Title
Effect of LiF on Creep of MgO

Authors
Hart, Patrick E
Pask, Joseph A

Publication Date
1971-02-01

Submitted to J. of the
American Ceramic Soc.

UCRL-19699 Rev.
Preprint C.2

EFFECT OF LiF ON CREEP OF MgO

Patrick E. Hart and Joseph A. Pask

February 1971

AEC Contract No. W-7405-eng-48

LAWRENCE RADIATION LABORATORY
UNIVERSITY of CALIFORNIA BERKELEY

DISCLAIMER

EFFECT OF LiF ON CREEP OF MgO

By

Patrick E. Hart* and Joseph A. Pask

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

February 1971

Observations are reported here on the compressive creep behavior of pore-free translucent polycrystalline MgO, containing approximately 0.25 wt% Li, whose density is approximately 99.4% of theoretical density on the basis of single crystal MgO. The Li is believed to be in a fluoride-rich phase along the grain boundaries.[1] Right rectangular parallelepiped specimens were used with length to width ratios of about 1.7. They were cut from disks hot pressed at 900°C and 2250 psi for 2 h. Initial concentration of LiF was 3 wt%. Grain size after hot pressing was less than 1 μm.[1]

Modified creep experiments were performed on a translucent hot-pressed specimen, and also on a specimen that had been additionally annealed at 1300°C for 3 h in air. The annealed specimen was transparent and had a density >99.9% TD. Strain behavior at 750 psi and constant heating rate of 250°C/hr is shown in Fig. 1. The transparent annealed specimen showed only thermal expansion. The strain of the hot-pressed specimen was observed to first deviate from the expansion curve of the annealed specimen at approximately 630°C with a large increase in the

---

This work was done under the auspices of the U.S. Atomic Energy Commission.
*Now at Battelle-Northwest, Richland, Washington.

strain rate occurring at approximately 830°C. After the test, the unannealed specimen was transparent, the Li was <500 ppm, and the bulk grain size was 10-20 μm. Development of transparency which is associated with LiF loss also occurs during annealing at 900°C and above.

The incremental stress technique was used to determine the stress dependence of the strain rate at constant temperatures of 770 and 850°C. Loads were changed after several percent strain. Specimen strain rates exhibited rapid response to changes in stress as shown in Fig. 2 for a series run at 850°C. A strain rate-stress dependence of 1.2 was determined at 770°C and 1.1 at 850°C. True strains in excess of 0.25 were reached without the onset of tertiary creep of specimen failure. Tests were discontinued when strain rates were observed to start to fall rapidly with time. This effect is attributed to the loss by evaporation of the LiF during the test.

These creep results may be compared with compressive creep data obtained by Langdon and Pask[3] at 1200°C on transparent annealed MgO specimens. They measured a strain rate of 1.1×10^{-3} h^{-1} at 10,000 psi as compared with a strain rate of 1.1×10^{-1} h^{-1} obtained at 242 psi and 850°C in the present work. A stress exponent of 3.3 was reported which suggests that creep of the annealed specimen was controlled by a dislocation climb mechanism. The presence of LiF, therefore, does not only enhance strain rate but changes the strain-rate stress dependence as well.

It has previously been deduced that densification during hot pressing of MgO containing low concentrations of LiF is controlled by flow of a liquid phase* along grain boundaries.[1] In this model, it was assumed that the liquid phase which serves as a mass transport medium behaved in a Newtonian or pseudo-Newtonian manner. Using simplified arguments, a strain-rate stress dependence of unity was derived for densification behavior. This mechanism may also account for the observed creep in the present work.

Morgan[4] has suggested that the effect of LiF additives may be a manifestation of superplasticity. In its narrowest usage, superplasticity refers to a creep process under tensile loading which is characterized by anomolously high elongation with no necking and is related to a phase transformation or small grain sizes. Therefore, in the narrowest sense, the present work should not be considered as an example of superplasticity because it does not meet all the criteria. However, in the broadest meaning of superplasticity, i.e., an exceptional deformation without cracking as indicated by no loss of transparency, the present work is an example of superplasticity.

---

* Although LiF melts at 842°C, liquid was believed to appear at approximately 600°C due to LiF reactions with adsorbed hydroxides and carbonates on the MgO particle surfaces.

REFERENCES

1. Patrick E. Hart, Robert B. Atkin and Joseph A. Pask, "Densification Mechanisms in Hot-Pressing Magnesia with a Fugitive Liquid," J. Am. Ceram. Soc., 53 (1970).

2. B. J. Skinner, "The Thermal Expansion of Thoria, Periclase and Diamond," Amer. Min., 42 (1 and 2) 39-55 (1957).

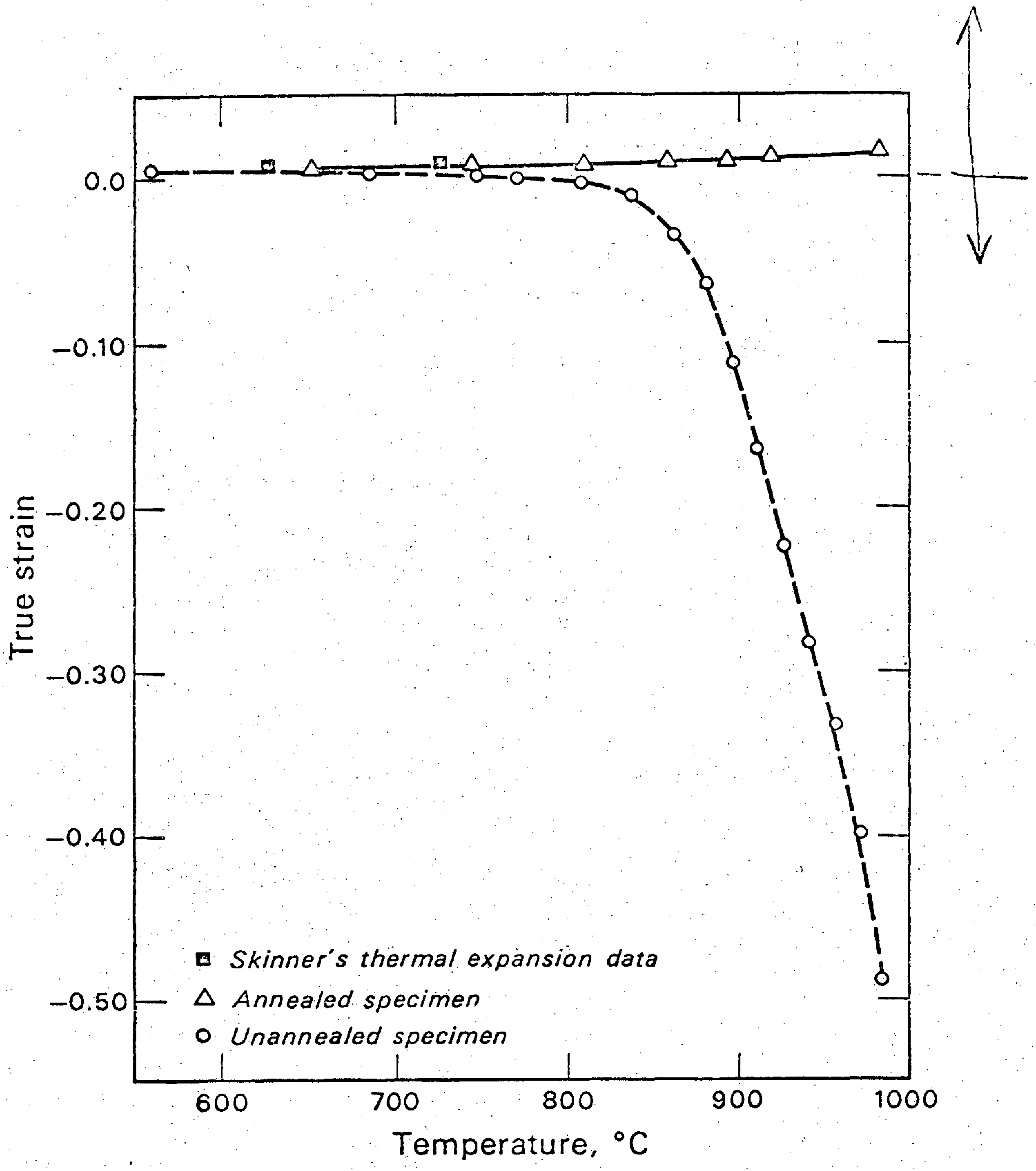
3. Terence G. Langdon and Jsoeph A. Pask, "The Mechanism of Creep in Polycrystalline Magnesium Oxide," Acta Met., 18, 505-510 (1970).

4. Peter E. D. Morgan, "Superplasticity in Ceramics," 251-270 in Ultrafine-Grain Ceramics, edited by J. J. Burke, N. L. Reed, and V. Weiss, Syracuse University Press, Syracuse, N. Y. (1970).

FIGURE CAPTIONS

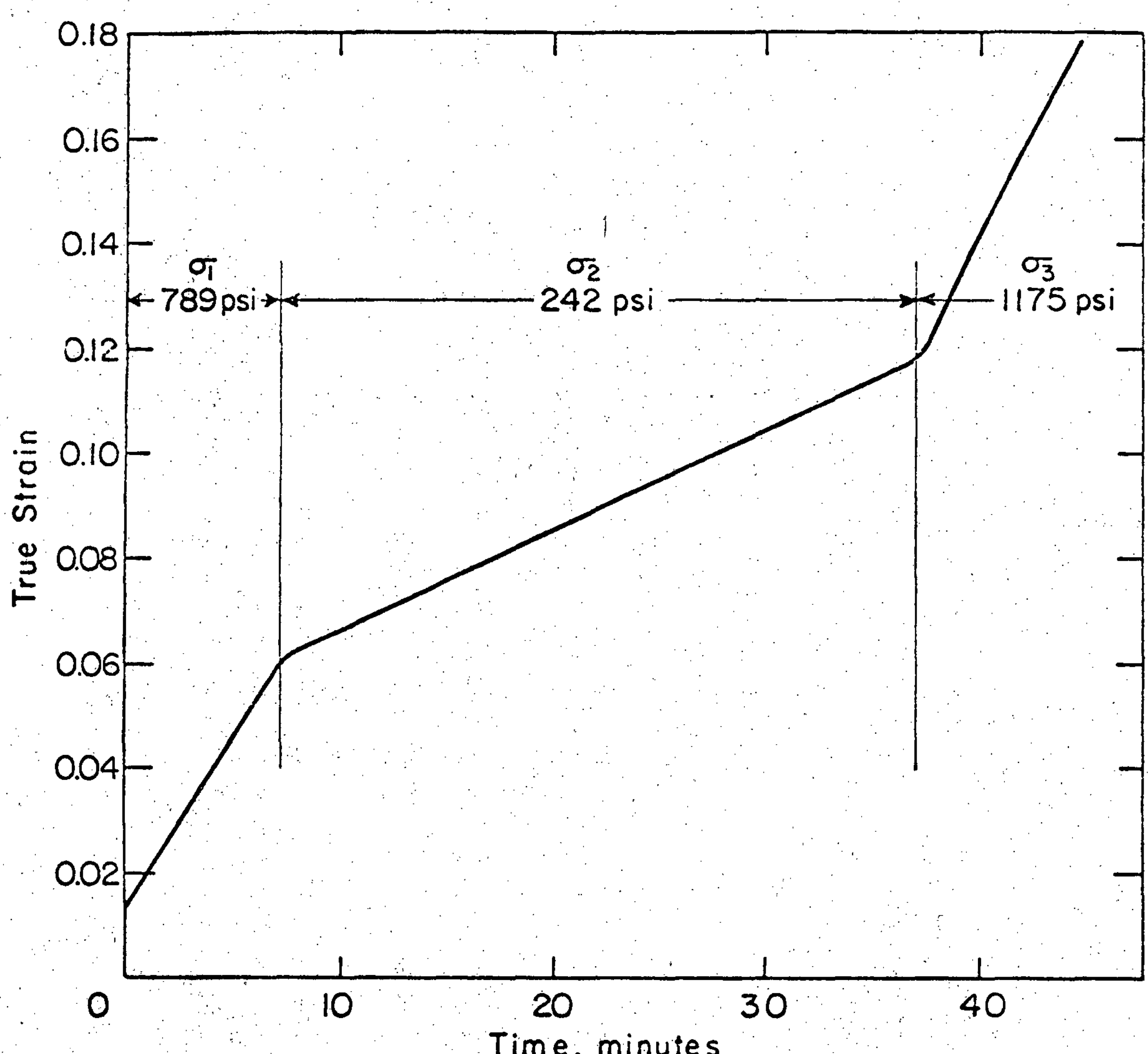
Fig. 1. True strain on heating annealed and unannealed specimens at a rate of about 250°C/h under a stress of 750 psi. Two points based on thermal expansion data of Skinner[2] are shown.

Fig. 2. Response of strain rate of specimen to changes in stress during test at 850°C.



XBL 6712-1984

Fig. 1. True strain on heating annealed and unannealed specimens at a rate of about 250°C/h under a stress of 750 psi. Two points based on thermal expansion data of Skinner[7] also shown.



XBL 708-1918

Fig. 2. Response of strain rate of specimen to changes in stress during test at 850°C.

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.

TECHNICAL INFORMATION DIVISION
LAWRENCE RADIATION LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720