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High temperature load–deflection behaviour of reaction bonded SiC (RBSC)

O.P. Chakrabarti *, P.K. Das

Central Glass and Ceramic Research Institute, Calcutta-700032, India

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Abstract

Preforms of commercial SiC (0.2 and 23.65 μm particle diameter) and petroleum coke powders were infiltrated with Si to prepare reaction bonded SiC (RBSC) with varying amount of residual Si phase (16.5–42.0% v/v). Load–deflection behaviour of the infiltrated materials was studied with the help of a 4-pt. bending strength tester at room temperature, 1300 and 1370°C. The high temperature deformation of the material showed marked difference from the room temperature behaviour. At 1300°C at a constant level of load (150 N), deflection increased from 0.16 mm to >0.58 mm with increase in residual Si from 16.5 to 42.0% v/v in the case of fine grained RBSC; for coarse grained material, the corresponding increase was observed to be from 0.11 to 0.24 mm with an increase in Si from 16.5 to 24.1% v/v. The room temperature load–deflection behaviour was found to be mostly elastic in nature. Reflected light optical microscopy (RLM) of deformed specimens and scanning electron microscopy (SEM) of fractured surfaces revealed the evidence of plastic flow that appears to be set in by high ductility of the residual Si causing deformation at high temperatures.

Keywords: A. Powders; B. Microstructure; D. SiC; Deflection; Load; Reaction bonding

1. Introduction

Reaction bonded SiC (RBSC) material is considered to be potentially important for various structural ceramic applications particularly at high temperatures. The material genetically contains a substantial amount of Si (m.p. 1410°C) which puts severe restriction on the practical-use-temperature up to around 1300°C [1,2] because of softening. Si differs considerably from SiC in melting temperature deformation and physical and chemical properties. The relative distribution of Si over SiC can also have a different character. Therefore, the deformation behaviour of such materials at around 1300°C is a matter of concern particularly for designers. Though high temperature creep data for RBSC are available [3–8], the studies on deformation behaviour of such materials have been the subject matter of very few investigations [9].

Bend test data can not suitably describe the stress–strain behaviour of material beyond its elastic limit. Hollenberg and co-workers [10] studied the stress–strain behaviour of ceramic samples under flexure on the basis of simple geometry. Those authors assumed the rectangular bar under flexure to be a part of a circle with the neutral plane normal to the direction of load and passing through the centroid of the sample. But during bend test, the stress distribution on the specimen cross-section is non-linear and depends on the stress–strain law of the material that must be known in advance. Also, in the case of ceramics, the behaviour in tension is different from that in compression and a shift of the neutral axis towards the compressed region continuously occurs during plastic straining. So, simple elastic stress–strain equations do not hold good. This aspect has been considered while developing modified equations describing stress–strain behaviour of brittle ceramic materials [6,11]. Caroll and Wiederhorn [12] also effectively dealt with a similar problem during creep testing of RBSC under tension.

The residial Si phase in RBSC plays a great role on the onset of ductile behaviour of the composited material, particularly at high temperatures, because of the fact that Si is more plastic than SiC. The aim of the present work is to study the role of residual Si phase upon high temperature deformation behaviour of RBSC material. Also the influence of starting SiC particle size on high

* Corresponding author.

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temperature deformation will be examined. Load-to-deflection measurements have been used as an effective tool for studying the deformation behaviour.

2. Experimental

2.1. Material

Carbonaceous SiC compacts used in this study for preparing RBSC material were made by pressing an intimate mixture of commercial raw SiC powder (98.20 wt.% SiC, 0.25 wt.% free C, 0.50 wt.% SiO_2, 0.04 wt.% Fe_2O_3, Grindwell Norton Ltd., India) of two different sizes, viz. 0.20 and 23.65 μm and petroleum coke (surface area of 4.03 m^2/gm; 0.68 wt.% ash content, Assam Carbon Products Ltd., India) powder. The green compacts were infiltrated with molten Si (-200 B.S.S.; 99.41 wt.% Si, 0.22 wt.% Fe, 0.08 wt.% Al, 0.004 wt.% Ca, 0.2 wt.% Mg and 0.09 wt.% Na + K, Indian Metals and Ferro Alloys Ltd., India). The fabrication details were described elsewhere [13]. The content of the residual Si in the infiltrated samples was measured by optical point count method and found to be varying within 16.5 and 42.0 vol.%. Microstructurally the material contained SiC, residual Si and a minor amount of porosity.

2.2. Sample preparation

Specimens (45 × 4.5 × 3.5 mm^3) were cut from the infiltrated samples; surfaces were finished by grinding and polishing up to 1 μm. The width and thickness of the samples were kept almost constant, i.e. 3.17±0.003 mm and 2.76±0.059 mm respectively. The edges of the bar were chamfered.

2.3. Deformation behaviour studies

A high temperature 4-pt. bending strength tester (422S, Netzsch, Germany) was used for this purpose. The instrument consists of a furnace capable of working up to 1450°C, a loading device and a four point bending fixer made of Si_3N_4 having a bending arm (d) of 10 mm. The sample was loaded by means of a double-lever loading device in which load is generated by the movement of a spindle-driven weight over the lever arm. The deflection of the centre portion of the specimen was measured by the movement of an alumina thrust rod monitor through a LVD transducer. Both the load and deflection were recorded in an X–Y recorder. Tests were conducted at room temperature, 1300 and 1370°C.

2.4. Microscopic examination

The tensile surface of the failed specimens was polished and viewed under optical microscope (Ortholux II pol-BK, Leitz, Germany). The fractured surfaces were examined in a scanning electron microscope (SE440, Leo-Cambridge, UK).

3. Results and discussions

3.1. Load–deflection behaviour

Fig. 1 shows the load–deflection curves of fine-grained RBSC at 1300°C with varying Si-content. The sample with the lowest amount of Si (16.5 vol.%) showed the lowest amount of deflection at any level of load. Initially at low load (up to ~ 80 N), the material with the lowest Si content (16.5 vol.%) behaved like a typical brittle ceramic material with a characteristic linear region of the load–deflection curve — elasticity of the material may play a dominant role in this region. Thereafter, non-linear deflection sets in due to high plasticity of the Si phase during further loading. Increase in the amount of Si (24.1 vol.%) is associated with the broadening of the non-linear region where a continuous increase of load was necessary in order to progress the deflection of the material leading to final failure. The effect was most pronounced when Si-content was very high (42.0 vol.%).

Similar trends were observed in the case of load-deflection behaviour at 1300°C for coarse-grained RBSC with varying Si-content (Fig. 2). The curves contained two distinct regions — one is the linear portion probably caused by the predominance of the elastic nature of the material. The other part is non-linear mainly caused by the influence of plastic Si. With increase in Si-content from 16.5 to 24.1 vol.%, the linear portion was shortened with gradual broadening of the non-linear part of

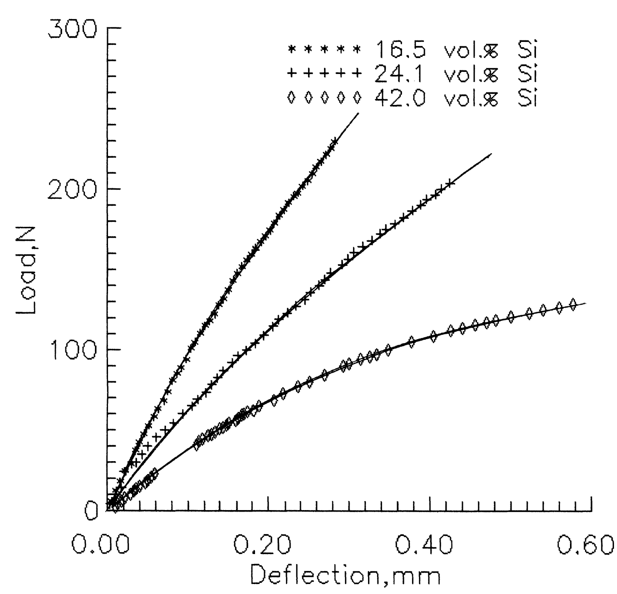

Fig. 1. Load–deflection behaviour of the fine-grained RBSC (0.20 μm) at 1300°C.

the load–deflection curves. Interestingly, the coarse-grained material showed a lesser amount of deflection at any level of load (say at 150 N) in comparison to its fine-grained counterpart, provided the Si-content was constant (Table 1).

Temperature has also a great influence on load–deflection behaviour. Fig. 3 shows the load–deflection curves of coarse-grained RBSC with 16.5 vol.% Si at RT, 1300 and 1370°C. With increasing test temperature, the plasticity also increased and the load involved during transition from linear elastic to non-linear plastic deformation decreased. This effect was observed to be more pronounced if Si was further brought down to a level of 24.1 vol.% (Fig. 4). Table 2 gives the comparative deflection at a particular load (150 N) for coarse-grained RBSC at different temperatures.

3.2. Microstructural observation

Figs. 5(a)–(c) shows the fracture surfaces of fine-grained RBSC failed at 1300°C, with varying amount of Si. At 16.5 vol.% Si [Fig. 5(a)] transgranular cleavage facets were mostly observed; fracture morphologies were smooth and planar with occasional wrapping of

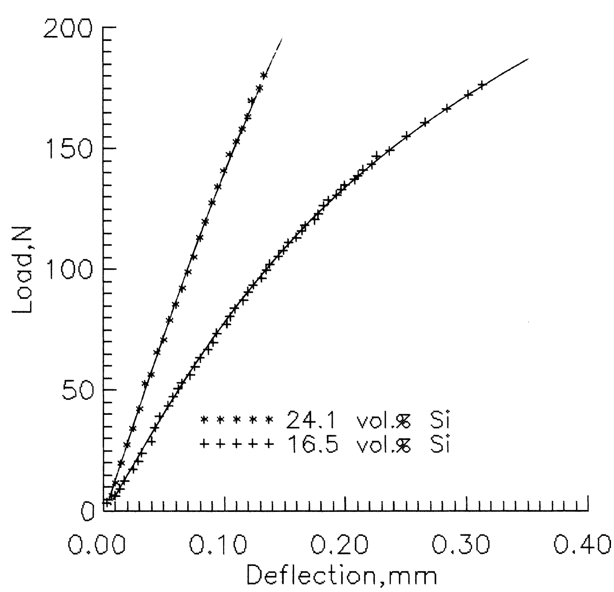

Fig. 2. Load–deflection behaviour of the coarse-grained RBSC (23.65 μm) at 1300°C.

Table 1
Deflection of coarse- and fine- grained RBSC at 150 N load at 1300°C

Volume % Si	Deflection (mm)	
	Fine-grained	Coarse-grained
16.5	0.16	0.11
24.1	0.28	0.24
42.0	> 0.58	–

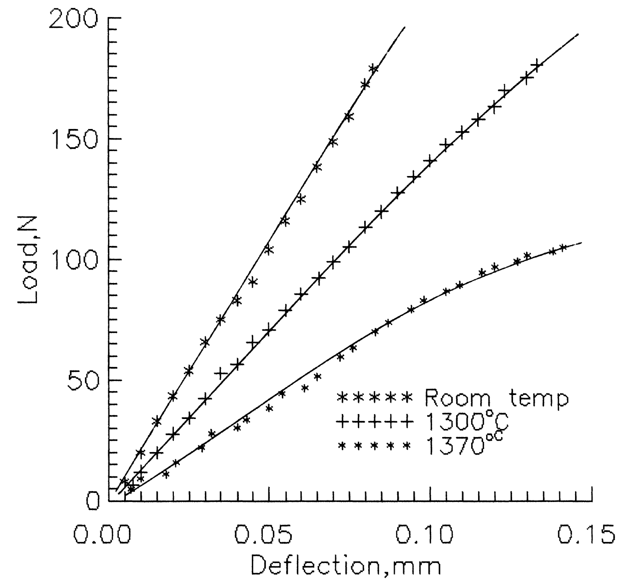

Fig 3. Load–deflection behaviour of the coarse-grained RBSC with 16.5 vol.% Si at different temperatures.

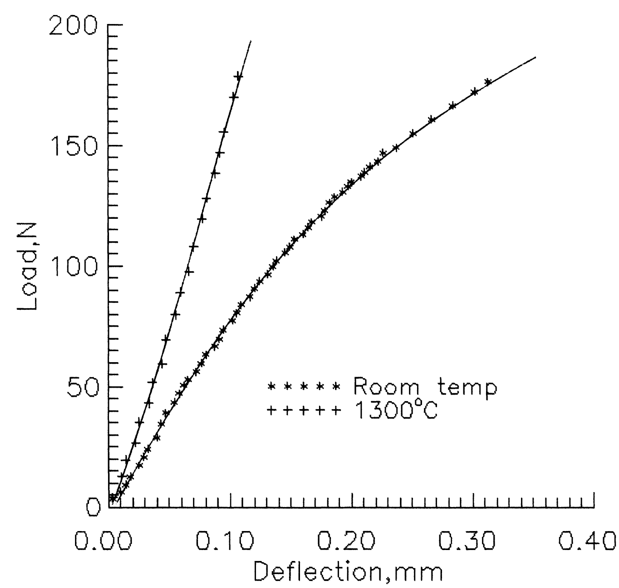

Fig 4. Load–deflection behaviour of the coarse-grained RBSC with 24.1 vol.% Si at different temperatures.

Table 2
Deflection of coarse-grained RBSC at 150 N load at different temperatures

Temperature	Deflection (mm)	
	16.5 vol.% Si	24.1 vol.% Si
Room temperature	0.07	0.09
1300°C	0.11	0.24
1370°C	> 0.14	–

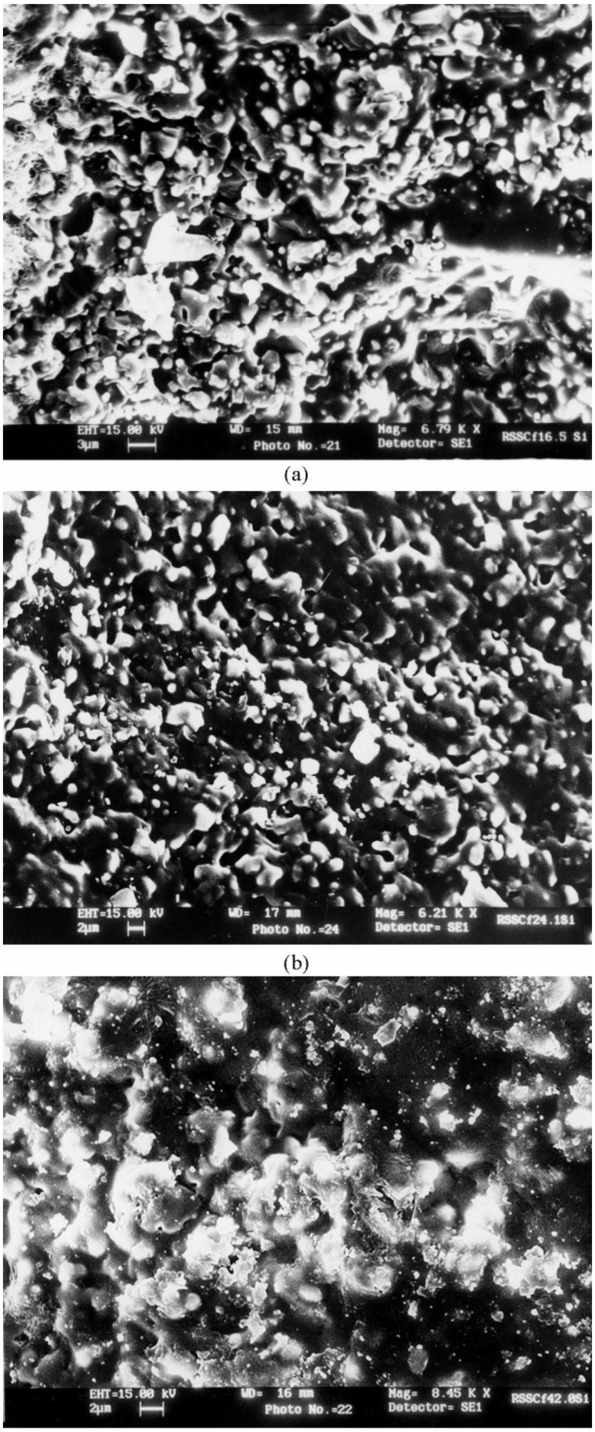

Fig 5. Fracture surfaces of fine-grained RBSC samples with varying amount of Si, failed at 1300°C; (a) with 16.5 vol.% Si, the fracture is mostly transgranular with less amount of wrapping of SiC grains, (b) with 24.1 vol.% Si, moderate wrapping of SiC grains with Si-melt, (c) with 42.0 vol.% Si, vigorous wrapping by Si-melt; bare SiC grains are not seen.

the melt over SiC grains. The wrapping of the SiC grains increased with increase in Si-content to 24.1 vol.% [Fig. 5(b)]. When the Si-content was very high (42.0 vol.%) bare SiC grains were never found and vigorous wrapping of the Si-melt was observed [Fig. 5(c)]. The small amount of impurity in Si used in the present study was enough to cause sufficient softening near 1300°C [14]. This effect was appreciably high particularly at high content of Si. This fact coupled with the high plasticity of Si at high temperature [15], might set in enough plastic flow in the material at 1300°C; consequently with increase in Si, gradual broadening of the non-linear segment of the load–deflection curve occurred (Fig. 1) with increased wrapping of SiC grains as observed in the fractured surfaces of the failed specimens [Fig. 5(a)–(c)]. Under similar conditions lower deflection was observed in coarse-grained material than fine-grained RBSC. This was probably because of the fact that during external loading, larger SiC particles gave greater obstruction towards deformation of the material. However, by the combined action of softening and plasticity that occurred in Si, particularly at high temperature (1300°C), larger SiC particles were observed to be aligned causing deformation (Fig. 6). Restriction towards deformation by the way of alignment of large SiC particles might be the cause for lesser deflection in the case of coarse-grained RBSC.

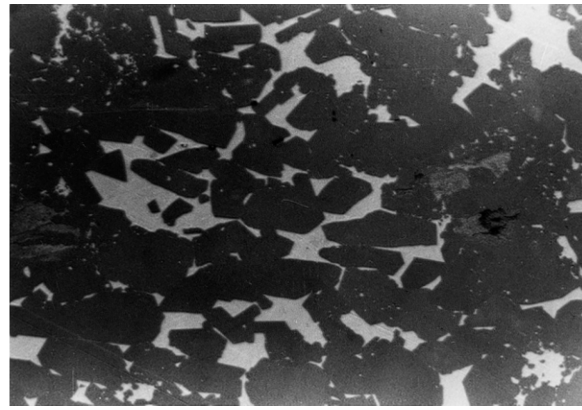

Fig 6. Optical photomicrograph of tensile surface of coarse-grained RBSC (16.5 vol.% Si) failed at 1300°C showing alignment of large SiC grains (× 500).

4. Conclusions

(i) The load–deflection curves of RBSC material at 1300°C contain two distinct portions — a linear elastic region followed by a non-linear deformation zone presumably caused by plastic Si.

(ii) For both coarse- and fine-grained RBSC, at a constant level of load deflection increases with increase in Si-content as well as temperature. At a load of 150 N deflection at 1300° C increases from 0.16 to > 0.58 mm for fine-grained material with increase in Si from 16.5 to 42.0 vol.%. For coarse-grained material the corresponding values are 0.11 mm to 0.24 mm with increase in Si from 16.5 to 24.1 vol.%. At 150 N load, the deflection increases from 0.08 mm at RT to > 0.15 mm at 1370°C for coarse-grained RBSC with 16.5 vol.% Si and from 0.09 mm at RT to 0.24 mm at 1300°C for same material with 24.1 vol.% Si.

Coarse-grained material showed less deflection than its fine-grained counterpart under a similar condition of loading.

(iii) The increased deflection of the RBSC material with an increase in Si-content and temperature may be explained in terms of enhanced plasticity and softening of the residual Si-phase. Microstructural observations revealed increased wrapping of SiC grains by plastic Si with increase in its content. Less deformation observed in coarse-grained material may be related to the difficulty of large SiC particles being aligned.

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