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CROSTRUCTURE STUDIES OF POLYCRYSTALLINE REFRACTORY OXIDES
QUARTERLY PROGRESS REPORT NO. 3

Prepared by

T. Vasilos
R. M. Spriggs
J. B. Mitchell

RAD-SR-63-80

Prepared under U.S. Navy, Bureau of Weapons
Contract Now 62-0648C

29 April 1963

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RESEARCH AND ADVANCED DEVELOPMENT DIVISION
AVCO CORPORATION
Wilmington, Massachusetts

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APPROVED


Charles Spencer, Manager
Materials Department

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ABSTRACT

Alumina and magnesia specimens were prepared having grain sizes from 20 to 300 microns. Transverse bend strength and elastic modulus determinations were made on 50-, 100-, and 300-micron grain-size alumina and 70- and 100-micron magnesia specimens.

The strength of alumina showed a strong grain-size dependence at all test temperatures. The strength at higher temperatures appeared to be more temperature-dependent in the finer-grain specimens. The strength of magnesia appeared to be essentially independent of grain size up to approximately 700 °C; effect of grain size on strength at higher temperatures is presently inconclusive. The effect of grain size on the elastic modulus is at present in doubt due to a possible calibration error in the recording instrument. The present data indicate that the high-temperature elastic modulus of alumina is more temperature-dependent in the finer-grain-size specimens. Observations of fracture surfaces of the 50- to 100-micron-grain-size specimens revealed a change in fracture appearance near 1000 °C. Similar observations, together with electron-microscope fractographs of 300-micron-grain-size specimens of alumina, revealed no observable changes in fracture characteristics from room temperature to 1500 °C.

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I. INTRODUCTION

This is the third quarterly report on the effect of microstructure on the elevated-temperature mechanical properties of selected ceramic-oxide refractory materials. The objective of the current investigation is to determine the effect of grain size on the elevated-temperature transverse bend strength and elastic modulus of pure, highly dense, polycrystalline alumina and magnesia.

Efforts in the third quarter period of this investigation have been concerned primarily with the preparation and testing of alumina specimens having grain sizes of approximately 20, 50, 100, and 300 microns and magnesia specimens having grain sizes of approximately 30, 70, 100, and 200 microns.

II. SPECIMEN PREPARATION

To obtain uniform specimens of a particular grain size of sufficient number for testing, batches of 50 specimens were heat-treated in an air-atmosphere furnace for times and temperatures previously determined to give the desired grain sizes. The alumina specimens were heat-treated at 1700 °C and the magnesia specimens at 1400 °C. (These temperatures were selected as the maximum temperatures at which bloating and warping of the specimens could be avoided.) During the grain-growth treatment, the specimens were contained in a closed impervious alumina tube centered in the furnace. The alumina specimens, which initially had a gray color, were pure white after heat treatment. The alumina specimens exhibited a slight increase in size during the heat treatment, probably due to anisotropic thermal growth and/or possibly some bloating. These specimens did not visually exhibit bloating.

Examination of the microstructure of the alumina specimens after grain growth revealed a nonuniform structure of columnar grains, characteristic of anisotropic grain growth of this pure material. A typical micrograph of this structure is shown in figure 1. The alumina specimens heat treated to obtain a grain size of approximately 300 microns were found to contain several transgranular microcracks. Heat treatment of the magnesia specimens generally produced a uniform structure of equiaxed grains. Grain growth, however, was severely inhibited to a depth of approximately 300 microns from the surface of the specimen. An example of this retarded grain growth in magnesia is shown in figure 2. These specimens were remachined to remove the abnormally small grains near the surface.

A slight discoloration was observed to be associated with the region of inhibited grain growth. A spectrographic analysis over the cross section of one of these specimens revealed no detectable difference in chemical composition between the center and surface regions.

The grain growth treatments changed the average surface roughness of both the alumina and magnesia specimens from approximately 22 μ inches of approximately 45 μ inches.

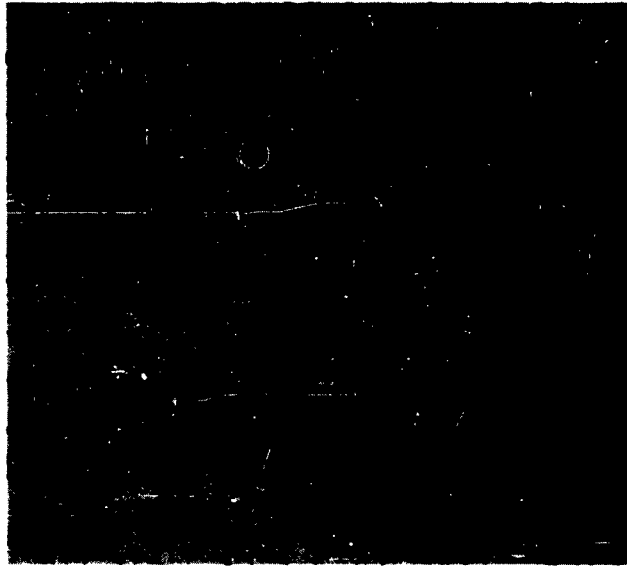


Figure 1 MICROSTRUCTURE OF Al_2O_3 AFTER GRAIN GROWTH TO APPROXIMATELY 300 MICRONS

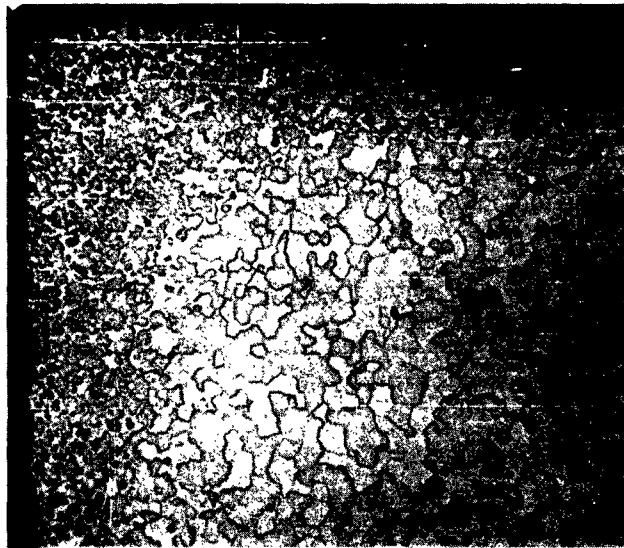


Figure 2 MICROSTRUCTURE OF MgO AFTER GRAIN GROWTH SHOWING INHIBITED GROWTH NEAR THE SPECIMEN SURFACE

III. MECHANICAL TESTING

During the third quarterly period, transverse-bend-strength and elastic-modulus tests were made on alumina specimens having grain sizes of approximately 50, 100, and 300 microns, and on magnesia specimens having grain sizes of approximately 70 and 100 microns. The mechanical test data obtained to date are shown in figures 3 and 4. The data points plotted in figures 3 and 4 for each grain size are the average of three or more individual specimen values, except for the strength of alumina plots in which individual specimen values are shown. The alumina specimens having a grain size of approximately 300 microns exhibited a lower elastic modulus than that previously determined for 1- to 2-micron-grain-size specimens up to approximately 1200 °C. At higher temperature, the elastic modulus of the fine-grained specimens exhibited a greater temperature dependence than did the 300-micron-grain-size specimens. This resulted in the large grain specimens having a higher elastic modulus above 1200 °C.

The 50- and 100- micron-grain-size alumina and 70- and 100- micron-magnesia specimens appeared to have a slightly higher modulus of elasticity than did the 1- to 2- micron specimens. This apparent increase in elastic modulus may have been due to a slight increase in density during the grain-growth treatments; however, post-grain-growth measurements of specimen density revealed no detectable increase in density during the heat treatments. There is reason to believe that the apparent increase in modulus of these specimens is due to a change in calibration of the data recording unit. This possible source of error will be checked during subsequent testing.

The transverse bend strength of alumina shows a strong grain-size dependence of all test temperatures. Above 1000 °C, the strength of the fine-grain alumina appears to fall off more rapidly than does the larger grain alumina.

The transverse bend strength of magnesia appears to be essentially independent of grain size up to approximately 700 °C. At higher temperatures, the larger-grain-size specimens exhibited higher strength than did the fine-grain specimens. Additional test are now being carried out, particularly at higher temperatures, to verify the mechanical-property trends observed to date.

The load-deflection curves for the larger-grain-size alumina and magnesia specimens did not exhibit the yielding and nonlinear behavior observed for the 1- to 2- micron-grain-size specimens at high temperatures. Inspection of the fracture of the 70- and 100- micron magnesia and 50- and 100- micron alumina specimens revealed a rough and irregular fracture up to approximately 1000 °C, generally occurring at a small angle to the tension axis. At higher temperatures, the fractures appeared to be slightly smoother and normal to the tensile axis. The change in fracture appearance with temperature was less obvious than that

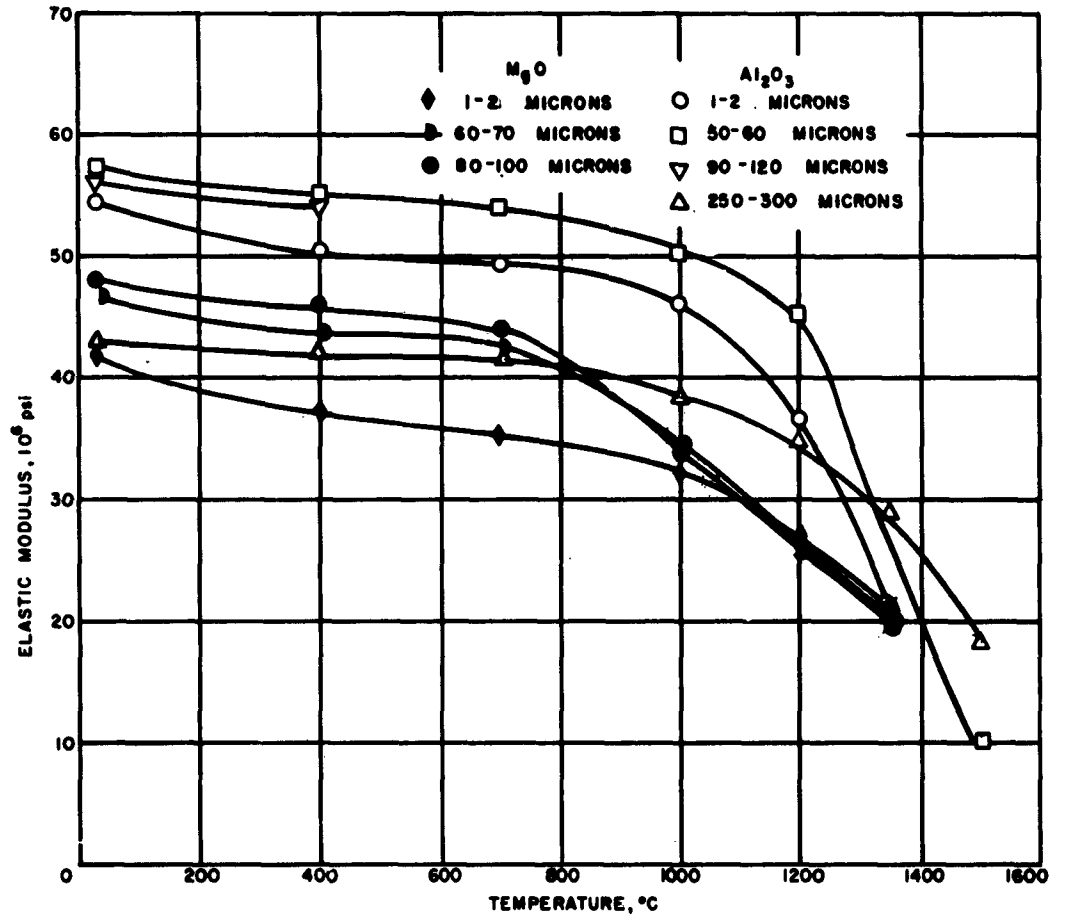


Figure 3 ELASTIC MODULUS OF DENSE MgO AND Al₂O₃ AS A FUNCTION OF GRAIN SIZE AND TEMPERATURE
63-3912

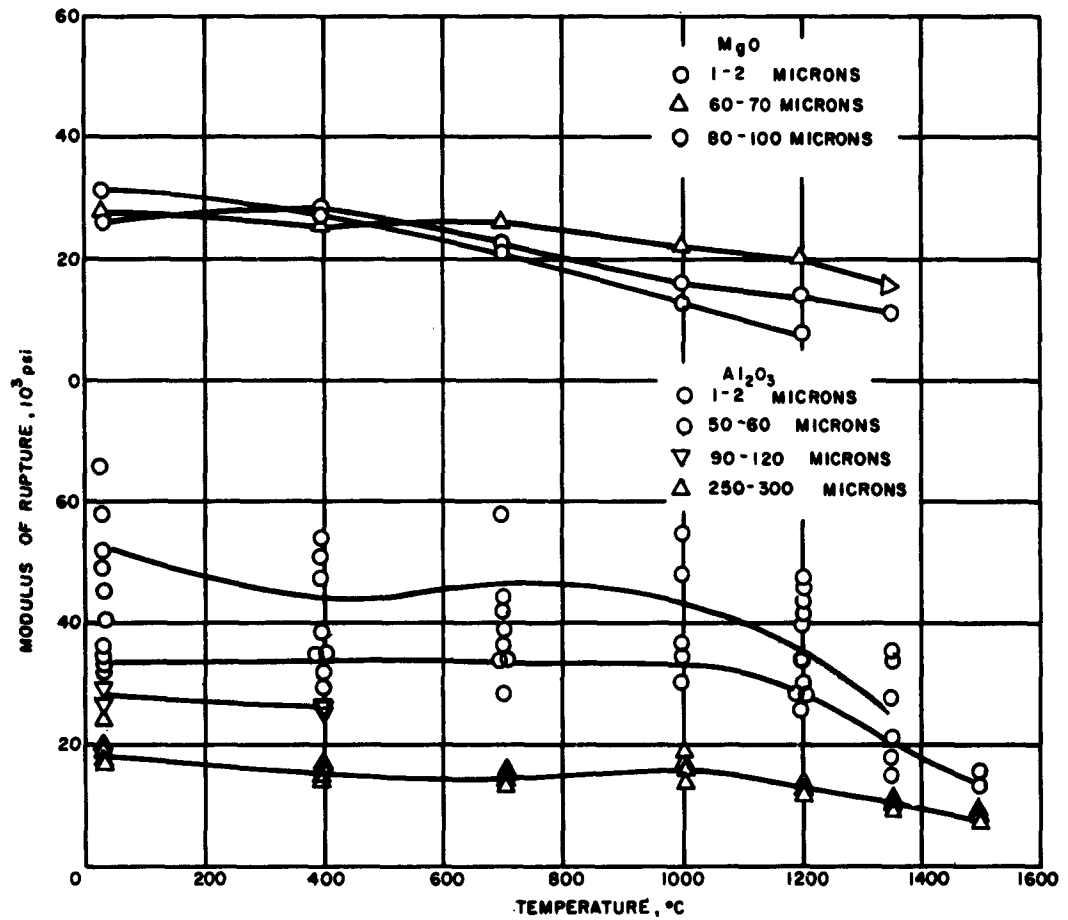


Figure 4 MODULUS OF RUPTURE OF DENSE MgO AND Al₂O₃ AS A
FUNCTION OF GRAIN SIZE AND TEMPERATURE
63-3913

previously observed for the 1- to 2- micron-grain-size specimens. The fracture surfaces of the 300-micron-grain-size alumina specimens were very rough with a granular appearance. These specimens exhibited the same fracture appearance at all test temperatures.

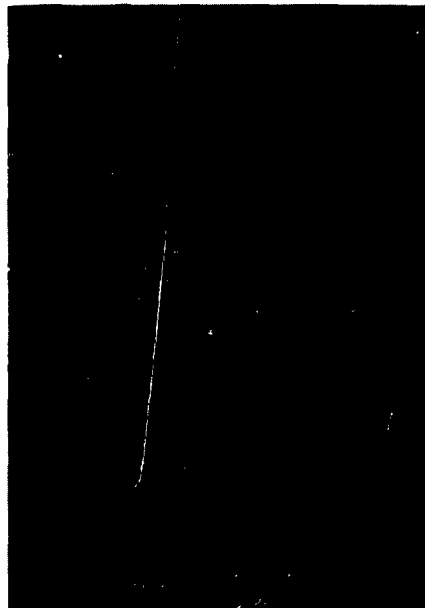
Electron-microscope observations of the fracture surfaces of the 300-micron-grain-size alumina specimens were made. Typical fractures observed in these specimens are shown in figure 5. Fracture in this material generally occurred by both transgranular and intergranular modes with intergranular fracture predominating at all temperatures. There was no evidence of an equicohesive temperature range, where the ratio of intergranular to transgranular fracture increased, as was observed in the alumina specimens having a grain size of 1 to 2 microns.



63C9

400°C

6700X



63C10

400°C

6700X



63D22

700°C

6700X



63D23

700°C

6700X

Figure 5 ELECTRON FRACTOGRAPHS OF 300-MICRON Al_2O_3 SPECIMENS
BROKEN IN TRANSVERSE BENDING AT VARIOUS TEMPERATURES
63-3914



63D29

1000°C

6700X



63D30

1000°C

6700X



63D14

1200°C

6700X



63D15

1200°C

6700X

Figure 5 (Cont'd)
63-3915



63D2 1350°C 6700X



63D3 1350°C 6700X



63D11 1500°C 6700X



63D12 1500°C 6700X

Figure 5 (Concl'd)
63-3916

IV. FUTURE WORK

During the remaining period of this project, it is planned to complete the mechanical test on all specimens prepared for this investigation. Additional tests are planned to verify certain aspects of the reported data and to better reveal the mechanical behavior in the higher-temperature region. An effort will also be made to correlate fracture modes more closely with the mechanical-property trends exhibited by various grain sizes as a function of temperature.

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