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High-Temperature Mechanical Testing and Properties of a Powder-based Low-Density Metal-Ceramic Material

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Abstract

Sustained hypersonic flight relies on critical technology innovations in extreme environment materials and associated test methods. This is because hypersonic flight vehicles experience high heat fluxes and sustained aerothermal loading for a significant period of time. Most materials cannot sustain such extreme operational environments. Ideally, candidate materials for hypersonic vehicle nose cap and leading edges should have the combined properties of both metals and ceramics including high strength, low density and ultra-high temperature erosion resistance. Today, materials that excel in each of these measures are not available commercially or simply do not exist. The associated testing technologies and methods also need to be developed and standardized for assessing the maturity of new materials and innovative hot structures technology. This paper presents high-temperature mechanical testing methods and results developed during research and development of a low-density extreme environment material for potential Defence hypersonic applications. A low-density metal-ceramic material was manufactured through a powder based processing technology. To assess the material's high-temperature performance, flexural strength testing was conducted at a range of temperatures up to 1200°C. Fracture toughness testing was completed also. Experimental results demonstrate this material has favorable high temperature mechanical properties up to 1200°C and maybe a suitable candidate material, with further refinement, to meet the demands of Defence hypersonic applications. The findings of this research will be used to facilitate the development of the next-generation metal-ceramic materials for the demands of sustained hypersonic flight.

Keywords: *High temperature mechanical testing; Metal-ceramic material; Flexural strength at high temperatures; Fracture toughness.*

Nomenclature

a – Notch depth
B – Specimen width
d – Specimen thickness
K_Q – Fracture toughness
L – Specimen length
P – Load at failure
S – Loading span
S_{th} – Flexural strength
T – Temperature

1. Introduction

Sustained hypersonic flight vehicles exposed to intense heat fluxes in extreme aerothermal heating operational environment for flight times measured in tens of minutes, depending on range and speed. The temperatures encountered in hypersonic vehicle structure and components such as the nose cap and leading edges are extremely high, posing significant material and structural challenges in design and manufacture of hypersonic vehicles [1,2]. Up to date, most materials and structures cannot sustain such extreme operational environments. Ideally, candidate materials for hypersonic vehicle nose cap, leading edges and propulsion components should have the combined properties of both metals and ceramics including high strength in extreme environment, low density and ultra-high temperature erosion resistance. Today, materials that excel in each of these measures are not available commercially or simply do not exist [3]. The associated extreme environment testing technologies and methods also need to be developed and standardized for assessing the maturity of new materials and innovative hot structures technology through the determination of mechanical properties of extreme environment materials under representative operational conditions in laboratory.

In this study, a low-density metal-ceramic was manufactured through a powder based processing technology [4,5]. To assess the material's high-temperature performance, flexural strength testing was conducted at a range of temperatures up to 1200°C. Fracture toughness testing was also completed at ambient temperature. Experimental results were then discussed and compared with published data.

2. Metal-Ceramic Specimens

Ten metal-ceramic specimens in total were fabricated and machined to approximately 4.0 mm × 4.0 mm × 20.0 mm (B ×d × L), as shown in Fig. 1. B is for the specimen width, d for the thickness, and L is the length of the specimen, while S is loading span. The specimens were polished to a surface finish with the roughness average of 1.0 µm using Silicon Carbide (SiC) papers. Eight specimens were used for high-temperature flexural strength tests.

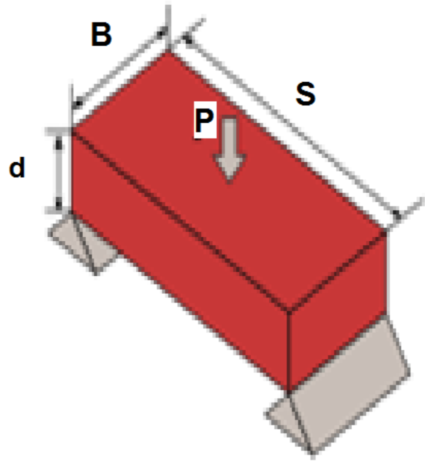


Fig 1. Schematics of flexural strength test specimen with loading span S=16.0 mm.

Two specimens were notched for fracture toughness testing. The pre-crack of the specimens were micro-notched using a 1.0 µm water-based diamond suspension with a razor blade to cut a notch root radii of 5.0 µm [6]. The ratio of notch depth (a) to specimen thickness (d) was 0.55 as required by ASTM standard E399-09 E2 [7] for valid fracture toughness testing. Fig. 2(a) shows fracture toughness specimen, while Fig. 2(b) is an optical image of a micro-notch made using a razor blade.

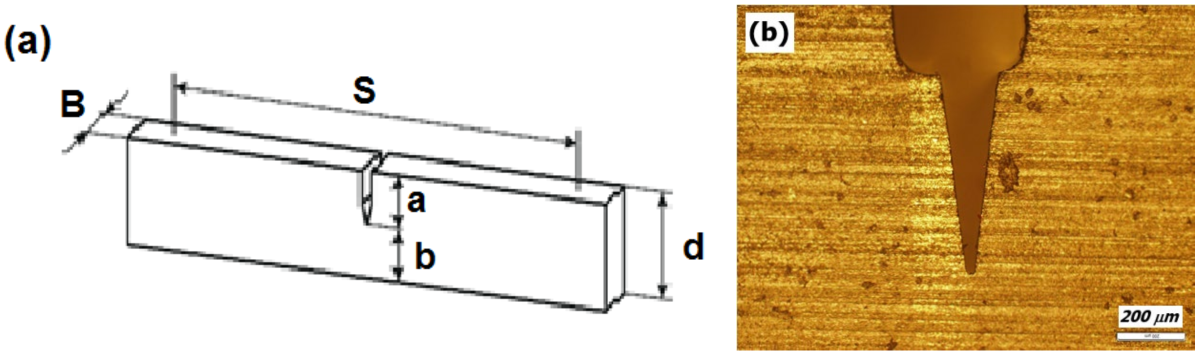


Fig 2. (a) Fracture toughness specimen, and (b) an optical microscopic image of the razor notch of a fracture toughness specimen.

3. Methods: High-temperature strength and fracture toughness testing

Flexural strength testing at high temperature was conducted at the micro-tomography beamline 8.3.2 of the Advanced Light Source at Lawrence Berkeley Laboratory, Berkeley, USA, as shown in Fig. 3(a) with a three-point bending fixture in Fig. 3(b). The beamline can collect 3D images of specimens subjected to different loads under different temperatures. The limited transmissivity of this metal-ceramic material to X-rays at this specific beamline prevented collecting micro-tomography data during testing. In this study, all specimens were tested using a loading span (S) of 16.0 mm under three-point bending. Detailed descriptions of the hot cell and set-up are described in References [8, 9]. All specimens were tested at different temperatures (two each at room temperature, 800°C, 1000°C, and 1200°C) under displacement control at a constant displacement rate of 1.0 μm.s-1 to failure. Both load and crosshead displacements were recorded simultaneously in accordance with ASTM C1161 [10].

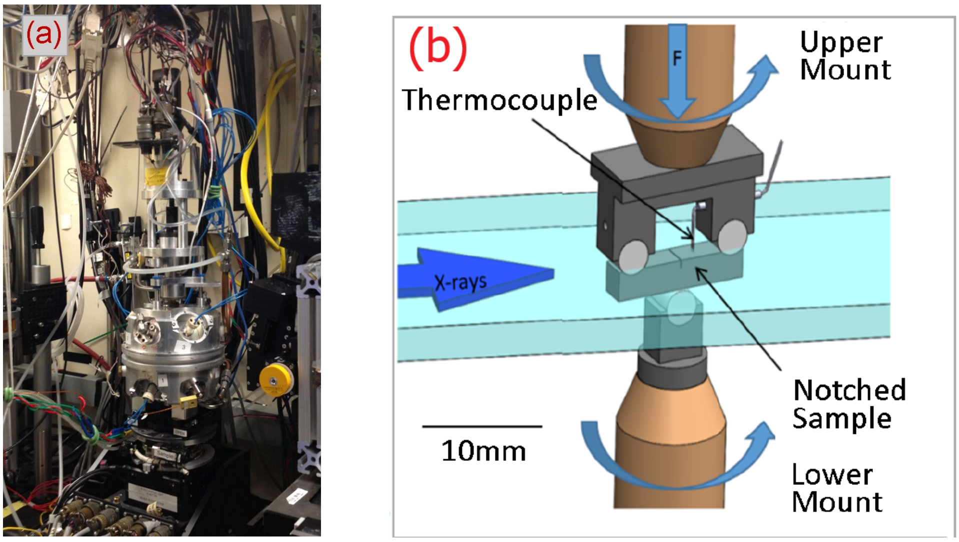


Fig 3. Images showing (a) a hot cell used in the ALS beamline 8.3.2 at the Lawrence Berkeley National Laboratory in Berkeley, CA, USA, (b) a three-point bend loading system [11].

The fracture toughness testing was performed in accordance with ASTM standard E399-09 E2 [7]. The tests were done under a controlled constant displacement rate of 0.83 μm.s-1 using a Gatan MicroTest 2kN bending stage (Gatan, Abingdon, UK), as shown in Fig. 4. The system was equipped with a 660 N load cell and a 3-point bending setup with loading span (S) of 16.0 mm and samples were loaded at room temperature to complete failure. The loads and displacements were recorded simultaneously.

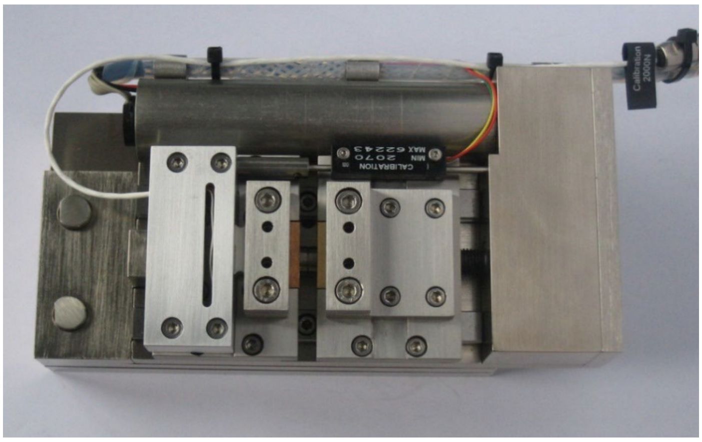


Fig 4. Gatan MicroTest 2kN bending stage (Gatan, Abingdon, UK) [7].

4. Flexural Strength Results at Different Temperatures

Table 1 presents the flexural strength data of the metal-ceramic material measured at room temperature (RT), 800°C, 1000°C and 1200°C respectively. Table 1 presents the specimen dimensions, testing temperatures, maximum loads applied during testing and the calculated flexural strength results based on the following Equation (1).

$$S_{th} = \frac{3PS}{2Bd^2} \tag{1}$$

where P is the load at failure. Compared with the flexural strength of metal-ceramic composites manufactured by stereolithography (≈100MPa at RT [12]) and electrodeposition (<300MPa at RT [13]), the strength of this powder-based metal-ceramic material has superior flexural strength with an average value of 396 MPa at RT. Table 1 presents the flexural strength results from RT to 1200°C, and indicated that the flexural strength at 1200°C is even higher than those reported counterparts at room temperature.

Table 1. Flexural strength results of the metal-ceramic material at different temperatures

Specimen ID	**Temperature** ***T* (°C)**	**Width** ***B* (mm)**	**Thickness** ***d* (mm)**	**Load** ***P* (N)**	**Strength** ***S_{th}* (MPa)**	**Average** ***S_{th}* (MPa)**
SES-11	20	4.0	4.0	827	310.6	396.2
SES-12	20	4.0	4.0	1322	481.9	
SES-13	800	4.0	4.0	1041	383.2	361.8
SES-14	800	4.0	4.0	905	340.4	
SES-15	1000	4.0	4.0	898	329.7	324.0
SES-16	1000	4.0	4.0	873	318.3	
SES-17	1200	4.0	4.0	713	263.4	284.8
SES-18	1200	4.0	4.0	835	306.1	

5. Fracture Toughness Results

The fracture toughness testing of two metal-ceramic specimens (ID: SES-10 and SES-19) were conducted at room temperature. Table 2 shows the fracture toughness results of this newly developed metal-ceramic material. The fracture toughness values were determined using Equation (2) with the test data. As can be seen, the average fracture toughness was 4.4 MPa√m and the range of measured fracture toughness was 3.9-4.8 MPa√m. The results are equivalent to that of a published metal–ceramic composites (4.0-6.0 MPa√m) at room temperature [14,15].

$$K_Q = \frac{PS}{Bd^{3/2}} f\left(\frac{a}{d}\right) \tag{2}$$

where

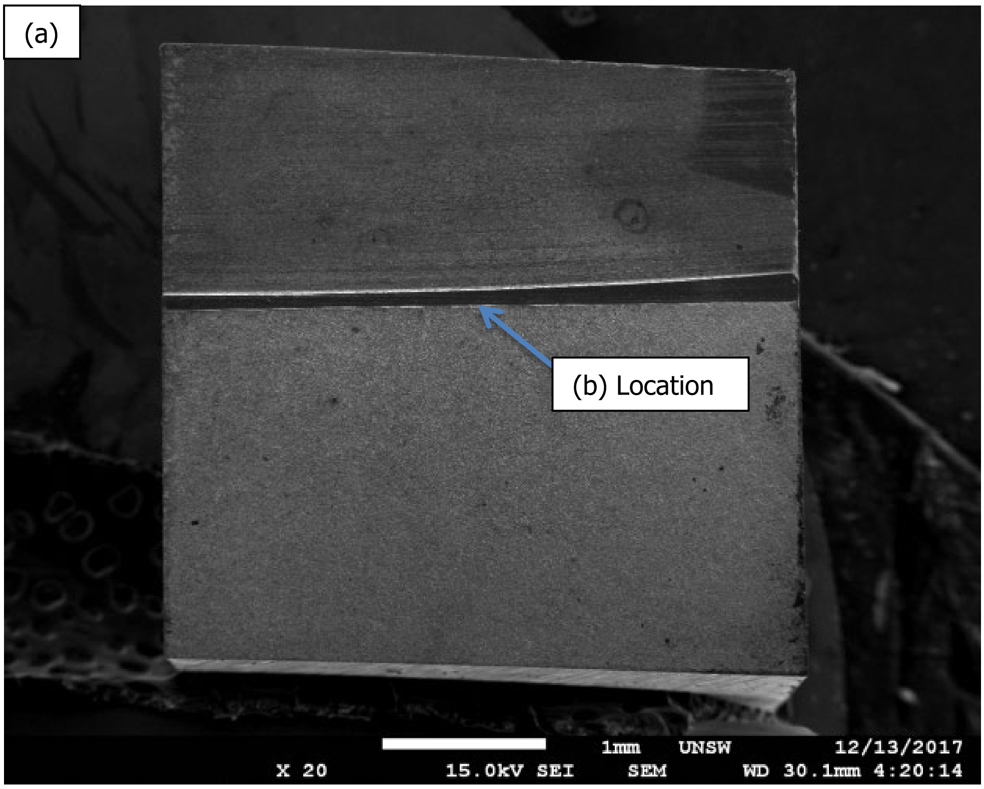
$$f\left(\frac{a}{d}\right) = 3\sqrt{\frac{a}{d}}\,\frac{1.99 - \left(\frac{a}{d}\right)\left(1 - \frac{a}{d}\right)\left[2.15 - 3.93\frac{a}{d} + 2.7\left(\frac{a}{d}\right)^2\right]}{2\left(1 + 2\frac{a}{d}\right)\left(1 - \frac{a}{d}\right)^{3/2}}$$

in which *a* is notch depth as shown in Fig. 2(a). Thus, fracture toughness can be determined from the loads at fracture failure of the notched specimens, the dimensions and the notch depth.

Table 2. Fracture toughness results of the metal-ceramic material

Specimen ID	**Width** *B* **(mm)**	**Thickness** *d* **(mm)**	**Notch depth** *a* **(mm)**	**Load** *P* **(N)**	**Fracture toughness** K_Q **(MPa.m$^{1/2}$)**
SES-10	3.8	3.9	1.5	135	4.8
SES-19	3.9	3.9	2.0	81	3.9

The notch depth was identified and measured, as shown in Fig. 5(a). Fig. 5(b) reveals the smooth and homogeneous fracture surface with a fine, micron-sized grain structure and only few large grains throughout the fracture surface. As can be seen in Fig. 6, a large sized grain can cause stress concentrations and lead to crack propagation from the notch.



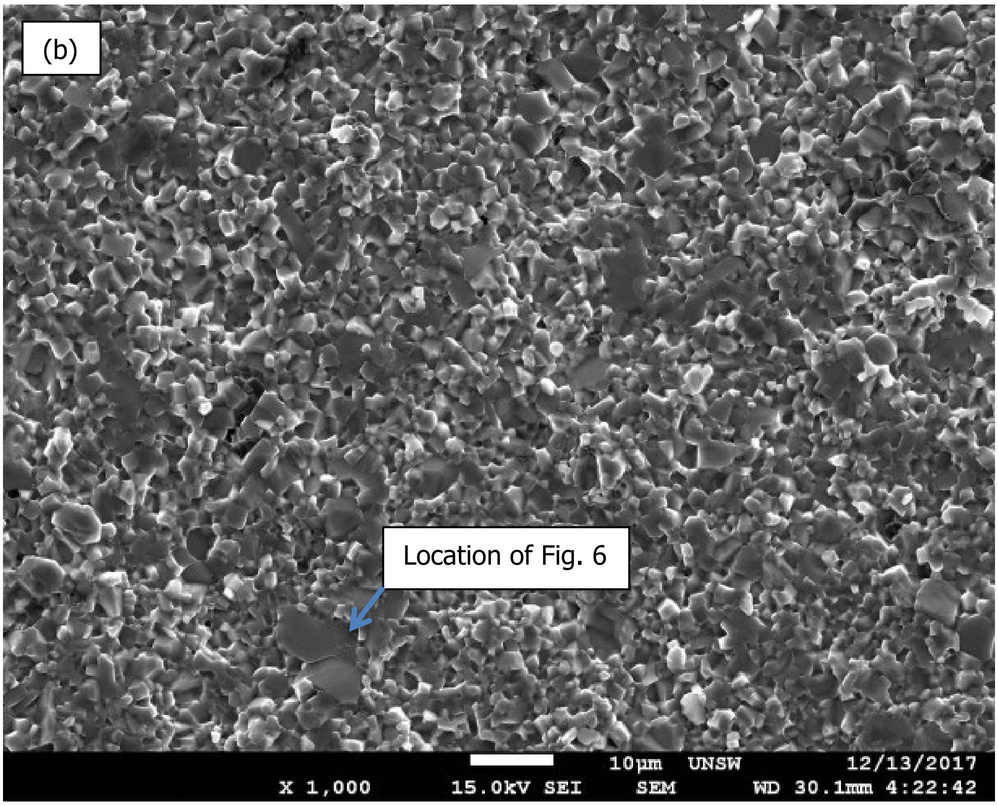


Fig 5. SEM images of the specimen (SES-10) fracture surfaces tested at room temperature showing (a) a smooth and homogenous fracture surface and (b) micron-sized grains from a magnified fracture surface image.

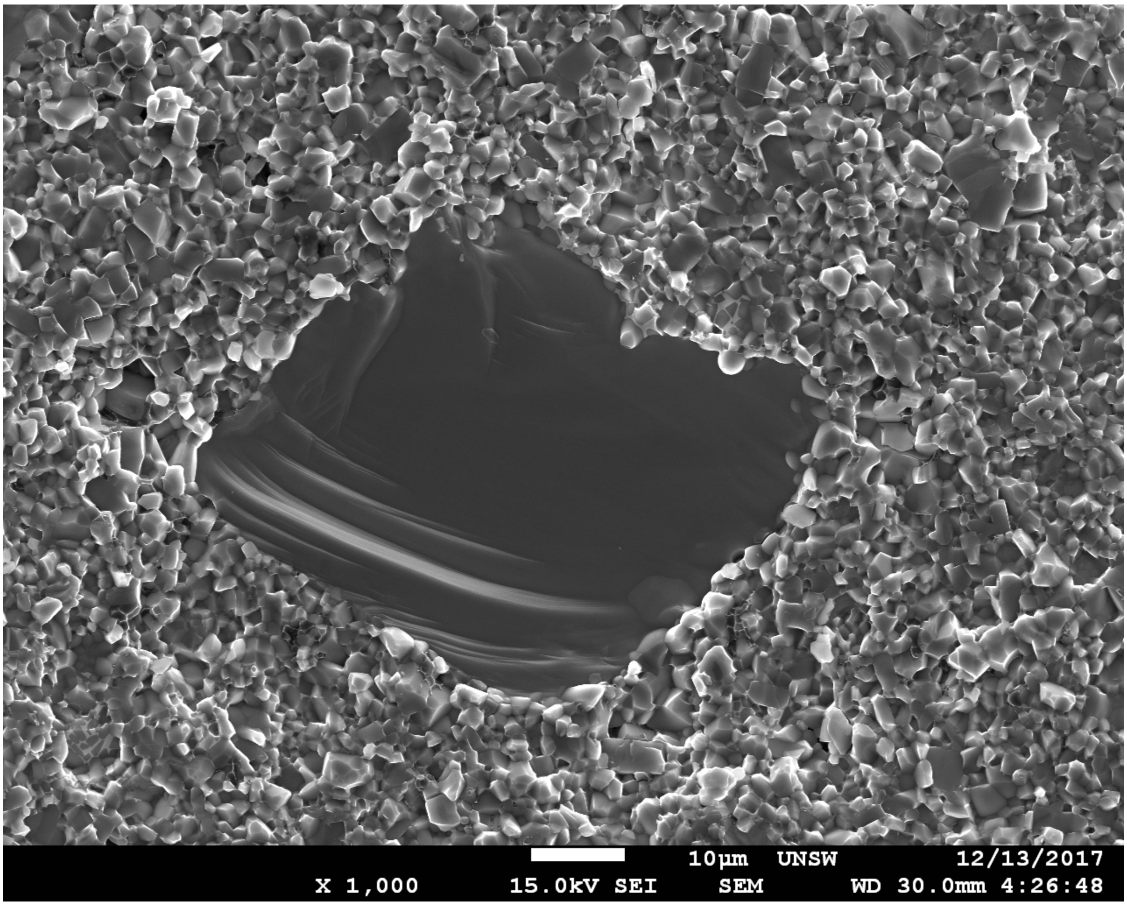


Fig 6. A large sized SiC grain on the fracture surface of specimen SES-10.

6. Conclusions

In this study, a novel low-density extreme environment metal-ceramic material for high-temperature applications was investigated experimentally. Based on the aforementioned experimental results and discussions in the previous sections, some conclusions can be drawn as follows.

1) The flexural strength test results from room temperature up to 1200°C in air indicate that the newly-developed metal-ceramic material possesses approximately high flexural strength close to 400 MPa at room temperature and degrades gradually with increasing temperature down to 300 MPa at 1200°C. The experimental result clearly showed that the material possesses superior mechanical properties at high temperature up to 1200°C, compared with the published data on metal-ceramic composites in Refs [12] and [13].

1) The fracture toughness of this metal-ceramic material, tested on micro-notched specimens at room temperature, was 3.9-4.8 MPa√m. The results so far demonstrated that the newly-developed material offers competitive performance in resistance to fracture when enduring a crack or defects, compared with published metal-ceramic materials in Refs [14] and [15].

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