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POLYCRYSTALLINE LITHIUM ORTHOSILICATE

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MECHANICAL PROPERTIES AND DEFORMATION OF POLYCRYSTALLINE LITHIUM ORTHOSILICATE*

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ABSTRACT

Room-temperature strength, fracture toughness, Young's modulus, and thermal-shock resistance were determined for 68 to 98% dense lithium orthosilicate (Li_4SiO_4) specimens. In the low-density regime, both strength and fracture toughness were controlled by the density of the specimen. At high density, the strength depends on grain size. Young's modulus values ranged from 30 to 103 GPa at densities between 68 and 98% TD. A critical quenching temperature difference in the range of 150-170°C was observed in thermal-shock tests of bar specimens. Steady-state creep tests indicated that 90% dense Li_4SiO_4 fractures at $T \leq 800^\circ\text{C}$ before reaching steady state and deforms plastically at 900°C. It is more creep-resistant at 900°C than Li_2O , about equal to Li_2ZrO_3 , and less than LiAlO_2 .

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1. INTRODUCTION

Lithium-bearing ceramics are being considered as blanket materials for the breeding of tritium in fusion power reactors [1]. Lithium orthosilicate (Li_4SiO_4) is a potential breeder material because of its high lithium content and high stability relative to other lithium-containing ceramics. Room-temperature strength and toughness and high-temperature creep resistance are important properties relative to installation and operation of breeder blankets, and are therefore essential for designing an efficient blanket. Li_4SiO_4 was prepared and fabricated by a conventional ceramic method [2]. Its mechanical properties were determined for different densities and grain sizes. The results of high-temperature tests were compared with those for three other candidate materials (Li_2O , Li_2ZrO_3 , and LiAlO_2).

2. EXPERIMENTAL PROCEDURES

Lithium orthosilicate powder was prepared by the solid state reaction of lithium oxide and amorphous silica. For details of the procedure, see Ref. 2. As-sintered rectangular bars (49×6×3 mm) with various densities and grain sizes were used in flexural strength and fracture toughness tests. Flexural strength was measured in a four-point bending mode with an Instron testing machine. The support span was 44 mm, the loading span was 22 mm, and the crosshead speed was 1.27 mm/min. The number of samples for fracture strength measurements was between 8 and 10 for each variation in microstructure. Fracture toughness was measured by the single-edge notched beam (SENB) technique [3] with a notch width of 0.6 mm. The specimens were ground to a

surface finish of 600 grit. For fracture toughness measurements, 3 to 5 specimens were used per condition. Elastic modulus was determined by ultrasonic velocity measurement [4]. Thermal-shock resistance was determined by measuring the flexural strength of 49×6×3-mm specimens before and after quenching from various high temperatures into silicone oil (5 centistoke viscosity) at room temperature. All the above experiments were done under laboratory conditions (room temperature, 60% relative humidity).

High-temperature constant-strain-rate tests were performed in a 99.9% argon gas atmosphere in direct compression with an Instron Model 1125 universal testing machine. 90% dense cylindrical pellets ($L = 5.0$ mm, $D = 3.6$ mm) were heated to temperatures ranging from 750 to 900°C (0.67 – $0.77 T_m$, where T_m is the melting temperature in Kelvin) and subjected to strain rates from 5×10^{-6} to $2 \times 10^{-3} \text{ s}^{-1}$. Specimens were weighed and their dimensions measured immediately upon removal from the apparatus in order to determine whether the density changed during testing. Each specimen was then intentionally fractured and scanning electron microscopy was used for grain-size determination.

3. RESULTS AND DISCUSSIONS

The sintering schedule, sintered density, average grain size, and elastic modulus for five batches of Li_4SiO_4 are listed in Table 1.

Figure 1 shows the flexural strength, and Fig. 2 the fracture toughness, of Li_4SiO_4 as a function of density and grain size. In the low-density (up to 90% TD) regime, the strength and toughness of Li_4SiO_4 are primarily controlled by the density of the specimen. The flexural strength is observed to increase from 33 to 58 MPa as the density increases from 68 to 90% TD. The corresponding increase in fracture toughness was observed to be from 0.6 to

1.1 Mpa/ $\sqrt{m}$. For high-density specimens (> 90% TD), the strength decreases with increasing grain size. This strength behavior is consistent with the observations made for many ceramic materials [6,7,8]. The critical stress intensity factor K_{IC} for an SENB specimen can be related to the critical applied stress σ_c and the critical crack length a_c by $K_{IC} = \sigma_c (a_c)^{1/2} Y(a/w)$, where Y is a dimensionless constant which depends on the geometry of the loading and the crack configuration, a denotes notch depth, and w denotes sample width for a four-point bend specimen [9]. It was observed that the strength could be correlated to fracture toughness by using the above equation for specimens with densities of up to 93% TD. This suggests that the critical flaw size does not change with density in the range from 68 to 93% TD (grain size from 4 to 10 μm). The Young's modulus of Li_4SiO_4 ranged from 38.2 to 103.2 GPa for densities between 68% and 93% TD (see Table 1) and, as expected, decreased with increasing porosity for specimens in the range from 68 to 93% TD.

The results of thermal-shock experiments, shown in Fig. 3, indicate that the strength of the material is not degraded for values of quenching temperature difference (ΔT) up to 150°C. At a critical value $\Delta T_c = 150-170^\circ C$, the strength decreases sharply from 85 MPa to a value of 10 MPa, after which it remains constant with further ΔT increases (up to 300°C). This strength behavior as a function of ΔT is consistent with the prediction of the fractural-mechanical theory for thermal shock developed by Hasselman [10]. The increase of the strength from 53 MPa at room temperature to 85 MPa at $\Delta T = 150^\circ C$ may be due to annealing of residual stress or microcrack formation during thermal quench. Further investigation is necessary to clearly understand the reason for the initial increase in strength of Li_4SiO_4 as ΔT increases from 0 to 150°C.

In high-temperature steady-state creep tests, 90% dense specimens fractured at 800°C and below before reaching steady state. For tests at 900°C in which fracturing did not occur, a clear steady-state stress σ_s (at which the work-hardening rate, $d\sigma/d\epsilon$, was zero) was established. It was found that no appreciable grain growth or densification occurred during deformation. For polycrystalline materials, the steady-state strain rate $\dot{\epsilon}$ for a given temperature is usually described by $\dot{\epsilon} \propto \sigma^n$, where σ_s is the steady-state stress and n is a characteristic exponent corresponding to the dominant creep mechanism. The stress exponent n can be obtained by plotting $\ln \dot{\epsilon}$ vs $\ln \sigma_s$ and was determined to be 4.8 at 900°C. This value is typical of many ceramics and of a deformation process whose rate is controlled by dislocation climb [11]. Additional tests are being performed at higher temperatures in order to determine the temperature dependence of the plastic deformation in Li_4SiO_4 [12]. The steady-state flow stress measured at 900°C for Li_4SiO_4 can be compared with those of other lithium-containing ceramics which are being considered for fusion blankets. As shown in Fig. 4, for a temperature of 900°C, 90% dense Li_4SiO_4 is more resistant to plastic flow than is Li_2O , about equal to Li_2ZrO_3 , and less than LiAlO_2 . (The value of flow stress for LiAlO_2 is interpolated from data at 800 and 1000°C [13].)

4. CONCLUSIONS

1. In the low-density regime, both the flexural strength and toughness of Li_4SiO_4 were controlled by the density of the specimen. The strength and fracture toughness increased from 33 to 58 MPa and 0.6 to 1.1 $\text{MPa}\sqrt{\text{m}}$, respectively, as the density increased from 68 to 93% TD.

2. The thermal-shock experiment indicated a critical quenching temperature difference in the range of 150 to 170°C.
3. At temperatures of 800°C and lower, Li_4SiO_4 fractured without exhibiting appreciable plasticity when deformed at slow strain rate.
4. Steady-state creep tests suggested that the deformation of 90% dense Li_4SiO_4 is controlled by dislocation climb at 900°C. At this temperature, Li_4SiO_4 is more resistant to plastic flow than Li_2O , about equal to Li_2ZrO_3 , and less than LiAlO_2 .

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FIGURE CAPTIONS

- Figure 1 Flexural strength as a function of (a) sintered density and (b) grain size. B1-B5 denote batches 1-5.
- Figure 2 Fracture toughness K_{IC} as a function of (a) sintered density and (b) grain size. B1-B5 denote batches 1-5.
- Figure 3 Flexural strength as a function of quenching temperature difference ΔT in thermal-shock tests.
- Figure 4 Comparison of steady-state deformation of 90% dense Li-bearing ceramics at 900°C: Li_4SiO_4 (■), Li_2O (Δ), Li_2ZrO_3 (x), and $LiAlO_2$ (□).

Figure 3

(K. Bar et al.)

Table 1 -- Sintering Schedule, Sintered Densities, Average Grain Size and Elastic Modulus for Five Batches of Li_4SiO_4

Batch	Sintering Temp. ($^{\circ}\text{C}$)*	Hold Time (h)	Density (% TD)**	Avg. Grain Size (μm)	Elastic Modulus (GPa)
I	900	0	68	4	38.2
II	950	0	77	5	57.1
III	1000	0.75	93	10	103.2
IV	1000	4	98	25	82.8
V	1000	8	96	50	94.9

* Heating rate was 100°C c/h.

** Theoretical density of Li_4SiO_4 was taken to be 2.39 g/cm^3 [5].