was measured continuously as a function of sliding distance (total sliding distance 1000 m). The tests were performed in a controlled environment (atmosphere with 40% ±5% relative humidity, and a temperature of 25–28 °C). Details of the tests can be seen in ref. 2.

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In addition to the coefficients of friction, the area of the wear scar of the discs was measured by stylus profilometry at four positions separated by 90° along the circular wear track. Furthermore, the wear volume of the counterpart, the ball, was measured by microscopy inspection.

2.2. Results from laboratory tests

For carbon implantation into steels, where the sputtering rate is relatively low, using the computer code "Profile Code" [3] it is possible to predict the resulting C^+ high dose concentration depth profile with remarkable precision. As an example, the Profile Code predictions of 1.0×10^{18} C^+ cm^{-2}, 100 keV and 2.8×10^{18} C^+ cm^{-2}, 100 keV implantations are shown in Fig. 1 and compared with experimental data obtained from Rutherford backscattering spectrometry (RBS) measurements [4]. The comparison shows that for the high dose carbon implantations investigated, the Profile Code data agree with experimental data to within 20% for the maximum carbon concentration, straggling and mean projected range.

Figure 2 shows the Profile Code concentration depth profiles for the energies and doses used in the present investigation. The carbon concentration is given both as atoms per cubic centimetre and as a relative concentration (in atomic per cent) calculated from these data. Carbon peak concentrations are also indicated in the figure and it is observed that at the lowest energy and highest dose the maximum carbon concentration is extremely high, between 80% and 90%. These high carbon concentrations were confirmed both by RBS and Auger depth profiling analysis.

The results of pin-on-disc wear measurements have

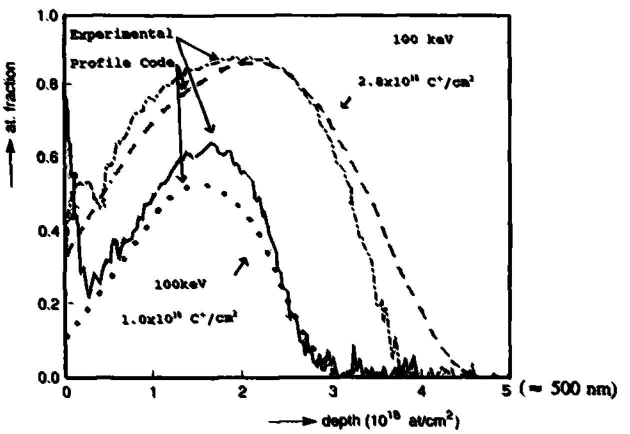

Fig. 1. A comparison between experimental data and computer calculated concentration depth profiles of 100 keV, 1.0×10^{18} C^+ cm^{-2} and 100 keV, 2.8×10^{18} C^+ cm^{-2} implantations into steel. Experimental data obtained by Kobs *et al.* [4] (RBS). Computer calculation from the Profile Code simulation program [3] where the atomic fraction of carbon was calculated from the atomic densities of the implanted carbon.

been discussed in detail in a previous publication [2]. However, the wear result after 1000 m sliding distance of the carbon implanted steel are summarized in Table 1, where the relative improvements in both volumetric ball wear and disc wear are shown together with the relative reduction in the coefficient of friction. For the hardened as well as for the unhardened steel, a considerable reduction in both wear and coefficient of friction is obtained by the implantations. The largest improvement is achieved at the highest doses, and relative reductions in ball wear of several orders of magnitude are obtained. For the disc wear, the data for the unhardened steels reveal an impressive reduction of up to two orders of magnitude. However, the improvement in wear resistance of the hardened Sverker 21 discs (up to a factor of 3) is not as pronounced as for the unhardened discs. It should be noted that both carbon implantation and steel hardening (prior to implantation) are observed to change the wear mode from adhesive to mildly abrasive, and in all cases the implantations reduce the scatter in the measured wear profiles significantly.

A comparison of the wear results in Table 1 and the carbon concentrations shown in Fig. 2 reveals that the wear resistance seems to be governed by the carbon concentration, *i.e.* increasing the dose and thereby the carbon concentration enhances the wear resistance. Furthermore, it is observed that the low energy, 75 keV, may be advantageous compared with the 150 keV implantation. This may be explained by the observation that for a given implanted dose a higher peak carbon concentration is obtained with a lower ion energy (see Fig. 2).

The carbon layers have been shown in a previous study (using transmission electron microscopy) to be partly amorphous and nanocrystalline [2]. In a study by Kobs *et al.* [4], where 100 keV high dose carbon implantation into AISI 52100 steel yielded up to 80%–90% carbon in the surface layers, traces of nanocrystalline graphite was observed by X-ray diffraction analysis. Thus, the excellent tribological properties of high dose carbon implanted steel may result from a combination of a hard wear resistant amorphous carbon layer and self lubrication, where the low friction results from the amorphous carbon [5] and/or the graphite [6]. Figure 3 shows the coefficients of friction for carbon implanted Sverker 21 steel both unhardened and hardened after 1000 m sliding distance. It is interesting to observe that for both energies at the highest dose, and for the low energy at the lower doses where the carbon is expected to be the dominating element near the surface, the coefficient of friction is about 0.4 for the unhardened and about 0.25 for the hardened steel. This could be explained by the fact that the hardened steel is approximately 2.5 times harder than the unhardened steel. Assuming the friction of the system is governed

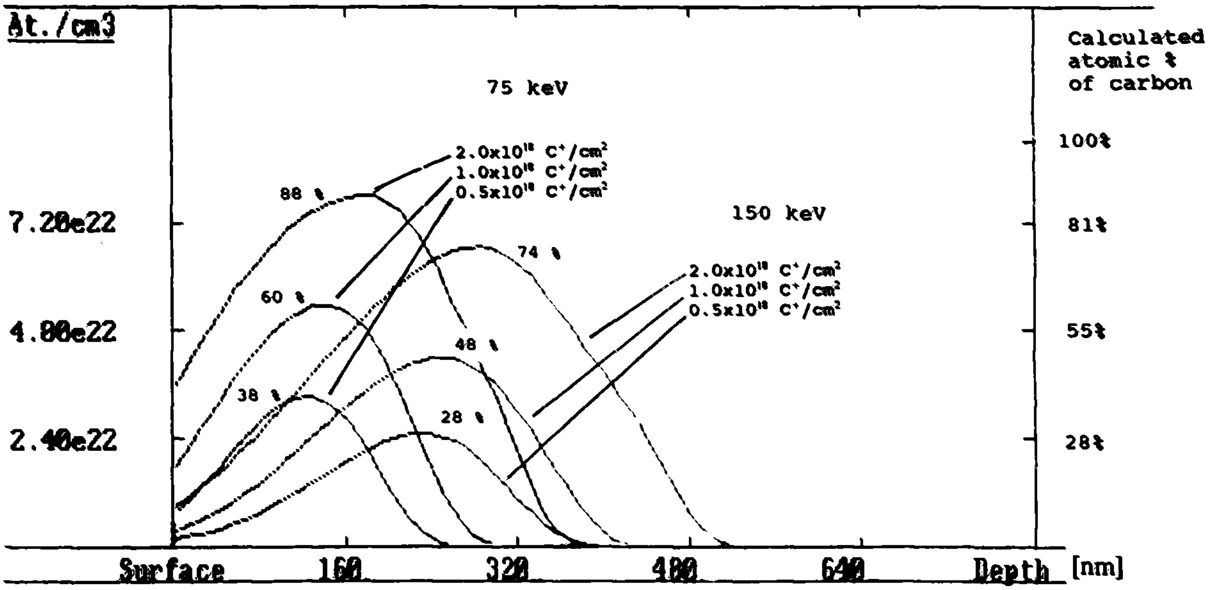

Fig. 2. Profile Code calculations of 75 and 150 keV high dose carbon implantation into steel at doses of 0.5×10^{18} C^+ cm^{-2}, 1.0×10^{18} C^+ cm^{-2}, and 2.0×10^{18} C^+ cm^{-2}. Carbon peak concentrations (in atomic per cent) are added to the curves.

TABLE 1. Relative improvement in wear resistance of high dose C^+ implanted Sverker 21 tool steel

Ion energy (keV)	Dose (10^{18} cm^{-2})	Relative improvement in ball wear	Relative improvement in disc wear[a]	Relative improvement in scatter in disc wear	Friction coefficient (% of unimplanted value)
Unhardened					
75	0.5	9.2×10^{0}	30	2×10^{1}	60
	1.0	4.7×10^{1}	22	1×10^{2}	60
	2.0	1.1×10^{2}	314	1×10^{2}	55
150	0.5	1.4×10^{0}	4.5[b]	5×10^{0}	80
	1.0	3.2×10^{1}	27	3×10^{2}	70
	2.0	1.5×10^{2}	18	1×10^{3}	50
Hardened					
75	0.5	2.5×10^{2}	0.39	2×10^{1}	35
	1.0	7.5×10^{3}	2.0	3×10^{1}	30
	2.0	7.5×10^{3}	3.2	6×10^{1}	30
150	0.5	7.1×10^{0}	0.16	1×10^{1}	70
	1.0	1.6×10^{1}	0.16	3×10^{0}	60
	2.0	9.6×10^{2}	0.7	4×10^{1}	30

[a]Indicates that the unimplanted and unhardened disc exhibits adhesive wear (material added to the disc). For implanted and/or hardened discs, adhesive wear is only seen in cases marked [b].

completely by a solid lubricating carbon layer on the steel substrate, the coefficient of friction μ can be related to the shear strength S of the lubricating layer and the hardness of the substrate H by the following relation [6, 7]

$$\mu = S/H + \text{constant}$$

According to this the coefficient of friction is lowered by the increased substrate hardness, as observed for the present system. This indicates that the system may to some extent act as a solid lubricating layer.

Furthermore, when comparing the volumetric wear of the ball for carbon implanted hardened Sverker 21 with the coefficients of friction from Fig. 3 (see Fig. 4), it is found that the wear of the ball seems to be strongly related to the coefficient of friction. Thus, the low wear of the ball may to some extent result from the low friction exhibited by a self lubricating system. However, it is not possible from the present results to draw any definite conclusions on whether the low wear of both the ball and the implanted disc results from the low friction itself, or whether the low wear rate results from

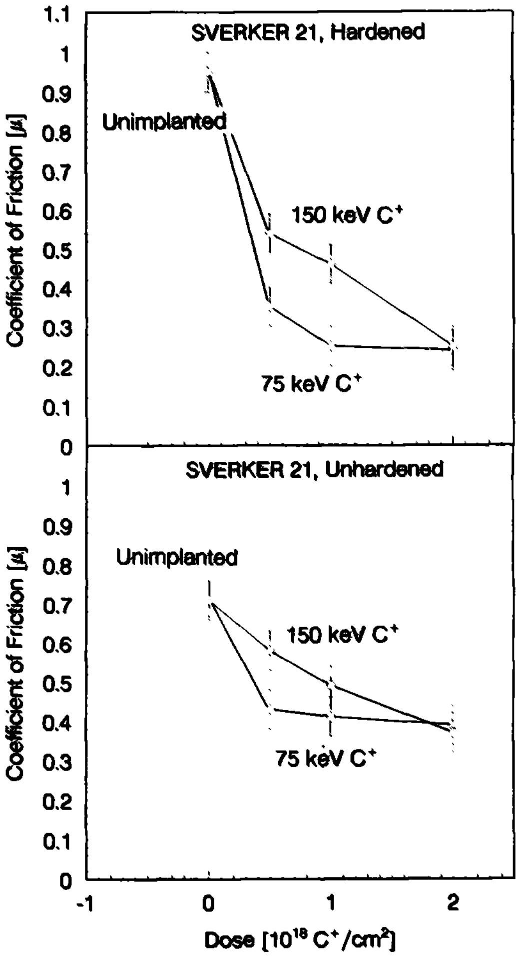

Fig. 3. Coefficient of friction for high dose C^+ implanted Sverker 21 steel. The measurements were taken on a pin-on-disc tribometer, where an AISI 52100 steel ball was made to slide against the flat Sverker 21 sample.

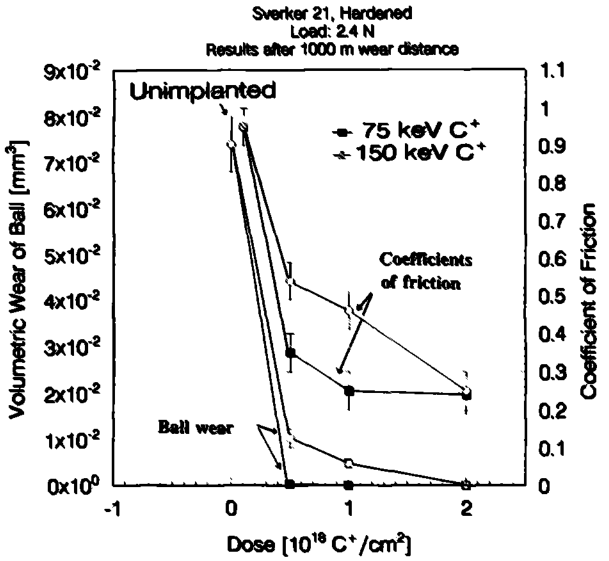

Fig. 4. The volumetric wear of the unimplanted AISI 52100 steel ball after 1000 m sliding distance against C^+ implanted and hardened Sverker 21 steel. The wear is shown as a function of carbon ion dose and energy. The corresponding coefficients of friction are also shown.

a hard wear resistant carbon layer which in addition to a high wear resistance exhibits low friction.

3. Case studies, N^+ implantation and others

The following case studies are examples taken from Danish industry. The implantations are performed on a commercial basis at the Danish Technological Institute in Aarhus, Denmark with the high current implanter model Danfysik 1090–200 developed by Danfysik A/S.

Although there is substantial knowledge of implantation of tools with nitrogen ions, a new tool from a customer still often presents a new and non-routine situation, and optimal implantation parameters have to be found. The first implantations for a new customer are therefore often performed as trial implantations, and the experience gained from the trials often results in routine work on a commercial basis.

Example 1

An example from routine production is blanking dies made of either AISI L6 or AISI H11 type steel hardened to HRC 57$\pm$1. The steels are nitrogen ion implanted at temperatures below 200 °C with 180 keV N_2^+ at a dose of 2×10^{17} N_2^+ cm^{-2}. The tools are working tin can material. Normally, the tools last on average around 3 weeks. After nitrogen implantation the lifetime is increased to more than half a year.

Example 2

V-shaped knives for cutting dried leaf material (with sand particles incorporated) can be improved substantially by ion implantation. The knives are made of spring steel hardened to HRC 59. The implanted knives have been tested against normal knives and TiN (physically vapour deposited) coated knives. Both standard nitrogen implantation and high dose carbon implantation have been tested. The knives perform differently on different testing machines. The improvements are therefore only comparable for tests performed on the same machine.

On machine I a comparison was made between standard, carbon implanted (100 keV C^+, 2×10^{18} C^+ cm^{-2}, implantation temperature below 200 °C) and TiN coated knives. On machine II a comparison was made between standard, nitrogen implanted (95 keV N^+, 4×10^{17} N^+ cm^{-2}, implantation temperature below 200 °C) and TiN coated knives. The knives were only implanted on one side of the cutting edge. After resharpening from the other side it was possible to maintain very good performance of the implanted knives. The results were as follows:

Machine I	
Carbon, new knives	4.0 times normal lifetime
Carbon, resharpened knives	4.0 times normal lifetime
TiN, new knives	2.5 times normal lifetime
TiN, resharpened knives	0.5 times normal lifetime
Machine II	
Nitrogen, new knives	3.5 times normal lifetime
Nitrogen, resharpened knives	4.0 times normal lifetime
TiN, new knives	1.0 times normal lifetime
TiN, resharpened knives	0.5 times normal lifetime

Example 3

Plastics can give severe abrasive wear on both nozzles, dies and moulds. In this example it was very important that the inlet nozzles retained their dimensions and sharp edges as long as possible. The nozzles were made of a Werkst. nr. 1.2363 steel, hardened to HRC 57. In the first test three sets of nozzles were implanted with nitrogen (100 keV N^+, 4×10^{17} N^+ cm^{-2}), carbon (100 keV C^+, 2×10^{18} C^+ cm^{-2}) and titanium (190 Ti^+, 4×10^{17} Ti^+ cm^{-2}) respectively at temperatures below 250 °C. The implanted nozzles all performed equally well and within the test period showed practically no wear. In the next phase a whole set of nozzles was implanted with nitrogen under the same conditions given above. For economical reasons this is the most advantageous of the three ions tested when the results are similar. The nozzles were removed from production for close inspection after having produced twice the normal amount of material. The investigation could not reveal any wear on the critical surfaces.

4. Conclusions

The present results indicate that substantial improvements in wear resistance and reduction in the coefficient of friction can be obtained by high dose carbon implantation. The improvements obtained in the laboratory tests of carbon implanted samples are in general substantially better than typical results obtained by nitrogen implantations. Similar tests of nitrogen implanted and hardened Sverker 21 steels reveal only marginal reductions in the coefficient of friction and a typical reduction in wear of the ball by around 30%. In contrast to this, the carbon implantations seem to give improvements by orders of magnitude. This large difference has not yet been seen in production tools, and it has been shown that nitrogen implantation has proved to be a very good and commercially viable process for industrial use in many situations.

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References

1 B. R. Nielsen, P. Abrahamsen and S. Eriksen, *Mater. Sci. Eng., A116* (1989) 193.
2 C. A. Straede, J. R. Poulsen, B. M. Lund and G. Sorensen, *Mater. Sci. Eng.*, in the press.
3 A. J. Armini and S. N. Bunker, *Mater. Sci. Eng., A115* (1989) 67.
4 K. Kobs, H. Dimigen, C. J. M. Dinissen, F. Gerritsen, J. Politiek, R. Öechsner, A. Kluge and H. Ryssel, *Nucl. Instrum. Methods*, (1991) in the press.
5 J. C. Angus, P. Koidl and S. Domitz, in J. Mort and F. Jansen (eds.), *Plasma Deposited Thin Films*, CRC Press, Boca Raton, FL, 1986.
6 B. Bhushan and B. K. Gupta, *Handbook of Tribology: Materials, Coatings, and Treatments*, McGraw-Hill, New York, 1991.
7 F. P. Bowden and D. Tabor, *Friction and Lubrication of Solids*, Part II, Clarendon Press, Oxford, 1964.