**MECHANICAL THINNING OF CERAMIC
MATERIALS FOR TRANSMISSION
ELECTRON AND OPTICAL MICROSCOPY**

T. E. BAUER AND R. H. BEAUCHAMP

APRIL 1968

**AEC RESEARCH &
DEVELOPMENT REPORT**

BATTELLE

NORTHWEST

BATTELLE MEMORIAL INSTITUTE

PACIFIC NORTHWEST LABORATORY

3000 STEVENS DRIVE, P. O. BOX 999, RICHLAND, WASHINGTON 99352

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

PACIFIC NORTHWEST LABORATORY

RICHLAND, WASHINGTON

operated by

BATTELLE MEMORIAL INSTITUTE

for the

UNITED STATES ATOMIC ENERGY COMMISSION UNDER CONTRACT AT(45-1)-1830

PRINTED BY/FOR THE U. S. ATOMIC ENERGY COMMISSION

3 3679 00060 7483

BNWL-652

UC-25, Metals, Ceramics
and Materials

MECHANICAL THINNING OF CERAMIC MATERIALS
FOR TRANSMISSION ELECTRON
AND OPTICAL MICROSCOPY

By

T. E. Bauer
Materials Research Section

R. H. Beauchamp
Materials Development Section

Materials Department

APR 11 1968

April 1968

PACIFIC NORTHWEST LABORATORY
RICHLAND, WASHINGTON

Printed in the United States of America
Available from
Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U.S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

MECHANICAL THINNING OF CERAMIC MATERIALS
FOR TRANSMISSION ELECTRON
AND OPTICAL MICROSCOPY

T. E. Bauer and R. H. Beauchamp

INTRODUCTION

Until very recently, there has been no way to apply the high resolving power of the transmission electron microscope to the investigation of multi-phase ceramic compounds and metal-ceramic composites. The need for this capability is especially acute for cermets and composites because the physical properties often depend critically on the interfacial structure.

The basic problem is one of achieving a sufficiently thin specimen. For good transmission electron microscopy, the specimen must be no thicker than $3000 \text{ \AA}$ ($3 \times 10^{-5} \text{ cm}$). Single-phase specimens can be prepared by chemical, electrochemical, and ionic bombardment techniques.⁽¹⁾ However, because etching rates may differ radically between materials in a multi-phase specimen, these techniques are not satisfactory for metal-ceramic composites or for most multi-phase ceramics. A standard technique suitable for the preparation of specimens from any material was needed.

In 1964, Doherty and Leombruno⁽²⁾ reported a mechanical-thinning technique for glass ceramics. After appropriate modification, this technique was successfully applied to UO_2 , forsterite, a stainless steel - UO_2 cermet, and a titanium - silicon carbide fiber-reinforced composite material. Unfortunately, the abrasive attack produces artifacts by disordering the crystalline lattice. This artificial structure, which masks the real nature of the material and makes interpretation difficult, sometimes can be removed by a light cathodic etch.

DESCRIPTION OF THE TECHNIQUE

Briefly, the mechanical-thinning technique consists of polishing one surface of a specimen, then grinding and polishing the opposite surface until the proper thickness is achieved. A wide variety of abrasives is available. A general technique will be described, and specific abrasive materials will then be listed for each of several materials that have been thinned.

In all cases, samples are first cut to approximately 1/2 in. square by 1/8 in. thick, mounted in Quickmount* casting resin, ground on a 120 grit wet-belt sander, treated further on a 15-micron diamond-impregnated wheel, polished on emery paper, and finally placed on a vibratory polisher. The choice of abrasives and polishing cloths used on the vibratory polisher depends on the sample material. The process and the polishing materials are described in the Appendix.

Mechanical polishing produces an extensive array of surface dislocations. Fortunately, this type of disturbance can be removed from some materials by a light cathodic etch. Though the cathodic etch is inherently preferential in its attack, only 100-500 $\text{\AA}$ are removed, and no significant disparity or relief is generated.

The specimens, polished and cleansed of artificial structure, are mounted in support blocks with their unpolished faces exposed, using highly overcatalyzed Maraglas 655**. Maraglas is an epoxy which, when approximately 100% overcatalyzed, is hard, strongly adhesive, and slightly soluble in acetone. The transparency of Maraglas permits the use of transmission optical microscopy to examine the specimens during the thinning process.

* *Fulton Metallurgical Products Corp., 4710 Ellsworth Avenue, Pittsburgh 13, Pennsylvania.*

** *Marblette Corporation, 37-31 30th Street, Long Island City, New York.*

The unpolished faces of the specimens are ground and polished with the same techniques applied to the first side. The specimens should be ground to about 1/16 in. thickness on the belt sander, abraded to approximately 0.001 - 0.002 in. on the diamond wheel, and made as thin as reasonably possible (about 0.0005 in.) on emery papers. The specimens are then placed on the vibratory polisher equipped with suitable abrasives and cloths, and periodically checked with the optical microscope to determine when the desired thickness has been achieved.

To accomplish transfer of the thinned specimens to the electron microscope, polyvinyl chloride (P.V.C.) is first applied over the thicker center portions of the specimens, leaving a 1 to 3-mm width of the thin edge uncovered. Next, 100-mesh copper screens, cut slightly larger than the specimens, are placed on the specimen surfaces while the P.V.C. is still liquid. When the plastic has dried to form a bond between the specimens and the screens, a 0.002-0.003 in. thick layer of polyvinyl alcohol (P.V.A.) is applied over the entire specimen and screen and allowed to dry overnight. The specimens, screens, and plastic are then cut with a scalpel into 1/4-in. squares, and the usable edges of the specimens centered in the squares. The scored assemblies are immersed in acetone until the samples float free. The squares are then placed in distilled water to clean off the P.V.A. which protected the sample from the slight swelling of the dissolving Maraglas. The P.V.C., insoluble in both acetone and water, binds the specimen to the support grid. The sample, after a final cathodic etch of the second face to remove surface deformation, is placed in the electron microscope.

RESULTS

Transmission electron micrographs of various materials thinned by mechanical grinding are shown in Figures 1 through 9 in the Appendix.

REFERENCES

1. *Howie Hirsch, et al. Electron Microscopy of Thin Crystals. Butterworth & Co., London, pp. 24-50, 1965.*
2. *P. E. Doherty and R. R. Leombruno, Transmission Electron Microscopy of Glass Ceramics, Journal of the American Ceramic Society, vol. 47, no. 8, pp. 368-370, 1964.*

APPENDIX
TRANSMISSION ELECTRON MICROGRAPHS

APPENDIX

TRANSMISSION ELECTRON MICROGRAPHS

Figure Number	Polishing Process, Materials, and Observations
1	Polished in slurry of Linde A* and 2% chromic acid for 12 hr on Met-cloth,** then in same slurry for 30 min on a micro-cloth, and a final polish with Met-polish #6** on a Gamal-cloth** for 15 minutes.
2	Same polishing as for Figure 1. Dark spots are uranium inclusions.
3	Polishing as per Figures 1 and 2. Bumps, removable by light cathodic etch, are polishing artifacts.
4	Polished 13 hr with Linde A on Met-cloth followed by 5 min with Met-polish #6 on a Gamal-cloth. Seven different phases identifiable in this material by electron-diffraction patterns.
5	Polishing as per Figure 4.
6	Polishing as per Figure 1, except chromic acid not added until last hour of 1st step. Dark phase is stainless steel, and white phase is UO_2 .
7	Polishing per Figure 6, but not cathodically etched. Interface is clean and sharp, implying little or no reaction between phases.
8	Polished 12 hr with 1/2-in. diamond slurry*** on a nylon cloth, and a 15-min final polish with Met-cloth #6 on Gamal-cloth. The SiC fiber appears lighter than the Ti matrix.
9	Polishing as per Figure 8. A hexagon-shaped SiC crystal is at one side of micrograph. The interface is not sharp, possibly reflecting reaction between the two phases.

* Linde Co. Crystal Products, Division of Union Carbide Corporation, East Chicago, Illinois.

** Buehler, Ltd., Evanston, Illinois.

*** Pennsylvania Scientific Product Co., Abington, Penn.

Neg 3579

60600X

FIGURE 1. Sintered UO_2

Neg 3578

60600X

FIGURE 2. Sintered UO_2 Showing Uranium Inclusions

Neg 3657

29200X

FIGURE 3. Single Crystal UO_2

Neg 3753

~40,000X

FIGURE 4. Forsterite-Bearing Mineral

Neg 3752

FIGURE 5. Forsterite-Bearing Mineral

~40,000X

58400X

FIGURE 6. Stainless Steel - UO_2 Cermet

Neg 3759

14600X

FIGURE 7. Stainless Steel - UO₂ Cermet

Neg 3745

43800X

FIGURE 8. *Silicon Carbide - Titanium Composite*

Neg 3747

73000X

FIGURE 9. *Silicon Carbide - Titanium Composite*

DISTRIBUTIONNo. of
Copies

2	<u>AEC, Chicago Patent Group</u> G. H. Lee R. K. Sharp
270	<u>AEC Division of Technical Information Extension</u>
2	<u>AEC, Richland Operations Office</u> C. L. Robinson Technical Information Library
1	<u>Lawrence Radiation Laboratory</u> Dr. James Hadley
54	<u>Battelle-Northwest</u> D. E. Baker R. J. Baker J. L. Bates T. E. Bauer c/o P. L. Farnsworth (10) R. H. Beauchamp (10) L. R. Bunnell D. C. Carlson T. D. Chikalla J. A. Christensen J. L. Daniel K. Drumheller E. A. Evans P. L. Farnsworth (10) R. L. Gibby S. S. Jones S. J. Mayhan R. E. Nightingale R. K. Robinson O. D. Slagle F. M. Smith E. A. Snajdr C. E. Vogel Technical Information Files (3) Technical Publications (2)