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Surface Effects of Corrosive Media on Hardness, Friction, and Wear of Materials

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SURFACE EFFECTS OF CORROSIVE MEDIA ON HARDNESS, FRICTION, AND WEAR OF MATERIALS

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ABSTRACT

Hardness, friction, and wear experiments were conducted with magnesium oxide exposed to various corrosive media including two acids, a base, and a salt and with elemental iron and nickel exposed to water and various concentrations of NaOH. Chlorides such as $MgCl_2$ and sodium-containing films were formed on cleaved magnesium oxide surfaces from their interactions with HCl-containing solutions. The $MgCl_2$ films softened the magnesium oxide surfaces and caused high friction and great deformation. Hardness was strongly influenced by the pH value of the HCl-containing solution. The lower the pH, the lower the microhardness. Neither the pH value of nor the immersion time in NaOH-containing, NaCl-containing, and HNO_3-containing solutions influenced the microhardness of magnesium oxide. NaOH formed a protective and low-friction film on iron surfaces. The film was broken up by sliding action at low concentrations but remained on the surfaces at high concentrations. The coefficient of friction and the wear for iron were low at concentrations of NaOH higher than 0.01 N. An increase in NaOH concentration resulted in a decrease in the concentration of ferric oxide (mainly Fe_2O_3) on the iron surface. It took less NaOH to form a protective, low-friction film on nickel than on iron. A small amount of NaOH on nickel gave a low coefficient of friction and low wear damage, and the values did not change with NaOH concentration. In water alone the nickel surface sustained considerable wear damage.

INTRODUCTION

The presence of surface-active agents on metals and nonmetals can alter the surface activity of these materials as well as their mechanical properties (1,2). Tribological properties such as adhesion, friction, deformation, wear, and lubrication of solid surfaces in contact are extremely dependent on the adsorbed surface-active ions or molecules (i.e., environmental constituents).

It was anticipated that the hardness of solids would be influenced by environment because hardness is one of the measurements used to indicate the extent of surficial plastic deformation. Many investigators have studied the effects of environment on the hardness of a variety of nonmetals such as CaF_2, Al_2O_3, and LiF (2-5). These effects include strengthening by dissolution of the solid surface, the Joffe effect (6); surface hardening, the Roscoe effect (7,8); surface softening, the Rehbinder effect (9); correlation between hardness and the zeta-potential (10,11); and the effect of adsorbed water on indentation creep (5,12).

Since environmental effects on the hardness of nonmetals are so surface dependent, the present authors took a different analytical approach to determining environmental effects on hardness. A surface analytical tool (i.e., x-ray photoelectron spectroscopy (XPS)) was used to study the surficial or outermost atomic layers of solids.

The first objective of the present paper is to examine the surface chemistry, hardness, friction, and wear of a ceramic (single-crystal magnesium oxide) exposed to various corrosive media including two acids, a base, and a salt.

Although corrosion is recognized as an important variable in the friction and wear of metals (13), its role is not well understood. In the sliding, rolling, or rubbing contact of materials the surfaces become strained as a result of the mechanical activity that takes place. A wear surface is different electrochemically from its surroundings. It contains metal that is highly strained and that reaches locally high temperatures at shearing points or asperities (14,15). Electrochemical potentials may be established locally to either impede or enhance corrosion; cyclic stresses may promote stress corrosion and corrosive fatigue (16).

Resistance to corrosion is often the result of the formation of some type of film on the metal. Sliding action can destroy such films - or it can develop better corrosion-resistant films by producing new surfaces. The coefficient of friction is, like corrosion resistance, highly sensitive to surface films.

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The second objective of this paper is to examine the surface chemistry, hardness, friction, and wear of elemental iron and nickel exposed to various concentrations of NaOH and to water, as well as to analyze their surface chemistry by XPS.

MATERIALS

The arc-melted, single-crystal magnesium oxide used was 99.99 percent pure as determined from the manufacturer's data. Magnesium oxide was selected as the material to examine for a number of reasons: its slip and fracture behavior are well understood, the Rehbinder effect has been observed with it, fresh, atomically clean surfaces can be prepared by cleavage, and it can be used as a bearing material (17).

The iron used was more than 99.99 percent pure and had been fully annealed. Its hardness was Rockwell B40. The nickel used was electrolytic and had been annealed to a hardness of Rockwell B30.

APPARATUS

The apparatuses used in this investigation have been described previously (18-22). The Vickers microhardness tester was fixed on a vibration-free table and measured indentation hardness in a controlled environment (18,19). Specimens were indented manually with a diamond indenter. The friction and wear apparatuses were basically a pin (rider) on a flat (e.g., Fig. 1). An ultra-high-vacuum system containing the XPS system and an ion gun (18-20) was used for ion sputter-etching.

EXPERIMENTAL PROCEDURE

Hardness, Friction, and Wear Experiments on Magnesium Oxide

The magnesium oxide bulk crystals were immersed in room-temperature aqueous solutions with various pH values and then cleaved to a specimen size of 5 mm by 5 mm along the {001} surface by using a knife and hammer. The submerged specimens were then mounted on the Vickers microhardness tester and the friction apparatus. Indentations and sliding friction experiments were made on cleaved {001} surfaces in the desired aqueous solution and in water without exposing the specimen surface directly to the air or to any other environment. For comparison tests the magnesium oxide bulk crystals were similarly prepared in laboratory air at room temperature.

The indentation diagonals were parallel to the <001> directions on the {001} surfaces. The time in contact was 20 s. A hemispherical diamond rider (20-μm radius) was made to slide in the <001> directions on the magnesium oxide {001} surfaces under a load of 0.25 N and at a sliding velocity of 10 mm/s.

Table I lists the aqueous solutions used in this investigation and their analyzed pH values. The solutions contained NaOH, NaCl, HNO_3, and HCl.

Hardness, Friction, and Wear Experiments on Iron and Nickel

Iron and nickel flats were metallographically lapped with 600-grit silicon carbide paper. They were then polished with diamond paste, first 6-μm paste and then 3-μm paste, or with 3-μm paste alone. Final polishing was with 0.3-μm alpha alumina on a flooded, flat polishing wheel. The polished specimen was cleaned with water and then with ethanol. The specimens were subsequently etched until the residual deformed region was removed and the grain structure was visible. For iron the etchant was 5 percent Nital, and for nickel it was 1 part nitric acid to 1 part acetic acid (glacial). The specimen was then lightly polished with the 0.3-μm alumina and cleaned with distilled water and ethanol.

Before the Vickers microhardness measurements drops of the desired solution were placed on the flat metal surface. After 1-hr exposure an indentation was made with a diamond Vickers indenter through the drops. The metal specimen and the diamond indenter were then cleaned with distilled water and ethanol. The indentation diagonals were measured right after cleaning. The hardnesses of iron and nickel were also measured in laboratory air. All indentations were made at a load of 0.1 N at room temperature. The time in contact was 20 s. Each hardness value is the average of 8 to 12 measurements.

In the sliding friction experiments the specimens were clamped in the vise of the apparatus. Several drops of the desired solution were placed under the rider and on the flat surface to form one large drop covering most of the surface of the flat specimen. The rider, a 6.4-mm-diameter Al_2O_3 (sapphire) sphere, was lowered through the drop until a strain gauge indicated that it was just in contact with the metal flat surface. The weight was then loaded onto the rider and sliding begun. The rider and its point of contact with the specimen were always in the interior of a large liquid drop. Furthermore a meniscus formed between the rider and the flat specimen provided a continuous supply of solution to wet the flat specimen in and near the wear track. The rider slid in reciprocating motion, on the iron and nickel flats at 1.5 mm/s, at a load of 2.5 N (250 g), at room temperature. Each experiment lasted 60 min. Thus the rider passed over the track 540 times (270 times each way) during an experiment.

To estimate the wear characteristics of iron or nickel, their wear tracks were scanned by a surface profilometer.

XPS Analysis

For XPS analysis (18-20) the specimens were placed in the vacuum chamber, and the system was evacuated and subsequently baked out to 26 nPa ($2x10^{-10}$ torr). The surfaces were examined with a magnesium kα x ray at a source power of 400 W (10 kV, 40 mA). The analyzer work function was determined by assuming the binding energy for the gold 4f 7/2 peak to be 83.8 eV; that is, the gold 4f 7/2 peak was used as the reference line.

RESULTS AND DISCUSSION

Hardness, Friction and Wear of Magnesium Oxide

The distribution of dislocation etch pits shown in Fig. 2 is typical for indentations made on magnesium oxide surfaces cleaved in air. Because magnesium oxide is highly ionic, slip occurs not on the close-packed {001} cubic planes but on the {011} cubic diagonals. The dislocation-etch-pit pattern around the indentation on the {001} surface (Fig. 2) contains screw dislocations in the <001> directions and edge dislocations in the <011> directions. The arrays of screw and edge dislocations were 4.9 and 7.7 times wider, respectively, than the average length of the two diagonals of indentation.

The Vickers microhardnesses of magnesium oxide surfaces cleaved in air and in water were essentially the same at 824 and 822, respectively. Indentation experiments were also conducted in corrosive solutions containing NaOH, NaCl, HNO_3, and HCl with various pH values. The microhardness as a function of the time that the magnesium oxide crystal was immersed in the corrosive solution is given in Fig. 3. Each hardness value is the average of 10 measurements. There were no changes in microhardness with immersion time for surfaces immersed in solutions containing NaOH (pH = 13.5), NaCl (pH = 6.7), and HNO_3 (pH = 1.0). The pH value had little effect on microhardness: the difference in microhardnesses of the surfaces immersed in these solutions was relatively small. However, the surfaces immersed in HCl-containing solutions with a pH of 1.0 exhibited both low microhardness and a slight change in microhardness with immersion time. These microhardness data could not be taken beyond an immersion time of 7200 s because the magnesium oxide surfaces were considerably roughened.

To gain an understanding of the surfaces of magnesium oxide in the HCl-containing solution, indentation experiments were conducted in HCl-containing solutions with pH values of 4.2, 2.5, and 1.0. The data (Fig. 4) indicate that the microhardness of a magnesium oxide surface immersed in HCl solutions is influenced by the pH value as well as by the immersion time. The stronger the acid, the lower the microhardness observed. The microhardness of the surface immersed in the HCl solution with a pH of 1.0 for 900 s was almost the same as those of surfaces immersed in solutions with pH values of 4.2 and 2.5 for $3.5x10^5$ s.

There was a significance in the differences in the distribution length of edge dislocations for the HCl solution (pH = 1.0), the NaCl solution (pH = 6.7), and the NaOH solution (pH = 13.5). The lengths of the rows of etch pits on the solid surface reflect the extent of deformation. The longer the rows, the greater the deformation and the lower the microhardness. The distribution lengths of the edge dislocations are consistent with the microhardness data shown in Fig. 3 (18,19).

XPS spectra of Cl_{2p}, Na_{1s}, Mg_{2p}, and O_{1s} were obtained from narrow scans taken on the magnesium oxide surfaces cleaved in 10^{-2} N aqueous NaCl solutions containing HCl (pH = 1.0) or NaOH (pH = 13.5) before and after argon-ion sputter etching for 30 min (18,19). Typical results are presented in Figs. 5 and 6. Chlorine, sodium, and magnesium peaks for the surfaces exposed to the HCl solution can be seen (Fig. 5) before and after sputtering. The sodium peak was larger after sputtering. Oxygen peaks were also evident on surfaces exposed to the acid solution before and after sputtering (19).

Figure 5 reveals that chemically reacted chloride- and sodium-containing films formed as a result of the HCl solution interacting with the magnesium oxide surface and deeply in the subsurface. Chlorine chemically reacted with the magnesium to form compounds such as $MgCl_2$ in the surficial layer. Even after 30 min of sputtering these chemically reacted films were not removed; that is, the interactions occurred not only on the surface, but also in the subsurface. The chloride films played a role in softening the magnesium oxide surface (Figs. 3 and 4).

A chlorine peak is clearly evident (Fig. 6(a)) in the spectrum of the magnesium oxide surface exposed to the NaOH solution (pH = 13.5) before sputtering, no chlorine is apparent after sputtering. Although sodium peaks appear both before and after sputtering (Fig. 6(b)), the amount of sodium was decreased by sputtering. No magnesium peak is observable before sputtering, but afterward a magnesium peak does appear in the spectrum.

The magnesium oxide surface cleaved in this alkali solution was covered with chloride- and sodium-containing compound films. Its outermost surficial layer consisted of mostly NaOH and NaCl films with no magnesium oxide exposed. However, argon ion sputtering completely removed the chloride films from the surface and partially removed the sodium films, exposing the magnesium oxide. These sodium-containing films played a slight role in softening or hardening the magnesium oxide surface (Fig. 3).

Sliding friction experiments were conducted with a diamond rider having a 20-μm-radius hemispherical tip in contact with magnesium oxide surfaces exposed to an HCl solution (pH = 1.0), an NaOH solution (pH = 13.5), water, and air. Plastic deformation occurred and permanent grooves were formed in the surfaces during sliding. The coefficients of friction and the groove widths were influenced by the solutions and environments (Table II). When magnesium oxide was exposed to the HCl solution, the $MgCl_2$ (as detected by XPS analysis) formed in its surficial layer caused greater plastic flow and plowing action and resulted in higher friction and greater groove width than for magnesium oxide exposed to the NaOH solution, water, and air. The differences for magnesium oxide exposed to the NaOH solution, water, and air were small. Thus, the friction and deformation of magnesium oxide, as well as its hardness are markedly affected by exposure to an HCl solution.

Hardness, Friction, and Wear of Iron and Nickel

The Vickers microhardnesses of iron and nickel were measured as a function of NaOH concentration. At 0.001 N NaOH the hardness of iron (Fig. 7(a)) was higher than that measured in laboratory air (81.5), increased to a maximum at 0.01 N NaOH, and continuously decreased with increasing NaOH concentration.

The Vickers microhardness of nickel exposed to NaOH solutions was also influenced by the concentration of NaOH. At 0.001 N NaOH the hardness of nickel was almost the same as that measured in laboratory air (88.8). The higher the concentration, the lower the microhardness observed.

Friction and wear experiments were conducted with iron and nickel flats in contact with 6.4-mm-diameter Al_2O_3 spherical riders in NaOH at a load of 2.5 N for 60 min. There were marked differences in the friction and wear behavior of the two metals. For iron (Fig. 8) the maximum coefficient of friction and wear track width occurred at 0.01 N NaOH. The coefficient of friction continuously decreased with increasing NaOH concentration, but the wear initially decreased and then leveled off at 0.4 N NaOH. On the other hand, the coefficient of friction and wear track width for nickel (Fig. 9) did not change with NaOH concentration over the entire range from 0.001 to 20 N. The coefficient of friction was low under all of these conditions, as is consistent with nickel being highly resistant to corrosion by NaOH.

Because the difference in wear between iron and nickel under corrosion conditions is important, iron and nickel wear surfaces were examined in more detail by using optical and scanning electron microscopy and a surface profilometer (Figs. 10 to 12). At 0.001 N and 0.01 N NaOH overall corrosive attack was low, but considerable wear was indicated on the iron. At 0.001 N NaOH the pitting and grain boundary attack inherent in caustic embrittlement were caused by the applied and residual stresses in the wear track. Pits were seen in the optical microscope as well as in the surface profile of the wear track (Fig. 10). Iron exhibited the highest friction and wear at 0.01 N NaOH. Although there was no evidence of deep pits, the wear track surface was severely damaged (Fig. 11).

The friction and wear of iron were high in dilute NaOH but low in concentrated NaOH. In 0.4 N to 20 N NaOH (Fig. 10) the wear surfaces were smooth, as also observed in optical and scanning electron micrographs (20,21). The sliding action left a permanent groove on the wear surface, with deformed iron piled up along the sides of the groove. The amount of iron removed, however, was much less than for dilute NaOH solutions. The tenacious film formed was not broken up by the sliding action, and this film acted as a lubricant.

Sliding the Al_2O_3 sphere on a nickel surface in 0.1 N NaOH solution (Fig. 12) resulted in a permanent groove on the nickel surface with a considerable amount of deformed nickel piled up along the sides of the groove. The wear track on nickel was rough and irregular, but the surface was not damaged as it was for iron.

Sliding the Al_2O_3 sphere on a nickel surface in water created a highly torn and distorted surface in the wear track (Fig. 13). Corrosion principles suggest that a tenacious film protects nickel from corrosion by water but that this film might be disrupted in the wear track.

XPS spectra for wear surfaces of iron in 0.001 N, 0.01 N, and 20 N NaOH were compared (Fig. 14). Oxygen bonded directly to iron (as in Fe_2O_3) had an oxygen 1s peak at 530 eV (Fig. 14(a)). The height of this peak decreased with increasing NaOH concentration. The oxygen peak at approximately 532 eV is generally attributed to a bond involving hydrogen as well as iron (i.e., Fe-O-H, such as in FeOOH). This peak is dominant in 20 N NaOH and gets relatively weaker with decreasing NaOH concentration. Fe_2O_3 peaks were also an oxidation product on iron in NaOH solutions (Fig. 14(b)).

No sodium peaks were detected in any scans between 1068 and 1082 eV with NaOH concentrations even up to 20 N.

CONCLUSIONS

The following conclusions were drawn from the data presented herein on the tribological characteristics of magnesium oxide, iron, and nickel in corrosive media.

Magnesium Oxide

1. $MgCl_2$ films are formed on cleaved magnesium oxide surfaces as a result of an HCl-containing solution interacting with the surface. The $MgCl_2$ films soften the magnesium oxide surfaces and cause higher friction and greater deformation. The pH value and the immersion time in HCl solutions strongly influence microhardness. The smaller the pH value and the longer the immersion time, the lower the microhardness.
2. There is almost no change in microhardness with immersion time on magnesium oxide surfaces immersed in NaCl solution containing NaOH with a pH of 13.5, in water containing NaCl with a pH of 6.7, and in water containing HNO_3 with a pH of 1.0. The difference in hardness between these surfaces is also relatively small.
3. Sodium-containing films are formed on cleaved magnesium oxide surfaces as a result of an NaOH-containing solution interacting with the surface. The sodium-containing films do not soften the magnesium oxide surface, and the pH value of the NaOH solutions does not influence its microhardness.

Iron and Nickel

1. In low NaOH concentrations, such as 0.01 N, the friction for iron is high and the wear track is torn up and considerably damaged. The hardness, friction, and wear of iron peak at 0.01 N NaOH and decrease with increasing concentrations of NaOH. This decrease is accompanied by a decrease in the concentration of ferric oxides on the iron surface and an increase in a tenacious film composed of complex iron-oxygen compounds.
2. The general conclusion is that NaOH forms a protective, low-friction film on iron that is destroyed by wear at low NaOH concentrations but remains at high concentrations.
3. Nickel behaves differently from iron in that only a little NaOH gives low values of hardness, coefficient of friction, and wear damage.

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TABLE I. - SOLUTIONS WITH VARIOUS pH VALUES

Solution	pH value[a]
HCl added to 10^{-2} N aqueous NaCl solution	1.0 2.5 4.2
10^{-2} N aqueous NaCl solution	6.7
NaOH added to 10^{-2} N aqueous NaCl solution	10.5 12.3 13.5
HNO_3 added to distilled water	1.0

[a] Potentiometric method was employed for pH determination.

TABLE II. - COEFFICIENT OF FRICTION AND GROOVE WIDTH FOR MAGNESIUM OXIDE IN SLIDING CONTACT WITH HEMISPHERICAL DIAMOND RIDER IN VARIOUS MEDIA

[Single-pass sliding; load, 0.25 N; sliding velocity, 10 mm/min; rider radius, 20 μm.]

Medium and environment	Coefficient of friction	Groove width, μm
HCl added to 10-2 N aqueous NaCl solution (pH = 1)	0.23	41
NaOH added to 10^{-2} N aqueous NaCl solution (pH = 13.5)	.11	28
Distilled water	.12	32
Laboratory air	.10	27

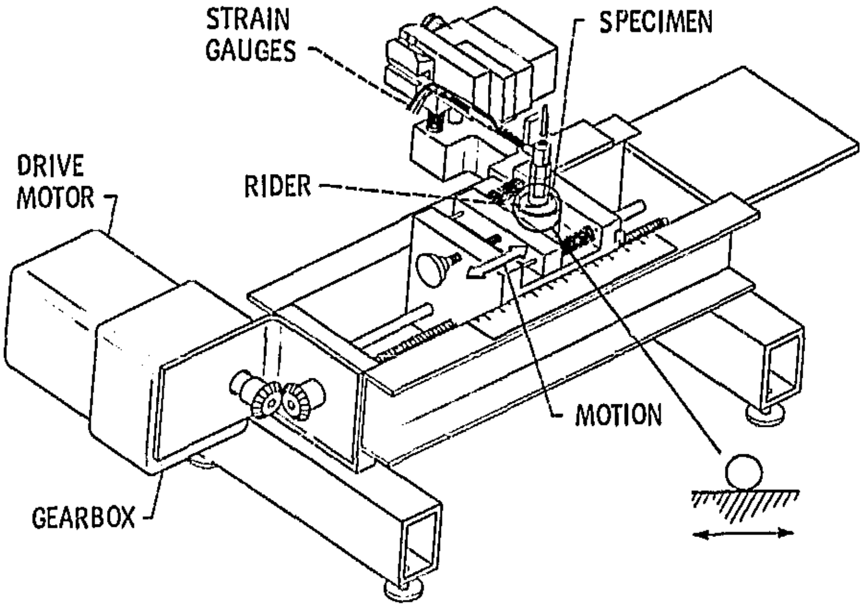


Figure 1. - Friction and wear apparatus.

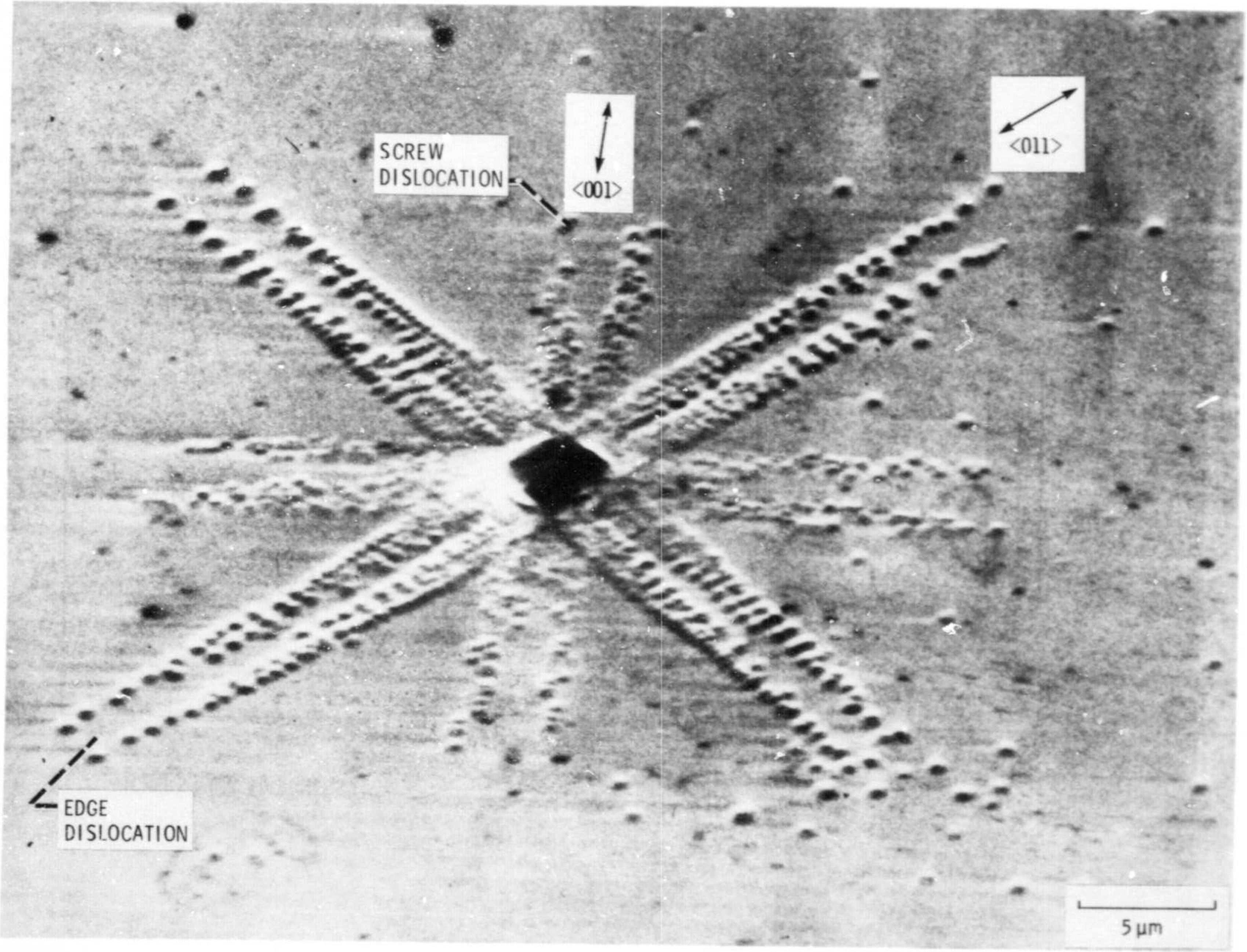


Figure 2. - Distribution of dislocation etch pits around Vickers indentation on magnesium oxide surface. Indenting surface, {001} cleaved in laboratory air; load, 0.1 N; indenter, diamond.

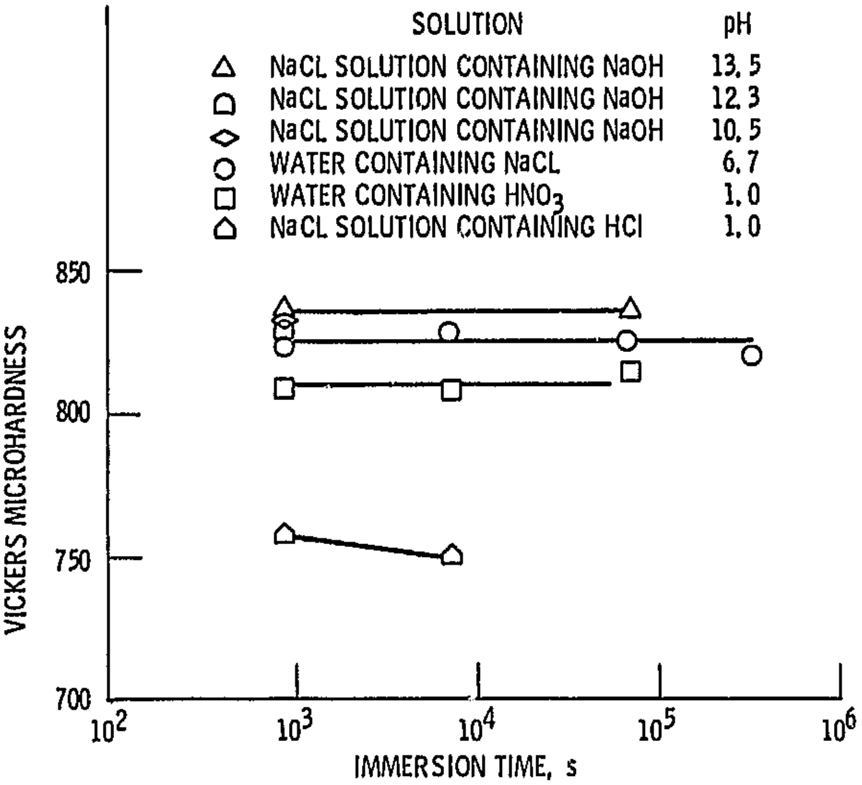


Figure 3. - Effects of immersion time on microhardness of magnesium oxide (001) surface. Load, 0.1 N; indenter, diamond; room temperature. (MgO crystals were cleaved in various solutions, and indentations were made on immersed surfaces.)

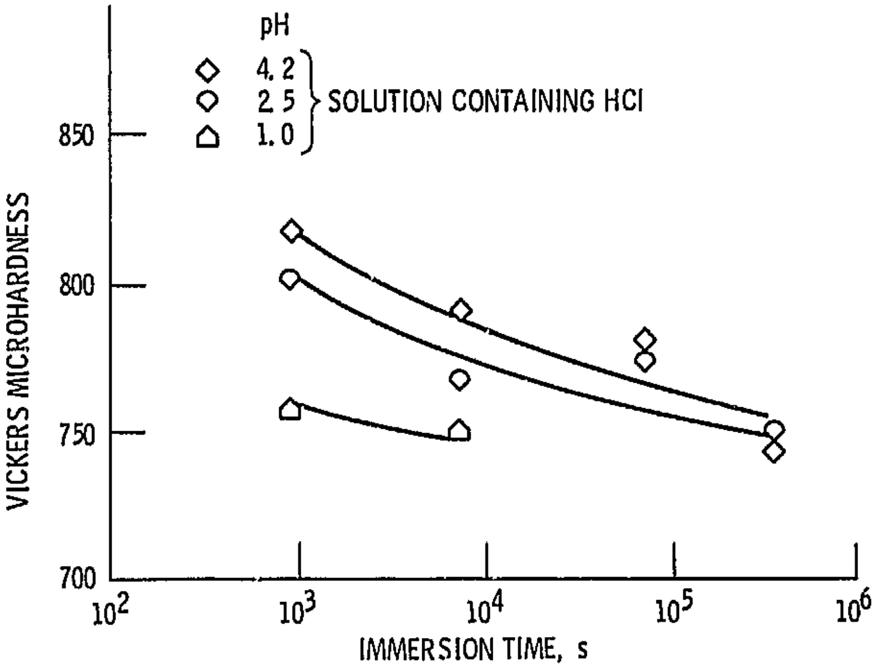
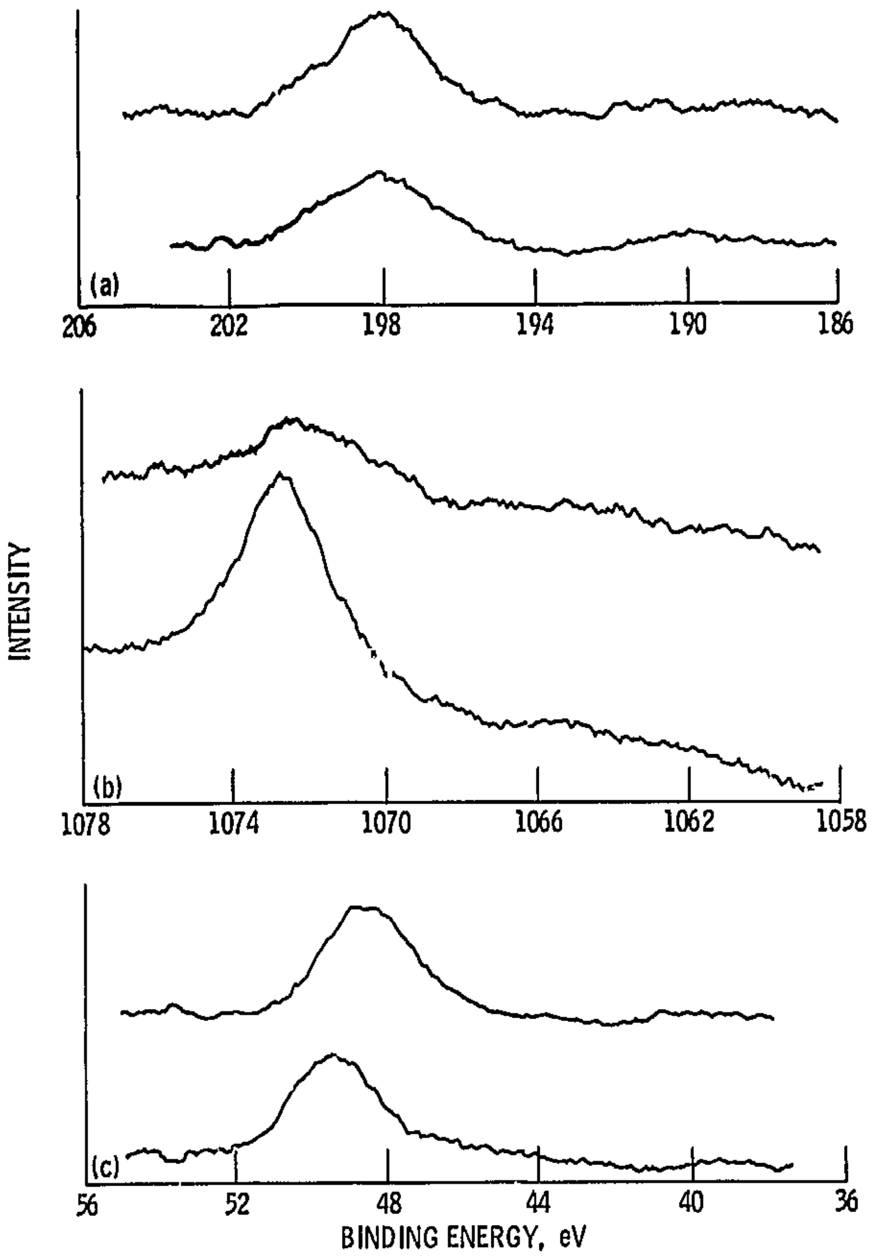
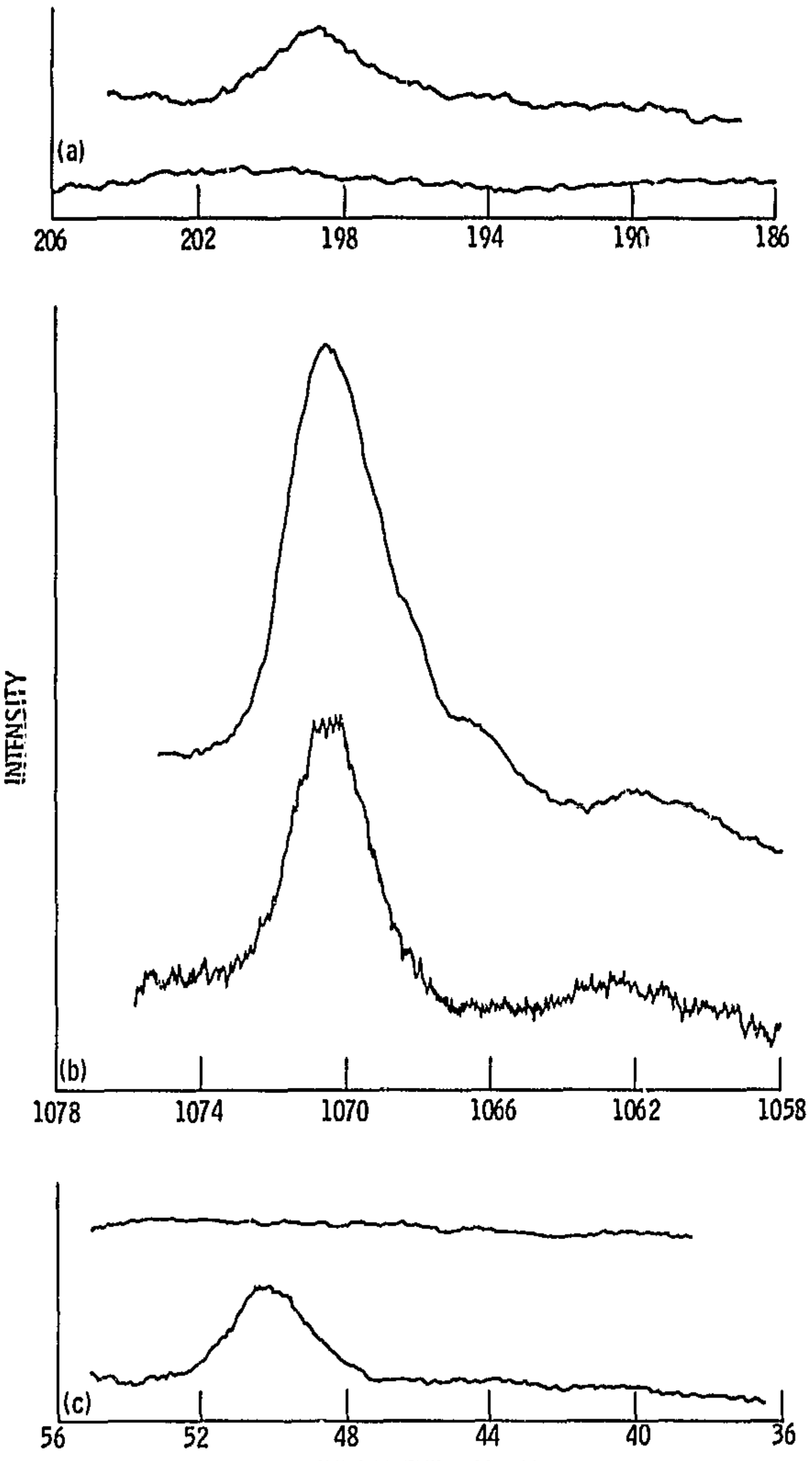


Figure 4. - Effects of immersion time on microhardness of magnesium oxide (001) surface immersed in HCl solutions. Load, 0.1 N; indenter, diamond; room temperature.



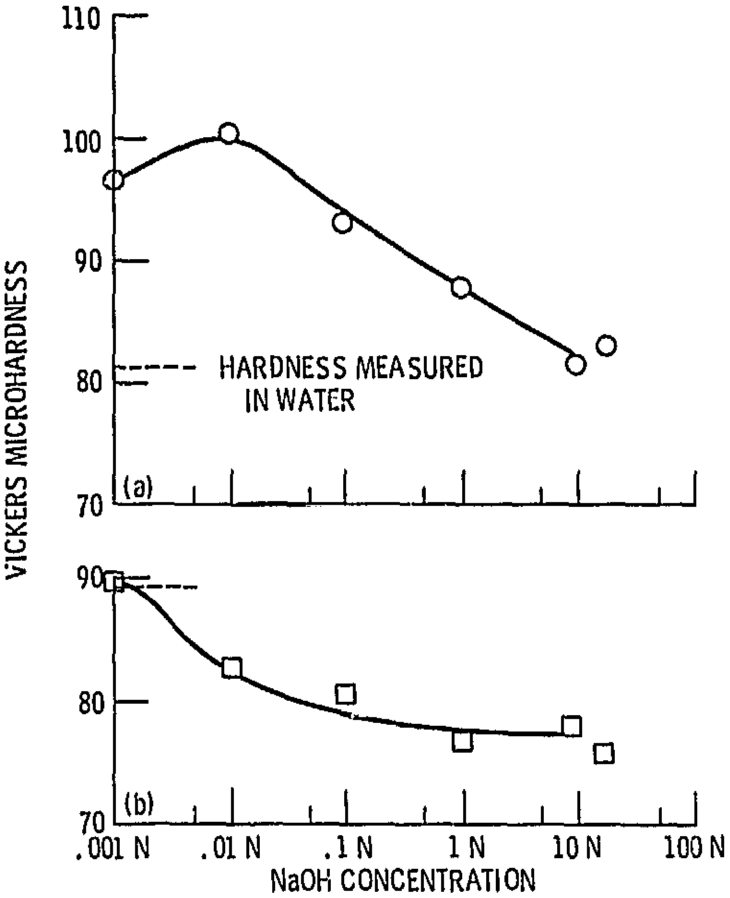
(a) Cl_{2p}.
(b) Na_{1s}.
(c) Mg_{2p}.

Figure 5. - Representative Cl_{2p}, Na_{1s}, and Mg_{2p} peaks on magnesium oxide surface cleaved in solution containing HCl (pH = 1). (Top traces taken before sputtering; bottom traces, after sputtering.)



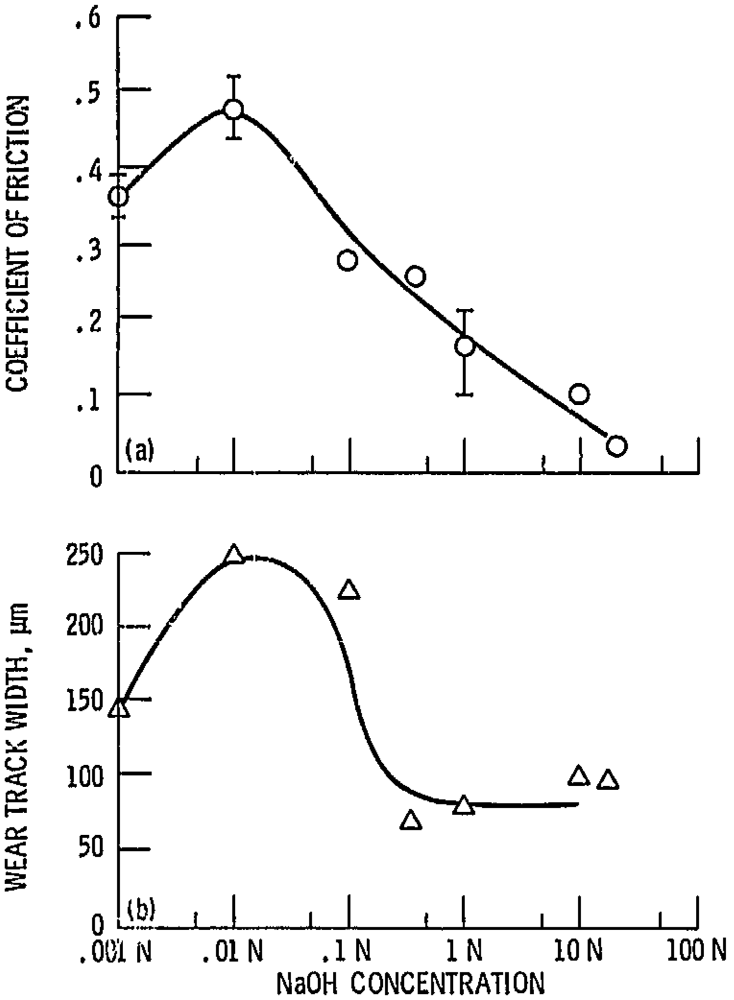
(a) Cl_{2p}.
(b) Na_{1s}.
(c) Mg_{2p}.

Figure 6. - Representative Cl_{2p}, Na_{1s}, and Mg_{2p} peaks on magnesium oxide surface cleaved in solution containing NaOH (pH = 13.5). (Top traces taken before sputtering; bottom traces, after sputtering.)



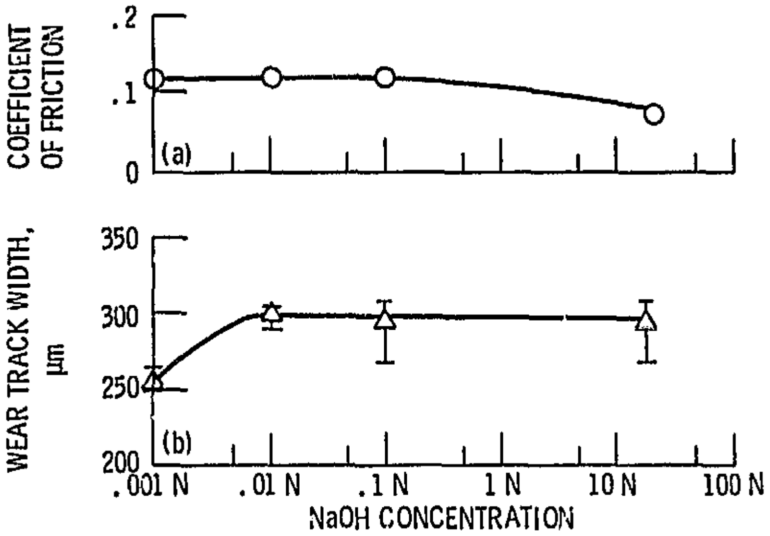
(a) Iron.
(b) Nickel.

Figure 7. - Effects of NaOH concentration on microhardness for iron and nickel in NaOH solutions. Load, 0.1 N.



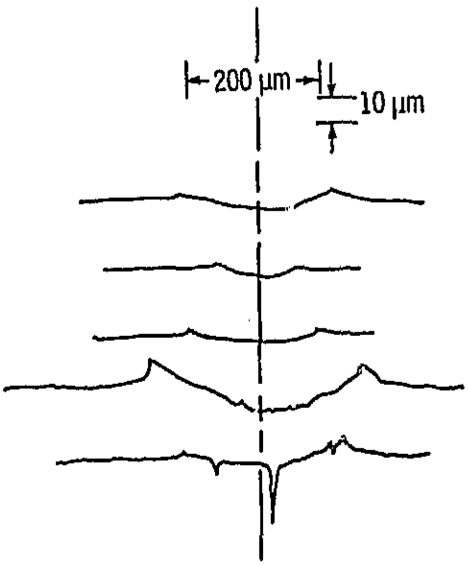
(a) Coefficient of friction.
(b) Wear track width.

Figure 8. - Effects of NaOH concentration on coefficient of friction and wear track width for iron sliding against Al_2O_3 sphere in NaOH solutions.



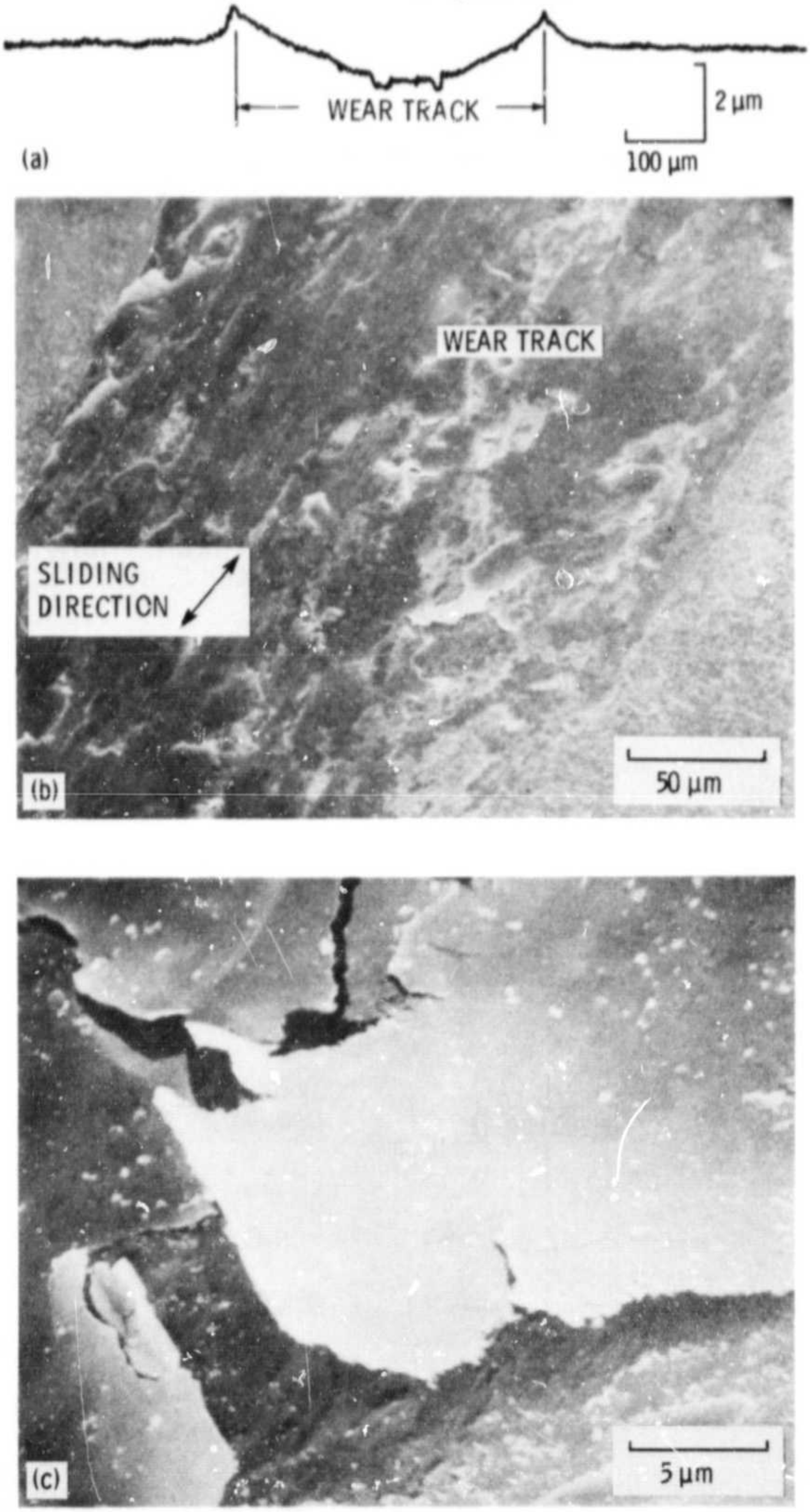
(a) Coefficient of friction.
(b) Wear track width.

Figure 9. - Effects of NaOH concentration on coefficient of friction and wear track width for nickel sliding against Al_2O_3 sphere in NaOH solutions.



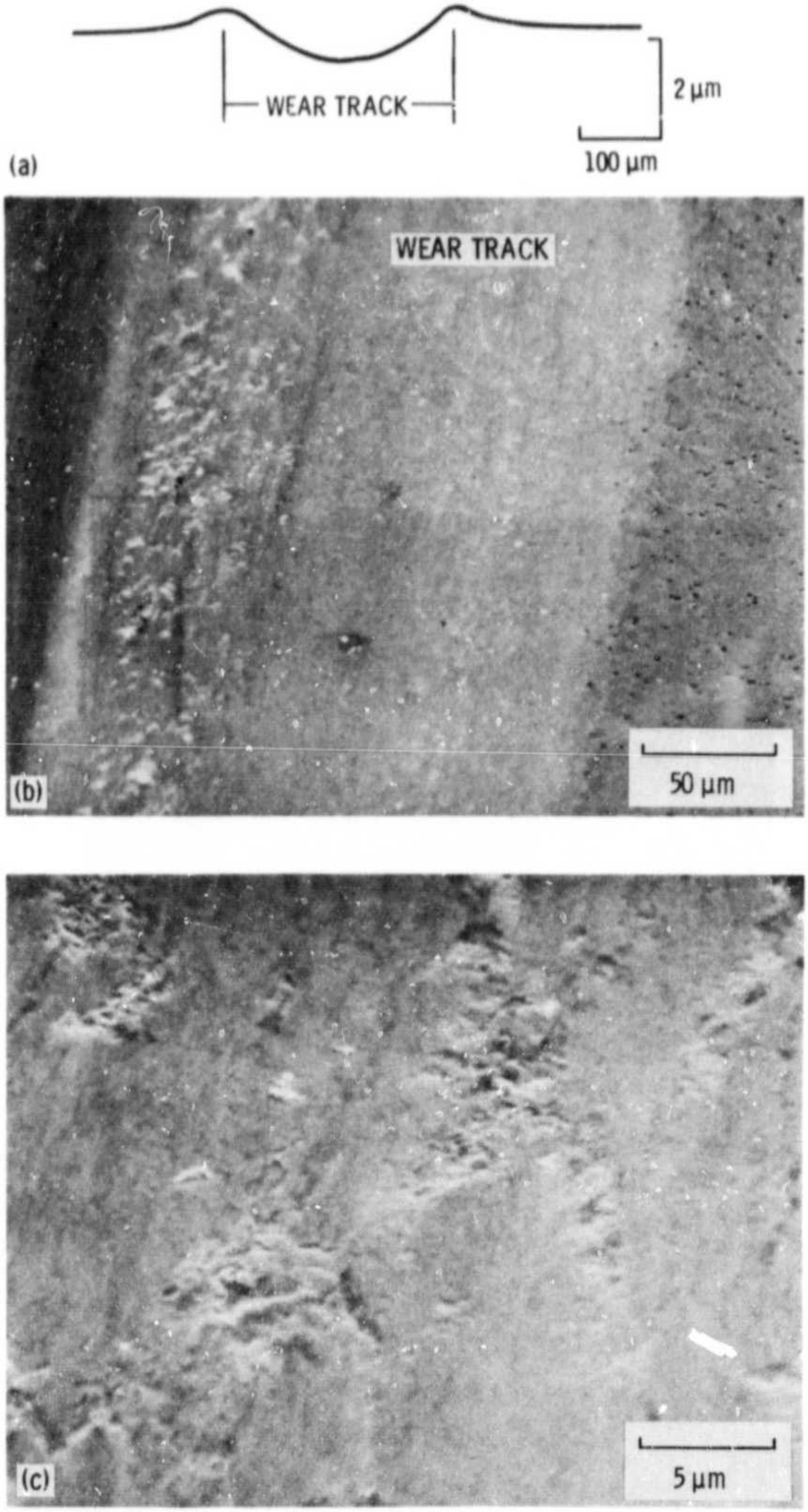
CORROSIVE MEDIUM	STATIC COEFFICIENT OF FRICTION, μ_s
H_2O	0.26
20 N NaOH	.17
0.4 N NaOH	.26
0.01 N NaOH	.52
0.001 N NaOH	.39

Figure 10. - Profilometer traverses across wear tracks generated by Al_2O_3 sphere sliding on iron flat in various media. (Wear tracks were made on iron flat.)



(a) Surface profile.
(b) Wear track (low magnific
(c) Wear damage (high magnifica

Figure 11. - Surface profile and scanning electron micrographs of wear track on iron surface sliding against Al_2O_3 sphere in 0.01 N NaOH solution.



(a) Surface profile.
(b) Wear track (low magnification).
(c) Slightly roughened surface (high magnification).

Figure 12. - Surface profile and scanning electron micrographs of wear track on nickel surface sliding against Al_2O_3 sphere in 0.1 N NaOH solution.

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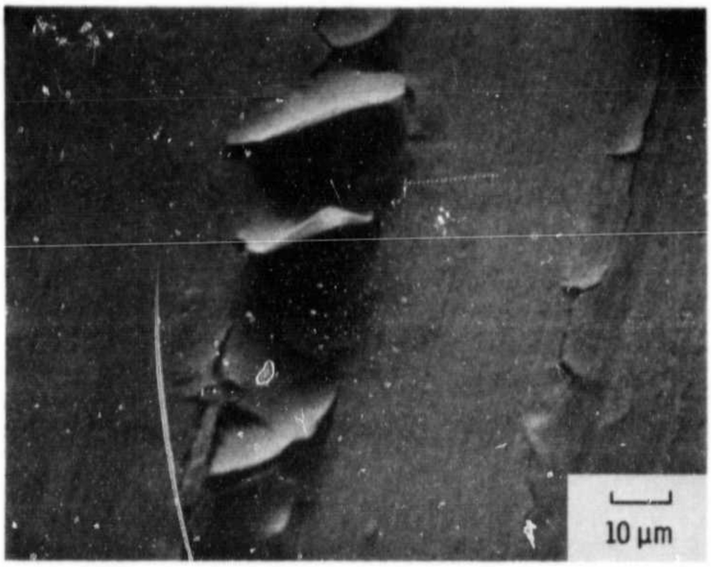
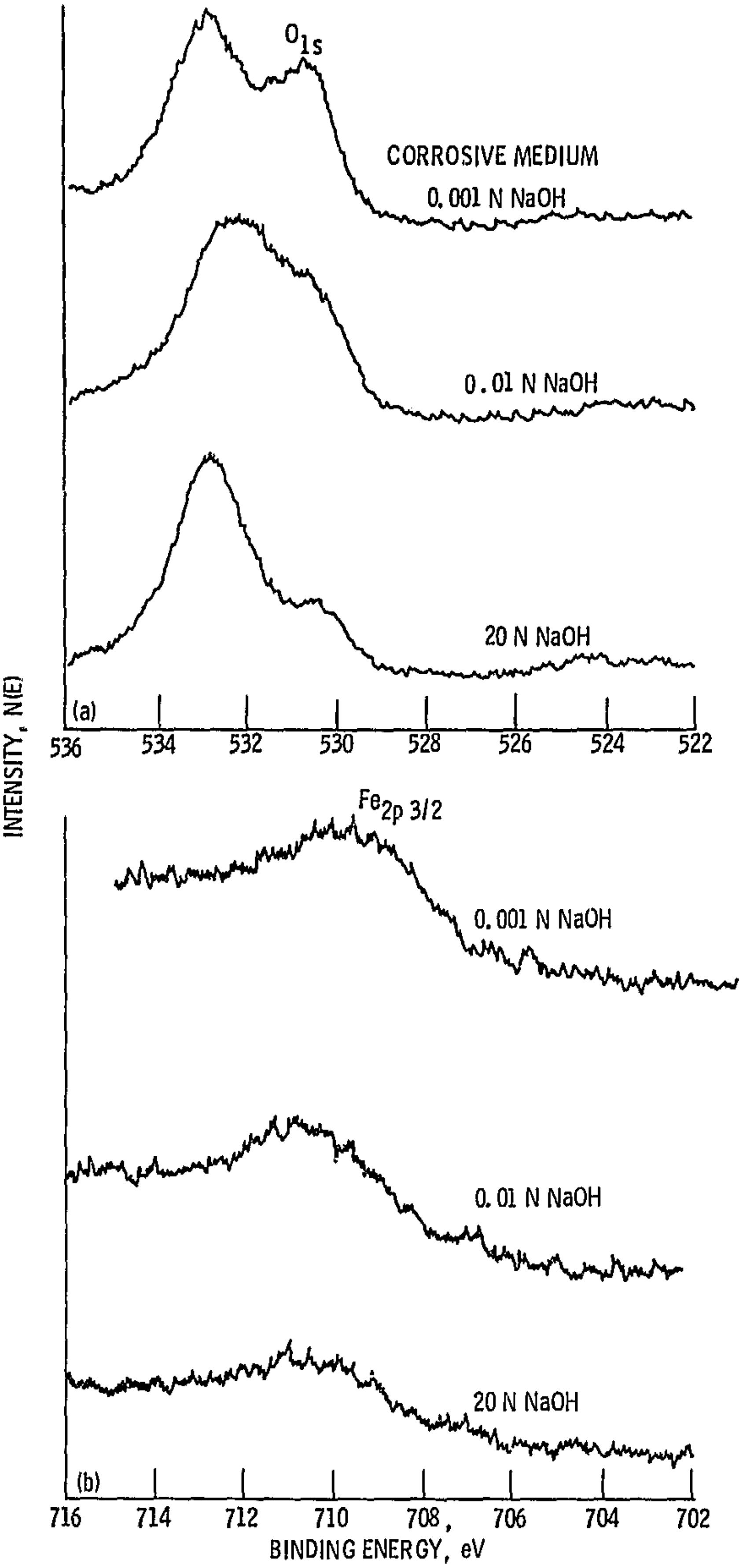


Figure 13. - Scanning electron micrograph of wear track on nickel surface sliding against Al_2O_3 sphere in water. Static coefficient of friction, 0.28.



(a) O_{1s}.
(b) $Fe_{2p\ 3/2}$.

Figure 14. - XPS data of wear surface of iron indicating effect of sodium hydroxide normality on surface chemistry.