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(NASA-TM-X-71659) DYNAMIC SEM WEAR STUDIES OF TUNGSTEN CARBIDE CERMETS (NASA) 35 p HC $3.75 CSCL 11B

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DYNAMIC SEM WEAR STUDIES OF TUNGSTEN CARBIDE CERMETS

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ABSTRACT

Dynamic friction and wear experiments were conducted in a scanning electron microscope. The wear behavior of pure tungsten carbide and composite with 6 and 15 weight percent cobalt binder was examined. Etching of the binder was done to selectively determine the role of the binder in the wear process. Dynamic experiments were conducted as the WC and bonded WC cermet surfaces were transversed by a 50 micron radiused diamond stylus. These studies show that the predominant wear process in WC is fracture initiated by plastic deformation. The wear of the etched cermets is similar to pure WC. The presence of the cobalt binder reduces both friction and wear. The cementing action of the cobalt reduces granular separation and promotes a dense polished layer because of its low shear strength film-forming properties. The wear debris generated from unetched surface is approximately the same composition as the bulk.

INTRODUCTION

The cemented tungsten carbide cermets have been in commercial use since the 1920's. Great impetus for the further development of cermet materials was the recognition of the need for materials with superior strength and high temperature utility for use in gas-turbine blading

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at the close of the second world war. Further development was prompted by the requirements of nuclear power systems. Considerable progress has been made both in concept and development of a wide range of cermet materials exhibiting superior strength and high temperature properties, (ref. 1).

The combination of strength and hardness found in these cermet materials made them an attractive choice for extensive applications in tooling such as wire drawing dies and rotary drilling tools. Further applications of these materials were based on their hardness and particularly wear resistance, notably in bearings, bushings and seal mating surfaces. In application such as dies or bushings, wear of the cermet, will to a large degree determine the effective life of such a component. It is thus important to understand cermet wear behavior. The wear behavior of cermets can be complicated by the multiphase nature of the material, and so macroscopic wear studies may not be sufficient to illucidate wear mechanisms. Microscopic studies of the wear of cermets as a compliment to existing macroscopic studies may prove useful.

The use of the scanning electron microscope (SEM) as a device to study the wear process on a microscopic scale is well recognized, (refs. 2 and 3), and has been applied in this laboratory to study the wear process dynamically (ref. 4). Dynamic studies are inherently better than static or post-testing examination because of the dynamic nature of the wearing process itself as well as affording considerable reduction in interpretive difficulties.

The objective of this investigation was to examine by means of in situ dynamic SEM studies the wear behavior of tungsten carbide and cobalt bonded tungsten carbide **cermets and to determine the role the** individual phase in the overall wearing process.

APPARATUS

The friction and wear experiments were conducted in the vacuum chamber of the SEM. A commercial SEM was modified by the addition of a friction and wear apparatus. The SEM is a field emission electron source type which provides for real time viewing on a closed circuit television system at 15 complete frames per second.

The specimen chamber is mechanically fore pumped and ion pumped to a pressure of 10^{-6} to 10^{-7} torr at which pressure the experiments were conducted.

The friction and wear apparatus used for these experiments is shown schematically in figure 1. A disk specimen 1.9 centimeters in diameter is mounted on an adapter to the rotary specimen feedthrough. The surface of the disk is inclined at approximately 70° with respect to the electron beam. This steep angle permits the interface to be viewed from a near side view. A variable speed electric motor and gear train is attached to the external rotary specimen feedthrough to provide rotation of the disk from 0.001 to 5 RPM. The rotation can either be clockwise or counterclockwise to provide for SEM observation of either the prow or wake of the rider disk contact. In addition, a side view of the wearing process can be observed, however, without the capability of friction force measurements.

The stylus is mounted in an arm which can be moved in and out as well as up and down and laterally by means of a bellows and gimbal system. The gimbal system is comprised of a precision optical orientor which is mounted on a translational stage. This stage and the optical orientor are micrometer controlled thereby allowing very precise positioning of the rider on the disk under the scanning beam.

The arm in which the stylus is mounted contains two flex bands of beryllium-copper upon which strain gages are attached. One band, mounted parallel to the disk surface is used to measure the normal load applied to the contact. The other band and strain gage which are mounted perpendicular to the disk surface are used to monitor the friction force. In addition, another strain gage is mounted on the bottom of the loading beryllium-copper band and can be used to measure the force of adhesion between the rider and disk. The normal loading is accomplished by allowing the magnets which are mounted on the optical orientor ring to pull the arm downward to the disk surface.

The strain gage output of the load sensing gage is amplified and displayed on a digital millivolt meter which with suitable calibration provides for a direct reading of the load being applied. The friction force gage is read out either on a strip chart recorder or oscilloscope to provide for the observation of the friction trace which is more transient in nature.

The entire friction test is viewed on the television monitor of the SEM and the video signal is recorded along with audio comments on video tape to provide for data recording. The tape can be played back in slow motion and stop action to facilitate interpretation. In addition kinescopic motion pictures can readily be made from the video tape.

To provide for an analysis of the wear track, an energy dispersive X-ray analyzer is mounted on the SEM. The analyzer has 400 channels and a resolution of 170 electron volts. Also provided is the capability of elemental mapping.

MATERIALS

The materials used in this investigation were purchased commercially. Sintered tungsten carbide (binderless) and sintered cobalt bonded tungsten carbide of 6 and 15 weight percent binder were obtained. The 6 percent cobalt bonded tungsten carbide had a density of 15.9 grams/cm^3, hardness of C-71 (Rockwell) and a mixed grain size. The 15 percent cobalt bonded tungsten carbide had a density of 14.4 grams/cm^3, hardness of C-78 (Rockwell) and an intermediate grain size. Disks of 1.9 centimeter diameter were electric discharged machined from larger stock. Following machining, the disks were heat treated for several hours at 450° C and then slowly furnace cooled. The disks were then polished with 6 and 3 micrometer diamond paste. After polishing, the disks were etched in nital, if desired. Figure 2 presents photomicrographs of the 6 and 15 percent cobalt binder cermets after a light etch. Prior to mounting on the rotating stage of the SEM, the disks were ultrasonically cleaned with acetone, then with a final rinse of ethyl alcohol.

The diamond styli were also commercially purchased. They consisted of small diamonds with 75° taper down to a radius of 50 micrometers. The diamonds were mounted in a steel shaft which was machined down on a lathe to 1 mm in diameter to accommodate it in the rider specimen holder of the SEM friction device.

RESULTS AND DISCUSSION

In order to provide a basis on which to separate out the role of the individual phases in the cermet wear process, a series of runs was made with a pure tungsten carbide specimen. The mode of wear of this hard phase could then be determined. The pure tungsten carbide was subjected to single and multipass sliding by a 50 micrometer radiused diamond stylus in the scanning electron microscope at loads up to 50 grams.

Figure 3 is a micrograph of the wear track being generated by the diamond stylus at a load of 50 grams during a single slow speed pass (~ 20 micrometers/second). The sliding direction is from right to left on this micrograph. Pronounced fracturing of the carbide surface occurs to the front and sides of the contact region. When observed dynamically, cracks can be observed to propagating outward from under the stylus. In certain instances, slip bands can be observed forming prior to the crack initiation. Both large (several microns in dimension) and small fragments (submicron in dimension) are generated indicating both inter and intragranular fracture are occurring. Figure 4 presents micrographs of the wear tracks after single passes at 50 grams load. It is obvious that a significant degree of plastic deformation occurs in what is generally considered to be a brittle material. Plastic behavior is indicated by the presence of slip bands which extend outward from the track. Cracks are also observed in the track propagating subsurface.

Plastic behavior of a covalent material, normally considered very brittle, is not unusual under conditions of sliding contact. Bowden and Tabor have observed similar plastic behavior of nominally brittle materials such as ionic salts, (ref. 5). Also, single crystal sapphire exhibits plastic behavior under sliding contact, (ref. 6).

It has been suggested that the plastic behavior of otherwise brittle materials is due to the high compressive stresses that exist during sliding and that if the stresses are too small, the material will behave in a brittle manner. With tungsten carbide, single passes made with loads down to 5 grams showed plastic behavior. No evidence of a change in mechanism with load could either be visually observed or inferred from friction force measurements.

The fracturing of the carbide surface is the result of cracks propagating and intersecting other cracks in grain boundaries. There are two distinct phases of the fracturing process, namely; 1) crack initiation and 2) propagation. It seems likely that plastic deformation of the carbide initiates fracture by the creation of microcracks during the slip process. Studies on the deformation behavior of tungsten carbide crystals show that slip can be nucleated by bending and micro-indentation. The operable slip system identified for tungsten carbide in $\{01\bar{1}0\}$ $\langle 2\bar{1}\bar{1}3 \rangle$, (ref. 7). Further microscopic studies by Luychx et al., have shown that during fracture tests with Co-WC alloys, plastic deformation of the carbide precedes and follows fracture on the microscopic scale, although not observable **macroscopically** (i.e., linear stress-strain curves), (ref. 8). Further evidence shows that intracrystalline fracture can originate from slip line intersections, and that cleavage often occurs, (ref. 9). Such cleavage cracks were observed in this study at the intersection of slip bands ahead of the moving diamond stylus.

Following the single pass experiments, multipass experiments were conducted to establish long term or equilibrium wear and friction behavior. Long term experiments at loads of 10, 20, and 50 grams were run. At 10 grams load very little surface disruption occurs. The surface damage is primarily plastic flow. At increasing loads, considerable fracture of the surface occurs with the consequent generation of debris. The repeated sliding over the same surface leads to increased fracture as newly generated cracks propagate to intersect existing cracks. Further fragmentation creates smaller and smaller particles. These small particles undergo extensive plastic flow. The net effect is to create a surface which is smooth and dense. Although the track may still contain occasional fracture sites. There is much less likelihood of fracture occurring as it did in the first pass. Plastic flow increases the area of contact between the sytlus and tungsten carbide causing a significant reduction in stress levels. During this polishing phase the friction coefficient increases to its equilibrium value of 0.6 to 0.7. The equilibrium wear behavior of tungsten carbide is in agreement with the observations of Billinghurst et al., for covalent crystalline solids (ref. 10).

After running the pure tungsten carbide, two cermets with cobalt binder of 6 and 15 weight percent were examined. Both cermets had approximately the same particle size. The cermets were examined in two ways, with the binder present and with the binder etched away prior to running. Etching the binder had the effect of creating an open skeleton of carbide particles. The etching away of the binder is of practical importance because depletion of binder materials in cermets can occur both by chemical attack and physical mechanisms (e.g., erosion in rotary drilling tools, (ref. 11)). Such binder depletion can be expected to markedly alter the wear behavior of a cermet material. Dynamic experiments were conducted with slow speed sliding of a diamond stylus on both types of surface at leads from 10 to 50 grams.

The etched cermet surfaces were examined first. In general the etched cermet surfaces wore in a manner comparable to the pure carbide. **Figure** 5 shows a micrograph of the stylus sliding over the etched cermet surface at a load of 50 grams. Again sliding direction is from right to left. Pronounced fragmentation of the carbide skeleton occurs at the front and sides of the contact region. The fracture is primarily intergranular in nature although considerable intragranular fracture also occurs. The fracturing is more extensive for both the cermets than for the pure tungsten carbide. This, of course, is related to the fact that the grains in open skeleton are more easily separated from adjacent grains due to the unbonded areas inherent in the open structure. **Figure 6** presents photomicrographs of wear tracks created by a single pass at loads from 3 to 50 grams. As with the pure carbide, plastic behavior is evident over all loads. The open regions have been smeared over by the plastic flow of the carbide. Note on the 30 gram track the carbide grain raised out of the surface some 2 micrometers from the edge of the track.

Passing over the same track produces more granular separation with the eventual development of a furrow and the accumulation of significant amounts of debris both on the stylus and on the edges of the track, figure 7. This process continues until the underlying cobalt binder material is exposed. When the binder material is reached, the rate of wear decreases drastically. Figure 8 presents surface profilimeter tracings of the wear tracks for the pure carbide and for the cermet both lightly and severely etched All the tracks were generated under the same conditions of load, speed, and time. Energy dispersive X-ray analysis was used to monitor the cobalt concentration in the wear track as wear occurred. The profile of the wear track on the lightly etched cermet is of the same magnitude as the pure carbide. Cobalt was observed in the tracks after several passes for loads of 10, 20, and 50 grams, indicating that the cobalt was etched to 1-2 micrometers depth. For the severely etched cermet, no cobalt was observed in the 10 gram track which was 20 micrometers deep. Cobalt was detected in the 20 gram track which was 24 micrometers deep. The 50 gram track was also 24 micrometers deep which indicated that once the cobalt was reached, the wear rate dropped drastically and the total wear for the 20 gram and 50 gram loads was comparable.

The effect of reaching the cobalt was also evident in the friction measurements. Figure 9 is a curve showing how the friction coefficient drops as more and more cobalt is exposed. The friction reaches an equilibrium value at a CoKα to background ratio of approximately 1.4. The same effect could be observed with time. Figure 10 is a plot of friction versus time for the etched cermet at loads of 10 to 50 grams. During the 10 gram load run, the friction starts near .7, rapidly increases to unity and remains there the entire 110 minutes. The cobalt ratio in the track at the end of the run was 1.0 indicating the cobalt level had not been reached. For the 20 gram load, the friction drops after 80 minutes and eventually decreases to the equilibrium value at the end of the run. The final cobalt ratio was 1.6. With the heavier load, sufficient wear occurred so that the underlying cobalt was eventually exposed. For the 50 gram load, the wear rate is sufficiently high such that the cobalt is reached in the first 30 minutes. The final ratio for the 50 gram load was 2.3.

A similar effect was observed with the 6 percent binder cermet as shown in figure 11. Because of the lower binder to carbide ratio, subsurface etching of the binder does not occur as readily. Therefore, even at a 10 gram load a small concentration of cobalt is present and the friction coefficient is slightly less than the unity value. The final ratio was 1.2 and this concentration is in the transition range of figure 10. Both the 20 and 50 gram load runs obtained the equilibrium friction coefficient from the start. Both the 20 and 50 gram showed ratios of CoKα to background of 1.4.

Following the runs with the etched cermets, unetched surfaces were used under the same conditions. The wear behavior of the unetched cermet is demonstrated by **figure 12.** In contrast to the etched cermet, very little granular fracture is observed to occur. The wear debris generated consists of thin sheets of small carbide particles held together by the cobalt binder. The single pass wear track is smooth and generally crack free. Similar behavior is observed with both the 6 and 15 percent binder materials. During repeated sliding both materials exhibited near identical friction behavior as is seen in **figure 13.**

The initial friction coefficient is highest for the 50 gram load and lowest for the 10 gram load in both cases. With time, all the curves converge to an equilibrium value of approximately 0.6 to 0.7. The heavier load runs reach this equilibrium value first. The friction value is the same as the equilibrium value obtained for the pure tungsten carbide. This suggests that although the cobalt must be present to achieve the lower equilibrium friction and wear, it may not be the dominant phase at equilibrium.

Both the wear tracks and wear debris from the unetched surfaces were examined by X-ray to determine if any appreciable deviation of binder to carbide concentration occurred. All wear tracks showed a slight increase in cobalt intensity (up to 20 percent) over the unrun surface but this small increase can be attributed to the smearing of the pockets of cobalt by plastic flow thus exposing more cobalt on the surface to the scanning electron beam. Cobalt Kα X-ray maps confirmed a uniform smearing of the binder in the tracks. The wear debris also showed higher cobalt concentration than the bulk, **figure 14.** In figure 14, the spectrum were taken at equal intensities of the tungsten Lα line at 8.39 Kev in order to better compare the cobalt peak at 6.92 Kev. The dots indicate the intensity from the debris while the bars represent the bulk surface. The integrated peaks show the debris to have about 22 percent higher cobalt intensity which is comparable to the track values. The debris has the same approximate composition as the bulk considering the 22 percent higher value equilibrium due to smearing. This is in agreement with the work of Golden, et al., who determined by autoradiographic methods the composition of wear debris generated by cobalt bonded tungsten carbide sliding on metal surfaces (refs. 12 and 13).

The presence of cobalt in the wear track is necessary for low wear to occur. Without the cobalt, microspalling of the carbide grains continues with each pass of the stylus. With cobalt present, however, there is considerably less tendency for carbide grains to separate from the surface. Rather the wear track will become polished in appearance with the formation of a dense plastically flowed film of both cobalt and tungsten carbide. This is illustrated in figure 15. Presented are the wear tracks produced by the diamond stylus on the severely etched 15 percent Co-WC cermet in loads of 10, 20 and 50 grams. In the track produced by the 10 gram load, the track consists of loose carbide particles which have been fragmented to an increasingly smaller size in comparison to the unrun areas, figure 16. The 20 gram and 50 gram wear tracks show the polished appearance that is obtained when the binder is reached.

It appears unlikely that the presence of the binder at these concentrations will significantly lower the stress levels in the contact region thereby reducing the tendency for crack initiation and propagation. Work on deformed Co-WC cermets have shown that with cermets up to 15 weight percent cobalt, the stresses still are highly localized and that most deformation is initiated in the carbide phase. For higher cobalt concentrations (> 25 percent) the binder phase begins to reflect the bulk of the deformation and stresses are distributed over a greater distance by three orders of magnitude. It is suggested that this behavior is due to the formation of a continuous skeleton structure of tungsten carbide up to binder concentrations of 10-15 weight percent and so long as there is continuity of this structure the load will be transmitted through it by the contact of one carbide given against another, (refs. 8 and 9). Other investigators claim that the carbide particles are separated from one another by an extremely thin layer of cobalt at concentrations as low as 0.5 percent and that the cobalt layer is restrained from plastic flow by the carbide particles, and it is this restraint which accounts for much of the increase in mechanical strength in the cemented carbides, (refs. 14 and 15). In actuality, the structure of cemented carbide probably lies between these two extremes; the predominance of one or the other strongly dependent on processing history, (ref. 16). The observable effect of the cobalt with regards to friction and wear behavior seems to be related more to the cementing action it affords rather than alteration of local mechanical properties. With the binder etched out, intergranular separation occurs more readily because of the smaller total bonding force acting on each carbide particle. With the cobalt present, less intergranular separation occurs and the wear debris generated consists more of fragments broken off of the carbide grains themselves, i.e., intragranular fracture. With subsequent passes of the stylus, these fracture grains are plastically flowed with the binder to form a dense film. Here in addition to the cementing action, the lower shear strength of the binder provides for easier slip of carbide particle over carbide particle so that the polishing can occur. Without cobalt present to retain the fragments in the wear track, subsequent passes will easily dislodge them and continually expose new surface leading to excessive wear rates.

SUMMARY

In view of the preceding discussion, the following observations can be summarized.

1. The wear behavior of pure tungsten carbide is characterized by both fracture and plastic flow. The evidence suggests that during sliding, fracture is initiated by plastic deformation of the tungsten carbide.

2. The wear behavior of the cemented carbides (6 and 15 weight percent cobalt binder) is similar to pure tungsten carbide but more severe in degree when the cobalt binder is etched away. When the cobalt is present the primary mode of wear changes from intergranular separation to intragranular fracture and plastic flow.

3. The effect of the binder (cobalt) on the friction and wear behavior of 6 and 15 percent bonded cermets is more related to the cementing properties and low shear it provides rather than changes in

local stress distributions or mechanical properties.

4. The wear debris generated from the unetched cermets surfaces, during abrasive wear had the same approximate composition as the bulk.

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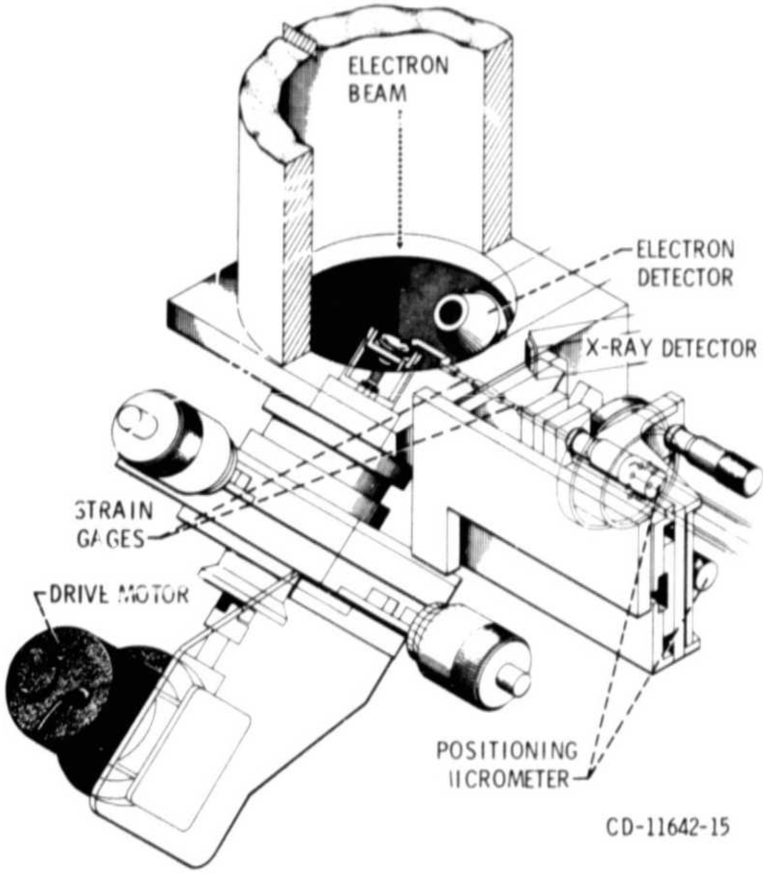

Figure 1. - Detailed drawing of friction apparatus mounted on scanning electron microscope.

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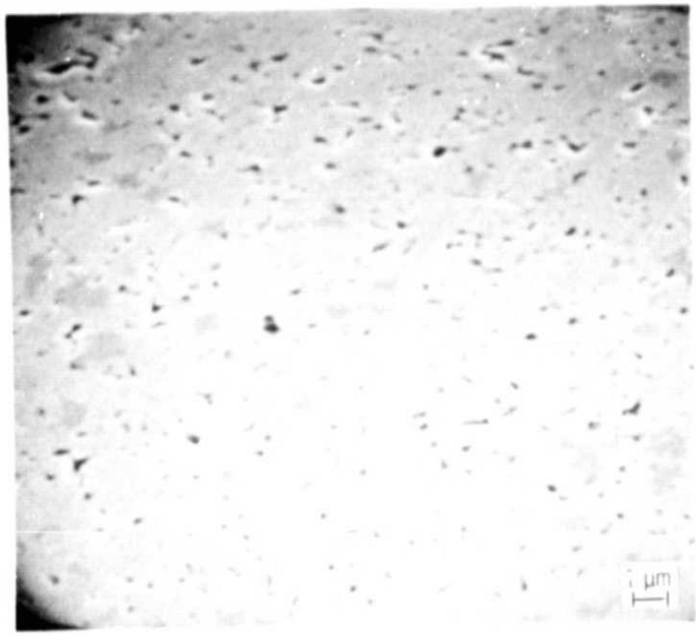
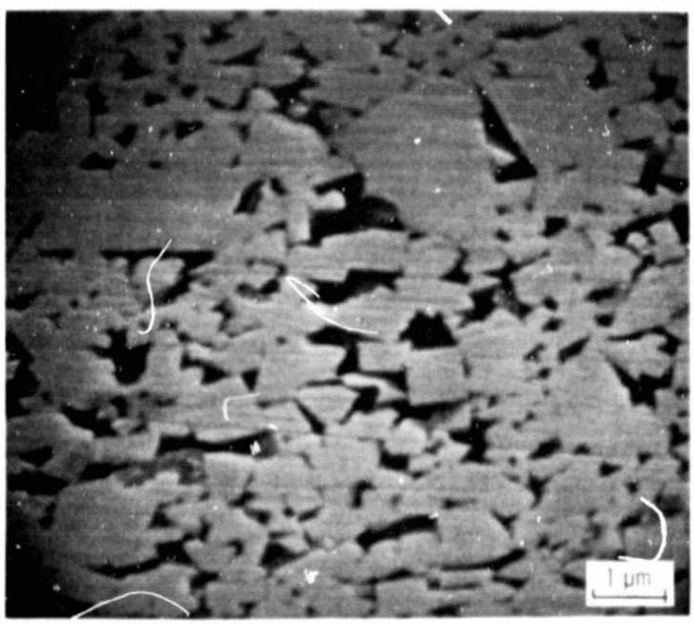


(a) 6 PERCENT COBALT BINDER.



(b) 15 PERCENT COBALT BINDER.

Figure 2. - Surface micrographs of polished and etched Co-WC.

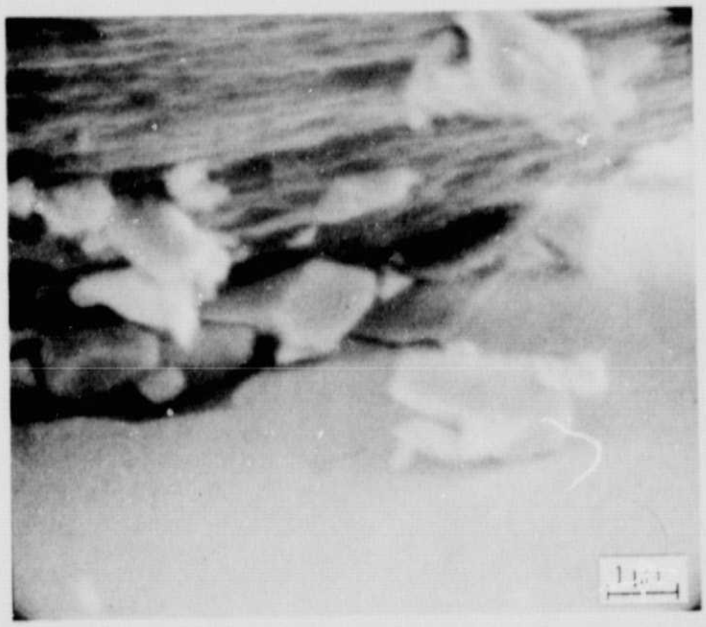


Figure 3. - Photomicrograph of tungsten carbide as 50 μm diamond stylus slides over it at 50 gram load.

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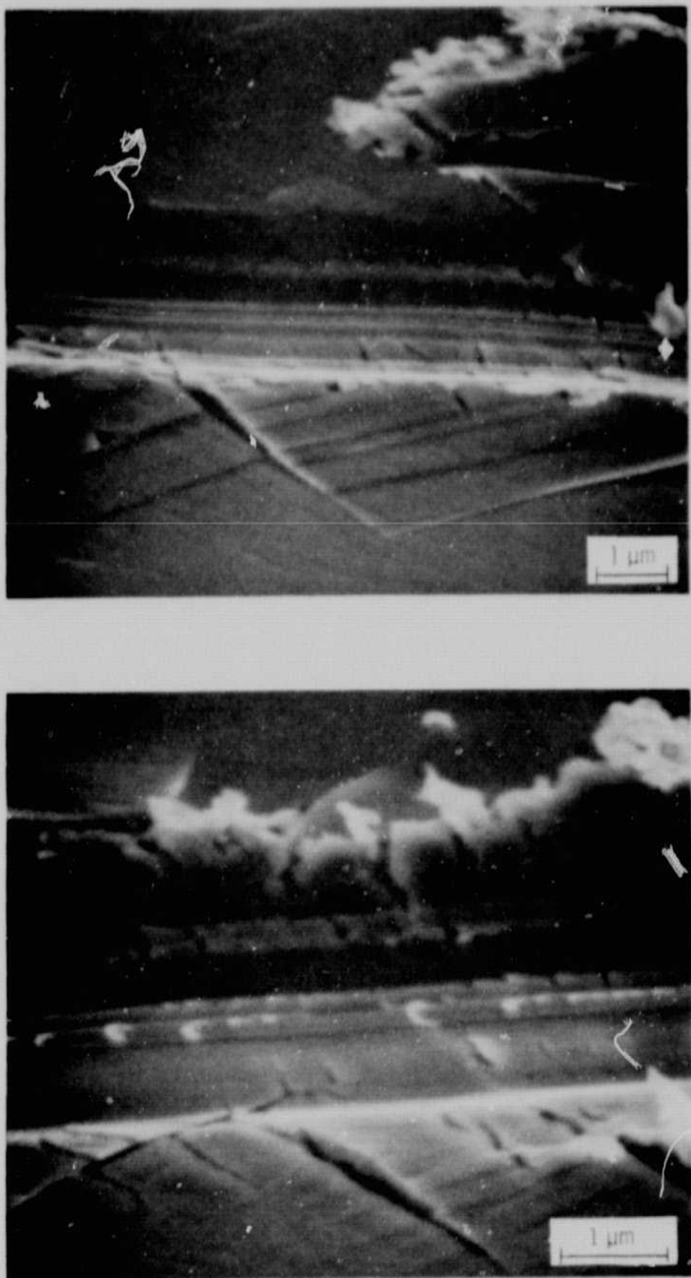

Figure 4. - Single pass of 50 µm diamond stylus on WC.

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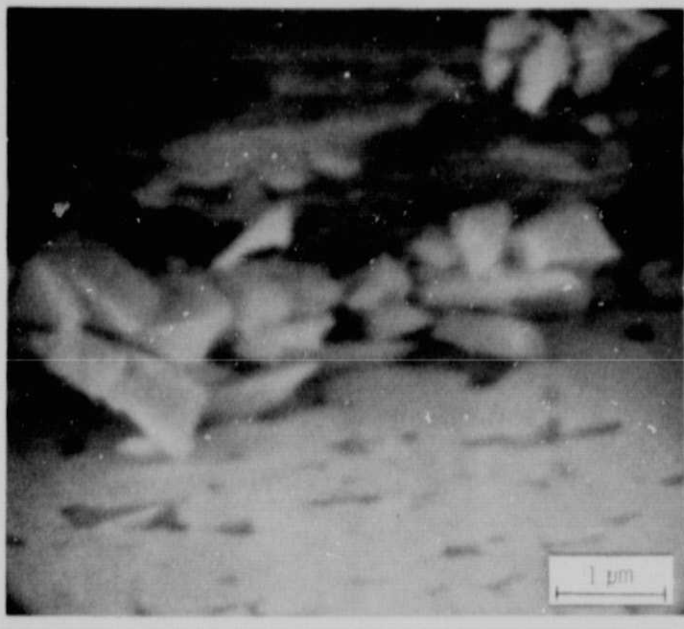

Figure 5. - Photomicrograph of 15 percent Co-WC as 50 μm diamond stylus slides over it at 50 gram load, surface etched.

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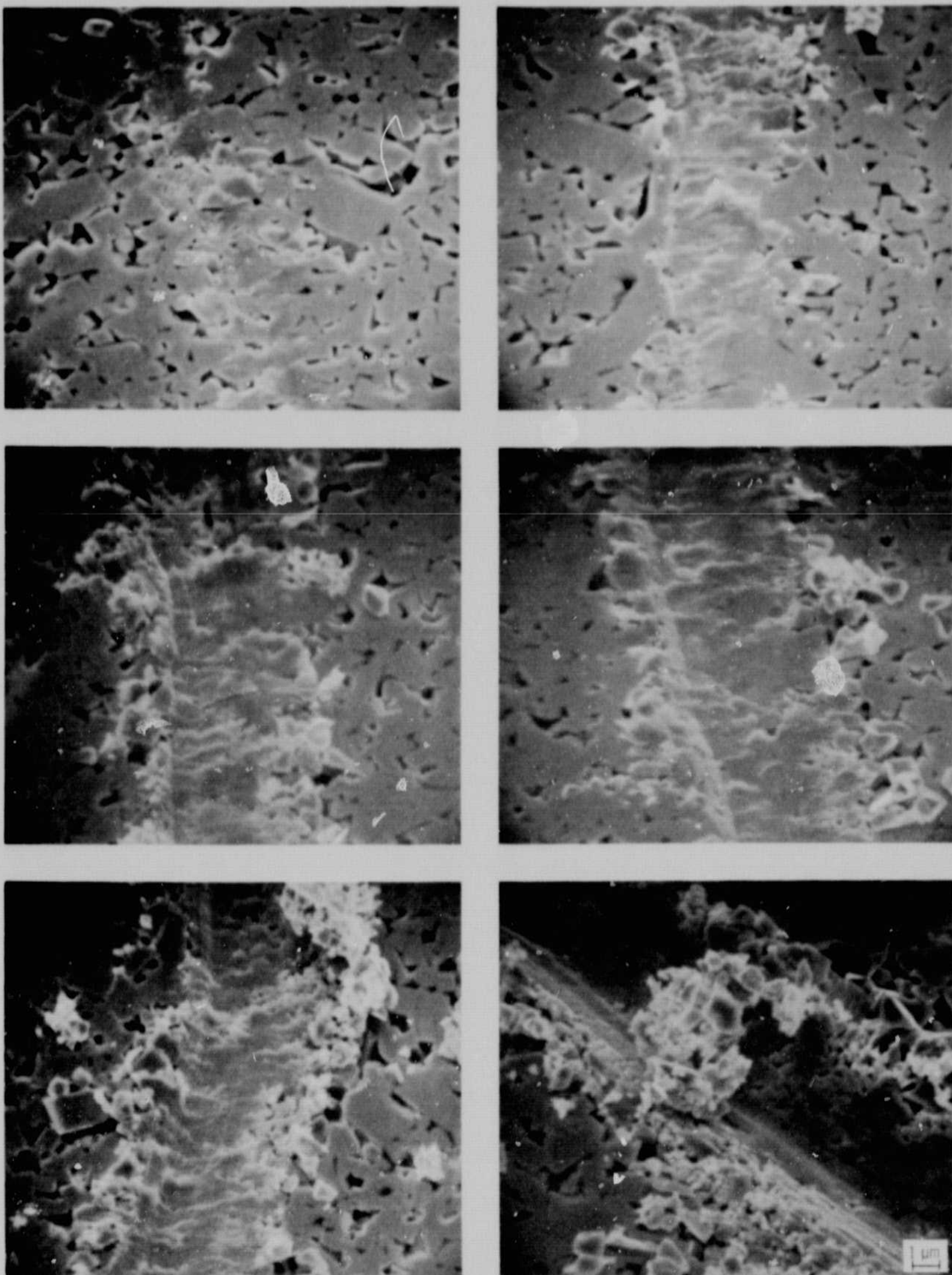

Figure 6. - Photomicrographs of single pass wear tracks.

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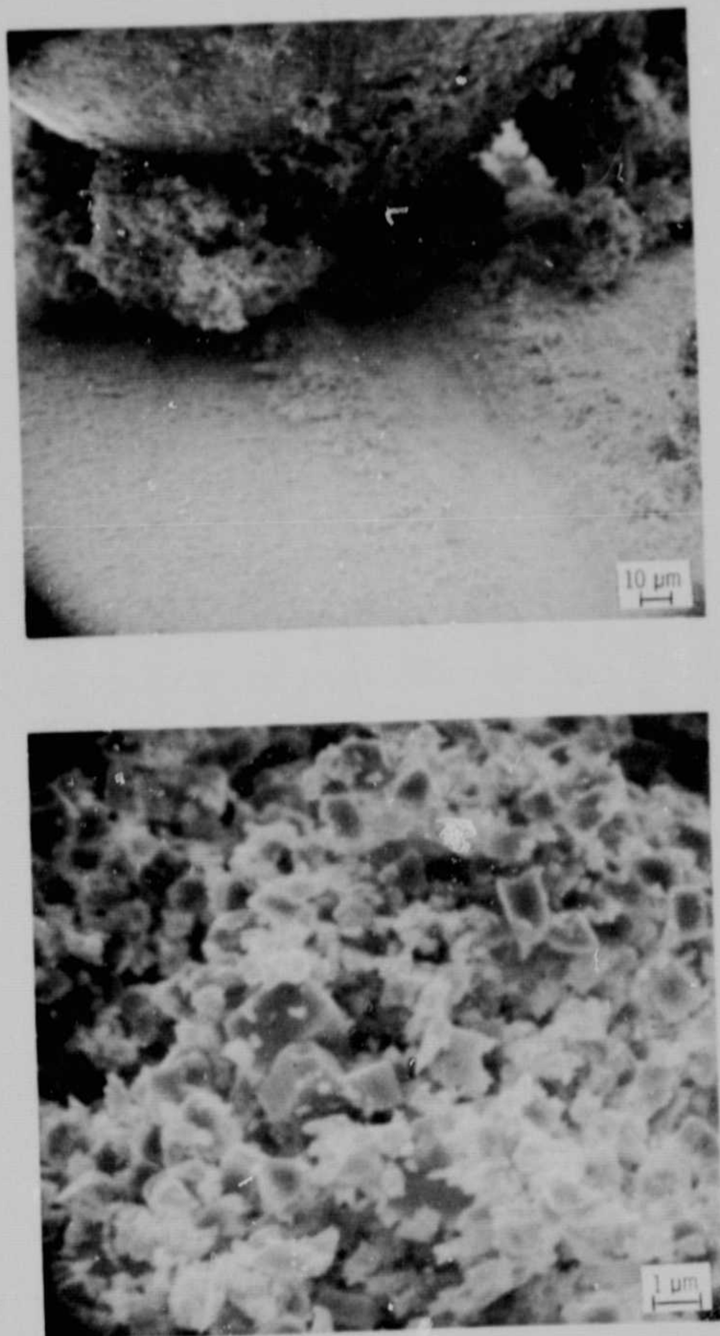

Figure 7.

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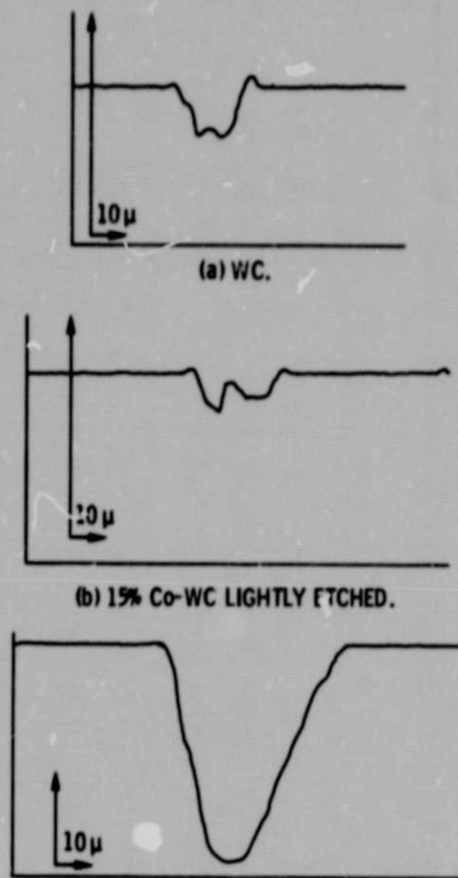

Figure 8. - Surface profile tracings of wear tracks made by diamond stylus at 50 gram load, 110 minutes duration.

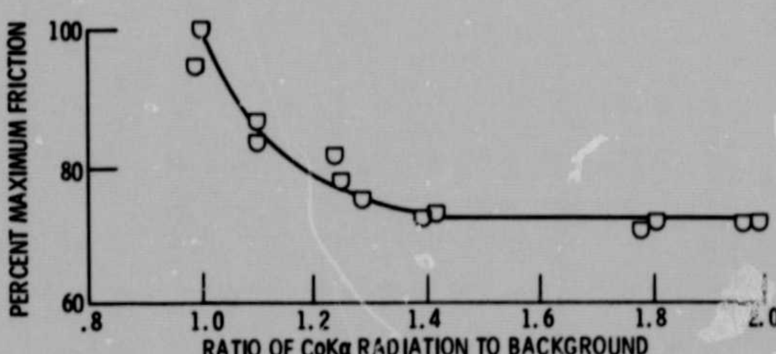

Figure 9. - Friction as a function of cobalt X-ray counts for 15% Co-WC with 50 gram load. Specimen polished and etched.

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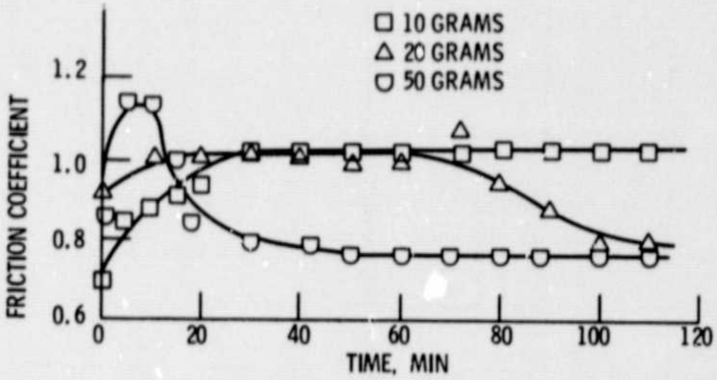

Figure 10. - Friction coefficient as a function of time for 15% Co-WC at 10, 20, and 50 gram loads. Specimen polished and etched.

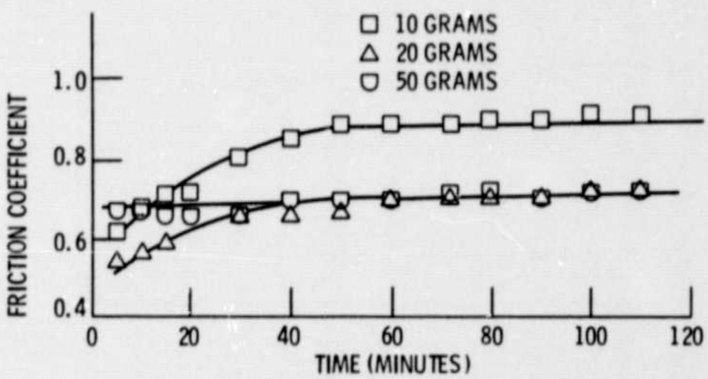

Figure 11. - Friction coefficient as a function of time for 6% Co-WC at 10, 20, and 50 gram loads. Specimen polished and etched.

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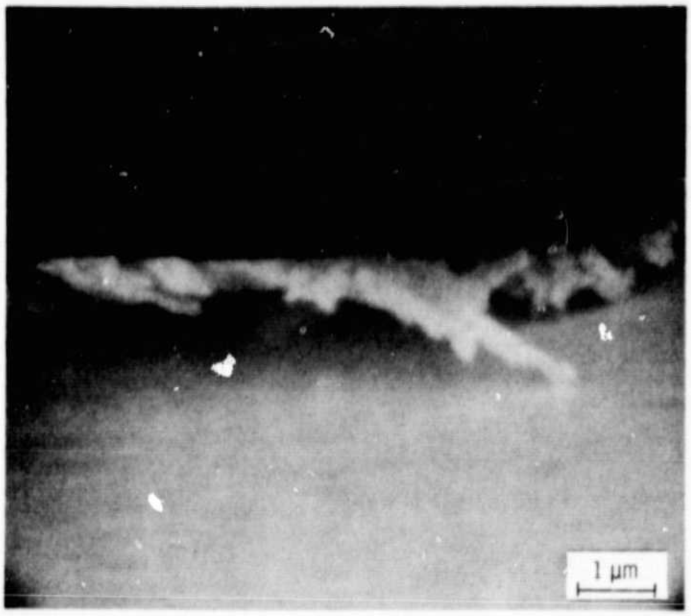


Figure 12. - Photomicrograph of 15 percent Co-WC as 50 μm diamond stylus slides over it at 50 gram load, no etching.

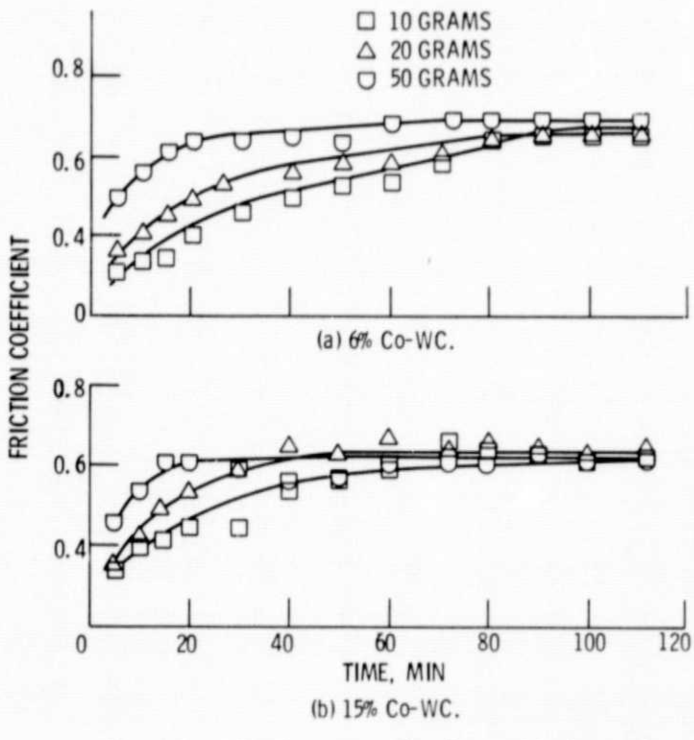
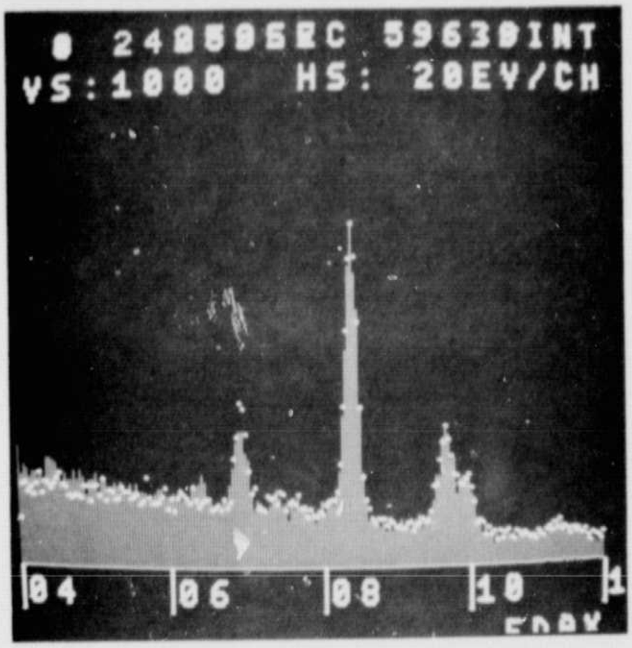
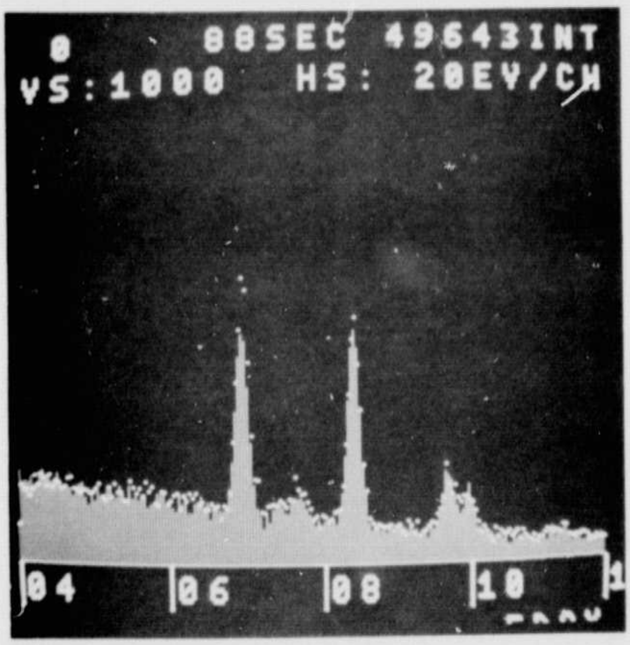


Figure 13. - Friction versus time for unetched cermets.



6 PERCENT Co-WC



15 PERCENT Co-WC

Figure 14. - Energy dispersive x-ray profiles of wear debris from unetched surfaces compared to bulk surface profiles.

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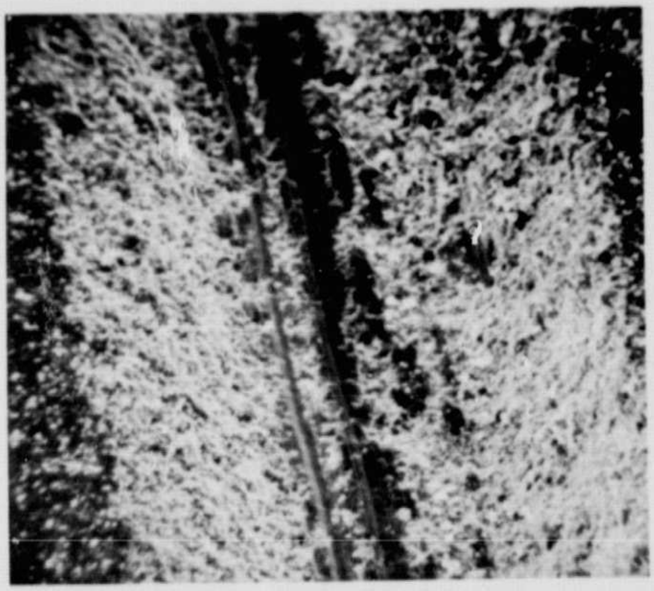
(a) 10 GRAMS.



(b) 20 GRAMS.

(c) 50 GRAMS.

Figure 15. - Wear tracks on 15 percent Co-WC after 110 minute runs at 10, 20, and 50 gram loads. Surface polished and etched.

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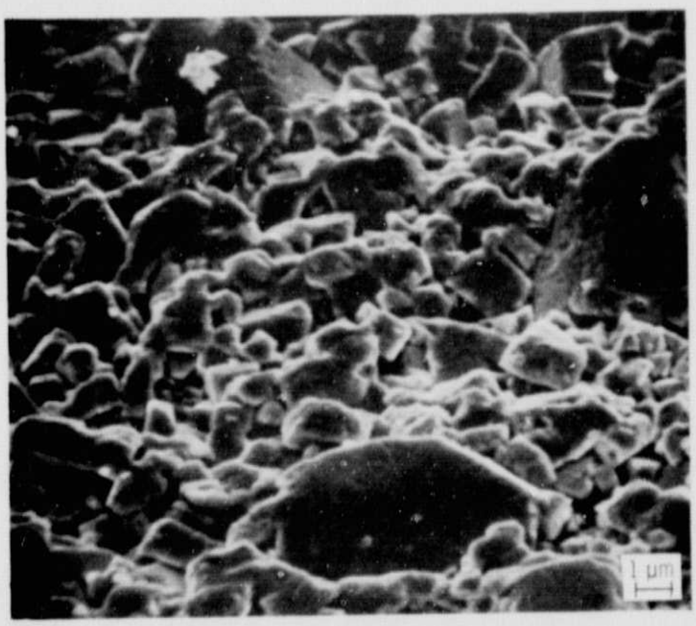
OF POOR QUALITY

(a) IN-TRACK.



(b) OUT OF TRACK.

Figure 16.