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EXCIMER LASER PROCESSING OF TOOL STEEL: TRIBOLOGICAL EFFECTS OF MULTIPLE PULSE PROCESSING AND TI ALLOYING

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ABSTRACT

Excimer lasers were used to modify the surface of AISI type A-7 tool steel, a high C, high V, high Cr material used in many cutting applications. Multiple pulses of laser radiation at 248 nm were used to alter the composition of the surface alloy. Hardness and modulus were not significantly affected by the treatment, but friction in dry sliding against an alumina pin was reduced. The reduction was small but persistent for multiply melted and resolidified surfaces. These surfaces showed a marked increase in the surface Cr concentration. Greater reductions in friction were obtained from a Ti rich surface layer formed by laser mixing an evaporated Ti layer into the material. The friction coefficient of the Ti alloyed surface deteriorated after approximately 1,000 cycles, indicating wear-through of the modified surface. The observed properties will be discussed in terms of the excimer laser modification process and the microstructure and composition of the resulting surfaces.

INTRODUCTION

AISI type A-7 is an air-hardening, medium-alloy, cold work tool steel used in cutting and forming dies and in applications that exploit its exceptional abrasive wear resistance. Alloying additions of C (2.3 wt %), Cr (5.25 wt %), V (4.75 wt %), and lesser amounts of Mo and W (1.1 wt % each) are present in the material. This material is hardened at a solution temperature of 830 C and tempered at 125 C for several hours to a final hardness. In its fully hardened condition, it is somewhat brittle so in many applications it is used in a tempered state.

Laser processing was approached to examine the effect of laser processing, specifically repeated melt-solidification cycles, and to examine the effect of Ti alloying of the surface. Both laser surface processing an laser Ti alloying with the rapid solidification attendant to excimer laser processing has been shown to improve tribological properties of other metal alloys and ceramics[1,2].

EXPERIMENT

Experiments were performed on steels tempered to Rockwell hardness of 64 (C-scale). Some experiments were performed on fully solution hardened materials (RC-68), but because these materials are subject to brittle failure in applications, we did not examine the tribological effects of surface alloying on these materials.

Sample materials were obtained in the fully solution hardened and in the tempered condition and were polished to a mirror finish. For alloying experiments, the polished samples were coated with 200 nm of Ti using an e-beam evaporation system with a base pressure of less than 10^{-8} Torr. The samples were then subjected to varying numbers of pulses of excimer laser radiation at 248 nm in air at a fluence of 1.1 $J\text{-}cm^{-2}$.

Pulses from an excimer laser operating at 248 nm were homogenized in order to produce uniform illumination on the surface. The homogenizer also produces a square beam spot. The sample was then moved in front of the beam so that the entire area experienced the requisite number of pulses, each overlapping to further ensure uniform coverage. Using repetition rates of 10 Hz, a beam spot size of 0.5 cm^2, and a scan speed of 1.4 $cm\text{-}sec^{-1}$, the surface is treated with 5 pulses per position at a rate of 1 $cm^2\text{-}sec^{-1}$.

Excimer laser light couples well to metallic surfaces and is absorbed very close to the surface (within 50 nm), increasing the efficiency of the process. The short pulse duration means that the energy is deposited in a time short compared to that required to diffuse the thermal energy away from the surface. The result is that the material melts rapidly to a depth of a few hundred nm and then refreezes within 50-100 nsec. At the repetition rates used in this experiment, each melting and refreezing cycle is completely independent and net heating of the sample is negligible. A further discussion of the methodology is given elsewhere[3]. The treatment conditions used in these experiments are summarized in Table I.

Sample #	Substrate Hardness (RC)	Coating	Laser Processing	Surface Hardness (GPa)
I	64	None	None	12.7 ± 2.0
II	64	None	5 pulses @ 1.1 $J\text{-}cm^{-2}$	12.5 ± 2.0
III	64	None	20 pulses @ 1.1 $J\text{-}cm^{-2}$	12.5 ± 2.0
IV	64	200 nm Ti	None	--
V	64	200 nm Ti	5 pulses @ 1.1 $J\text{-}cm^{-2}$	10.8 ± 2.0
VI	68	None	None	17.1 ± 2.0
VII	68	None	5 pulses @ 1.1 $J\text{-}cm^{-2}$	12.5 ± 2.0
VIII	68	200 nm Ti	5 pulses @ 1.1 $J\text{-}cm^{-2}$	14.8 ± 2.0

Table I. Sample descriptions, substrate hardness, and measured surface hardness of samples of A-7 tool steel used in wear and friction measurements.

Redistribution of alloying additions were examined by Auger spectroscopy and by Rutherford backscattering spectroscopy (RBS). Auger spectroscopy was used to determine surface concentrations of trace alloys before and after laser processing and RBS was used to examine the redistribution of Ti due to laser processing.

Surface hardness and modulus measurements were performed using a commercially available nanoindenter[4]. This instrument directly measures the load on a triangular pyramid diamond indenter tip as a function of displacement from the surface. Hardness is determined from the load data using the relation:

$$H = \frac{L(h)}{A(h)},$$

where L(h) is the measured load, and A(h), the area function, is the projected area of the indent as a function of the plastic depth, h. The area function is determined by an iterative process involving indents into materials of known and isotropic properties[5]. Measurements were made under constant load rate of 80 $\mu N\text{-}sec^{-1}$ to a nominal depth of 100 nm. Typically nine indents were made on each sample and the data averaged. The depth of the indents was chosen to be less than 30% of the thickness of the treated material[6]. As discussed by Doerner and Nix[7], the actual depth of the indent includes the plastic depth as well as the elastic recovery of the material as the indenter is removed. This analysis presumes a homogeneous material, rather than a layered surface structure, but the limitation on the depth of the indentation should validate this assumption.

Wear and friction measurements were made using a pin-on-disk tribometer using alumina (Al_2O_3) balls as pins. The pin surface was therefore initially spherical. The diameter of the pin was 6. 0 mm and the measurements were made without lubrication in room air at a relative humidity of 30%. A Hertzian load stress, calculated based on the properties of the ball and the substrate material, of 0.11 GPa was used for all experiments. The tribological performance was evaluated from the behavior of the friction coefficient during the tests, which were run at sliding speeds of 1.8 cm-sec^{-1} for a total of 20 minutes, and by examination of the wear tracks by scanning electron microscopy.

RESULTS

The measured hardness of the untreated and treated surfaces is given in Table I. The scatter in the data is large, a function of the inhomogeneities in the base material. Each indent is approximately 0.3 μm across, a dimension much smaller than the typical grain size in the substrate material. It is apparent that laser processing has no significant effect on the tempered material and that the result of laser processing on the fully hard material is to reduce its hardness to about that of the tempered material.

As previously observed in the multiple pulse laser processing of AISI 304 stainless steel[8], samples of A-7 steel treated with multiple pulses of laser radiation show enrichment of Cr on the surface. For samples treated with 5 pulses, the Cr Auger signal was approximately 3 times that for untreated surfaces. This enrichment comes as a result of "zone refining" of Cr, which is more soluble in the liquid than the solid state, and hence is driven to the surface by the moving melt front. With multiple melt-solidification cycles, substantial segregation can occur.

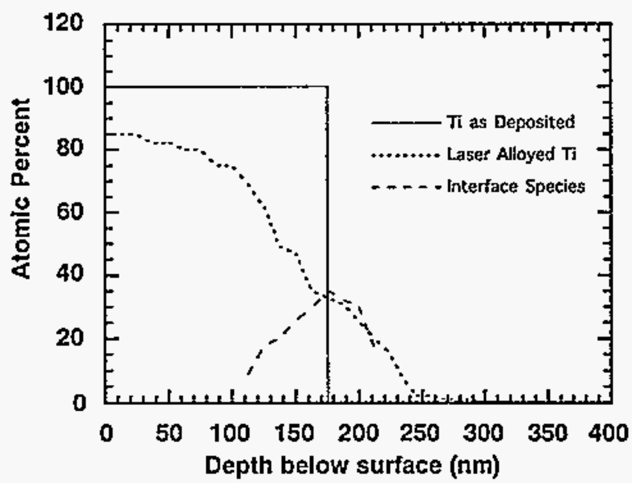

Figure 1. Reconstruction of Ti concentration in as deposited and laser alloyed surface as determined by Rutherford backscattering spectroscopy. Ti is well mixed into the surface by 5 pulses of laser radiation.

Figure 1 shows the results of RBS analysis of the mixing of Ti layers on the steel substrates. After 5 pulses, the Ti has diffused into the substrate and Fe has diffused to the surface, forming a well mixed alloy layer. For 5 melt-solidification cycles, the total time in the molten state is about 500 nsec, consistent with the time required for liquid state diffusion at rates of the order of 5×10^{-5} cm^2-sec^{-1} to a hundred nm. Also shown in the figure is the presence of a buildup at the interface of an unknown material. Both C and Cr are potential candidates, since these are known to segregate from the melt, although any of the constituents of the steel are also possible. RBS analysis was unable to identify the exact composition.

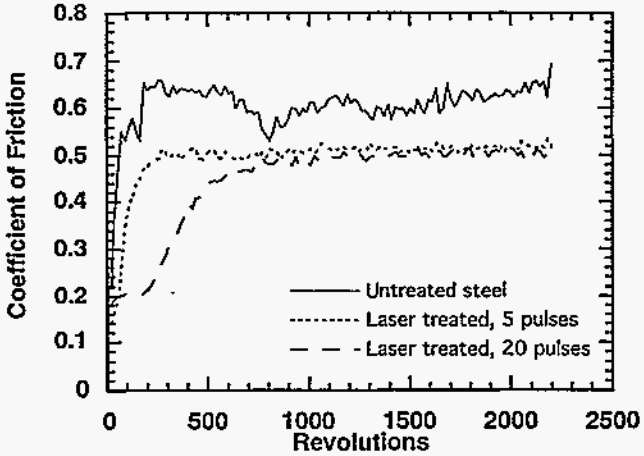

Figure 2. Friction as a function of number of revolutions for the untreated surfaces and samples treated with 5 and 20 pulses of excimer laser radiation at 1.1 J-cm-2 (samples I, II, and III). The duration of the initial low friction period is precisely related to the number of pulses.

Figure 2 shows the friction history of laser processed samples I-III tested at a Hertzian stress of 100 MPa and a sliding speed of 1.8 cm-sec^{-1} against an alumina pin. Two features are apparent. One is a transient period of very low friction at the beginning of the test. The half-life of these periods is 200 revolutions for sample II treated with 5 pulses of radiation and 800 revolutions for sample III treated with 20 pulses of radiation. The linear relation between the lifetime of the low friction transient and the number of pulses is consistent with earlier observations of the development of an oxide film on AISI 304 stainless steel. The second feature is the persistent friction reduction of approximately 20% which lasts for the duration of the test.

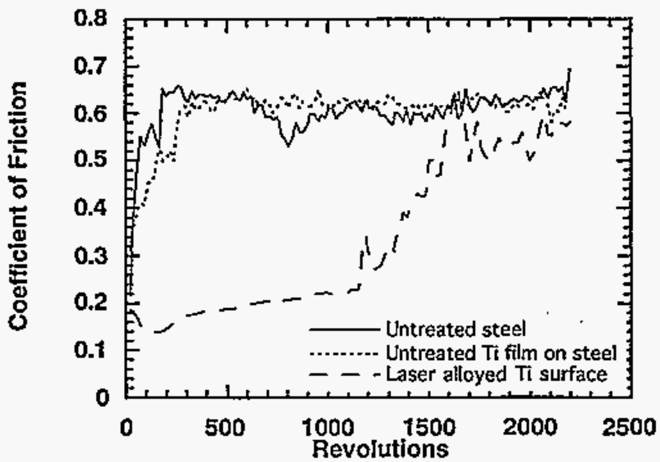

Figure 3. Friction as a function of number of revolutions for untreated, Ti coated, and laser alloyed surfaces. Ti coating by itself does not change the friction behavior, while alloying results in an extended period of low friction.

To demonstrate the effect of Ti alloying, figure 3 shows the friction history of samples I, IV, and V tested at a Hertzian stress of 100 MPa and a sliding speed of 1.8 cm-sec^{-1} against an alumina pin. The Ti coated sample has the same friction behavior as that of the untreated sample. The laser alloyed sample, on the other hand, shows very low friction for an extended period and then gradual failure approaching the friction level of the untreated surfaces.

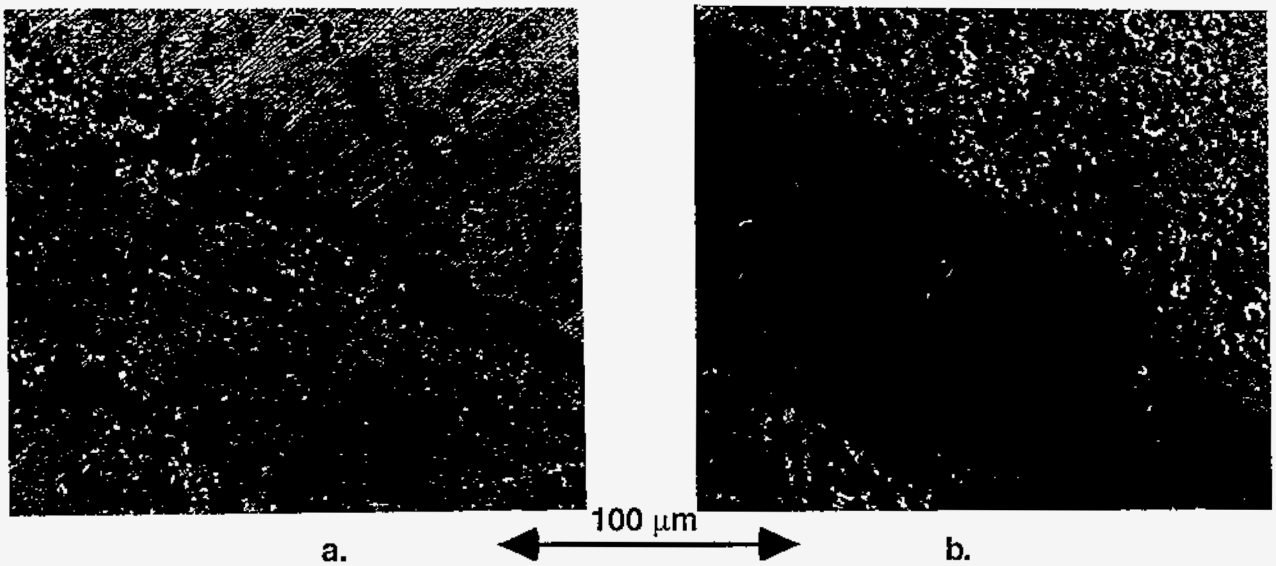

Figure 4. Scanning electron micrographs of the wear track in untreated (a) and laser surface processed with five pulses (b) cases. The adhesive wear apparent in a is entirely absent in b.

Figure 4 shows scanning electron microscope images of the wear track for the as received (a) and laser processed with 5 pulses (b) (samples I and II respectively). The unprocessed surface shows substantial buildup of wear debris typical of adhesive wear and the high friction seen in figure 2. In the laser surface processed case, there is much less damage to the surface even though it is clear from the friction history that the low friction surface layer has been consumed. Examination of the surface of the Ti alloyed sample (V) again showed evidence of substantial adhesive interactions and Ti rich wear debris. The scale of the fundamentally inhomogeneous nature of the parent material is not altered by the wear process.

DISCUSSION

The principal conclusion from this work to date is that both laser surface processing and laser Ti alloying show promise for reducing friction. Nanoindenter measurements indicate that the modified surfaces are in general not significantly different in hardness than the unprocessed surfaces. The uniformity of the hardness in all cases suggests that there is no significant change in the mechanical properties of the modified surfaces. However, Auger and RBS spectroscopy both indicate significant changes in the chemical composition of the modified surfaces. Although we have not yet determined the microstructure of the surface layer, these findings suggest that changes in tribochemistry, not changes in the mechanical properties of the surfaces, are causing the changes in the friction behavior.

For the laser processed surfaces, an initial period of low friction is almost certainly due to the formation of a low friction oxide film. The dependence of the duration of this low-friction on the number of pulses is consistent with previous work on the thickness and low friction of oxide films on AISI 304 stainless steels[9]. The thickness of this film can be increased arbitrarily by further laser processing. However, although this feature may be of use in tool applications, the relatively short lifetime is a disadvantage. Of greater interest is the long-term reduction in the friction of these materials. While not such a dramatic decrease in friction, the reduction of about 20%, coupled with the obvious change in the wear illustrated in figure 4, suggest a significant change in the nature of the tribological interaction.

Ti alloying results in a dramatic reduction in friction and may be of use in cutting tool applications. Because the tests far exceeded the lifetime of the low friction surface structure, the damage and debris seen in the wear track are not characteristic of the wear process in the low friction regime. Further work on this system is therefore required to determine the mechanism for low friction in the alloyed surfaces.

REFERENCES

1. T. R. Jervis, J-P. Hirvonen, & M. Nastasi, Lubrication Engineering **48** 141 (1992)
2. T. R. Jervis, M. Nastasi, J-P. Hirvonen, & K. M. Hubbard, J. Am. Ceram Soc. **76** 350 (1993)
3. T. R. Jervis, M. Nastasi, & J-P. Hirvonen, Mater. Res. Soc. Symp Proc. **279** 665-678 (1993)
4. J. B. Pethica, in *Ion Implantation into Metals*, V. Ashworth, W. A. Grant, and R. P. M. Proctor, Eds, (Pergamon Press, Oxford, 1982), pp 147–156.
5. W. C. Oliver & G. M. Pharr, J. Mater. Res. **7** 1564 (1992).
6. B. D. Fabes, W. C. Oliver, R. A. McKee, & F. J. Walker, J. Mater. Res. **7** 356-364 (1992).
7. M. F. Doerner & W. D. Nix, J. Mater. Res. **1** 601 (1986).
8. T. R. Jervis, D. L. Williamson, J-P. Hirvonen, & T. G. Zocco, Materials Lett. **9** 379 (1990)
9. T,. R. Jervis & J-P. Hirvonen, Wear **150** 259 (1991)