UCRL-17072

University of California

Ernest O. Lawrence Radiation Laboratory

THINNING OF POLYCRYSTALLINE MgO FOR TRANSMISSION ELECTRON MICROSCOPY

TWO-WEEK LOAN COPY

This is a Library Circulating Copy which may be borrowed for two weeks. For a personal retention copy, call Tech. Info. Division, Ext. 5545

Berkeley, California

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

Submitted to Rev. Sci. Instr.

UCRL-17072
Preprint

UNIVERSITY OF CALIFORNIA

Lawrence Radiation Laboratory
Berkeley, California

AEC Contract No. W-7405-eng-48

THINNING OF POLYCRYSTALLINE MgO FOR TRANSMISSION ELECTRON MICROSCOPY

T. G. Langdon

September 7, 1966

THINNING OF POLYCRYSTALLINE MgO FOR TRANSMISSION ELECTRON MICROSCOPY

T. G. Langdon

Inorganic Materials Research Division, Lawrence Radiation Laboratory, and Department of Mineral Technology, College of Engineering, University of California, Berkeley, California

September 7, 1966

Since the early work of Washburn and co-workers,[1,2] showing that it was possible to use transmission electron microscopy to observe dislocations in MgO in a manner similar to that for metals, a number of papers have been published dealing with the examination of ceramic materials by electron microscopy. These have, however, dealt almost exclusively with work on single crystals. Recently, Hulse and Tice[3] presented a fracture method for obtaining thin foils of a number of polycrystalline ceramics, but a disadvantage of their technique is that it yields only extremely small foils and these rarely contain even low-angle grain boundaries. In this note, a simple method is presented for obtaining thin foils of polycrystalline MgO; the difficulty of thinning material containing a high degree of porosity, and the possibility of introducing mechanical damage into the foil during the preliminary handling, is also discussed.

Two types of material were used in this work. The first, obtained commercially (Honeywell Inc., Minneapolis, Minn.), was opaque, with a porosity of $\sim$1.5%, and an average grain size of $\sim$20μ. The majority of pores in this material were located on the grain boundaries. The second material was produced in this laboratory by hot-pressing MgO

powder with a 3% LiF additive; full details of this technique are given elsewhere.[4] The resultant specimens had an average grain size of ∿15μ, and no observable porosity; the measured density was better than 99.5% theoretical.

To prepare thin foils, small pieces of the material (approx. 3 x 3 x 0.6 cm) were stuck to a micarta block, and carefully sectioned with a diamond saw to produce strips of ∿0.3 mm thickness. The edges of the strips were coated in a masking lacquer, and the strips were then chemically thinned by immersing in ortho-phosphoric acid at 150°C. During this initial procedure, each strip was examined periodically with a binocular eye-piece, and, when the first hole appeared, the strip was removed from the acid and washed thoroughly in distilled water, methyl alcohol and ethyl ether. Additional lacquer was then applied around the hole, and the final thinning carried out by dipping by hand in a fresh solution of ortho-phosphoric acid at 150°C. It was often necessary to repeat this stopping-off process a number of times before the strip was sufficiently thin that a number of small holes appeared in the center. Finally, the specimen was washed thoroughly, and examined under the optical microscope.

Very thin areas suitable for use in the electron microscope (∿1000 Å thickness) were located by the existence of interference fringes at the edges of the holes. A similar technique has also been used with single crystals of MgO[2] and Al_2O_3.[5] A typical example is shown in Fig. 1 for the high-density material; in this case the situation is particularly favorable for electron microscopy, since there is a

wide area of very thin material between the two converging holes. With this material, the edges of the holes were fairly smooth, and there was no preferential attack at the grain boundaries. In contrast, the pores of the lower density material opened up during the chemical thinning, with the result that whole grains were often removed from the edge of the foil. This gave rise to a very irregular edge around the holes, as shown in Fig. 2 where large pores are also visible within the foil, both along grain boundaries (A) and at triple points (B). It is therefore clear that good results, particularly involving studies of boundary structure, can only be obtained with material approaching theoretical density.

An examination of specimens in both the undeformed and deformed states[6] revealed the ease of introducing mechanical damage into the foils during the preliminary handling. In the undeformed condition, the high-density material was relatively dislocation-free, but dislocations could be introduced by the cutting of the foil, as shown in Fig. 3. In practice, best results were obtained by removing very thin portions from the strips with a needle point, but even this may introduce some damage, and it is therefore essential to distinguish between mechanical damage introduced during handling and the genuine properties of the material.

The author is indebted to Larry Ernst for technical assistance.

This work was performed under the auspices of the United States Atomic Energy Commission.

REFERENCES

1. J. Washburn, A. Kelly and G. K. Williamson, Phil. Mag., 5, 192-193 (1960).

2. J. Washburn, G. W. Groves, A. Kelly and G. K. Williamson, Phil. Mag., 5, 991-999 (1960).

3. C. O. Hulse and W. K. Tice, J. Am. Ceram. Soc., 49, 190-194 (1966).

4. M. W. Benecke, N. E. Olson and J. A. Pask, Report No. UCRL-16368, Lawrence Radiation Laboratory, Berkeley, California (1966). (Submitted for publication to the American Ceramic Society.)

5. N. J. Tighe, Rev. Sci. Instr., 35, 520-521 (1964).

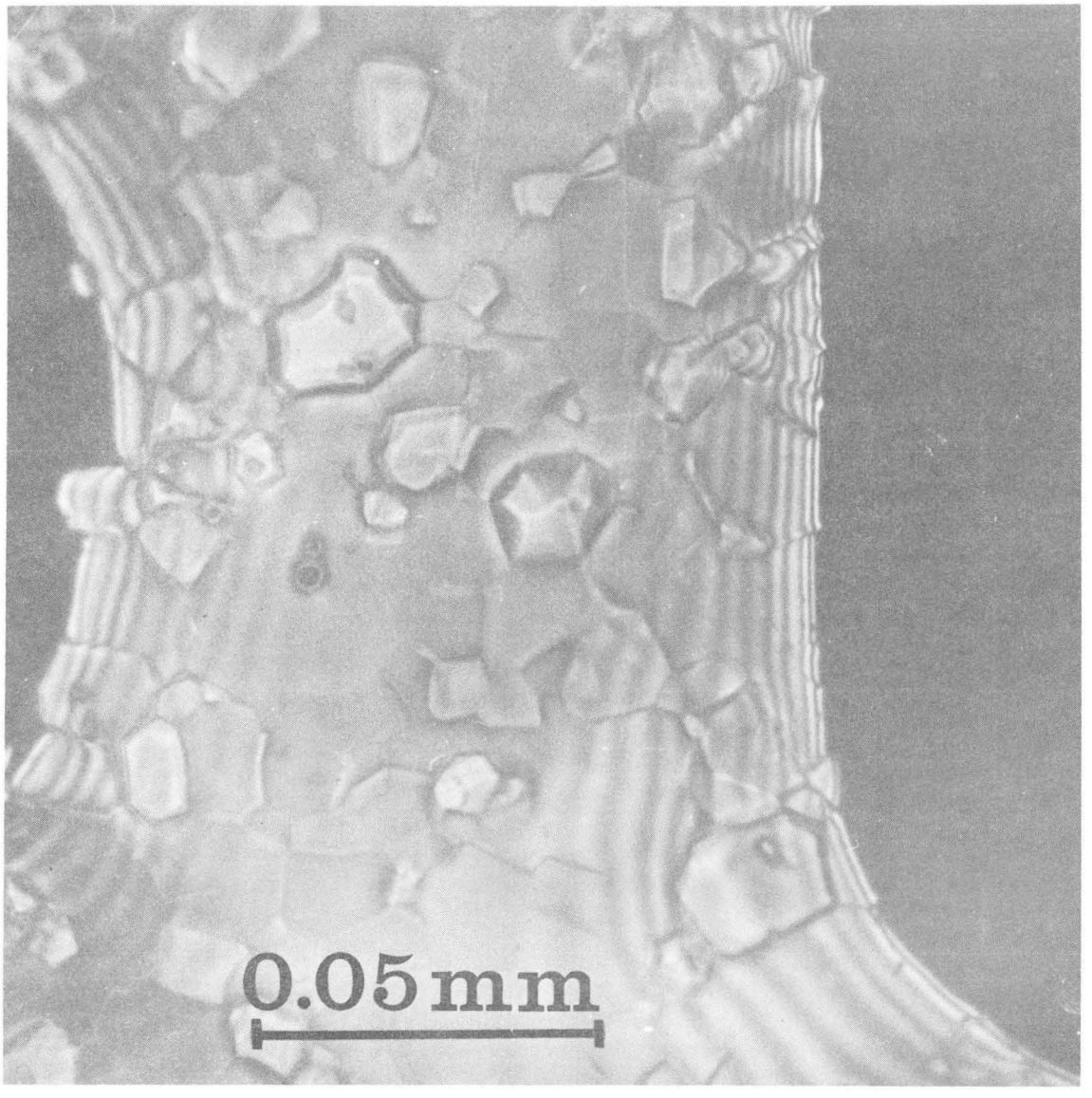
6. T. G. Langdon and J. A. Pask, Proc. Third International Materials Symposium, University of California, Berkeley, June 1966 (John Wiley and Sons, New York), to be published.

FIGURE CAPTIONS

Fig. 1. Optical micrograph of thinned high-density polycrystalline MgO, showing interference fringes in regions suitable for transmission electron microscopy.

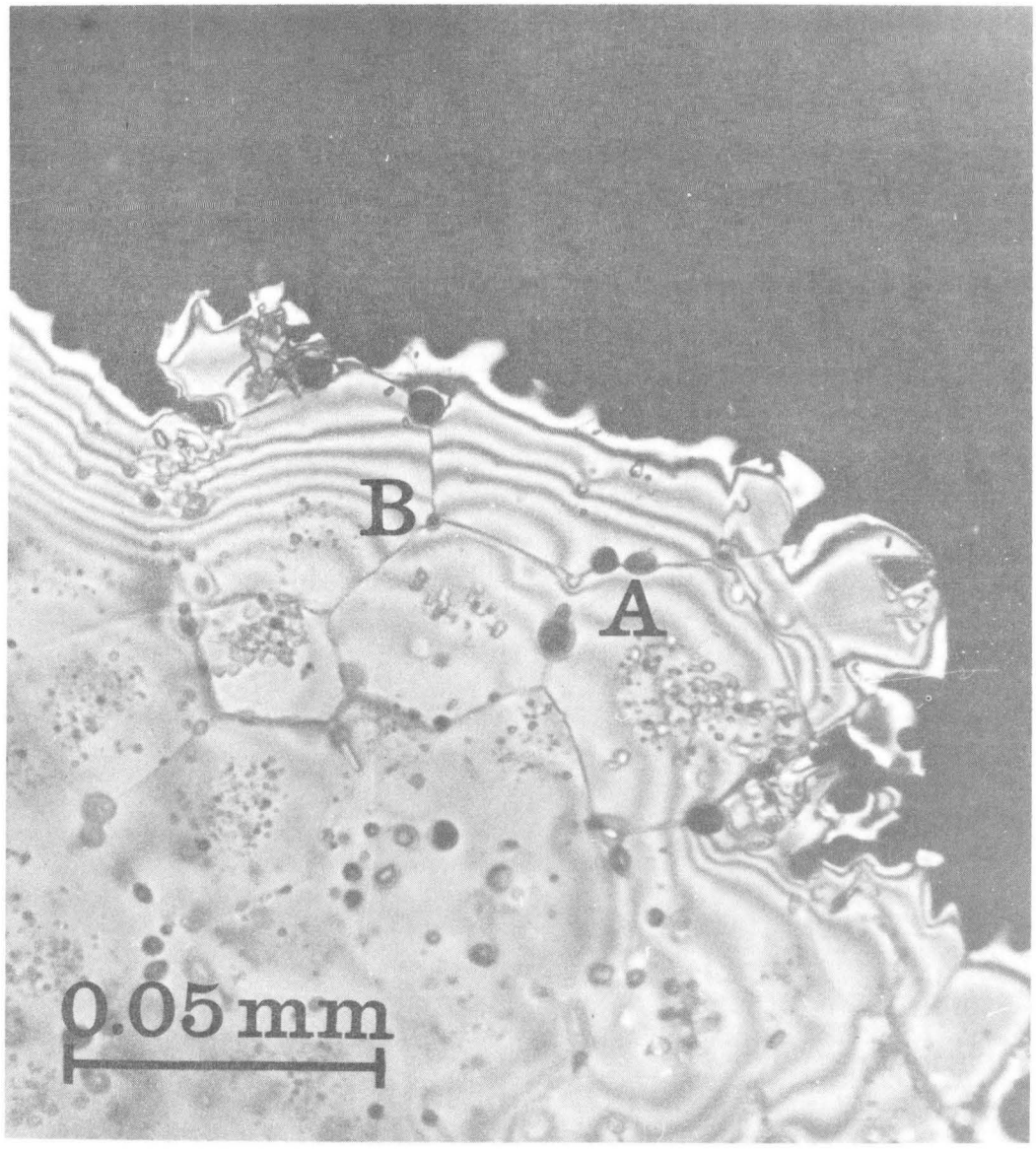
Fig. 2. As for Fig. 1., for lower-density material; pores are visible within the foil along grain boundaries (A) and at triple points (B).

Fig. 3. Transmission electron micrograph of polycrystalline MgO, showing introduction of dislocations into dislocation-free grain by cutting of the foil at point A.



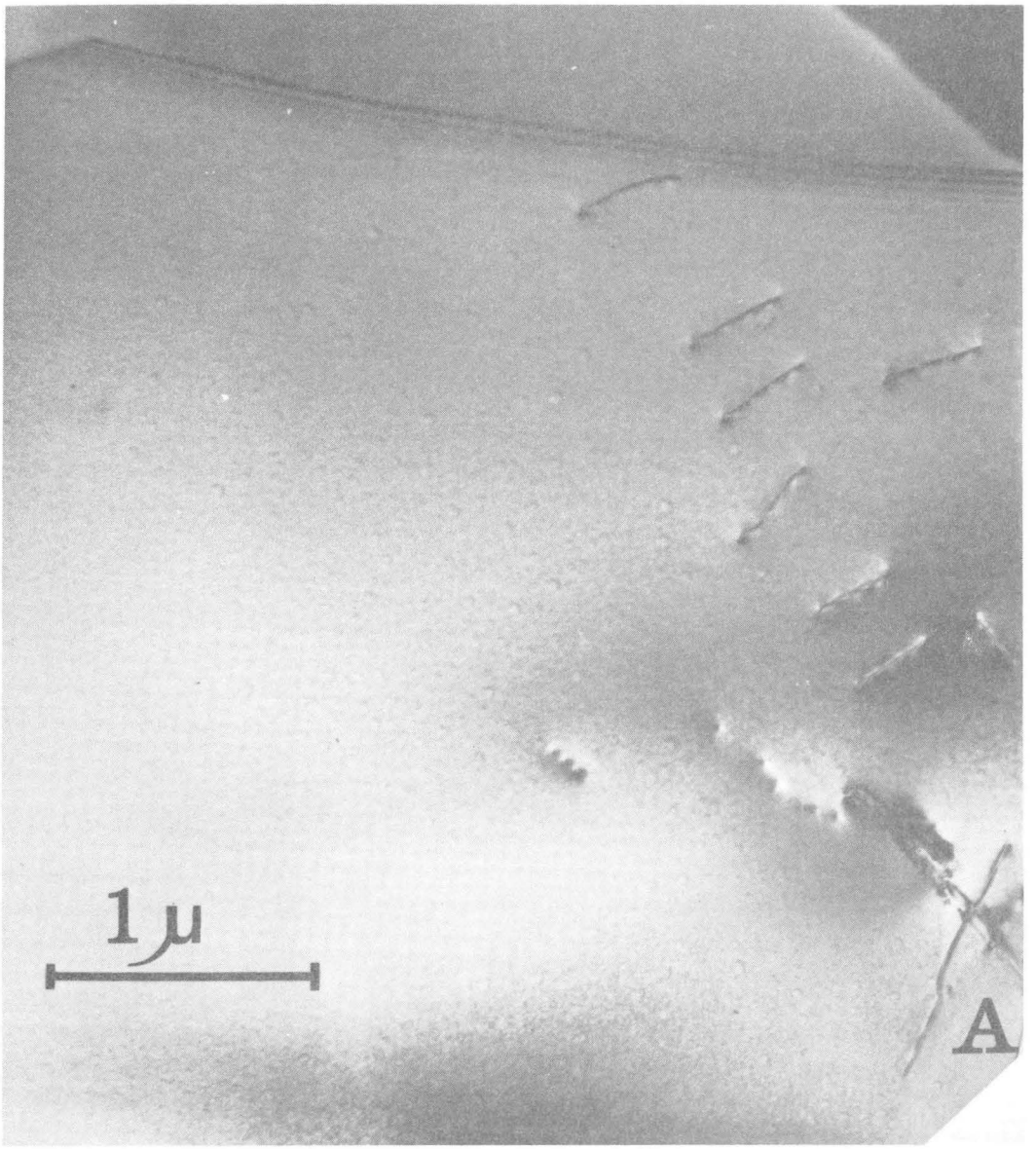
ZN-5920

Fig. 1



ZN-5921

Fig. 2



ZN-5922

Fig. 3

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.