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Deformation behavior and joining of a MgF_2 optical ceramic

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Abstract

Compressive deformation behavior of a polycrystalline magnesium fluoride (MgF_2) ceramic was investigated at temperatures ranging from 760 to 830 °C in an argon atmosphere at strain rates between 2×10^{-6} and 4×10^{-5} s^{-1}. Steady-state flow stresses increased with increasing strain rates and ranged between 2 and 38 MPa. Stress exponents of $\approx 1.4 \pm 0.2$ were determined at temperatures >760 °C, indicative of a viscous diffusion-controlled deformation mechanism. Activation energy, determined from flow stress as a function of temperature, at a constant strain rate, was $\approx 476 \pm 60$ kJ/mol. Self-joining by plastic deformation of MgF_2 was demonstrated at 830 °C at a strain rate of 5×10^{-6} s^{-1}. The joined samples were characterized by optical transmission measurements and their transmittivity was ≈80% of the unjoined sample in the 2.5–8 μm wavelength range.

Keywords: Magnesium fluoride; Optical properties; Creep

1. Introduction

Polycrystalline magnesium fluoride (MgF_2) is almost transparent in the infrared (IR) wavelength regime (2–8 μm).[1,2] Due to this unique property, MgF_2 has applications for IR windows and coatings in optical instruments, space vehicles, and frontal domes in heat-seeking missiles.[1–3] Magnesium fluoride is an excellent candidate for thermal lensing because it has one of lowest temperature dependent refractive index distortions.[3] As compared to IR transparent single crystals, polycrystalline MgF_2 is relatively simple and inexpensive to fabricate. However, fabrication of complex shaped engineering components using polycrystalline MgF_2 still remains a major challenge.

Polycrystalline MgF_2 is fabricated using typical ceramic processing routes by hot-pressing magnesium fluoride powder in a die in a controlled environment.[1] This process allows fabrication of simple shapes. One approach that is being considered is to join multiple simple-shaped parts to produce a complex part. In this regard, development of a joining process for optical materials such as MgF_2, without degradation of optical characteristics, becomes important.

In the literature, self-joining of MgF_2 has been demonstrated by a diffusion bonding process without an interlayer[4] or using a lithium fluoride (LiF) interlayer.[5] Diffusion bonding entailed applying a thin film of LiF on the mating surfaces of the pieces being joined and maintaining a constant compressive stress for >3 h at 550–575 °C. Without any interlayer, diffusion bonding was accomplished by applying constant pressure at 800–830 °C for about 6 h[4]. Limitations of this process are two-fold: (a) long hold times at elevated temperatures which can result in grain growth and (b) use of an extraneous interface material. Both factors can cause degradation of the optical and mechanical properties of the joined component. Thus, plastic deformation, as discussed in this paper, presents an approach to self-joining MgF_2 that precludes the limitations imposed by the diffusion bonding process.

High-temperature plastic deformation process has been demonstrated for joining various structural ceramics and composites.[6–9] The joining process requires heating the components to elevated temperatures and deforming them at a constant displacement rate such that total strains are typically <10%. The mechanism by which pore-free joints are obtained is due to the interpenetration of grains at the interface as the result of grain boundary sliding (GBS).[6–9] There are several attributes that make the plastic joining process attractive: (a) it requires no surface preparation or interlayer material, (b) permanent deformation is minimal, and (c) the process occurs at lower temperatures and for shorter times relative to the conventional diffusional bonding.[10] The interface formed by plastic deformation is indistinguishable from the bulk material.[9] This attribute

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is particularly significant when optical properties are of concern as in the case of joining of polycrystalline MgF_2.

High-temperature deformation behavior of polycrystalline MgF_2 has not been reported. Because of the importance of deformation in processing and fabrication of components, a study of high-temperature deformation behavior of a polycrystalline MgF_2 ceramic was undertaken. In addition, self-joining of MgF_2 using plastic deformation was explored, so that complex shaped parts could be fabricated by joining simple geometries. Finally, the quality of joined MgF_2 components was established by microstructural and optical characterizations.

2. Experimental details

2.1. Polycrystalline magnesium fluoride

Polycrystalline magnesium fluoride samples were procured from a commercial source (Superconix Inc., Lake Elmo, MN). Samples were fabricated by hot-pressing MgF_2 powders. As-received samples were 10 mm × 10 mm × 40 mm. The density of as-received samples was determined by the Archimedes method. The microstructure of the sample was examined on the fracture surface using a Hitachi Model S-4700-II (Tokyo, Japan) field emission scanning electron microscope (FE-SEM). Grain size was measured by the standard linear intercept method.[11] Phase analysis was conducted by room-temperature X-ray diffraction (XRD) measurements using a Phillips X-ray diffractometer (Model X'PERT, Almelo, The Netherlands) with a Cu $K\alpha_1$ radiation. Data acquisition was conducted using a step-scan program run at 100 s/° with a step size of 0.01° over a 2θ range of 20–75°. In addition, stability of the MgF_2 phase was characterized by conducting XRD analysis on samples that were deformed and compared with the as-received samples.

2.2. Deformation study and joining

For deformation and joining experiments, right parallelepiped specimens (≈3 mm × 3 mm × 5 mm) were cut from as-received MgF_2 samples using a low speed diamond saw. Surfaces were as-cut. Specimens were subjected to uniaxial compression, at a constant cross-head displacement rate on an Instron machine (Model 1125, Canton, MA) equipped with an atmosphere-controlled high-temperature furnace.[9] All deformation and joining tests were conducted in an argon atmosphere. The furnace chamber was flushed with argon atmosphere prior to joining or deformation. Deformation tests were conducted in the temperature range from 760 to 830 °C at strain rates varying from 2×10^{-6} to $4 \times 10^{-5}\ s^{-1}$. For joining tests, two samples of MgF_2 were mated and compressed. To ensure plastic deformation of MgF_2 during joining, strain rate and joining temperature were selected on the basis of results from the deformation study of MgF_2. The joining temperature selected was 830 °C at a strain rate of $5 \times 10^{-6}\ s^{-1}$. All joinings were completed within 0.5 h. The total permanent strains introduced during the plastic joining process ranged from ≈0.05 to 0.08.

Microstructure of the as-received and deformed samples was examined by transmission electron microscopy (TEM). TEM foils were prepared by first sectioning thin slices using a diamond blade and then further thinning by mechanical polishing. Thereafter, samples were ion milled using a Gatan (Pleasanton, CA) Model 691 precision ion polishing system. TEM was conducted using a Phillips CM-200 electron microscope operating at 200 kV.

After joining, samples were sectioned, polished, and the joint interface was examined using an SEM (Hitachi S-4700, Tokyo, Japan). In addition, efficacy of the joint was established by conducting optical transmission measurements on samples before and after joining. Optical transmission measurements were conducted using a Brucker (Billerica, MA) Vertex 70 Fourier Transform Infrared (FTIR) spectrometer running OPUS software. This spectrometer has a resolution of $0.5\ cm^{-1}$. Prior to evaluating the optical transmission, sample surfaces were polished to a mirror finish and cleaned in acetone.

3. Results

The density of the as-received MgF_2 measured by the Archimedes method was $3.1\ g/cm^3$, which was approximately 98% of the theoretical density of MgF_2 taken as $3.17\ g/cm^3$.

Fig. 1 is a photomicrograph of a fractured surface of as-received MgF_2. The microstructure was uniform with an average grain size of 0.1–0.2 μm. In addition, the fracture morphology was intergranular. Fig. 2 shows the XRD pattern of an as-received MgF_2 sample for which the major peaks correspond to magnesium fluoride.

Fig. 3 shows the typical stress-strain behavior obtained during compressive deformation of polycrystalline MgF_2 at different strain rates. Initially, the stress increases linearly (elastic regime) with applied strain followed by deviation from linearity, indicative of plastic deformation. With further increase in strain, a steady-state stress was reached which is referred to as the flow stress. However, at the highest strain rates such as $1 \times 10^{-5}\ s^{-1}$ at 828 °C (Fig. 3), there was evidence of work hardening. In such situations, it was difficult to determine the appropriate steady-state flow stress. In addition, there was an increase in the flow stress at a given strain rate after some strain had accumulated

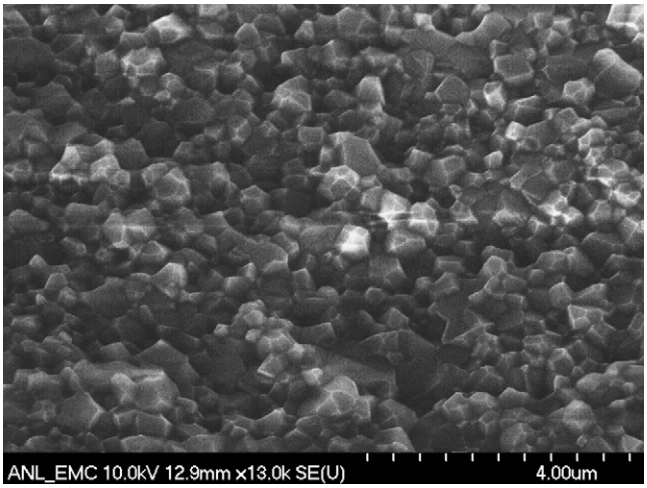

Fig. 1. Fracture surface of the as-received polycrystalline MgF_2.