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"INTERACTION OF CRACKS WITH MICROSTRUCTURE IN POLYCRYSTALLINE CERAMICS"

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This research has been directed toward the development of a suitable technique for monitoring simultaneously the stresses and the detailed path produced by a crack proceeding through a polycrystalline ceramic. The ultimate goal is to relate stress changes with microstructure. The technique to be developed was to be compatible with a wide variety of experimental techniques such as optical microscopy, electron microscopy, etc., and was to permit a crack to proceed under a relatively controlled manner. The proposed work sub-divided the activity into six phases, all related to this single problem. The progress achieved and difficulties encountered in each of these phases will be discussed individually.

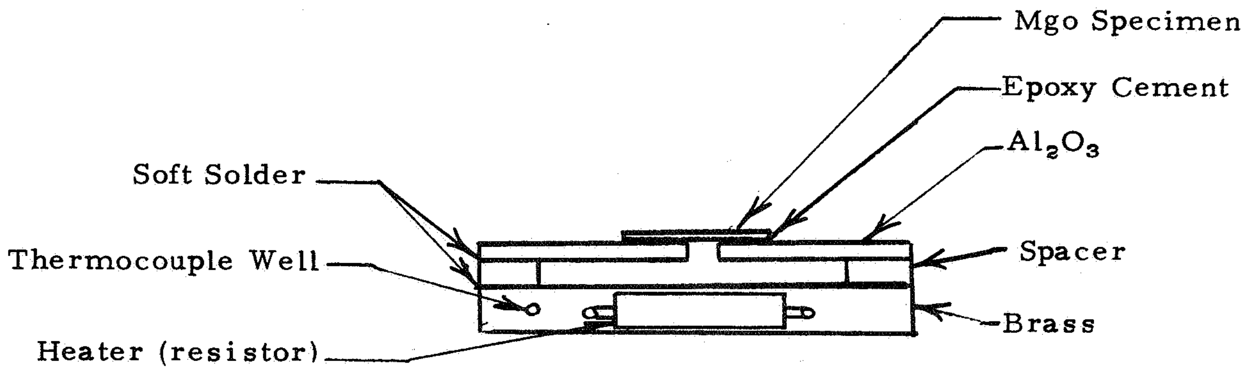
Phase 1. Develop a technique permitting controlled propagation of a crack through a polycrystalline ceramic.

The requirements for such a crack propagations system may be summarized as follows:

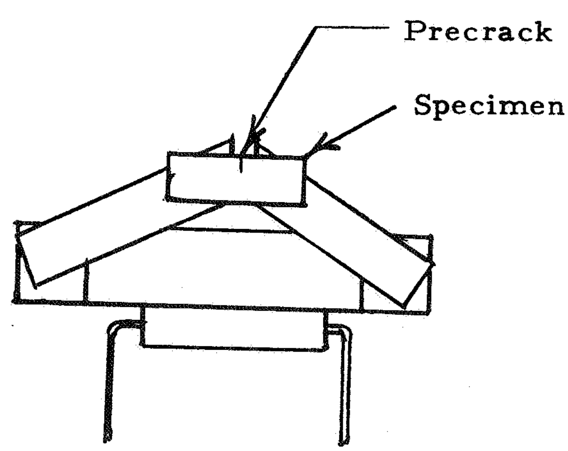
1. small size
2. high stiffness
3. capability of very small strains

Two approaches were apparently capable of meeting these requirements. The first employed differential thermal expansion between a reference body and a piece of polycrystalline ceramic under test. The second used the strains developed in a piezoelectric crystal under an applied electric field. Both techniques have been attempted and both have shown some success.

The first of these approaches, differential thermal expansion, is represented by Figure 1, sideview. With the materials and sizes shown here, strains in the specimen of approximately 10 μin /^{o}C are obtainable. With magnesium oxide and a 0.50 gap, this corresponds to several thousand PSI per ^{o}C. Such a system was readily capable of propagating a crack across a precracked glass cover slip (0.1 millimeter thick). Glass cover



SIDE VIEW



TOP VIEW

Fig. 1 Strain Application Device Activated by Thermal Expansion (shown two times actual size) slips are being used in preliminary work as a convenient low-cost model. It was also necessary to provide a means of reducing the applied force quickly as the crack propagated to control crack propagation. A self-regulating system was developed by placing the support arms at a slight angle as shown in Figure1 topview, and permitting the crack to propagate from the outside in. In this manner the moment arm producing the crack propagation is reduced as the crack proceeds. The crack will proceed at relatively slow rates or in most cases stop periodically until the stresses are increased by additional thermal expansion. The temperature changes required for failure were higher than calculated ($\sim 20^{\circ}C$) probably because of deformation in joints. This method appears to be entirely satisfactory for the crack propagation requirements and is compatible in size for use in microscopes and other analytical tools.

The second method of propagating a crack investigated is depicted in Figure 2. Calculations of the deformation available from either x-cut quartz crystals, or from properly polarized barium titanate indicate a sensitivity of approximately 0.01 μ in/V for a single piezoelectric element. To produce the necessary strains for fracture in MgO (approximately 10-20 microinches) excessively high voltages would be required. Consequently, the strain per volt figure was increased by stacking piezoelectric elements back to back as shown in Figure 2. In the case of quartz, crystals were placed in a + to- configuration, while with barium titanate, the elements were polarized after assumbling. In this manner the strains could be added to give approximately 0.1 μ in/V with ten elements per unit. Several fixtures of this type were constructed but difficulty was encountered with reliability of the silvered faces. These continually peeled during subsequent assembly and consequently, units repeatedly required reassembly. However, ultimately several units were successfully assembled, and evaluated for their crack propagation ability. Insufficient strain was generally developed by the piezoelectric units to propagate a crack across the glass cover slips, in disagreement with the calculated strain. The explanation appears to lie in minor amounts of deformation occuring in the solder

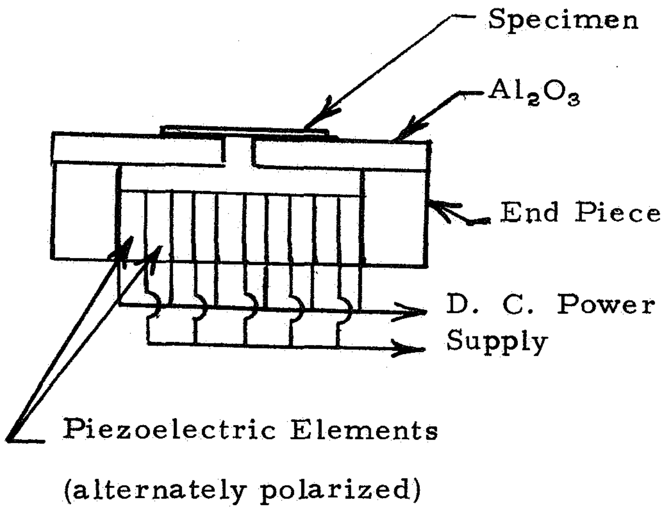


Fig. 2 Strain Application Device Activated by Piezoelectric Effect (shown two times actual size)

joints and adhesives in the unit, although attempts had been made to minimize these. This is consistent with the larger strains required in the thermal expansion units. Even during these unsuccessful tests the silver contacts frequently peeled, apparently from the applied stresses, requiring reassembly.

Because of the extensive difficulties encountered with construction of these piezoelectric stressing units, and because of their frequent failure to properly propagate cracks in the ceramic when assembled, further development of this approach has been temporarily abandoned. Attempts will be made however, to obtain information for improved silvering techniques. If this can be obtained, further attempts will be made to use this technique since it does offer the advantage of better control of stress rate changes than does the thermal expansion technique.

Phase 2. Develop quanitative stress monitoring during propagation.

To avoid introducing significant amounts of drformation in the crack propagating system for measurements of the applied loads an extremely rigid force transducer must be used. Here again, piezoelectric crystals are ideally suited because the deflections required for usable signals are very small. They also offer the advantage of very fast response. Consequently, assemblies such as that shown in Figure 3 were constructed with the piezoelectric elements providing an output proportional to the shear on the crystal faces. To provide sensitivity to shear loads, quartz AT cut crystals were used. The signal from the quartz transducer was fed into a charge amplifier, Kistler model 503 and finally to a Sargent model SR-1 Recorder. Ultimately an oscilloscope will be used for improved frequency response. Using this configuration, the changes in stress as a crack proceeds through the glass samples have been observed.

Two sources of spurious signals were noted in the output. The first of these was simply stray pick-up steming from the extremely high input impediances required for piezoelectrics (greater than 10^{10} ohms) and the relatively low signal levels (fractions of a volt thru volts). This problem was reduced by proper shielding of the piezoelectric crystal by grounded shields on either side, (Figure 3) and by an electrostatic shield surround-

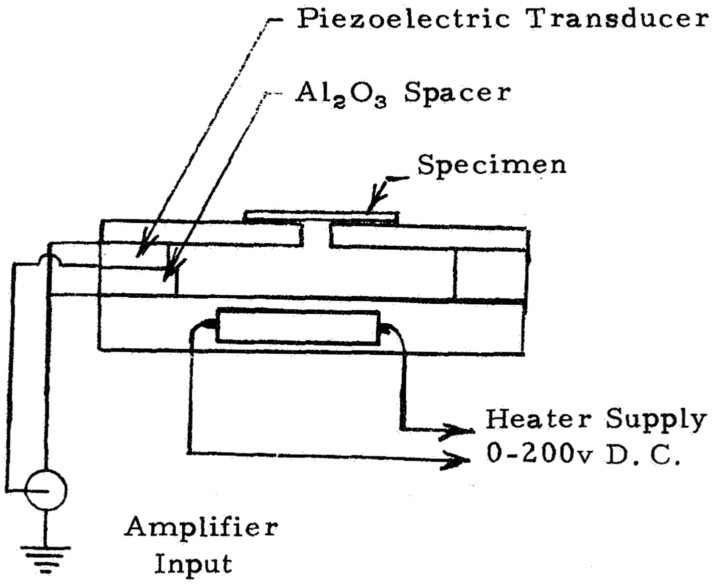


Fig. 3 Strain Application Device (Thermal Expansion Type) with Stress Transducer (shown two times actual size)

ing the entire stressing device. In this manner, stray pick-up may be kept to less than 40 millivolts. The second source of spurious signal arises from the stresses produced on the crystal from the thermal gradient existing across it. Note that the heat is introduced into the brass member, Figure 3, and consequently, the face of the crystal attached to it will be at a higher temperature. This signal takes the form of a very low frequency DC drift, and no satisfactory means of eliminating it has been found. However, it may be circumvented by noting that even slow rates of crack propagation are considerably faster than the changes in output due to drift. Thus by selection of appropriate electrical time constants, stress changes due to crack propagation may be separated from drift. Appropriate time constants are under study and development.

Phase 3. Make initial evaluation of technique with commercial ceramics in which some reasonable knowledge of character is available and,

Phase 4. Investigate the role of impurities with a well characterized polycrystalline ceramic (MgO) in which controlled grain boundary impurity additions have been made.

The selection, development, and characterization of materials for study as part of this program have been combined for discussion. Because of the failure to obtain facilities for the fabrication of ceramics suitable for these studies from previous NASA programs, no direct fabrication of specimens has been attempted. However, success has been achieved in obtaining specimens for study in which some knowledge of grain boundary composition is available. Five types of material, all polycrystalline MgO have been obtained, see Table I. They are being refired to produce equilibrium impurity distributions and increase grain size for ease of observation of the crack. About 20 specimens of the JP material have been cut to the appropriate size and have polished awaiting testing. The other samples are in process of sample preparation. They will all be characterized as to final density and grain size. These five types of material are expected to provide the necessary range to evaluate the effect of these particular compo-

TABLE I

Polycrystalline MgO Materials for Studies of Crack Interaction with Grain Boundaries

Type	SiO_2	CaO	Source
JP	100	50	Ref. 1
JF	1000	1000	Ref. 1
KK	300	500	Kaiser Ref. 2
KJ	500	1700	Kaiser Ref. 2
N	25	30	Norton polycrystals

sitional variables on crack propagation. This is based on the knowledge that the particular impurities do reside at grain boundaries after appropriate thermal treatment[3] and that mechanical behavior at higher temperatures is influenced by the relative-proportions of these impurities[2].

Phase 5. Extend technique of crack monitoring to the electron microscope with the emphasis on reflection techniques.

The use of reflection techniques in standard electron microscopy has been little applied to ceramics and consequently, little has been reported on the experimental procedure. It has recently been determined that such studies have been attempted elsewhere[4], and in the course of the studies, it was noted that extremely high electron beam currents were required to obtain proper image. These beam currents resulted in high specimen temperatures (> 800°C). Since such temperatures are incompatible with studies of low temperature crack propagation, this approach (reflection) has been abandoned and consideration given to more conventional electron microscopy. Some attempts have been made to apply the method of Doherty[5,6] for preparation of thin ceramic pieces for investigation of crack tip conformation. However, it has been found that because of the lack of control over the area viewed the technique would offer little value in these studies. This is in agreement with other investigators[7]. Two other techniques are under consideration for investigation of crack tip conformation. These are ion beam thinning for transmission studies, and replication of crack tip shapes by standard techniques. Further attempts will be made to evaluate the applicability of both of these techniques to this particular study.

Phase 6. Interpret fracture data in terms of new surface area developed and existing data for surface energy.

No effort has been expended on this phase of the activity awaiting the development of appropriate fracture data by the previously described studies.

Conclusions The experimental approach of monitoring the path of a crack proceeding thru a polycrystalline ceramic in a manner to permit correlation of microstructural features with stress changes appears feasable and will be further developed. The use of reflection technique in electron microscopy with ceramic oxides does not appear practical and emphasis will be places on other techniques.

Future Plans The crack monitoring procedure will be further developed as a quantitative wxperimental procedure. Data will be obtained on reasonably well-characterized processed materials of varying grain boundary cation composition to investigate the role of possible crack blunting in crack propagation.

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