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Experimentally Determined Wear Behavior of an Al_2O_3-SiC Composite from 25 to 1200 °C

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ABSTRACT

The sliding wear behavior of a self-mated alumina-silicon carbide whisker toughened composite was studied using optical, scanning electron (SEM) and transmission electron (TEM) microscopy. Because of its excellent strength and toughness properties this composite material is under consideration for use in heat engine applications for sliding contacts which operate at elevated temperatures. The composite's wear behavior and especially its wear mechanisms are not well understood.

Pin-on-disk specimens were slid in air at 2.7 m/sec sliding velocity, under a 26.5-N load, at temperatures 25 to 1200 °C. Pin wear increased with increasing temperature. Based upon the microscopic analyses, the wear mechanism seems to be loosening of the reinforcing whiskers due to frictional and bulk heating. This leads to whisker pullout and increased wear.

INTRODUCTION

The successful application of ceramics as triboelements at elevated temperatures depends upon a full understanding of their wear behavior. This is especially true when ceramics are applied in high temperature sliding contacts such as cylinder liners for Low Heat Rejection (LHR) diesel engines or gas turbine seals (Ref. 1). In order to predict and possibly improve friction and wear characteristics of ceramics at high temperatures, the wear mechanisms must be understood.

Much work has been done to model the wear of ceramics (Refs. 2 to 5). However, these models usually assume room temperature behaviors, i.e., wear occurs solely or predominantly through brittle fracture and subsequent particle removal. When ceramics are slid at high temperatures or at high speeds and loads, which result in high surface temperatures (Ref. 6), other effects may become important. Some of these effects are: plastic deformation, tribochemical reactions, thermal-mechanically induced phase or structure transformations or other behaviors which alter the wear behavior. Unfortunately, it is very difficult to analytically predict which effects will dominate wear behavior during sliding under anticipated use conditions. Therefore experimental techniques are frequently employed to determine the wear behavior of materials.

Determination of the wear mechanism becomes much more complex when composite ceramics are considered because more than one material system is present.

There may also be reactions between constituents in the composite itself. It is, however, beneficial to study such complex, composite systems due to their superior tribological and mechanical performance. One such complex system is an alumina-silicon carbide (Al_2O_3-SiC) whisker composite (Ref. 7).

Al_2O_3-SiC is a ceramic composite made from an alumina matrix reinforced matrix with up to 30 vol % single crystal SiC whiskers. The whiskers act as barriers to or deflectors of crack propagation thereby improving the composite's physical properties, namely strength and toughness (Ref. 8).

The tribological properties of this composite material are also superior to monolithic alumina (Refs. 9 and 10). Since this composite has good mechanical strength and exhibits low wear, it is a candidate for heat engine sliding applications and metal cutting machining operations. Little is known, however, about the mechanisms responsible for its high temperature wear behavior.

Therefore, this paper describes a research program to experimentally determine the wear mechanisms of an Al_2O_3-SiC composite using a pin-on-disk tribometer from 25 to 1200 °C. By studying the wear surfaces with optical, scanning electron (SEM) and transmission electron microscopy (TEM), the most probable wear modes or wear mechanisms for the material will be determined. This information will be helpful in further improving this composite's high-temperature wear properties and in more accurate wear modeling.

MATERIAL

The composite material studied contains 75 vol % Al_2O_3 with 25 vol % SiC whiskers. Table I gives the material's detailed composition and manufacturer's strength property data.

The composite is made by hot pressing high purity (>99.5 purity) alumina powder mixed with single crystal SiC whiskers. During consolidation, most of the whiskers preferentially align themselves in a plane perpendicular to the pressing direction (Ref. 8). The pins and disks tested in this study have their rubbing surfaces parallel to the whisker planes. See Fig. 1.

The whiskers are single crystal SiC with length-to-diameter ratios of 10 to 60 and diameters of ≈0.75 μm. The matrix grain size is ≈2 μm and the material is hot pressed at ≈1600 °C. The maximum tribological test temperature used in the present study is 1200 °C.

Figure 2 shows some TEM micrographs of the virgin material. From this figure it can be seen that there is little residual porosity and that there is good contact between the matrix and the whiskers, i.e., there are no large voids between the whiskers and the matrix. Figure 2(a) shows a lengthwise cross-section of a whisker and Fig. 2(b) shows a whisker end on.

Wear pins, 0.476 cm in diameter and 2.5 cm long are made from the composite. Hemispheres of 2.54 cm radii are machined on the pin ends and are diamond polished to ≈0.1 μm rms surface finish.

The wear disks are 6.35 cm in diameter and 1.25 cm thick. The faces are diamond polished to an ≈0.1 μm rms surface finish.

APPARATUS AND PROCEDURE

The specimens were tested in a high temperature pin-on-disk tribometer. With this apparatus, the pin is held in a torque tube and is loaded against a rotating disk. The disk specimens are mounted on a ceramic spindle which is rotated by a variable speed DC motor. Sliding velocity during these tests was 2.7 m/sec (1000 rpm). The rig has a well insulated SiC glowbar furnace capable of heating the specimens to 1200 °C. The test atmosphere was ambient air with

a relative humidity which ranges from 40 to 65 percent at 25 °C. Reference 11 gives a detailed description of the tribometer.

The torque tube which holds the pin specimen is connected to a temperature compensated strain gauge load cell which is connected to the rig table through a flexure. The pin is loaded against the rotating disk by a ceramic load rod. Like the torque tube, the load rod is connected to a load cell and then to a flexure which is connected to the rig support. The flexures allow for minute displacement of the load cells so that the friction and load forces can be measured. See Fig. 3. With this design, the actual applied load and friction force are measured without any parasitic losses encountered with pivots or support bearings. Thus the data from the rig is very accurate.

Test temperature is measured with four proximity thermocouples located ≈1 mm from the disk surface. The test temperature is computed as the average of the four readings. After the furnace temperature stabilizes, the thermocouple readings are within 5 percent of one another. Therefore, temperature readings are considered accurate to within 5 percent. Temperature measurement calibration using contact thermocouples and two color infrared pyrometry indicate that ±5 percent is a reasonable temperature measurement uncertainty.

Prior to testing, the specimens are cleaned with pure ethyl alcohol, rinsed in deionized water, and dried before being mounted in the rig.

To begin a test, the pin is slowly loaded against the rotating disk and data acquisition begins. Since initial hertzian contact stresses for this material combination and geometry at the test loads can be as high as 698 MPa, the first 30 sec of sliding was done at a much lower load, typically 1 N. This lowered the initial contact stress and allowed a wear scar to form on the pin so that at no time did the nominal contact stress exceed the compressive strength (≈500 MPa) of the material which would have caused cracking due to overloading. The final test load, after the brief run-in at 1 N, was 26.5 N.

The tests were typically run for 1 hr for a total sliding distance of 9.7 km. The specimens were then unloaded and the furnace was turned off and allowed to cool. The specimens were then removed from the rig to make wear measurements and to analyze the wear surfaces. The pin wear measurements were made by using optical microscopy to measure the wear scar diameter and then calculating the wear volume. The disk was measured using stylus surface profilometry of the wear track to get an average track cross-section area. The disk wear volume was then calculated by multiplying the average cross-section area by the average track diameter.

To investigate the wear mechanisms of the Al_2O_3-SiC ceramic composite, samples of the pin wear surfaces were prepared and analyzed using both SEM and TEM techniques. For the SEM, the pin samples from the 25, 600, and 1200 °C tests were coated with carbon to prevent charging and then analyzed. For the TEM, thin foils were prepared from the pin wear scars themselves by slicing the worn tip from the pin, then ion mill thinning the foil until a hole was created in the center of the wear scar. Due to the tediousness of fabricating the TEM samples, only pin wear scars from the room temperature tests and the highest temperature tests, 1200 °C, were examined. Standard TEM procedures were then used to examine the pin wear scar. Disk surface specimens were not prepared due to geometry complications which made it too difficult to prepare foils. Also because the pin surface is under continuous sliding it suffers more frictional heating and severe wear conditions than the disk and may provide more information regarding wear mechanisms than the disk surface.

RESULTS: FRICTION AND WEAR - The friction and wear data (for both the pin and the disk) are given in Table II and plotted as a function of temperature in Figs. 4 to 6. The data indicate that only the pin wear significantly varies as

a function of test temperature. Both the disk wear factor and the friction coefficients, remain relatively constant as the test temperature is increased from 25 to 1200 °C. The reasons for this behavior are not clear but may be due to a variety of factors including the development of glassy surface layers and wear debris.

In general, average friction coefficients for the Al_2O_3-SiC composite sliding against itself vary from a low of 0.58 to a high of 0.72 in the range of 25 to 1200 °C. Although these friction coefficients are high, the wear is relatively low when compared to steel sliding against steel or alumina sliding against alumina which have wear factors in the range of 10^{-3} to 10^{-4} $mm^3/N-m$ (Refs. 9 and 12). Disk wear factors for the alumina composites tested here are in the range of 4 to $9x10^{-7}$ $mm^3/N-m$. Pin wear factors show an increase with temperature from $2x10^{-7}$ at 25 °C to $12x10^{-7}$ at 1200 °C. The pin wear factor at 600 °C is highest, $15x10^{-7}$ $mm^3/N-m$. This may represent normal wear data scatter, however, since only two specimens were tested at this temperature whereas more than two specimen sets were tested at the other test temperatures.

RESULTS: ELECTRON MICROSCOPY - TEM analyses of the room temperature pin wear scar show large regions of brittle fracture, individual wear particles, and many cracks. Little or no evidence of plastic behavior (dislocations) was seen. Figures 7(a) to (c) show typical TEM photomicrographs. These micrographs suggest that, under these conditions at room temperatures, the wear mode is conventional, i.e., generalized brittle fracture and subsequent removal of material.

TEM analysis of pin wear scars at 1200 °C exhibit markedly different features. Very few cracks were found, no wear particles were discovered, and some dislocations were detected. See Figs. 8(a) and (b) for representative photomicrographs. Clearly the wear behavior at 1200 °C differs from that at 25 °C.

TEM analysis of the virgin material indicates that it is mostly free from voids, cracks, and dislocations. Therefore, changes in the material after testing can be attributed to effects from the sliding.

SEM analysis of the specimens also indicated that the wear mode is much different at 25 °C than at 1200 °C. At 25 °C, the wear debris outside of the pin wear scar is characterized by short broken SiC fibers and Al_2O_3 matrix particles. Also present are large areas of compacted fine particles, which, at lower magnification, look like plastically deformed areas. See Fig. 9. In general, the room temperature wear surface indicates that the wear mode is predominately brittle fracture of both the matrix and the whiskers.

In contrast, the SEM analysis of the pin wear surface and wear debris from the elevated temperature tests indicates a radically different wear mode. At 600 °C, the pin wear scar shows evidence of whisker pullout. This can be seen as empty whisker pockets or troughs on the pin wear scar. See Fig. 10. Also detected at 600 °C, but not at 1200 °C, were occasional wear debris particles that were in the form of wear rolls. See Fig. 11. These rolls are probably the remnants of a glassy Al_2O_3/SiO_2 surface layer which forms as the oxidation product of the SiC whiskers and the Al_2O_3 matrix. As wear takes place, this glassy layer debonds and is rolled upon itself to form the needle-like roll debris. This type of behavior with ceramics has been observed by other authors (Ref. 13). At 1200 °C, large areas of long, unbroken whiskers, many as long as in the original material and devoid of Al_2O_3 matrix particles, are found outside the wear scar. See Fig. 13. Near the scar, long whiskers and matrix particles are present. The long whiskers indicate whisker pullout. Since the whiskers are largely unbroken it is plausible that they are somehow debonding from the matrix.

Figure 14 shows a TEM micrograph of a whisker which has reacted with impurities in the matrix, possibly iron, when slid at 1200 °C. The reaction product, identified by the TEM diffraction pattern is iron silicide. Since iron silicide is liquid at 1200 °C the remaining unreacted part of the whisker may be debonding and, hence, easier to pull out. Thus, at 1200 °C the wear mode seems to be whisker loosening, pullout and breakup of matrix with possible whisker/matrix reactions.

DISCUSSION

The pin wear data indicate an increase of wear with test temperature. To determine the reason for this behavior, pin wear surfaces from room temperature and 1200 °C tests were examined using scanning and transmission electron microscopy.

The TEM analyses indicate that at room temperature, the predominant failure mode is crack initiation and growth and subsequent delamination and removal of fractured particles. The wear debris consists of alumina particles and broken SiC whiskers. TEM diffraction analyses indicate that, during room temperature sliding, no significant reactions between the alumina matrix and the whiskers occur.

The TEM analyses also detected cracking in samples prior to particle removal. This observation further supports the theory of a brittle fracture wear mode. See Fig. 7(a). Analyses of unslid samples showed no pre-existing cracks or large voids.

The SEM analyses of the room temperature pin wear surface showed short pieces of broken whiskers and matrix particles. At low magnifications, the pin wear debris resembles plastically deformed areas. However, at higher magnifications, these areas are actually found to be compacted fine particles. These fine particles were probably produced by communition in the sliding contact during the wear process. See Fig. 9 for representation of "pseudo plastic deformation."

The pin wear rate at elevated temperatures was much higher than at room temperature, yet the friction coefficient was relatively stable. TEM analyses of the pin surface from the 1200 °C tests indicated that some dislocations in the alumina matrix were present. See Fig. 8. The presence of dislocations supports the theory that plastic behavior may be playing an important role in high temperature wear. However, too few dislocations were found to have a significant effect on the wear rate. Thus, the theory that plastic deformation and subsequent particle removal is dominating the high temperature wear behavior is not likely.

This conclusion, when coupled with the detailed SEM analysis, yields an alternative theory. The SEM photomicrographs of the wear debris outside the wear scar from 1200 °C samples show large areas of debris which are predominantly long whiskers with little or no matrix material debris. Many of the pulled out whiskers are 30 to 50 μm long and have very high aspect ratios. That is, these whiskers in the wear debris are as long as those in the virgin material indicating that they have been dislodged rather than broken off. It is very unlikely that these loose whiskers could be the result of matrix cracking alone. Rather, it is more plausible that the long whiskers have been pulled out because they were loosely held by the matrix.

One strong argument for a whisker loosening wear mode is that the whiskers may be loosening at elevated temperatures due to differences in the thermal expansion coefficient between the Al_2O_3 matrix and the SiC whiskers. The

thermal expansion coefficient for the alumina matrix is twice that of the SiC whiskers (Table I). Hence, as the material is heated, the whiskers loosen.

During hot consolidation or rather the initial production of the composite, at about 1600 °C, there is no thermal stress between the whiskers and the matrix. Upon cooling, however, the matrix contracts more than the whiskers thus "clamping" the whiskers in compression. At temperatures lower than the consolidation temperature, the whiskers are in compression and the alumina matrix is in tension.

When sliding occurs, the friction force is tangential to the surface creating the tendency to pull or tug at the whiskers in the matrix. At low temperatures the whiskers are held in the matrix by the thermal compression. However, during elevated temperature testing or during high speed and load tests, which exhibit high frictional heating, the difference in thermal expansion lowers the "clamping" force on the whiskers. This allows them to more easily be pulled out of the matrix. Then the matrix, which is riddled with empty whisker pockets, cracks up and wears easily.

Evidence for this wear mode is found by examining the large numbers of unbroken, long whiskers outside the wear scar after testing at 1200 °C. Wear debris from 25 °C tests show only short whisker pieces and matrix presumably due to the fact that at lower temperatures the whiskers are strongly held by the matrix and are being broken by the wear process rather than being pulled out.

TEM diffraction analyses indicate that at 1200 °C some reaction between whisker and matrix may be occurring. The reaction products may have an effect on the friction and wear especially since there appears to be a glassy layer forming at the sliding surface at 600 °C as evidenced by the roll debris observed. Also, perhaps a low shear strength reaction product such as mullite or iron silicide due to iron impurities in the Al_2O_3 is forming at the matrix whisker interface. If the reaction products are liquid at the sliding temperatures they may be allowing easier whisker pullout. SEM Energy Dispersive analyses (EDS) was inconclusive for this material because the analysis area was larger than the whisker diameter and spacing between whiskers.

From the results of this research study, several ways to improve the high temperature wear resistance of whisker toughened ceramics may be suggested. One is to consolidate the matrix at as high a temperature as possible to prevent thermal expansion loosing of the whiskers. Also, the whisker matrix material choices could be made to prevent thermal expansion mismatch. One material choice is zirconia (ZrO_2) instead of SiC. Because the thermal expansion coefficient of ZrO_2 is higher than Al_2O_3, with ZrO_2 toughened alumina one would expect a decrease in pin wear as the temperature increases because as the sliding temperature increases the whiskers would be under a higher "clamping" force (Table I). Irregularly shaped whiskers could also be employed to mechanically interlock the whiskers to the matrix, preventing pullout. These methods may reduce wear, especially at high temperatures. More experimental work needs to be done to verify the wear process and explore these improvement techniques.

CONCLUSIONS

The following conclusions can be drawn from this study:

1. The wear of the composite pin increases with test temperature. This seems to be due to whisker loosening, pullout, and subsequent matrix cracking.

2. Although dislocations in the matrix occur at elevated temperatures they do not appear to contribute significantly to the wear process.

3. Chemical reactions between the whiskers and the matrix and the test environment may be occurring and may promote whisker pullout at elevated temperature and may have an influence on friction.

4. Methods to reduce whisker pullout such as matched thermal expansion materials or mechanically interlocking whiskers may improve high temperature wear resistance.

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TABLE I

[Data taken from manufacturer's literature.]

Material	Four-point bend, MPa	Young's modulus, GPa	Vickers hardness, kg/mm^2	Density, g/cc	Coefficient of thermal expansion		Toughness, $MPa/m^{1/2}$
					$10^{-6}/°C$	Poisson's ratio	
Al_2O_3-SiC	641	393	2125	3.74	6.0	0.23	8.8
Al_2O_3	344	386	2000	3.9	8.0	.23	4.2
SiC	448	406	2800	3.1	4.0	.12	3.8
ZrO_2	650	200	1100	5.78	9.5	.23	9.0

TABLE II

[Uncertainties represent data scatter band.]

Test temperature, °C	Coefficient of friction, μm	Wear factor,[a] K_{pin} mm^3/Nm	Wear factor,[a] K_{disk}	Load, kg
1200	0.58±0.15	$(1.1\pm0.5)\times10^{-6}$	$(5.1\pm2.0)\times10^{-7}$	2.7
800	0.72±0.22	$(6.1\pm1.0)\times10^{-7}$	$(4.2\pm2.0)\times10^{-7}$	2.7
600	0.60±0.10	$(1.5\pm0.5)\times10^{-6}$	$(7.0\pm2.0)\times10^{-7}$	2.7
25	0.74±0.10	$(2.4\pm0.5)\times10^{-7}$	$(7.7\pm4.0)\times10^{-7}$	2.7

[a]May not be linear, only run at one set of conditions.

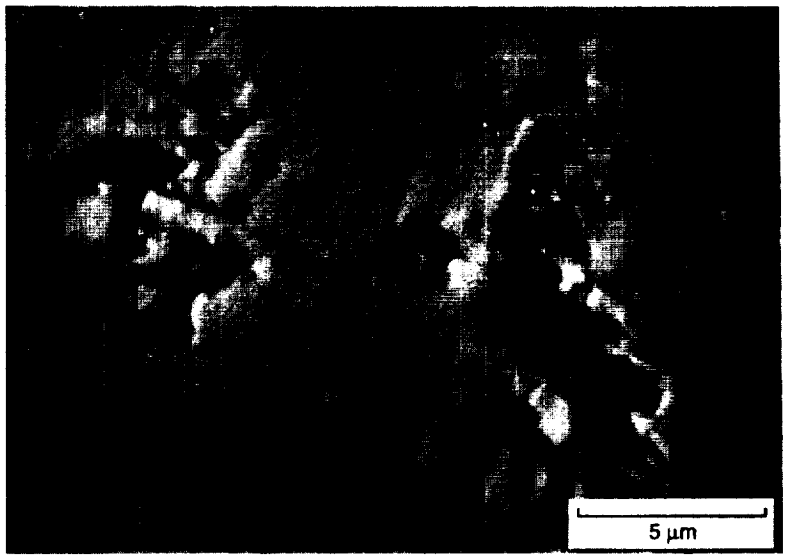


Figure 1. - SEM micrograph of ceramic surface showing orientation of whiskers in planes parallel to surface and sliding plane.

(a) Interface between whiskers and matrix is free from inclusions, large scale asperities, voids, etc. Alumina matrix grains visible.

(b) Whisker cross-section, some small enclosed pores, matrix grain structure.

Figure 2. - TEM micrographs of virgin (unslid) material.

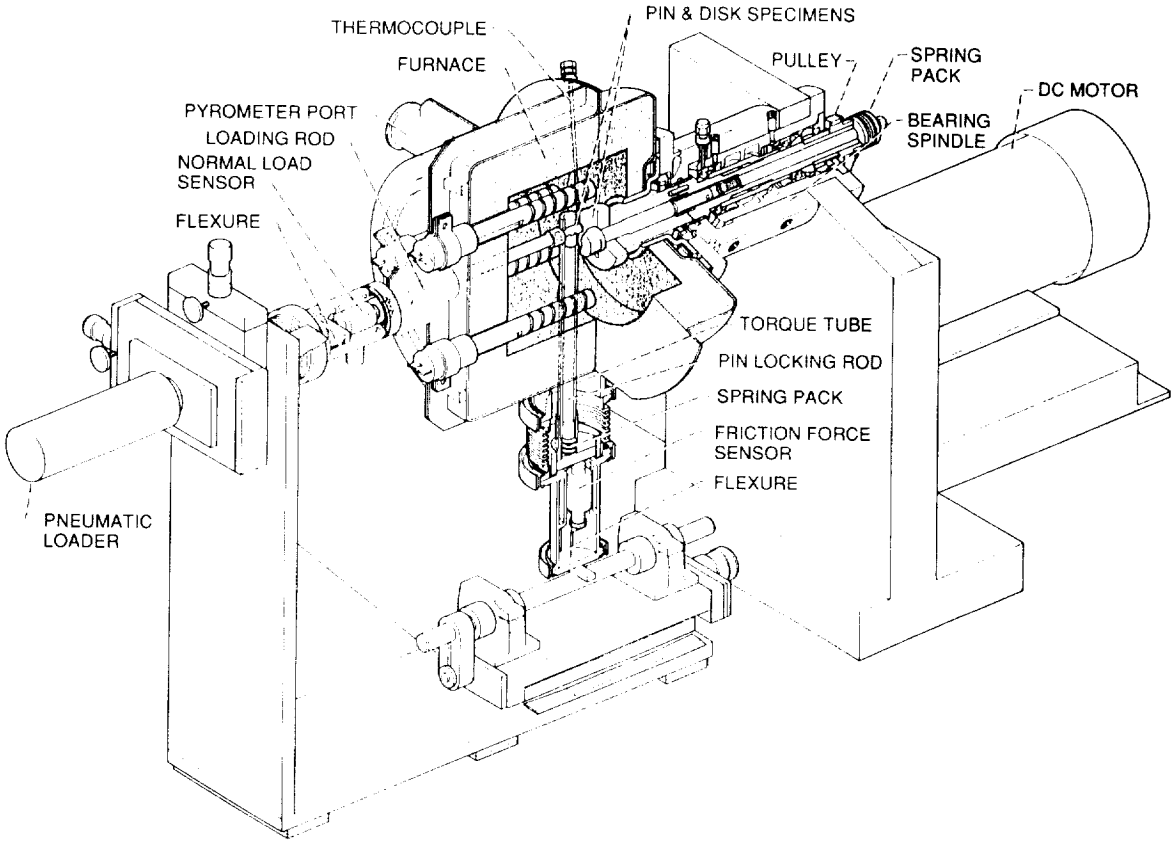


Figure 3. - High temperature friction and wear apparatus.

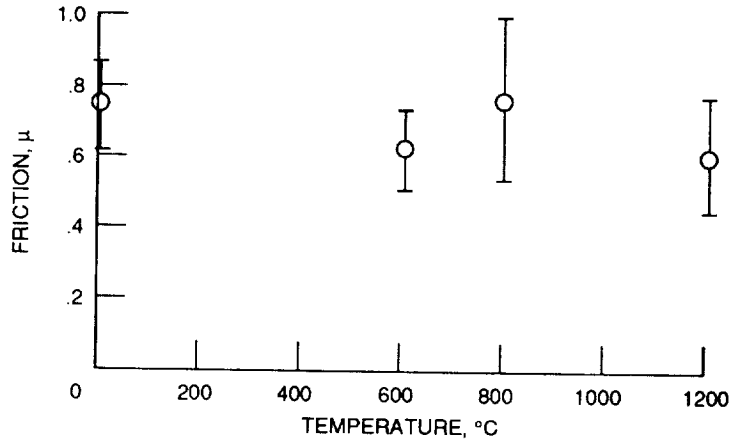


Figure 4. - Friction coefficient versus test temperature for the Al_2O_3-SiC composite sliding against itself in air at 2.7 m/s, 26 N load.

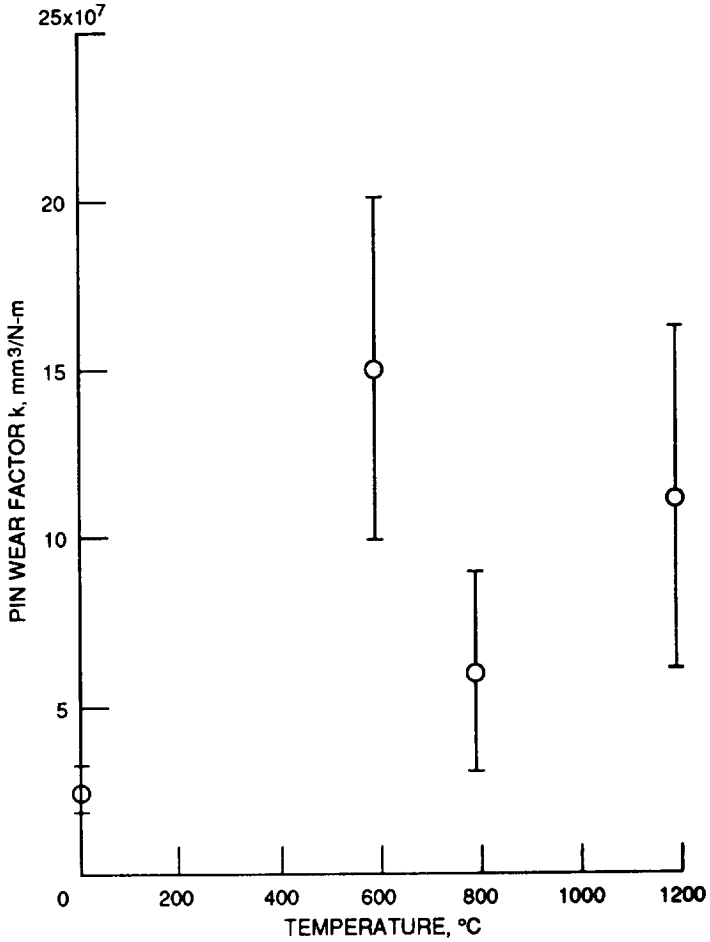


Figure 5. - Pin wear factor, k, versus temperature for the Al_2O_3-SiC composite sliding against itself in air at 2.7 m/s, 26 N load.

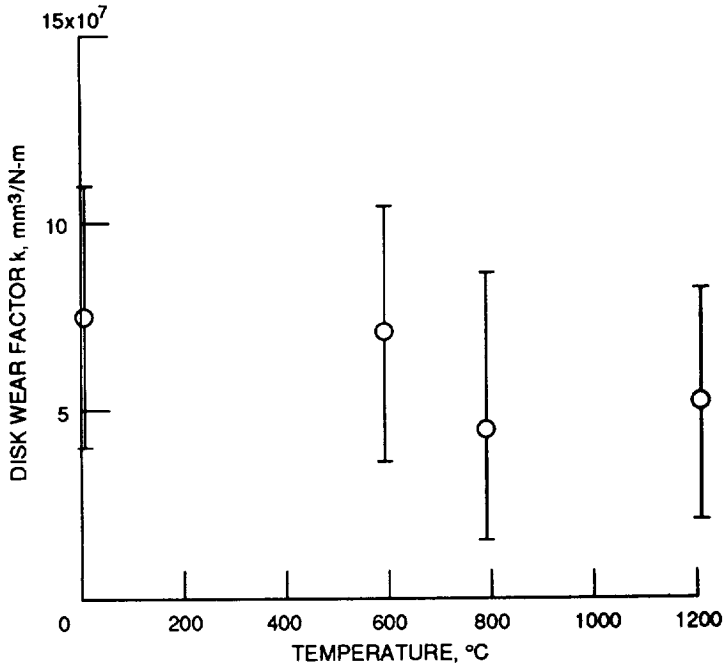
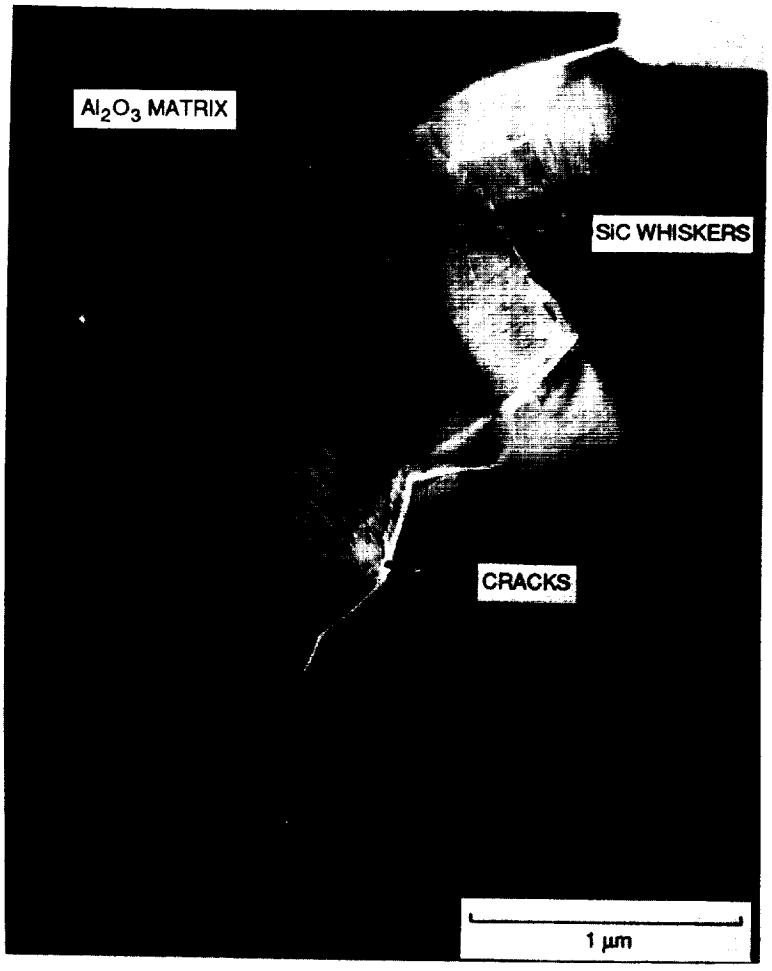
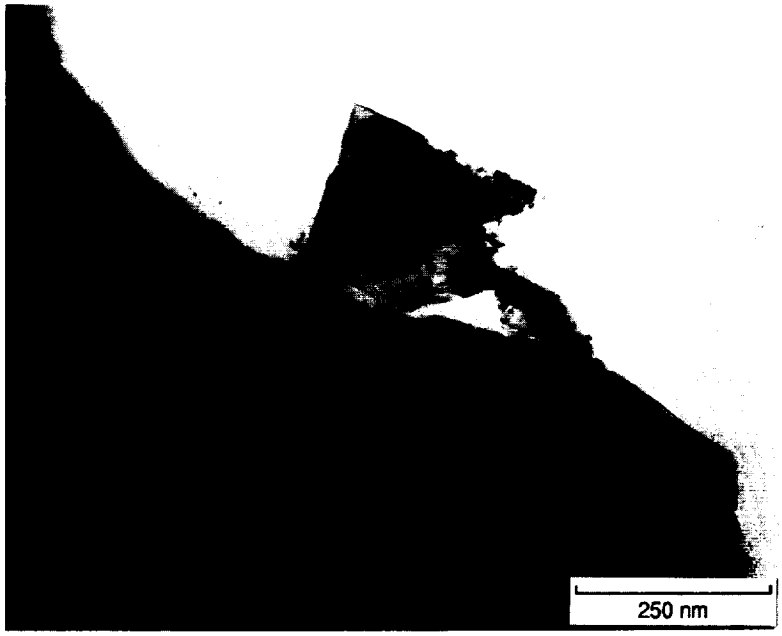


Figure 6. - Disk wear factor, k, versus temperature for the Al_2O_3-SiC composite sliding against itself in air at 2.7 m/s, 26 N load.

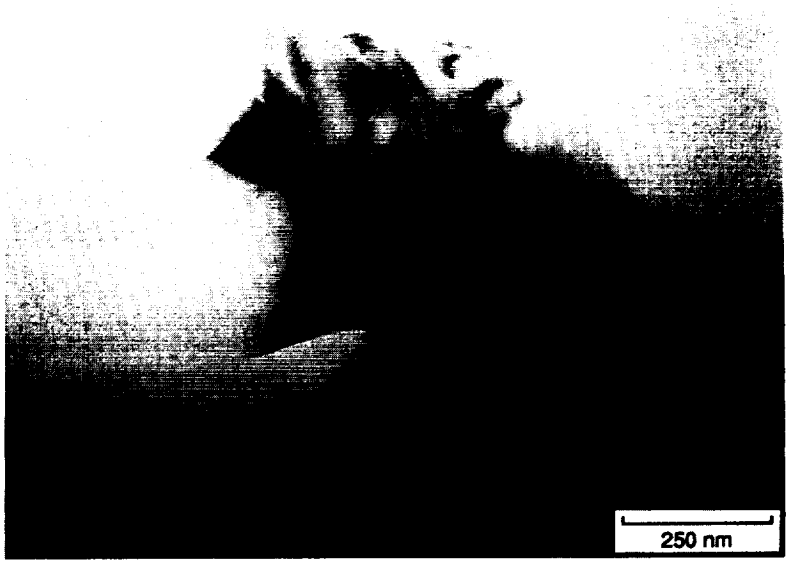


(a) Photo shows cracking in matrix and whisker as evidence for brittle behavior.

Figure 7. - TEM micrographs of pin wear surface from room temperature test.

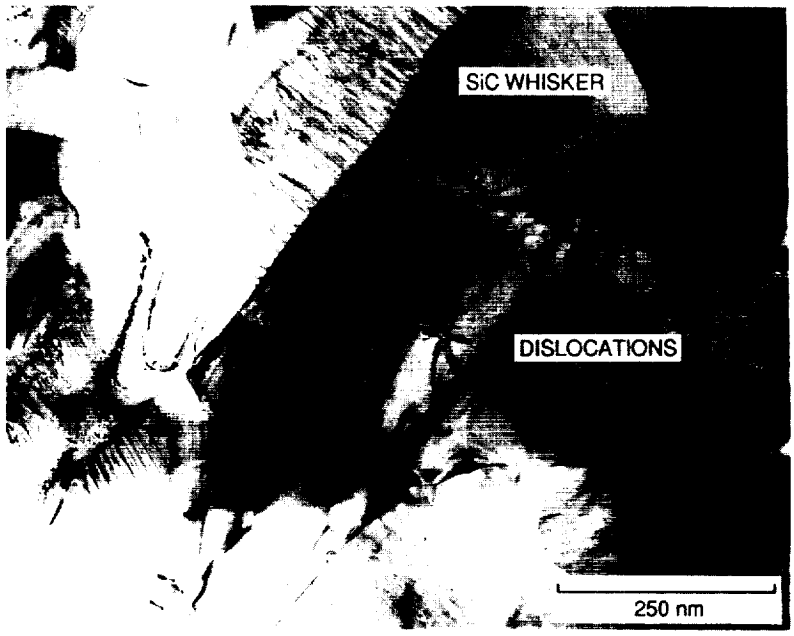


(b) Brittle fractured wear debris particle.



(c) Brittle fractured wear debris particle. Note no dislocations or evidence of plastic behavior detected.

Figure 7. - Concluded.



(a) Whiskers shown lengthwise.

(b) Whisker shown head on.

Figure 8. - TEM micrograph of pin wear surface region from 1200 °C test sample. Note dislocations induced from sliding. No wear debris detected on wear surface area.

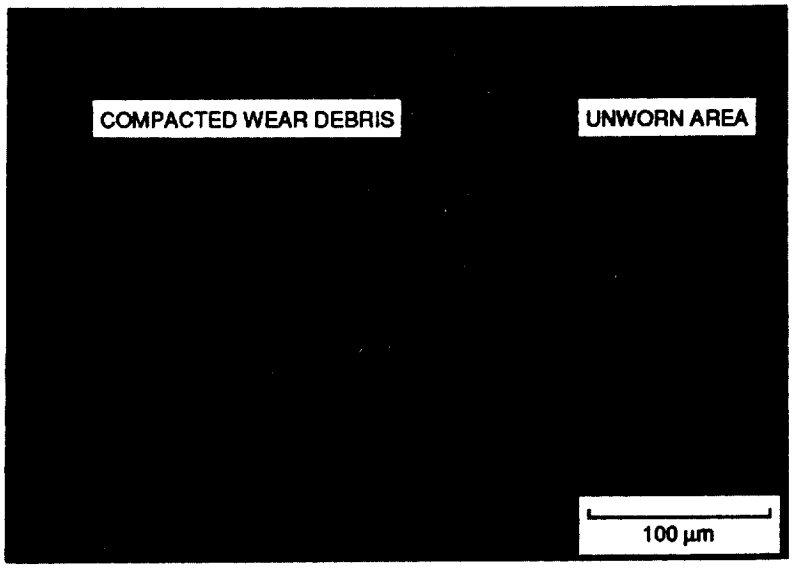


Figure 9. - SEM micrograph of wear scar area from room temperature test. Wear debris is compacted into larger regions.

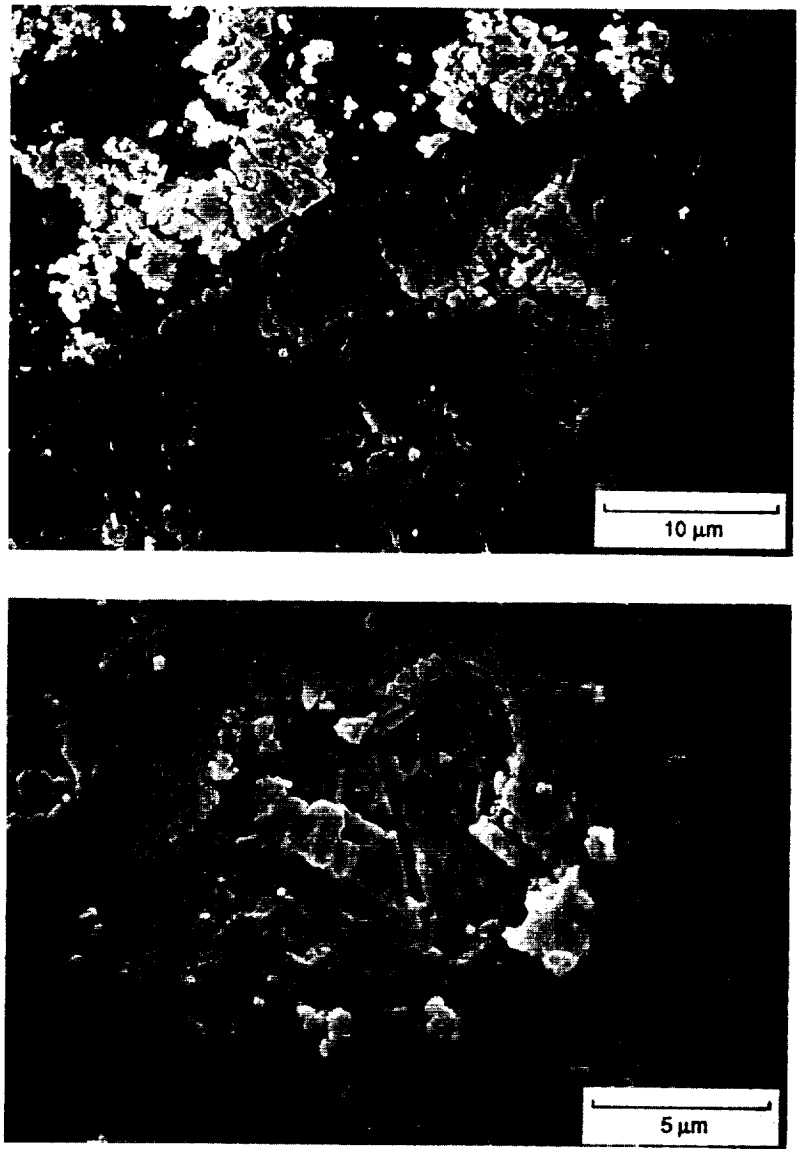


Figure 10. - Whisker pockets left behind by pulled-out whiskers on pin wear surface from 600 °C test.

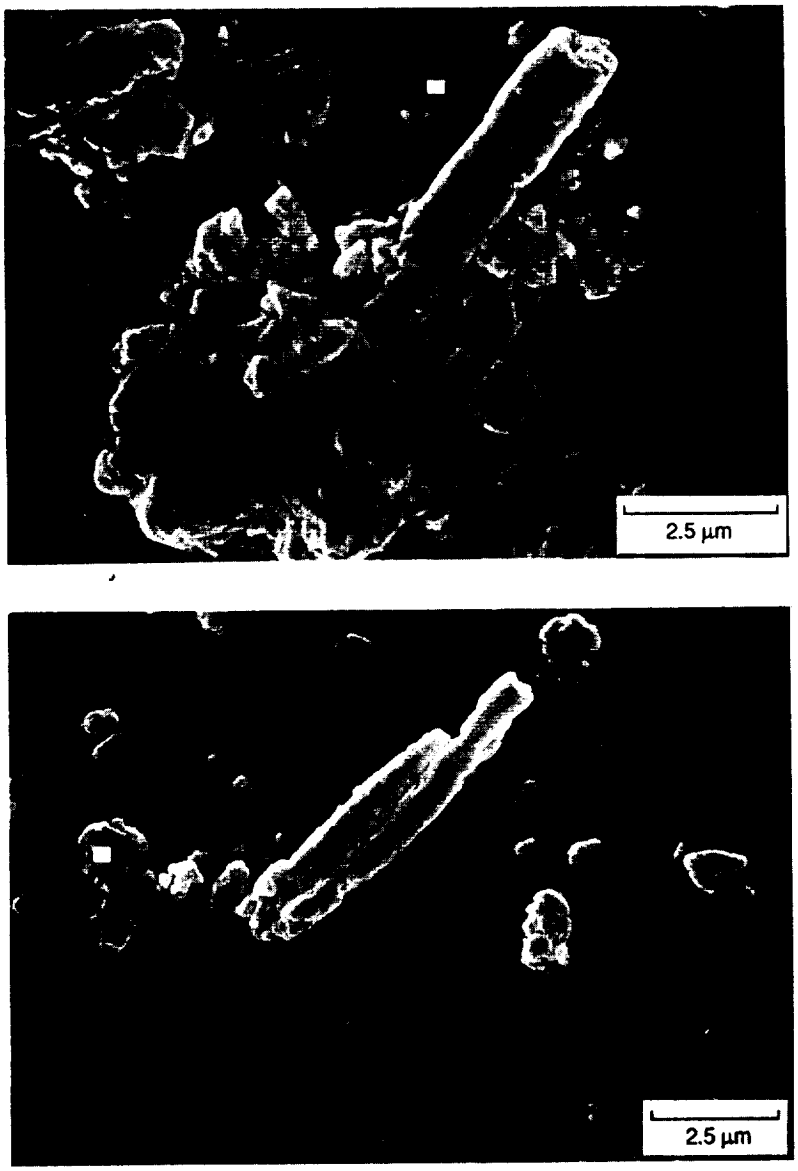
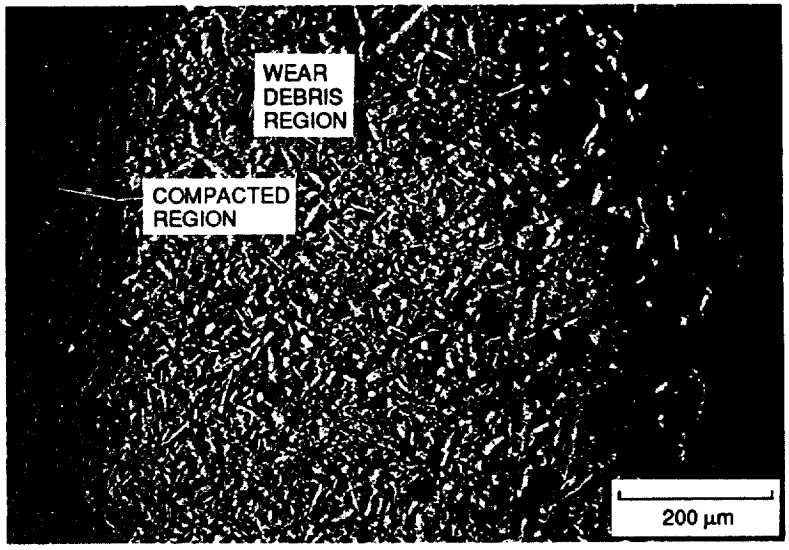
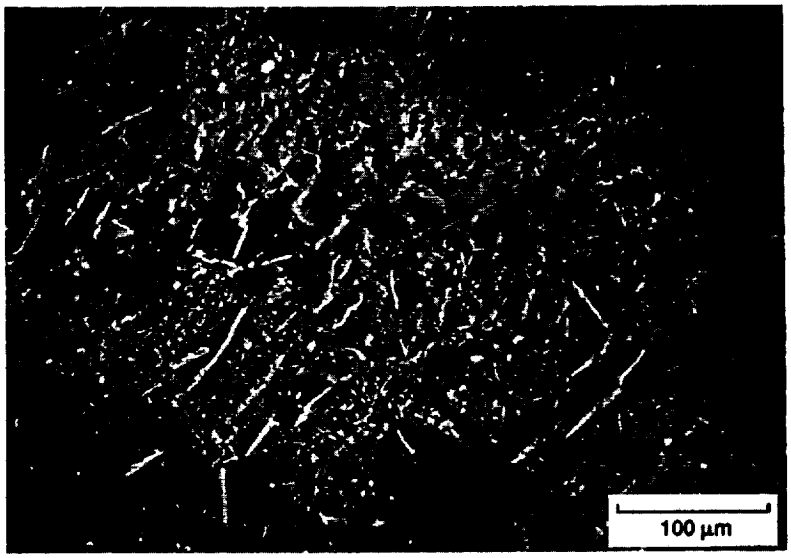


Figure 11. - Wear debris "rolls" or needles from 600 °C test specimens. Note seam along needles and small diameters (≈ .4 µm) distinguish these from whiskers.

Figure 12. - Pulled-out whiskers from 1200 °C test. Note uniform whisker diameters and lack of seams distinguish these whiskers from debris "rolls".



(a) Debris at 105x magnification.



(b) Debris at 200x maginification.

Figure 13. - SEM micrograph of "pulled-out" whiskers outside wear scar from 1200 °C sample. Note that most of the whiskers are 10-75 µm in length.

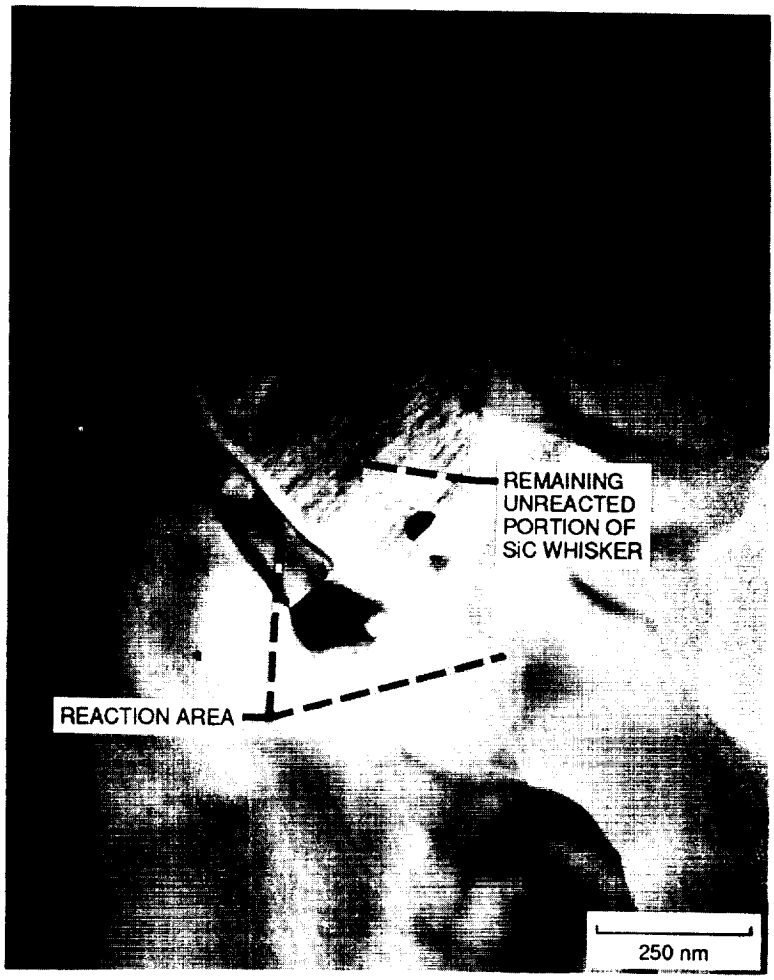


Figure 14. - TEM micrograph of pin wear scar from 1200 °C test. Figure shows a SiC whisker which has partially reacted with impurities and matrix. Reaction products may be promoting whisker loosening and pull-out.

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15. Supplementary Notes

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16. Abstract

The sliding wear behavior of a self-mated alumina-silicon carbide whisker toughened composite was studied using optical, scanning electron (SEM) and transmission electron (TEM) microscopy. Because of its excellent strength and toughness properties this composite material is under consideration for use in heat engine applications for sliding contacts which operate at elevated temperatures. The composite's wear behavior and especially its wear mechanisms are not well understood. Pin-on-disk specimens were slid in air at 2.7 m/s sliding velocity, under a 26.5-N load, at temperatures 25 to 1200 °C. Pin wear increased with increasing temperature. Based upon the microscopic analyses, the wear mechanism seems to be loosening of the reinforcing whiskers due to frictional and bulk heating. This leads to whisker pullout and increased wear.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
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