Mechanical Spectroscopy of High-Purity MgO

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Mechanical spectroscopy on high-purity MgO at temperatures of 20-1300 °C and oscillation periods between 1 and 1000 s results in a significant viscoelastic deformation at temperatures above 800 °C. At lower temperatures the distortion of the sample is essentially elastic. No dissipation peak superimposed on the dissipation background is measured in this high-purity MgO sample.

1. Introduction

The nature of the breakdown of elasticity in materials stressed at sufficiently high temperatures has important implications for structural applications of ceramic materials (e.g. refractories for steel-making and substrates for electronic devices), yet remains poorly understood. In this study, mechanical spectroscopy experiments on an MgO polycrystalline material have been performed at high temperatures and pressures to clarify these fundamental aspects of elasticity breakdown in ceramics.

2. Sample preparation and mechanical testing

A high-density MgO sample ($\phi < 1\%$) with a grain size of 10 μm has been fabricated from > 99.98 wt. % purity MgO powders with an initial grain size of 50 nm size (supplied by Ube Materials Industries Ltd.) by a four-stage fabrication process: (1) Cold isostatic pressing of the powder at 200 MPa confining pressure for >30 minutes in a pressure vessel with kerosine as confining medium followed by (2) Firing at 1100 °C for 2 hours at 1 atmosphere and in N_2 an rich environment to evapourate residual CO_2 and H_2O from the material (3) Hot-isostatic pressing of the fired cylindrical pellets in a gas-medium pressure apparatus at 300 MPa and 1100 °C for 24 hours to decrease the porosity to < 1%, (4) Continued hot-isostatic pressing at 1300 °C for another 24 hours to facilitate grain growth to a mean grain size of 10 μm.

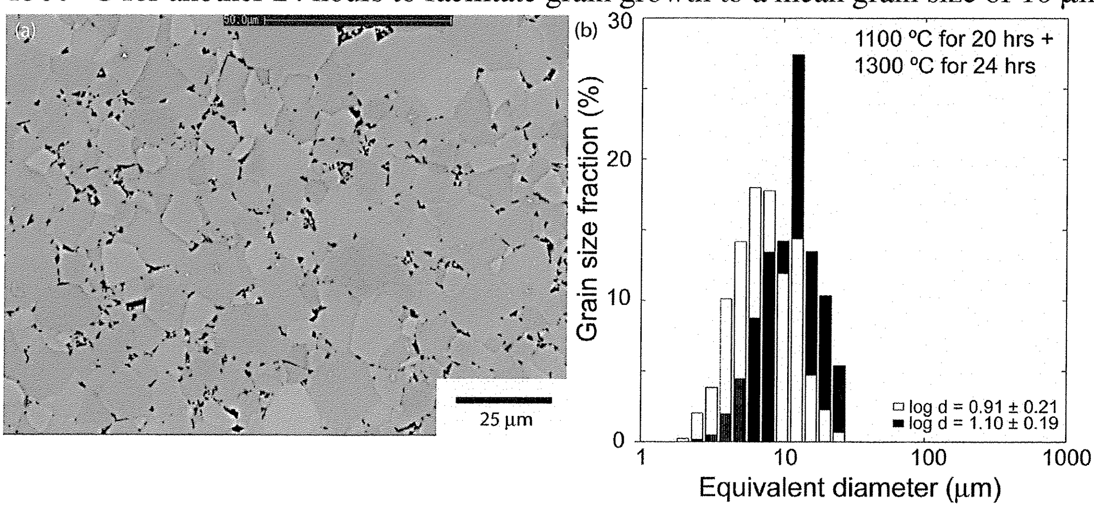

Fig. 1. (a) Backscatter electron microscope image (Note the equant grains and widely distributed small pores) and (b) grain size frequency histogram (number fraction; white bars, area fraction; black bars).

The observed low porosity at 1100 ºC hot-isostatic pressing conditions is similar to previous results on fabrication of high-density MgO [1]. The cylindrical sample of 11.5 mm in diameter and 30 mm in length was tested in a torsional forced-oscillation apparatus at a confining pressure of 200 MPa [2]. Shear modulus (G) and dissipation (Q^{-1}) were determined as functions of oscillation period ranging from 1 to 1000 s at temperatures of 20 to 1300 ºC. At each temperature, shear modulus and dissipation data were fitted to the Andrade creep function J(t) [3] to describe the elastic, transient and steady-state Newtonian response during deformation (Fig. 1).

$$J(t) = J_u + \beta t^n + t/\eta \tag{1}$$

J_u is the unrelaxed compliance describing the elastic deformation, β and n are constants describing the recoverable anelastic deformation and η is the steady-state viscosity.

3. Results

3.1 Breakdown of elasticity

Torsional forced oscillation experiments on MgO result in a shear modulus *G* at 20 ºC of 124 GPa, which is only slightly lower than the theoretical MgO shear modulus of 132 GPa[4], reflecting a low residual porosity. At temperatures up to ≤ 800 °C for all oscillation periods (1-1000s) deformation is accommodated elastically, i.e absence of any frequency dependent shear modulus *G* and no significant dissipation Q^{-1} (Fig. 2). At temperatures of ≥ 900 °C, both *G* and Q^{-1} become frequency dependent. *G* decreases ~70% from an oscillation period of 1s to 1000s at 1300 °C, indicating a large contribution of anelastic and viscous behaviour.

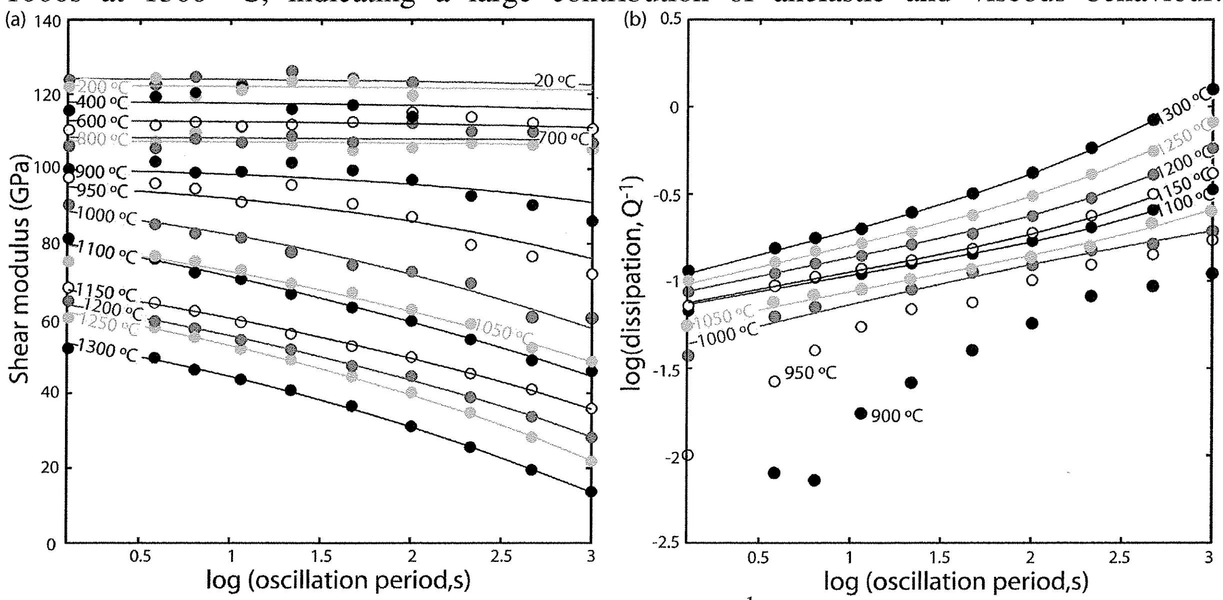

Fig. 2. (a) Shear modulus (G) and (b) log (dissipation,Q^{-1}) versus log(oscillation period) for temperatures between 20-1300 ºC. Symbols are measured values, solid lines represent Andrade model fits for each temperature.

The dissipation data with variable slopes (Fig. 2b) is not well fitted by a simple dissipation background model. The strongest frequency dependences occur at short periods at 900-950 °C and at long periods at 1250-1300 °C. These trends may reflect the presence of a very broad dissipation peak on top of the dissipation background. However, global modeling of the available *G* and Q^{-1} data with the extended Burgers model for a superimposed dissipation peak [5] doesn't fit the data any better than a model involving a dissipation background approach only. Thus, there is no statistically significant evidence for the dissipation peak in this study as observed in a previous study on polycrystalline MgO [6].

3.2 Effect of material purity

Fig. 2 shows the comparison of mechanical spectroscopy data of log dissipation versus 1/T for MgO samples for two studies (this study and [6]). A smooth monotonic decrease in dissipation with temperature in this study (a) deviates strongly from results obtained in a previous study (b), [6], in which a distinct dissipation peak is present. The samples differ in particular in purity of the material, grain size distribution and porosity. Impurities leading to the presence of secondary phases on grain boundaries regions including ~ 1 wt.% CaO, SiO_2, H_2O and CO_2 of the previous study [6] are likely to have enhanced grain boundary sliding processes potentially accounting for the presence of a dissipation peak in addition to the frequency-dependent dissipation background. In the pure MgO sample (this study) enhanced grain-boundary-sliding processes may be inhibited explaining the absence of a dissipation peak.

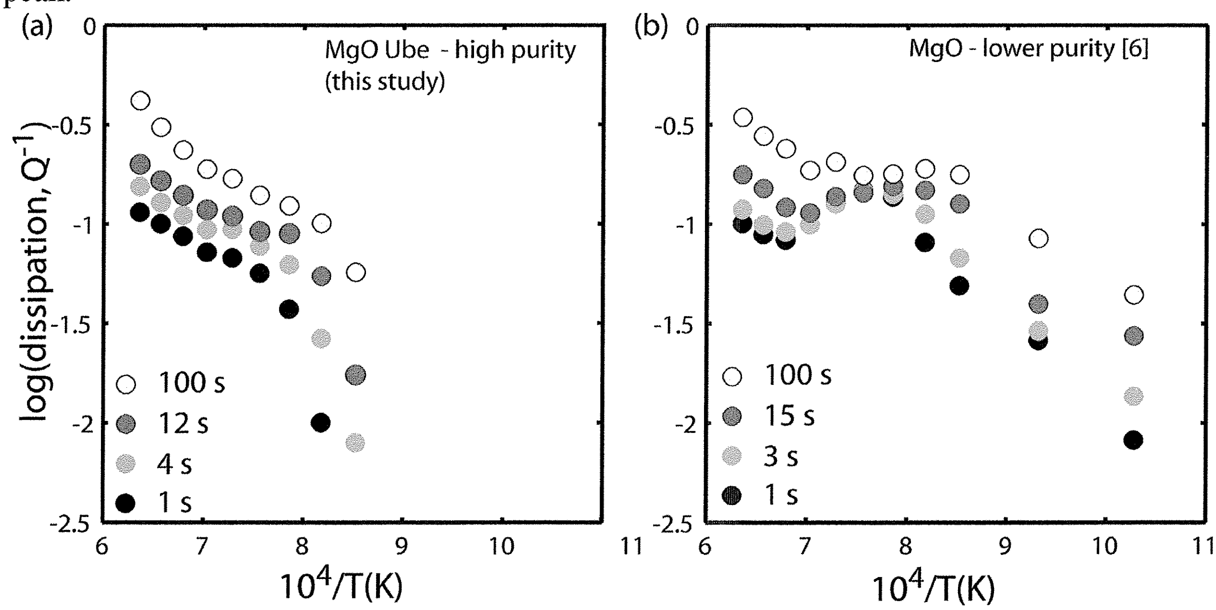

Fig. 3. Effect of MgO purity on formation of a dissipation peak.

Acknowledgments

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References

[1] K. Itatani, R. Yasuda, F. Scott Howell and A. Kishioka, *J. Mat. Sc. Lett.* **32**, 2977 (1997).

[2] I. Jackson and M.S. Paterson, *Pure Appl. Geophys.* **141**, 445 (1993).

[3] K.E. Amin, A.K. Mukherjee and J.E. Dorn, *J. Mech. Phys. Solids* **18**, 413 (1970)

[4] O.L. Anderson and D.G. Isaak, In: *Mineral Physics and Crystallography: A Handbook of Physical Constants,* 64 (1995).

[5] I. Jackson, In: *Advances in High-Pressure Technology for Geophysical Applications*, 95 (2005).

[6] S. Webb and I. Jackson, *Phys. Chem. Min.* **30**, 157 (2003)