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Slow-crack-growth and indentation damage in calcium magnesium aluminosilicate (CMAS) glass from desert sand

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ABSTRACT

Desert sand from a Middle East country was melted into calcium magnesium aluminosilicate (CMAS) glass. Its chemical composition was analyzed to be 25.2CaO-2.6MgO-8.2Al_2O_3-59.8SiO_2-1.6Fe_2O_3-1.5K_2O weight % using inductively coupled plasma-atomic emission spectrometry. The CMAS glass powder was hot pressed into billets. Slow-crack-growth (SCG) and indentation deformation/fracture of the CMAS glass was investigated. The SCG susceptibility parameter (n) was found to be 25 ± 3 which is within a range of n = 15–35 that has been observed in many silicate glasses and glass ceramics. A similarity in indentation hardness and toughness was found between the CMAS glass and the low-silica content (50–70%) glasses. However, an exception was that significant lateral cracking was typified in the CMAS glass, as quantified via stress analysis in the vicinity of an indent.

1. Introduction

Sand dust, prevalent on the earth's crust, converts into calcium magnesium aluminosilicate (CMAS) on melting. The CMAS has shown some deteriorating effects when ingested into hot section components of advanced aero-engines. Due to its low melting temperature (≥ 1150 °C), CMAS melts and solidifies under varying engine operating conditions. This results in clogging of the cooling passages in critical turbine components, degradation of the protective coatings on hot section components, and/or premature failure of related substrate hardware.

In light of the significance of CMAS issues in aero-engines, a large amount of work has been done during the past decade to characterize CMAS and its effects as well as to develop appropriate mitigation strategies/methodologies. The effects of CMAS have been explored in thermal barrier coatings (TBCs) with regards to thermo-chemical interactions, thermo-mechanical aspects, radiative transmission phenomenon, and mitigation methods e.g., [1–7]. Similarly, attempts have been made to explore CMAS interactions with environmental barrier coatings (EBCs) [8–11] and with ceramic matrix composites (CMCs) [12]. In a recent study, Bansal and Choi investigated phase composition and thermal stability of desert sand [13]. This study also included fabrication of CMAS glass from desert sand and assessment of various properties of this glass, including chemical composition, dilatometry, elastic modulus, hardness, and fracture toughness [13]. The crystallization behavior of this CMAS glass was also studied at various temperatures [13].

At elevated temperatures, molten CMAS deposits on the surface of the hot section components of turbine engines which converts into glass on cool down to room temperature. Fracture behavior of CMAS glass would influence the integrity of the underlying TBC/EBC layers. The primary purpose of the present work, as an extension of the previous study [13], was to assess the slow-crack-growth (SCG) behavior of the CMAS glass that was melted from desert sand. Slow crack growth of many silicate glasses and glass ceramics has been extensively studied for the past several decades, establishing a significant amount of databases. However, a question arises as to what extent this new class of CMAS glass would exhibit a susceptibility to SCG, which is the motive of the present work. To the authors' best knowledge, no earlier work seems to exist regarding the assessments of SCG behavior of CMAS glass directly processed from desert sand. Some additional work was also performed to characterize the behavioral aspects of Vickers-indentation deformation and fracture and to quantify cracking in terms of stresses induced during the loading and unloading sequences of indentation.

2. Experimental procedures

2.1. Glass melting and test specimens

Glass melting process was essentially the same as described in a previous study [13]. Briefly speaking, desert sand contained in a platinum crucible was heated in a box furnace at a rate of 10 °C/min with

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Table 1
Basic properties of CMAS glass [13].

Bulk density (g/cm^3)	Glass transition point (°C)	Softening point (°C)	Coefficient of thermal expansion (/°C)	Elastic modulus, E (GPa)[a]	Shear modulus, G (GPa)[a]	Poisson's ratio, ν[a]	Microhardness H (GPa)[b]	Fracture Toughness, K_c (MPa $m^{1/2}$)[c]
2.69	706	764	9.8×10^{-6}	92	36	0.28	6.3 ± 0.4	0.64 ± 0.05

[a] By the impulse excitation of vibration method, ASTM C1259.
[b] By the Vickers hardness method, ASTM C1327.
[c] By the indent-crack size measurement technique [18].

step-wise holds of 30 min each at 150 °C, 790 °C, and 1275 °C. After holding at 1550 °C for about an hour for homogenization, the glass melt was quenched in water. The resulting glass frit was ground to powder in a planetary mill using a corundum grinding bowl and zirconia milling media. The glass powder was loaded into a graphite die and hot pressed in vacuum at about 800 °C under 17 MPa for 10–15 min using a Centorr hot press. The applied pressure was released before onset of cooling. Grafoil sheets were used as spacers between the samples and the punches. Two billets having final dimensions of approximately 25 mm × 25 mm × 5 mm were fabricated.

The billets surfaces were ground using a #600 diamond grinding wheel and machined into flexure test specimens measuring 25 mm × 4 mm × 2.5 mm in length, width and thickness, respectively. A prospective 4-mm wide tensile face of each flexure test specimen, which was parallel to the hot press direction, was polished using 0.1 μm alumina compound. The long edges of the flexure specimens were also ground using #600 grit SiC paper to remove any spurious damage. A total of 12 flexure test specimens were prepared from the two billets. The basic properties of the CMAS glass as reported in a previous study [13] are presented in Table 1. From inductively coupled plasma-atomic emission spectrometry, the chemical composition of the CMAS glass was found to be 25.2CaO-2.6MgO-8.2Al_2O_3-59.8SiO_2-1.6Fe_2O_3-1.5K_2O weight % [13]. Both the glass powder and the as-processed CMAS glass billets were found to be amorphous by x-ray diffraction [13].

2.2. Slow crack growth (SCG) testing

Dynamic fatigue, also called constant stress-rate testing, was conducted to determine the SCG behavior of the CMAS glass. This test method has been used for several decades in glass and ceramics at both ambient and elevated temperatures and has also been established as ASTM test standards [14,15]. The underlying principle of the test method in the case of no residual stresses present in samples has been developed based on the fracture mechanics approach and can be formulated as follows [14–16]:

$$\log \sigma_f = \frac{1}{n+1} \log \dot{\sigma} + \log D_d \quad (1)$$

where σ_f is failure stress (or failure strength), n is SCG parameter, $\dot{\sigma}$ is applied stress rate, and D_d is a parameter associated with flaw geometry and material properties. Eq. (1) was derived based on the following empirical, power-law, stress-corrosion controlled crack velocity formulation [16,17]

$$v = \frac{da}{dt} = A\ [K_I/K_{IC}]^n \quad (2)$$

where v, a and t are crack velocity, crack size and time, respectively. K_I and K_{IC} are mode I stress intensity factor (SIF) and mode I critical SIF (or fracture toughness), respectively. A is a material-environment dependent parameter. Therefore, the SCG parameter n and the value of D_d can be determined from Eq. (1) via a linear regression analysis of log σ_f–log $\dot{\sigma}$ once failure strength is obtained as a function of applied stress rate from experiments. It can be readily implied from Eqs. (1) or (2) that the magnitude of n represents the degree of SCG susceptibility for a given material/environment system: lower the value of n, the greater susceptibility, and vice versa.

The SCG experiments using the CMAS test specimens were conducted in ambient-temperature distilled water in flexure using a 10/20 mm-span test fixture in accordance with ASTM C1368 [14]. Due to limited availability of test material, test specimens were indented to reduce their variability or scatter of failure strength, as customarily done for brittle solids in similar situations. Vickers indentation (via Model Wilson-Wolpert Tukon 2100B, Instron, Norwood, MA) was made with an indentation force of 10 N at the center of the polished surface of each test specimen with the indent aligned normal (or parallel) to the direction of prospective applied tensile stress. The indentation generated reasonably well-developed indent crack configurations, similarly observed in soda-lime glass. A total of four different stress rates ranging from 0.003 to 10 MPa/s were used through an Instron test frame (Model 5982, Norwood, MA). Inert strength of indented test specimens was also determined in silicon oil for a baseline reference. Normally, three test specimens were used at each test rate as well as in inert strength testing. For comparison, SCG experiments were also conducted in soda-lime glass microslides measuring 75 mm × 25 mm × 1.7 mm (thickness) in flexure using a 20/40 mm span test fixture. The indentation force, test environment, the number of test specimens, and the test frame used in the soda-lime glass were the same as those employed in the CMAS glass. Three stress rates from 0.01 MPa/s to 10 MPa/s were utilized in the soda-lime glass.

2.3. Vickers indentation

Vickers indentation testing was carried out in the CMAS glass flexure test specimens to assess and characterize its deformation (hardness), fracture (fracture toughness), and cracking behavior. A total of nine different indentation forces of P = 0.1–10 N were used with five indents employed at each indentation force. As done in the SCG testing, the soda-lime glass microslides were also used in this testing for comparison. The hardness and fracture toughness were assessed in accordance with ASTM C1327 and the indent crack size-measurement technique [18], respectively. All indentations were made in ambient air at room temperature with a relative humidity of about 55%, by keeping a duration of indentation for 12 s. Measurements and observation of all indent cracks were made right after indentation.

3. Results and discussion

3.1. Slow crack growth

The results of the SCG testing for the CMAS glass are presented in Fig. 1, where failure stress was plotted as a function of applied stress rate. The figure also includes the results of the soda-lime glass experiments. The solid lines represent the best-fit based on Eq. (1), with the correlation coefficients of r_{coef} = 0.880 and 0.989, respectively, for the CMAS glass and the soda-lime glass. It is important to note that failure stress decreased with decreasing stress rate, which represents a susceptibility to SCG of the material. Hence, judging from the slopes of the curves, the susceptibility to SCG is lower in the CMAS glass than in the soda-lime glass counterpart. Overall failure stress in either water or