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TITLE UNLUBRICATED SLIDING PROPERTIES OF ION BEAM AND EXCIMER LASER MIXED Fe-Ti-C MULTILAYERED FILMS

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SUBMITTED TO Fall 88 MRS Meeting
Boston, MA
November 28 through December 3, 1988

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UNLUBRICATED SLIDING PROPERTIES OF ION BEAM AND EXCIMER LASER MIXED Fe-Ti-C MULTILAYERED FILMS

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ABSTRACT

Multilayered Fe-Ti-C films consisting of eleven sublayers were vacuum deposited onto an AISI 304 stainless steel substrate and subsequently mixed using either 400 keV Xe ions or an excimer laser, operating at a wavelength of 308 nm. Ion mixing was accomplished in a two step process: the multilayers were first irradiated with 1×10^{17} Xe/cm^2 at 520 C, after which half of the sample was irradiated with 5×10^{15} Xe/cm^2 at 0 C. Laser mixing was carried out at both 1.1 and 1.7 J/cm^2 with the number of pulses varied between 1 and 10. Pin-on-disc studies revealed only slight differences between the two kinds of ion beam mixed samples, whereas the dry sliding properties of laser mixed samples were strongly dependent on the total fluence used. In the optimum conditions similar friction coefficients were obtained on both kinds of samples.

INTRODUCTION

As an alternative to titanium implantation into steels the ion beam mixing of multilayer Fe-Ti structures [1] or laser mixing of a single titanium layer [2] have been studied. Ion beam mixing is generally a non-equilibrium process and entirely independent of the substrate, if multilayered structures of several different elements are used [1]. In this sense ion beam mixing is unique and different e.g. from ion implantation. Laser treatment based on the surface melting and subsequent rapid solidification can also be used with multilayered structures or to produce mixing between a single surface layer and a substrate. The quench rate of laser melting is, however, slower than that of ion implantation or ion beam mixing, which may restrict the possible available microstructures. The processing time of laser surface treatment is the shortest, and no vacuum is required, which is an advantage. Despite the differences in these three methods they provide a novel technique to tailor surface sensitive properties and are of considerable interest from a tribological point of view.

The ion beam mixing of a multilayer Fe-Ti structure results in an amorphous microstructure around the equal atomic composition [1] whereas the microstructure of laser mixed surfaces depends on the total fluence. In the case of laser melted Ti on AISI 304 stainless steel an amorphous microstructure is obtained at a low fluence [2]. A crystalline structure is, however, maintained by increased fluence. Minor constituents of a substrate as well as the substitution of titanium by iron on a one to one basis is also observed in the modified surface [2].

The tribological properties of ion or laser beam induced Ti alloys on AISI 304 stainless steel have been reported elsewhere [3-6]. Ion beam mixing of the multilayered Fe-Ti structure produces a lowered friction, although in a test of 1000 passes the modified surface layer wore partially through [3]. In that work samples with a linearly varying concentrations of the constituents in a lateral direction were employed. It was observed that the tribological properties were the best around the composition of 50 at. % of Ti. The most striking feature of the surface mechanical properties of the ion beam mixed Fe-Ti alloy was the apparently improved ductibility [4]. In general these results were parallel to those of Follstaedt et al. [7], who implanted titanium into 304 stainless steel. Improvement in the tribological properties was observed with increasing titanium fluence, though this improvement never lasted for the 1000 revolutions of the pin-on-disc test. Presumably the titanium concentration in our previous work [3] was higher than the highest one used in ref. [7] probably giving a slightly longer lasting improvement.

The surface mechanical properties of laser melted Ti alloys on AISI 304 stainless steel are very similar to those of ion beam mixed Fe-Ti or titanium implanted steel explained above [5]. Some special features, however, can be noticed [6]. The optimum tribological behavior was obtained at a certain total fluence, and this fluence was dependent on the initial thickness of the single titanium layer on the steel. Moreover, the microhardness measurements performed with a nanoindentation technique revealed that the relative hardness of the laser melted surface was lower than that of the untreated material.

The beneficial effects of carbon on the mechanical properties of the surface in titanium implanted steel are well established [7-9]. Carbon implantation into an ion beam mixed Fe-Ti alloy also significantly improves tribological behavior of AISI 304 stainless steel [10]. Consequently, the natural extension of ion and laser beam mixing of Ti with iron or steel is the mixing of ternary multilayer structures consisting of titanium, iron, and carbon. In this work we report the first results of the tribological measurements of ion or laser beam mixed Fe-Ti-C alloys on 304 stainless steel.

EXPERIMENTAL METHODS AND MEASUREMENTS

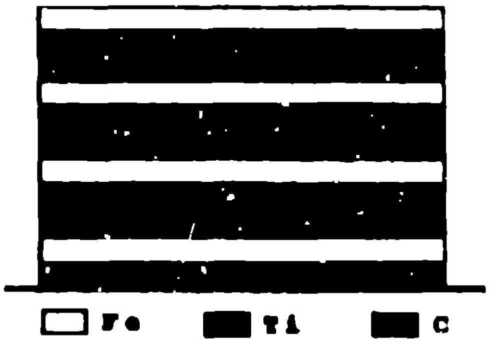

Fig.1. A schematic picture of a mutilayered structure.

The multilayer structure consisting of eleven sublayers, as illustrated in Fig. 1, was evaporated in vacuum using an e-gun thin film deposition system onto AISI 304 stainless steel substrate of commercial quality. The Vickers hardness of the substrate was 2.21 GPa. The vacuum at the beginning of the deposition was 5×10^{-7} torr and increased to 10^{-6} torr during evaporation. The entire ternary multilayer sample was deposited without breaking vacuum between the evaporation of the individual sublayers. Before deposition the substrates were mechanically polished with a diamond paste down to a grade of 0.3 µm and subsequently cleaned in an ultrasonic bath. The total thicknesses of the evaporated iron and titanium layers were determined as 550 and 600 Å by Rutherford backscattering spectroscopy (RBS). The total carbon concentration after deposition was determined as 5.1×10^{17} C/cm^2 using the nuclear reaction $^{12}C(d,p)^{13}C$ at a deuterium energy of 900 keV.

The ion beam mixing was carried out using Xe^{++} ions at an energy of 400 keV. Based on our earlier studies on ion beam mixing of iron-carbon and titanium-carbon which showed mixing at room temperature to be minimal [11,12], the sample was first bombarded at 520 C at a fluence of 1×10^{17} Xe/cm². At this temperature the mixing process has been proved to be controlled by radiation enhanced diffusion [12]. Subsequent to the high temperature mixing half of the sample was irradiated at 0 C with 1×10^{15} Xe/cm². Mixing between different sublayers was found to be complete after this treatment. Because of the high irradiation dose sputtering of the surface layer occurred, and the final thickness was 1000 Å. The corresponding carbon concentration was 3.6×10^{17} C/cm².

Laser mixing of the multilayer structure and alloying with the stainless steel substrate was performed with an excimer laser operated at 308 nm. The treatment was carried out in air at both 1.1 and 1.7 J/cm² with the number of pulses varied between 1 and 10. The repetition rate of pulses was about 1 Hz so that the sample was entirely cooled to the room temperature between the separate pulses. RBS revealed considerable mixing even after the mildest treatment, e.g. 1 pulse at 1.1 J/cm². Mixing in this case occurred presumably between individual sublayers. On the other hand at the highest total fluence deep diffusion of titanium and probably carbon into the substrate was observed. The carbon concentration in the surface layer had decreased to $4.6 - 2.7 \times 10^{17}$ C/cm² depending on the total fluence.

No detectable increase in oxygen concentration was found during either ion beam mixing or laser alloying as determined with the reaction $^{16}O(d,\alpha)^{14}N$ at a deuterium energy of 900 keV.

Tribological properties were tested utilizing a pin-on-disc apparatus. As a pin, hardened steel with a radius of curvature of 3 mm was employed. The friction force was monitored continuously during the measurements. The tests were terminated either after 1000 revolutions or after the increase of the friction indicating the penetration of the pin through the modified surface layer. The load on the pin was 31.2 g. This load produces a Hetzian stress of 440 MPa on the untreated substrate. This is comparable to a yield strength 310 MPa of the base material. The sliding speed was 22 mm/s. Friction and wear measurements were performed in room air without lubrication.

The wear tracks were investigated with the scanning electron microscope (SEM).

RESULTS AND DISCUSSION

The friction coefficients as the function of a number of revolutions are shown in Fig. 2 for both types of ion beam mixed samples. As compared to the friction coefficient of the untreated steel ($\mu = 0.9$) the steady state friction of the ion beam mixed surfaces are significantly lower. Within the error bars, which represent the oscillation of the friction during the measurements, the friction coefficients are the same in this regime. After about 2500 revolutions the friction coefficient of both samples begins to rise indicating the surface layer being worn through. The friction coefficient of the sample bombarded only at 520 C increases faster than that of the sample also bombarded at 0 C. This may indicate a slightly better wear resistance of the latter sample.

In Fig. 3 the friction coeffients of two laser mixed samples are shown. The corresponding total fluences are 1.1 and 11 J/cm^2, respectively. The general behavior of these curves differs from those shown in Fig. 2, as no steady state regime can be found. Instead the friction coefficients increase slowly but continuously. In the case of the high total fluence there is also an abrupt change after 2500 revolutions.

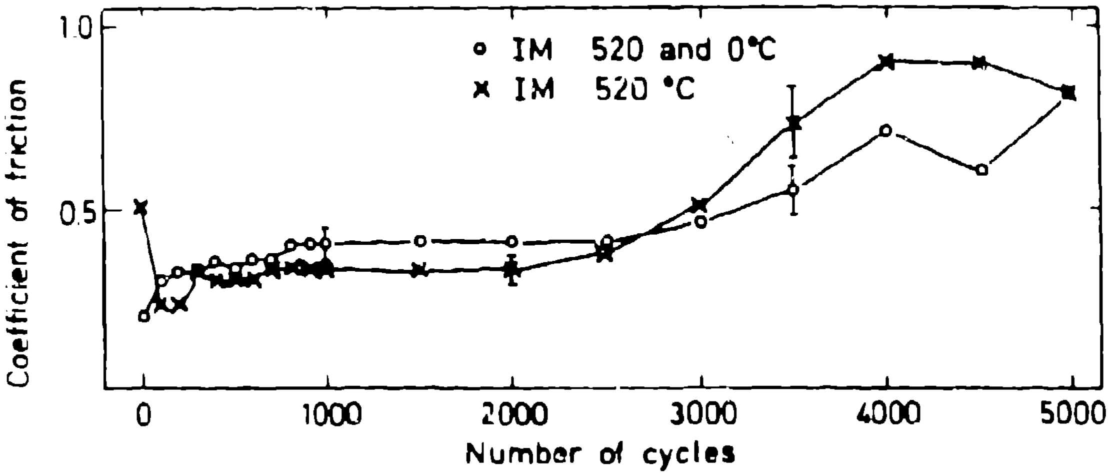

Fig. 2. The friction coefficient of the ion beam mixed samples as the function of a number of revolutions. The mixing temperatures used are shown in the figure. The friction coefficient for untreated steel was 0.9.

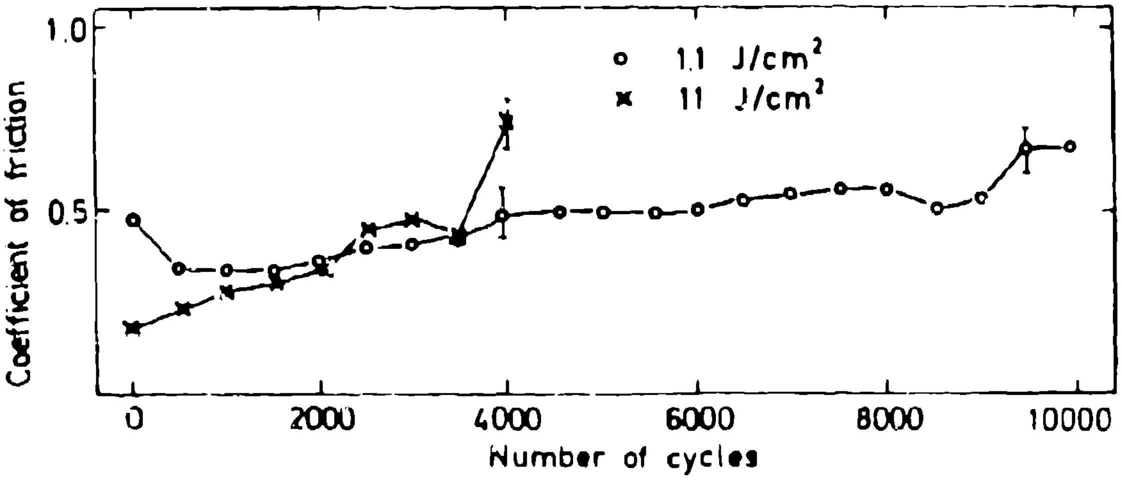

Fig. 3. The friction coefficient of two laser mixed samples as the function of a number of revolutions. The total fluences used are shown in the figure.

Scanning electron microscopy also revealed remarkable differences between the samples. The wear scar of the untreated substrate material exhibited severe damage even after only 1000 revolutions as shown in Fig. 4A. This damage can be characterized as a ductile fracture under the high surface pressure. The wear scars of the ion beam mixed samples after the identical test consist of smooth parallel grooves typical to wear tracks also in our previous studies of ion beam mixed materials [4,13]. Comparing further the sample ion beam mixed only at 520 C (Fig. 4B) to that received the postbombardment at 0 C (Fig. 4C) shows less and shallower grooves on the latter sample. This indicates that the wear resistance was slightly improved by the 0 C irradiation. This was also confirmed by SEM of the samples after the test of 5000 revolutions. Contrary to the untreated sample the wear tracks on both the ion beam mixed samples exhibited no sign of a ductile fracture after 1000 revolutions and the initiation of the fracture type damage on the surface seems to be extremely well inhibited.

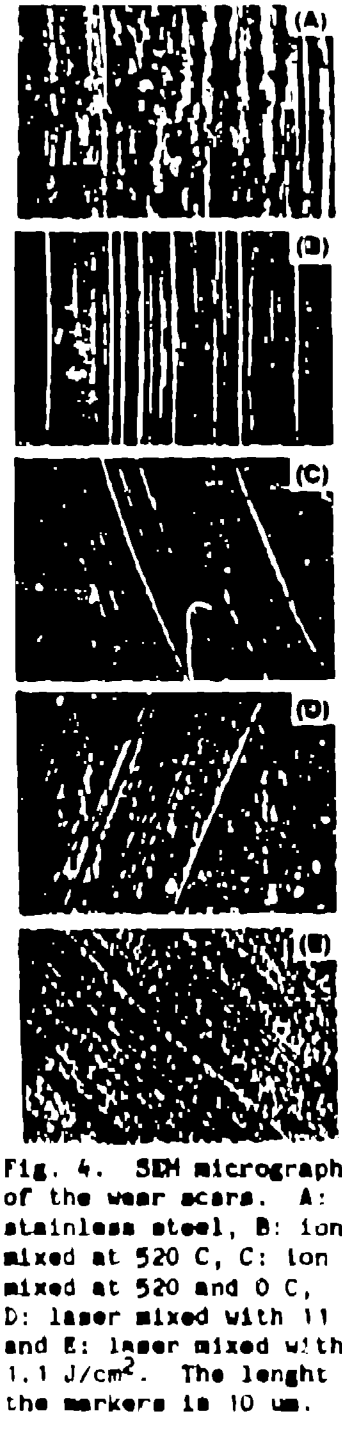

Fig. 4. SEM micrographs of the wear scars. A: 304 stainless steel, B: ion beam mixed at 520 C, C: ion beam mixed at 520 and 0 C, D: laser mixed with 11 J/cm^2, and E: laser mixed with 1.1 J/cm^2. The lenght of the markers is 10 um.

The most striking feature of the SEM micrograph of the laser mixed sample is the dispersion of precipitates or inclusions on the surface. This is especially clear in the sample mixed with the low, 1.1 J/cm^2 total fluence, Fig. 4E. The precise microstructure is not known at this moment. Because this kind of surface was not found on AISI 304 stainless steel mixed only with a single titanium layer [5] the structure must be caused by carbon in the present samples. The morphology of the wear scar on the laser mixed samples also differ from that of the ion beam mixed ones. The grooves or scratches are more finely distributed and no deep grooves can be observed in the case of low total fluence. The wear resistance of this sample was also excellent and a great deal of the original modified surface was still left in the wear track after 10000 revolutions. On the other hand, the wear scar of the sample mixed with the high total fluence, though relatively smooth, has signs of a collapse at the edge of the track. This is probably caused by the softening of the base material during the high fluence treatment.

The wear resistance of the best of laser mixed samples in this work seems to be better than that of ion beam mixed samples. This does not necessarily indicate a real difference. Because of sputtering the ion beam mixed surface layer was significantly thinner than the laser mixed surface. The impractical high dose of Xe ions required in the ion beam mixing of the present samples is, however, a fact that can be reflected in some tribological properties.

CONCLUSIONS

It is concluded that ion or laser beam mixed multilayered Fe-Ti-C structures on AISI stainless steel exhibited significantly altered tribological properties as compared to those of an untreated substrate material. Except for the sample laser mixed with the highest total fluence all samples possessed a lowered friction and increased wear resistance. In addition, all surface modified samples revealed many characteristics of their own which cannot yet be explained on the basis of the present measurements. A common feature to all was the absence of the adhesive interaction between the sliding steel pin and the modified surface and the inhibition of the fracture type damage at the surface.

ACKNOWLEDGEMENTS

Ion implantations and RBS analysis were carried out in the Los Alamos Ion Beam Materials Laboratory. This work was supported in part by the United States Department of Energy.

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