LEGIBILITY NOTICE

A major purpose of the Technical Information Center is to provide the broadest dissemination possible of information contained in DOE's Research and Development Reports to business, industry, the academic community, and federal, state and local governments.

Although a small portion of this report is not reproducible, it is being made available to expedite the availability of information on the research discussed herein.

1

CONF- 100400- -

LA-UR -90-1524

LA-UR--90-1524

DE90 011949

TITLE TRIBOLOGICAL AND MECHANICAL PROPERTIES OF Fe/Ti MULTILAYERED FILMS

AUTHOR(S) Michael Nastasi, MST-7
Juha-Pekka Hirvonen, MST-7
Thomas R. Jervis, MST-7
Soumendra Nath Basu, MST-7

SUBMITTED TO Thin Films: Stresses and Mechanical Properties II
Materials Research Society

TRIBOLOGICAL AND MECHANICAL PROPERTIES OF Fe/Ti MULTILAYERED FILMS.

M. Nastasi, J-P. Hirvonen, T.R. Jervis, and S.N. Basu

Los Alamos National Laboratory, Los Alamos, NM 87545

ABSTRACT

A 900 Å as-deposited multilayered structure of alternating Fe and Ti on hardened tool steel has been examined for tribological and mechanical properties. The multilayer in contact with a 52100 steel ball counter surface possessed a 0.16 friction coefficient and showed good wear character after 1000 cycles in pin-on-disk experiments. Nanoindentation experiments show a 20 % increase in hardness for material in the wear track. Electron microscopy shows that the as-deposited bcc-Fe plus hcp-Ti multilayer composite transforms to a nanocrystalline alloy plus bcc-Fe by solid state reaction during pin-on-disk friction and wear tests test. The low friction observed in these surface films is attributed to the formation of a hard nanocrystalline alloy and improved interlayer and film/substrate adhesion which result during solid state reaction.

INTRODUCTION

Novel alloy surface films of Fe-Ti have been shown to possess good friction and wear properties. These surface alloys have been formed by ion beam techniques such as Ti ion implantation into steels [1,2] and ion beam mixing of Fe/Ti multilayer on steel substrates [3,4], by laser mixing of Ti and steel [5], and by the coevaporation of Ti and Fe on steel substrates[6]. In all instances the good tribological properties observed in these materials were attributed to the formation of an amorphous phase and/or refined microstructure which evolved during processing.

In addition to microstructure considerations, Fe/Ti ion mixing experiments have shown that the composition of the mixed layer is also important [3,4]. From composition dependent friction studies it was concluded that a minimum in friction coefficient occurs at approximately $Fe_{50}Ti_{50}$. Transmission electron microscopy of the ion mixed layer at this composition revealed a completely amorphous microstructure. However, an additional interesting observation was that unirradiated as-deposited Fe/Ti multilayers also possessed a composition dependent friction profile with a minimum also occurring at $Fe_{50}Ti_{50}$. In fact, the friction coefficient of the $Fe_{50}Ti_{50}$ sample in its as-deposited state was equivalent to the ion mixed amorphous state, ~ 0.23 [4]

These observations indicate that thin multilayers of Fe/Ti, with an overall composition of $Fe_{50}Ti_{50}$ and an initial crystalline structure (hcp Ti and bcc Fe), have very similar tribological properties as an ion mixed amorphous alloy of the same composition. In the present study we reexamine the tribological properties of Fe/Ti multilayers as well as examine the microstructure and hardness which evolves during sliding in an attempt to explain its behavior.

EXPERIMENTAL

A 900 Å multilayered film composed of 4 Fe/Ti bilayers, each with a Fe thickness of 90 Å and a Ti thickness of 135 Å, was synthesized by sequential electron beam evaporation in a vacuum of 5×10^{-8} torr. Heat treated tool steel (1.55% C, 0.3% Si, 0.3% Mn, 12% Cr, 0.8 % Mo and 0.8% V) was employed as a substrate. The microstructure of the substrate consisted of tempered martensite, carbides, and retained austenite. The hardness was 7 GPa. Prior to deposition the substrates were mechanically polished, finishing with 0.25-μm diamond paste.

Wear and friction tests were carried out using a pin-on-disk tester. A 52100 steel ball 6 mm in diameter was used as the pin. The diameter of the wear track was 2 mm. In some experiments the sample was moved back and forth in order to obtain a 3 mm-wide, donut-shaped track for hardness and microstructure analysis. The average sliding speed was 10 mm/s. A load of 62.6 g used in all measurements corresponds to a maximum Hertzian surface pressure of 540 MPa. The friction force was monitored continuously with a calibrated force sensor. All tests were carried out without lubrication in air at a relative humidity of 45%.

Changes in surface hardness resulting from pin sliding was examined using a commercially available nanoindenter that directly measures the load on a Vickers type diamond indenter tip as a function of displacement from the surface. We chose to eliminate the effect of tip shape by normalizing the date to measurements of the steel substrate taken under identical conditions. Measurements were made under a constant load rate of 250 mN/s to a depth of 250 nm. Sixteen indents were made on each sample and the data were averaged in 10-nm increments. As discussed by Doerner and Nix [7], the actual depth of the indent must take into account the elastic recovery of the material as the indenter is removed. Elastic recovery was approximately 15% in these samples. Since indents were made well through the modified surface, the effect of this recovery on the measurements was small.

The surface morphology of the wear track was investigated using scanning electron microscopy (SEM). The microstructure of the Fe/Ti multilayers before and after pin-on-disk tests was examined with a transmission electron microscope (TEM).

RESULTS AND DISCUSSION

Friction data from pin-on-disk tests are presented in Fig. 1. These data show that after a short run-in period a steady state friction coefficient of 0.16 is achieved. This behavior is maintained throughout the 1000 cycle test.

The wear track morphology after 1000 cycles is presented in the low and high magnification SEM micrographs displayed in Fig. 2. These data show that the wear track has been partially worn through with the majority of the surface still covered by the deposited film. Two types of wear damage are apparent; severe large area pitting due to sudden fracture (a) and small area pitting due to a continuous wear mechanism. The effect of pin transfer on wear damage is currently under investigation.

Nanoindentation surface hardness data from the wear track are presented in Fig. 3. These data, which have been normalized to the as-deposited film, show that the material exposed to pin-on-disk testing are 20 % harder

A TEM examination of the as-deposited Fe/Ti multilayer showed it to be composed of bcc-Fe and hcp-Ti with an average grain size between 100 and 150 Å, Fig. 4a. After pin-on-disk sliding experiments changes in microstructure were apparent. The diffraction pattern displayed very broad diffuse rings suggesting the formation of a very fine grained or amorphous alloy. Also present was a sharp ring pattern corresponding to bcc-Fe. Dark field TEM imaging from the first broad ring showed a very fine grain structure with an average diameter of 30 Å, Fig. 4b.

These results show that a Fe/Ti multilayer structure, with individual layer thickness of approximately 100 Å, and an average composition of $Fe_{50}Ti_{50}$ will react under sliding conditions to form a nanocrystalline alloy. While these are the first reported results for metastable phase formation by solid state reaction in the Fe/Ti system, they are consistent with the mechanical deformation and thermal induced amorphization phenomena observed by others [8].

The present data also shows that the hardness of nanocrystalline $Fe_{50}Ti_{50}$ is greater than the composite hardness of as-deposited Fe and Ti. A comparison with other metastable forms of $Fe_{50}Ti_{50}$ shows the nanocrystalline layer to be harder than coevaporated amorphous Fe/Ti [6] and significantly harder than ion mixed amorphous Fe/Ti [9].

These data suggest that the low friction coefficient observed for as-deposited Fe/Ti multilayers can be attributed to the evolution of a nanocrystalline alloy by solid state reaction during pin-on-disk tests. The frictional properties are in part the result of surface hardening which occurs upon formation of the nanocrystalline alloy and in part due to improvements in interlayer and film/substrate adhesion which must also occur during solid state reaction. While the actual physical mechanism responsible for the low friction is not known at present it is not unreasonable to suspect that a film of surface oxygen formed in the high humidity of the testing environment are playing a role [6]. This subject will be the topic of a future publication [10].

CONCLUSIONS

The low friction observed in as-deposited Fe/Ti multilayers results from the formation of a hard nanocrystalline alloy and improved interlayer and film/substrate adhesion. These material changes are the result of a solid state reaction which occurs during pin-on-disk friction and wear tests.

ACKNOWLEDGEMENTS

This work was supported the U.S. Department of Energy and by the U.S. Air Force Wright Aeronautical Laboratory under contract DIO-90-2 through B.D. McConnell.

REFERENCES

1. I.L. Singer and R.A. Jeffries, Appl. Phys. Lett. **43**, 925 (1983).
2. D.M. Follstaedt, J.A. Knapp, L.E. Pope, F.G. Yost, and S.T. Picraux, Appl. Phys. Lett. **45**, 529 (1984).
3. J-P. Hirvonen, M. Nastasi, J.R. Phillips, and J.W. Mayer, J. Vac. Sci. Technol. **A4**, 2997 (1986).
4. J-P. Hirvonen, M. Nastasi, and J.W. Mayer, Appl. Phys. Lett. **49**, 1345 (1986)
5. T.R. Jervis, J-P. Hirvonen, M. Nastasi, T.G. Zocco, J.A. Martin, G.M. Pharr, and W.C. Oliver, in New Materials Approaches to Tribology: Theory and Application, eds., L.E. Pope, L.L. Fehrenbacher, and W.O. Winer (Materials Research Society, Pittsburgh, 1989) p. 189
6. J-P. Hirvonen, M. Nastasi, T.R. Jervis, and T.G. Zocco, Thin Solid Films, in press.
7. M.F. Doerner and W.D. Nix, J, Mater, Res., **1** (1986)601
8. R.B. Schwarz and W.L. Johnson, eds., "Solid State Amorphizing Transformations", Journal of the Less -Common Metals, **140** (1988).
9. J-P. Hirvonen, M. Nastasi, T.G. Zocco, and T.R. Jervis, J. Appl. Phys., in press.
10. J-P. Hirvonen, M. Nastasi, and T.R. Jervis, in preparation.

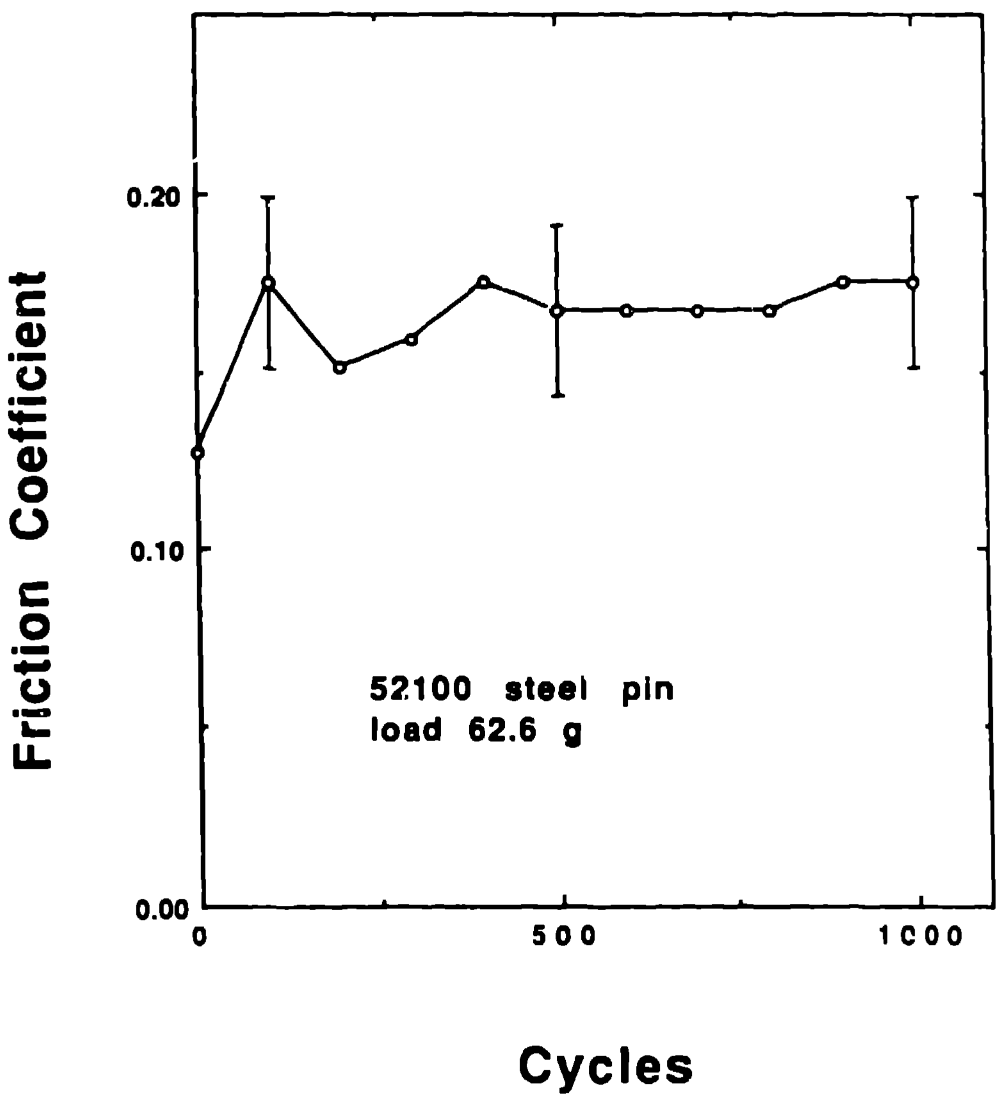
FIGURE CAPTIONS

FIGURE 1. Friction Coefficient from Fe/Ti multilayers on tool steel at a load of 62.6 g.

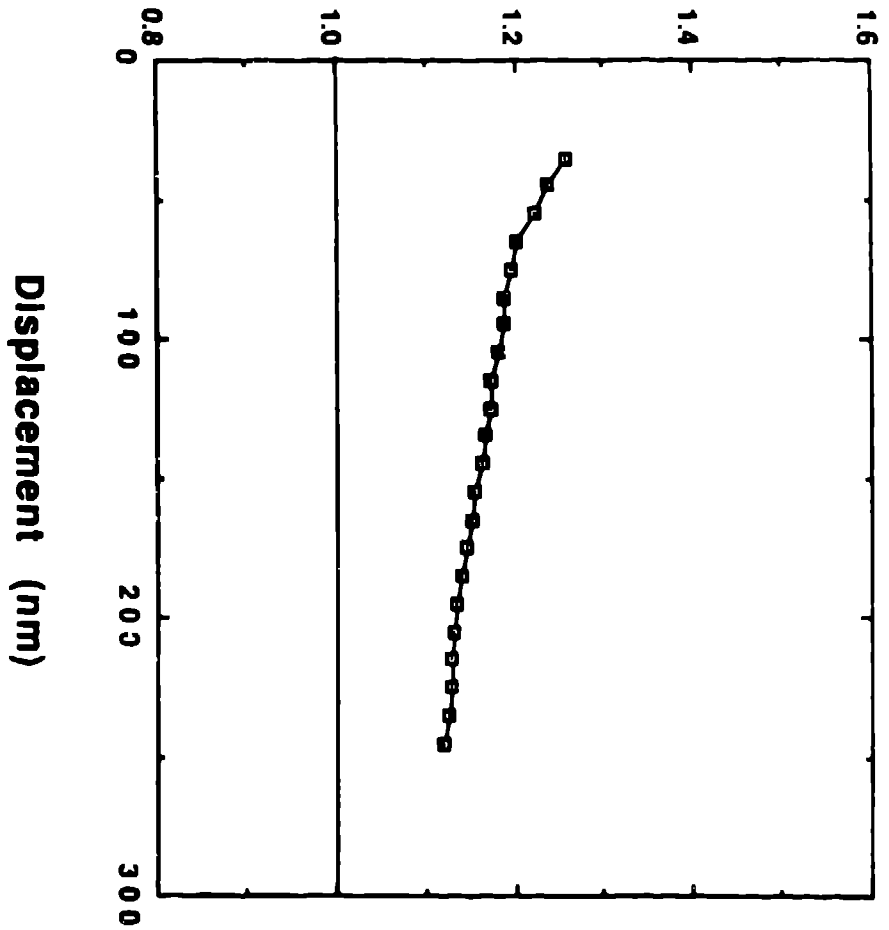
FIGURE 2. SEM micrographs of the wear track following 1000 pin-on-disk cycles.

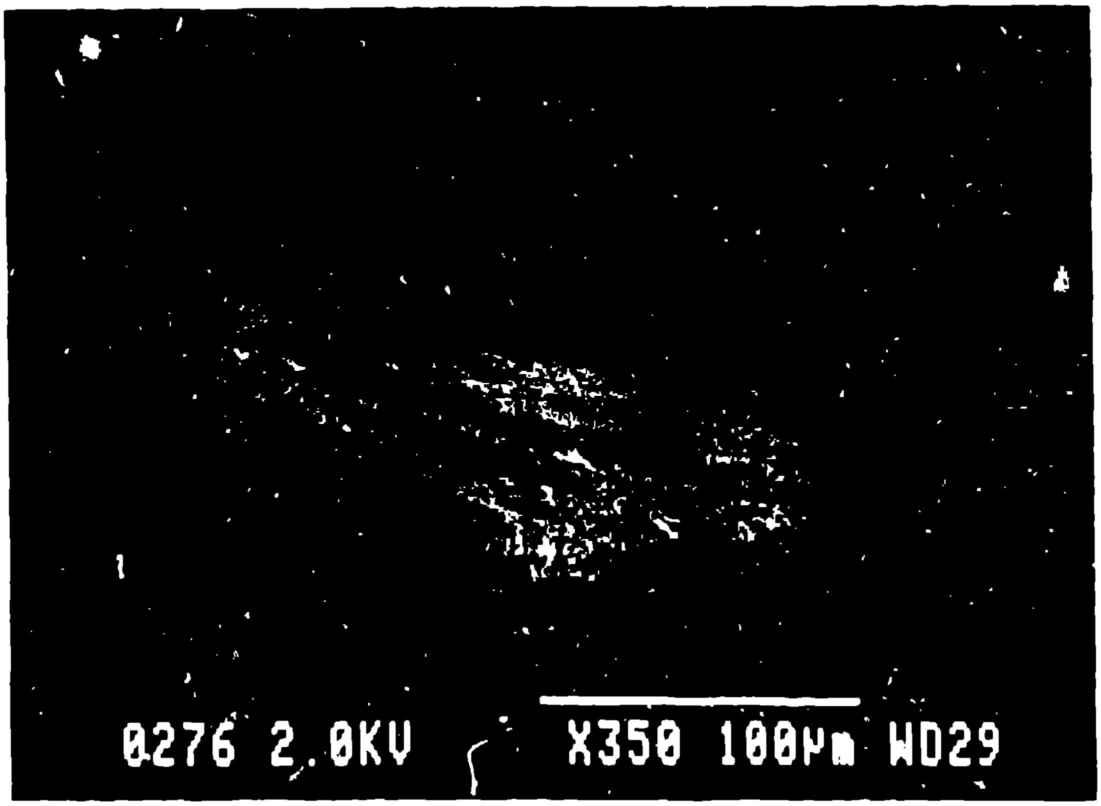
FIGURE 3. Wear track hardness data normalized to as-deposited Fe/Ti multilayer hardness.

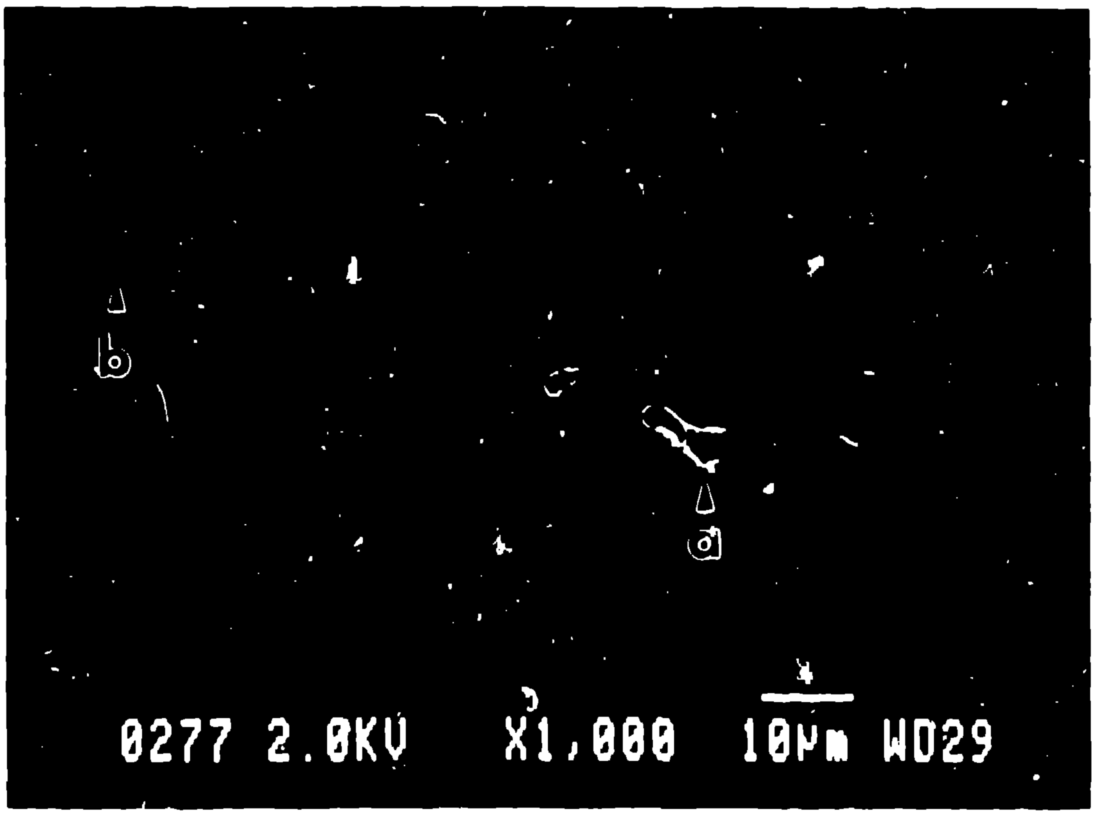
FIGURE 4. High magnification TEM micrographs before and after pin-on-disk test.

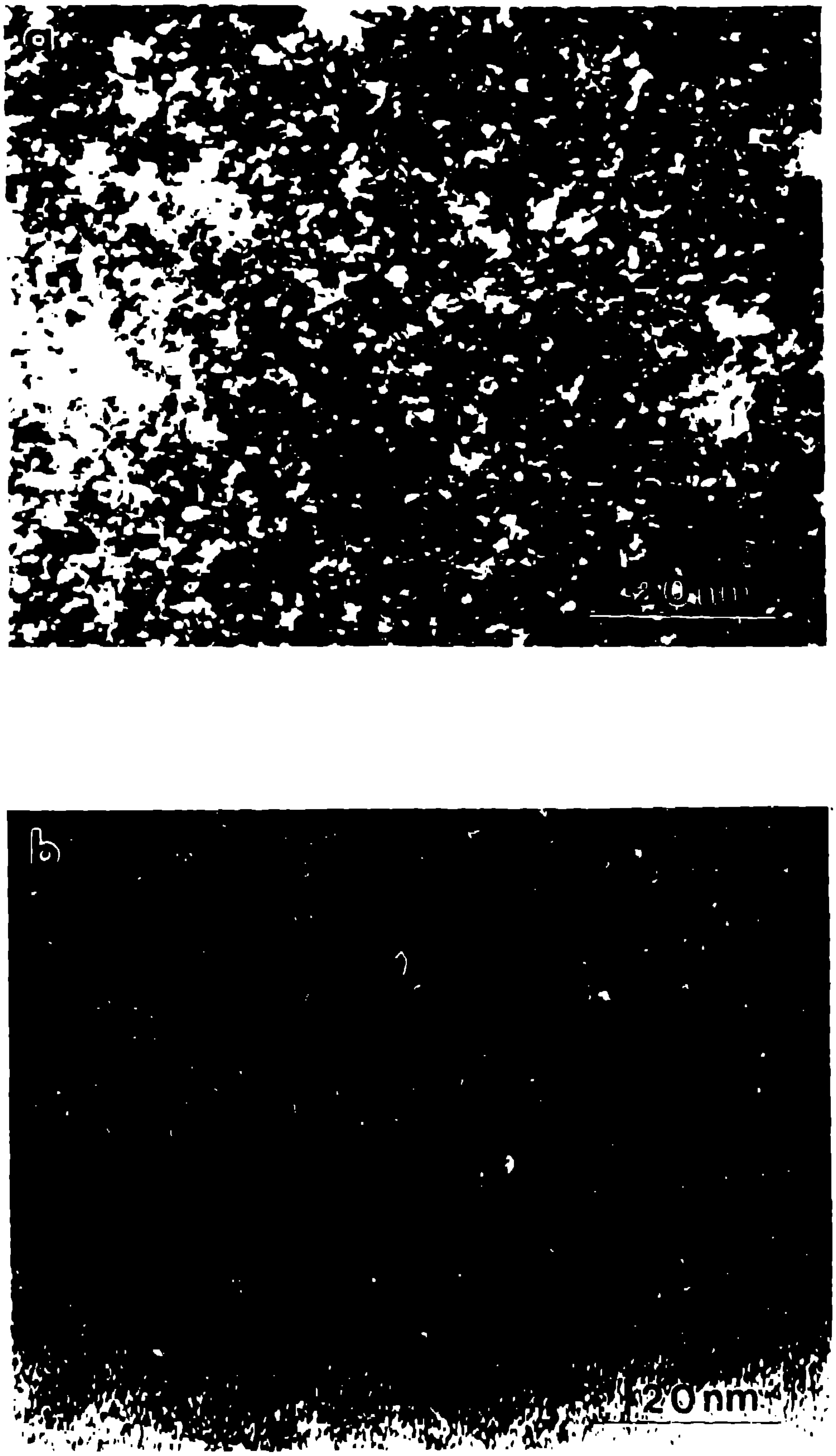

Friction Coefficient
0.20
0.10
0.00
0
500
1000
52100 steel pin
load 62.6 g
Cycles

Relative Hardness of Wear Track




0276 2.0KV X350 100µm WD29


0277 2.0KV X1,000 10µm WD29



a
b
20 nm