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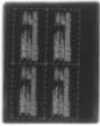
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MOLYBDENUM-REINFORCED ALUMINUM OXIDE SINGLE CRYSTALS.(U)
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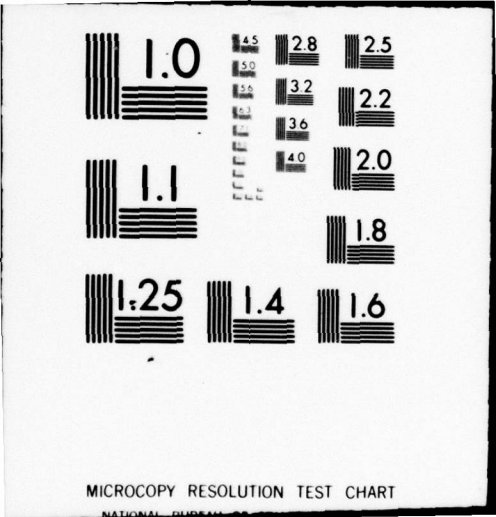
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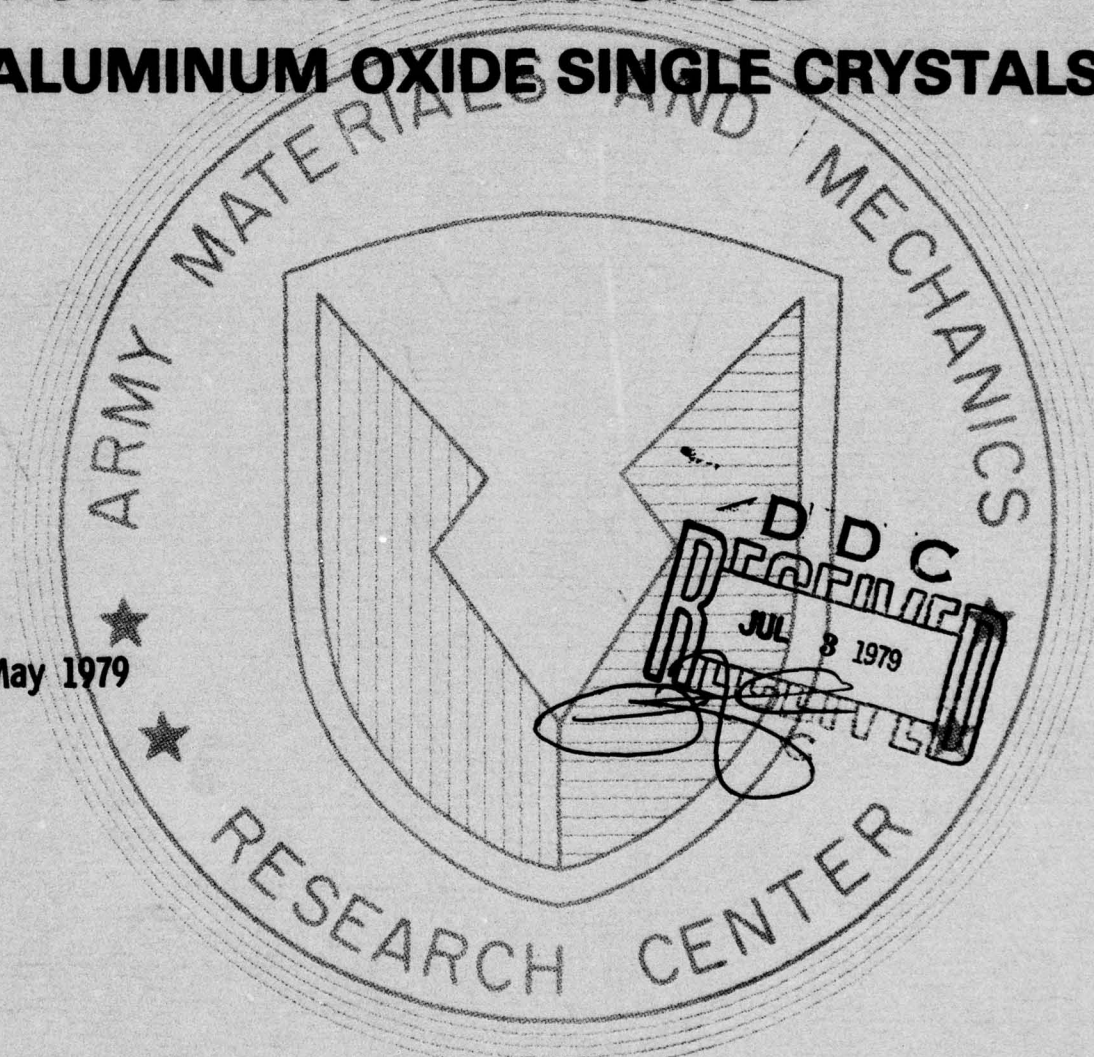
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**MOLYBDENUM-REINFORCED
ALUMINUM OXIDE SINGLE CRYSTALS**

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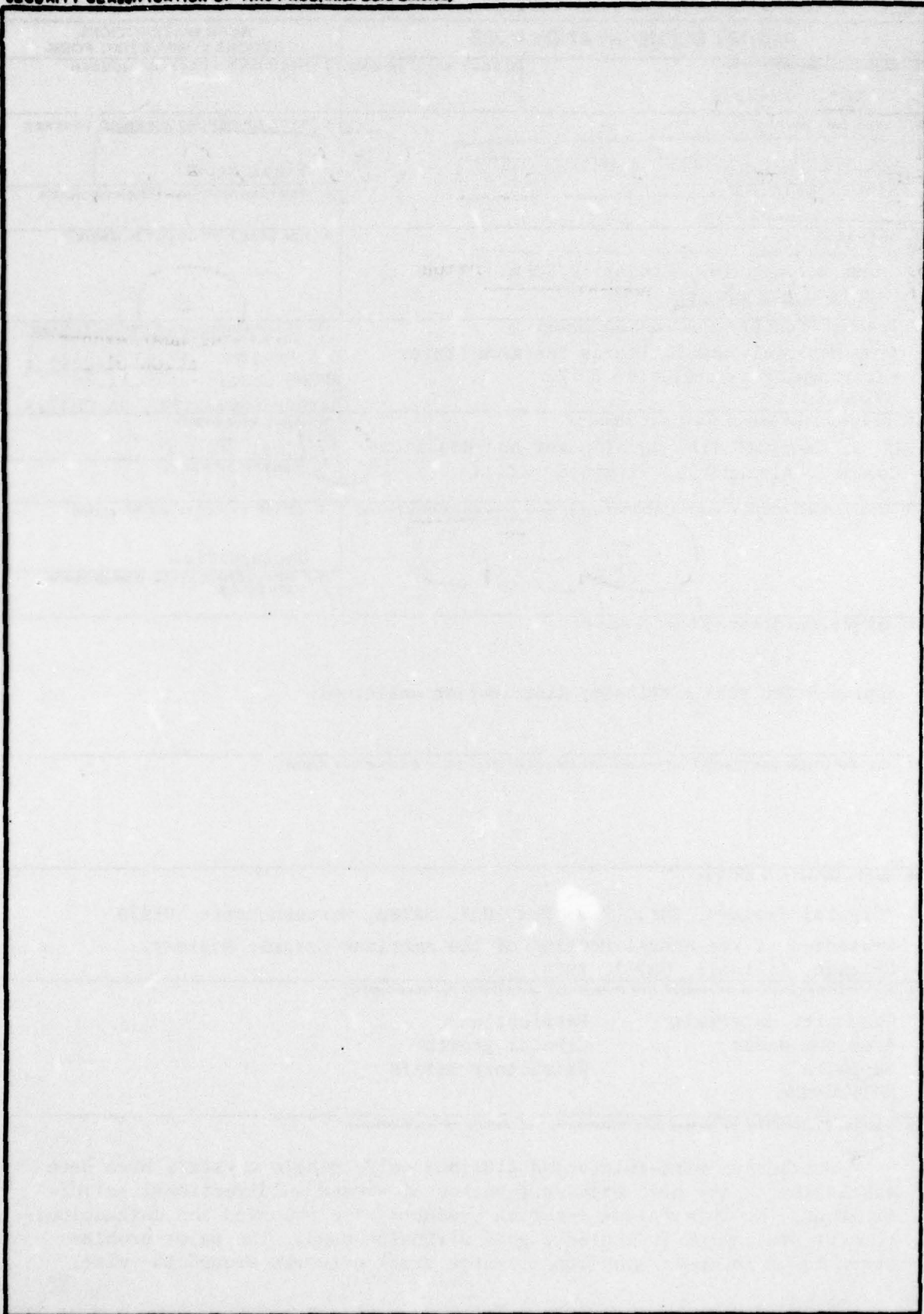
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INTRODUCTION

Metal fiber (wire) - crystalline ceramic matrix composites are potentially important electronic and structural materials. The beneficial effects of introducing metal fibers into ceramics (cermets) have been reviewed in many articles.¹⁻⁷ Solid state processing,^{3,4} plasma spraying,⁵ and melt techniques² have been variously used. Chapman et al. have reported a method for the production of UO₂-W composites by a eutectic unidirectional solidification technique. This procedure, however, limits composite fabrication to ceramic-metal systems which have eutectic or near eutectic phase equilibrium relationships. An attractive alternative to this technique is the physical incorporation of refractory metals into less refractory ceramic single crystals by vertical solidification. This paper describes preliminary results from a study involving the adoption of the heat exchanger method⁸ of seeded unidirectional solidification for the fabrication of refractory metal/single crystal Al₂O₃ composites. A review of the thermal expansion characteristics of various refractory metals⁹ and sapphire^{10,11} suggested that tungsten, molybdenum, and niobium wire could be successfully incorporated into a sapphire matrix.

EXPERIMENTAL

The general procedure for crystal growth by the heat exchanger method (HEM) has been discussed previously.⁸ Incorporation of refractory metals into the growing crystal involves only routine modifications of these procedures. Figure 1 is a schematic illustration of one arrangement showing a 2-inch-diameter by 2-inch-high molybdenum crucible into which has been placed an appropriately oriented and situated Al₂O₃ seed single crystal, premelted Al₂O₃ (crackle*), and a molybdenum wire screen† (20 × 20 mesh of 0.007-inch-diameter wire). The molybdenum wire screen was suspended from the top of the crucible and completely surrounded by crackle. Since molybdenum has a melting point of about 2610 C in vacuum (560 C

*Adolf Mellor Co., Providence, Rhode Island.

†Newark Wire Cloth Co., Newark, New Jersey.

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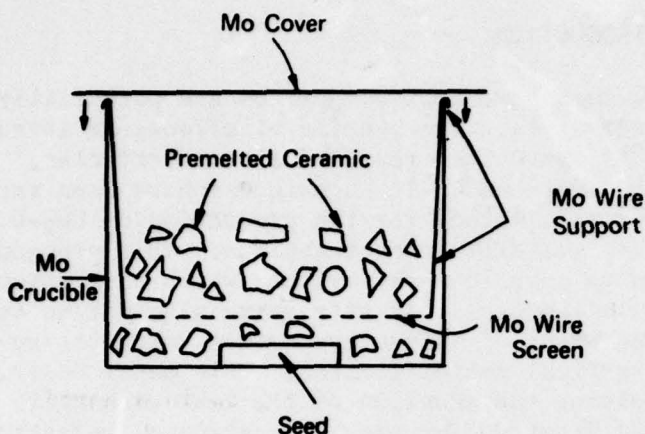


Figure 1. Schematic drawing of experimental configuration for incorporating molybdenum wire into single crystal Al_2O_3 .

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higher than Al_2O_3) the growth procedure is carried out without melting the molybdenum wire simply by maintaining the maximum temperature below the melting point of molybdenum. The seed does not melt because of its position above the heat exchanger and growth is controlled at a rate of 0.2 inch per hour through and around the molybdenum wire screen. After completion of the run, the crucible has to be peeled from the solidified ingot.

RESULTS AND DISCUSSION

In Figure 2 it can be seen that the Al_2O_3 single crystal did grow through the molybdenum screen, although shifting it somewhat from its original horizontal position. Laue photographs of the ingot above and below the screen showed no change in the $[10\bar{1}2]$ seed orientation. Observation of the ingot in a modified polariscope¹² confirmed this conclusion and indicated no re-nucleation of grains at the wire/ Al_2O_3 interface, although molybdenum fragments are occasionally eroded from the wire.

Molten alumina wets molybdenum in vacuo¹³ allowing for complete infiltration into and around all the wires. This was further confirmed in another experimental configuration where a section of wire screen was placed in a crucible perpendicular to the bottom, rather than parallel. When the alumina was molten, the screen was totally immersed. As the crystal grew and the liquid level dropped, the top of the screen became exposed, yet some of the molten Al_2O_3 still adhered to the wire screen. Figure 3 shows a sectioned ingot with about a third of the wire screen exposed above the sapphire ingot and covered with a film of aluminum oxide. Other configurations of the molybdenum wire in the crucible are possible, as well as sheets of molybdenum, as long as they permit the molten ceramic to fully wet the metal and solidify without severe distortions in the ceramic or the metal incorporation.

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The molybdenum/single crystal Al_2O_3 composites contained a large network of cracks. Some seemed to originate where the molybdenum screen was supported by molybdenum wire near the crucible wall. Others seemed to originate in the wire screen network itself; these can be seen in Figure 4, which are probably the result of intersecting residual strain fields in the Al_2O_3 . The strain seems to be due to both the mismatch in thermal expansion of the two components and the perturbation of the growth of the Al_2O_3 as the liquid-solid interface passes by the wires in the screen. Observation of polished sections transverse to the plane of the screen in reflected polarized light revealed a network of residual strain fields around the wires. The shape and location of these darkened strain fields (bowed out between the wires in the direction of the growing crystal) suggest that the liquid-solid interface of the crystallizing Al_2O_3 was momentarily pinned by the screen before it broke through and continued to grow in a normal fashion. Incorporation of tungsten wire into single crystal Al_2O_3 resulted in much more extensive cracking because of the larger difference in thermal expansion⁹ between the two components.

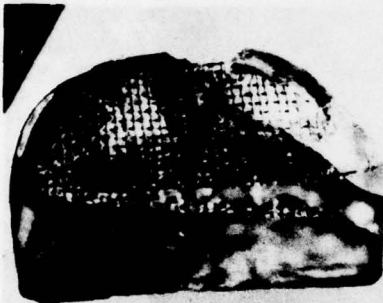


Figure 2. Halved molybdenum/single crystal Al_2O_3 ingot; diameter is two inches.

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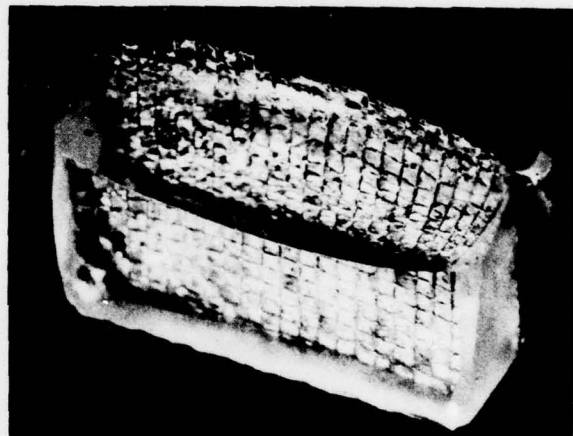


Figure 3. Cut and polished composite; large edge is one inch.

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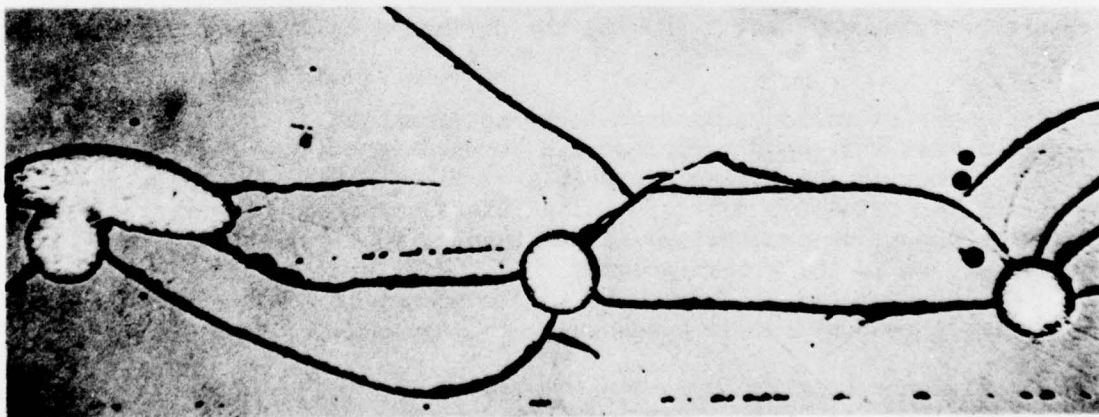


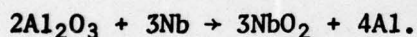
Figure 4. Reflected light photomicrograph of a transverse section of molybdenum/ Al_2O_3 ; wire diameter is 0.007 inch.

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A careful analysis of the crack network and an evaluation of the properties of molybdenum and Al_2O_3 suggests several means of eliminating the cracks:

1. appropriate adjustment of crystal growth rate,
2. a more careful consideration to the geometry of the incorporations (spatially) to minimize overlap of residual strain fields, and
3. more closely matching the bulk thermal expansions of the incorporation and the matrix; this could possibly be accomplished by alloying the refractory metal or by appropriate independent control of the temperature of the wire during solidification.

No reaction products were found at the molybdenum/ Al_2O_3 interface by electron microscope observation. Emission spectroscopy analysis of Al_2O_3 single crystals with and without molybdenum wire incorporations indicate that higher amounts of molybdenum and other metallic impurities are incorporated into the Al_2O_3 matrix when the wires are present. Certain impurities like iron and silicon are even preferentially dissolved from molybdenum and therefore should be removed prior to use. However, the kinetics of the molten Al_2O_3 -molybdenum reaction are slow enough as not to hinder the formation of the composite. However, this is not the case with niobium as the Al_2O_3 is totally and rapidly reduced at temperatures greater than 1500 C:



Electron microprobe analysis of areas in Al_2O_3 close to an interface indicated that diffusion of molybdenum into Al_2O_3 occurred to a depth of about 20 μm . A cathodoluminescent analysis¹⁴ of identical areas confirmed the probe results. The cathodoluminescence stimulated by electron bombardment¹⁵ occurred as distinct purple colored halos or borders around the molybdenum wires - the extent of the luminescence being a rough indication of diffusion distance. The normal cathodoluminescence from relatively pure single crystal Al_2O_3 is a bright red. The exciting ion in this work is probably molybdenum; however, selectively dissolved impurities such as iron or silicon may also be involved.

Figure 5 is a photographic mosaic of four individual large-area electron beam irradiated interface areas, showing the diffusion halo around the molybdenum wire.

In the course of cathodoluminescent characterization of these ingots it also became obvious that this same technique can be used to study remnant liquid-solid interfaces in properly doped single crystals of Al_2O_3 . Figure 6 illustrates an example of luminescent remnant liquid-solid interface bands. Apparently, small changes in atmosphere or temperature in the furnace during crystal growth will cause perturbations in the growth process. If this effect results in a valence change of the incorporated molybdenum or in the amount of molybdenum incorporated, this could result in luminescent bands where the anomaly occurred.

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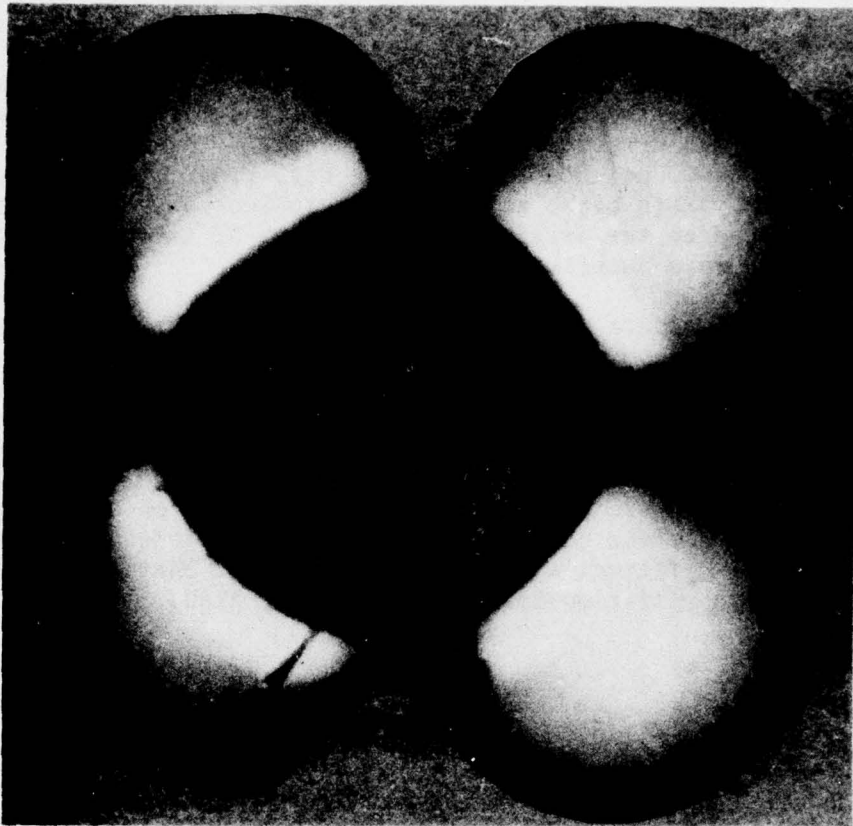


Figure 5. Mosaic photograph of cathodoluminescent (black and white of color print) halo around molybdenum wire in single crystal Al_2O_3 , excited in four areas by electron beam in electron microprobe; wire (dark center portion) is 0.007 inch in diameter.

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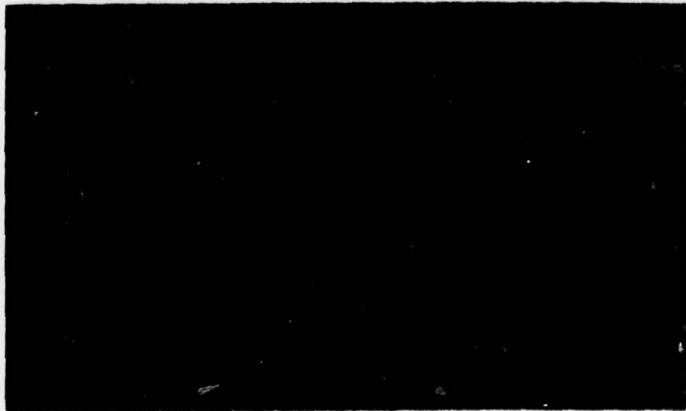


Figure 6. Cathodoluminescent photograph (black and white of color print) of Al_2O_3 single crystal slice showing distinct remnant liquid-solid interface luminescent bands (purple on red).

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Two composite bend bars were cut from a molybdenum/single crystal Al_2O_3 ingot so that the wire screen was in the center of the bars parallel to their long axes. These were surface ground with a diamond wheel to final dimensions of $0.191'' \times 0.191'' \times 1.000''$. They were then fractured on an Instron testing machine in four-point loading using a strain rate of 0.002 inch/minute. The modulus of rupture of the two bars were 30,500 psi and 30,800 psi. Catastrophic failure did not occur in these samples. Both broke at the levels indicated above but still held loads of 1000 psi (based on the original cross section of the bar) until the molybdenum wire failed in a ductile fashion.

Figures 7 and 8 illustrate fracture surfaces perpendicular and almost parallel to the plane of the molybdenum screen. Figure 7a is a macrophotograph of the fracture surface of a bend bar; Figure 7b is an SEM photograph of an area immediately surrounding one of the broken molybdenum wires. Figures 8a and 8b are SEM photographs of fracture surfaces almost in the plane of the molybdenum wire screen. This photograph also shows that the molybdenum/ Al_2O_3 interface is clean; cleavage planes of Al_2O_3 can be observed in Figure 8b. Observations on other fracture surfaces also indicate that the characteristics of the molybdenum wire is very important as apparently different wires exhibit different characteristics (e.g., pitting, etc.) after incorporation into single crystal Al_2O_3 .



a. Normal reflected light photograph of fracture surface; sides = $0.191'' \times 0.191''$



b. SEM of molybdenum wire (labeled 2) fracture; wire = $0.007''$ diameter

Figure 7. Fracture surfaces of molybdenum/single crystal Al_2O_3 bend bar.

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a. General view of surface



b. Magnified view of area 1

Figure 8. SEM photomicrograph of molybdenum/single crystal Al_2O_3 parted along the molybdenum wire screen plane.

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CONCLUSIONS

It is feasible to make molybdenum/single crystal Al_2O_3 composites by the heat exchanger method. The two components are chemically and physically compatible and form a good, reaction-free, interfacial bond. A crack network occurs which might be eliminated by a more careful consideration of growth rates, geometry of the wire incorporations, and means for more closely matching the thermal expansion of the two materials. The molybdenum wire reinforcement prevents catastrophic failures without seriously decreasing the strength of Al_2O_3 single crystals. Cathodoluminescence is discussed as a valuable new characterization procedure for studying diffusion bonds and remnant liquid-solid interface in single crystals.

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