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Strengthening and proof testing of siliconised SiC

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Experimental tests were conducted to show the degree of strengthening that can be produced in siliconized SiC by a 10 h or less, 1200° C, prestress that also serves as a proof test. A 1200° C, 10 h prestress at 86% of the room-temperature strength results in more than a 25% increase in the room-temperature strength. A similar strengthening effect occurs for bonded siliconized SiC butt joints. The Weibull distribution can be used to treat the strength populations including those that are truncated by the prestress/proof test or additional proof testing.

1. Introduction

In the application of SiC ceramic materials in heat exchangers and heat engines, the fracture strength must be evaluated and proof test techniques must be developed. In an evaluation of the strength of siliconized SiC it was observed that the strength can be increased by about 25% by a 1 h–1200° C treatment and can be further strengthened by exposures to 1200° C and stress for a period of 300 h [1]. However, to be useful, the strengthening with stress (prestressing) must exist for times much less than 300 h. In addition, the strengthening with stress can be considered a proof test since some of the weak specimens will fail upon or during the application of the prestress. Joining of SiC ceramics is important in the applications of SiC. The strength of siliconized SiC butt joints can also be increased by a 1 h–1200° C exposure and can be further strengthened by exposures to 1200° C and stress for a period of 300 h [1]. The objective of this paper is to show the degree of strengthening that can be produced in siliconized SiC (with and without joints) by a 10 h, or shorter, 1200° C prestress that also serves as a proof test.

2. Materials and experimental procedure

Strengthening of a siliconized SiC and a siliconized SiC joint material was evaluated. The siliconized SiC (NC430, supplied by the Norton Company, Worcester, Massachusetts) has a two-phase composition with about 10% silicon metal and a continuous matrix of SiC with a bimodal grain size distribution. The fine fraction is smaller than 10 μm and the coarse fraction is approximately 150 μm [2]. Siliconized SiC specimens with butt joints are produced with a bonding material similar to the composition of the base material in that it also has a bimodal grain-size distribution [2]. The major difference is that the bond material has a finer size for the coarse fraction. The bonding material is applied to the joint area in a manner similar to mortaring a brick. The pieces are then joined and the entire assembly refired to give a high-temperature bond. Pieces may be bonded while green, prior to final furnacing, or after they are fired. The bonded specimens that were tested were fired, bonded and then refired [2].

Small bars were loaded in three-point bending with the tensile surface of all of the specimens in the as-fired condition. The three-point bend specimens were of dimensions 2.5 mm × 2.5 mm and were tested over an outer span of 22.5 mm The butt joint was in the centre of the length of the bonded specimens. The bend tests were performed in air at room temperature and 1200° C. All of the specimens were initially exposed to 1200° C for 1 h. This treatment has been shown to increase the room-temperature strength by about 25% [1]. The prestress/proof test was applied at 1200° C at constant stress for periods of time of up to 10 h.

TABLE I Strengthening of SiC for 184 MPa prestress/proof test, obtained from an average of 12 specimens

Specimens failed during prestress/proof test	Time at 1200° C With prestress (h)	Time at 1200° C Without prestress (h)	Fracture strength (MPa)	Weibull modulus	Test temperatures (° C)
–	0	0	213	14.2	23
0	1	0	252	14.8	23
1	10	0	268	12.4	23
1	1	1	240	14.3	23
1	1	9	239	8.6	23
1	1	1	264	9.6	1200
–*	0	0	204	9.6	23
1*	1	0	239	11.3	23

*Butt joints.

3. Results and discussion

The results of all of the strength measurements are summarized in Table I as averages of 12 specimens for each of 8 conditions. All prestress/proof testing was performed at 1200° C and at 184 MPa which is 86% of the base-line strength of 213 MPa. Significant strengthening was observed for 1 h and 10 h prestress (see Fig. 1). Two other groups of 12 specimens were prestressed for 1 h followed by 1 h and 9 h at 1200° C with no stress. A small reduction in the strengthening was observed (Fig. 1).

The strength measurements can be plotted with the Weibull distribution [3, 4]

$$P = 1 - \exp(-R), \quad (1)$$

where P is the probability of failure and R is the risk of rupture. For the two-parameter distribution (σ_0, m), R is defined [3, 4] as the integral of the stress, σ, over the volume, V,

$$R = \int \left(\frac{\sigma}{\sigma_0}\right)^m \mathrm{d}V, \quad (2)$$

where m is the Weibull modulus and σ_0 is a normalizing constant. The constant σ_0 is linearly proportional to the average fracture strength, σ_f, for a constant m and specimen size and is equal to σ_f when there is no scatter ($m \to \infty$). The Weibull modulus is a measure of scatter in the strength distribution, a small m value indicating a large amount of scatter. The Weibull modulus for a material can be determined from a set of strength measurements by a number of methods. One

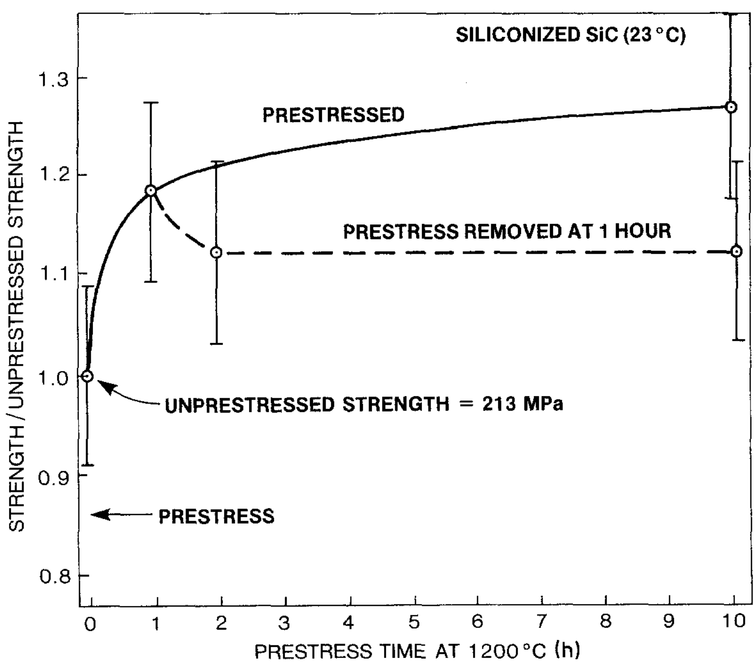

Figure 1 Time-dependence of strengthening by prestressing.

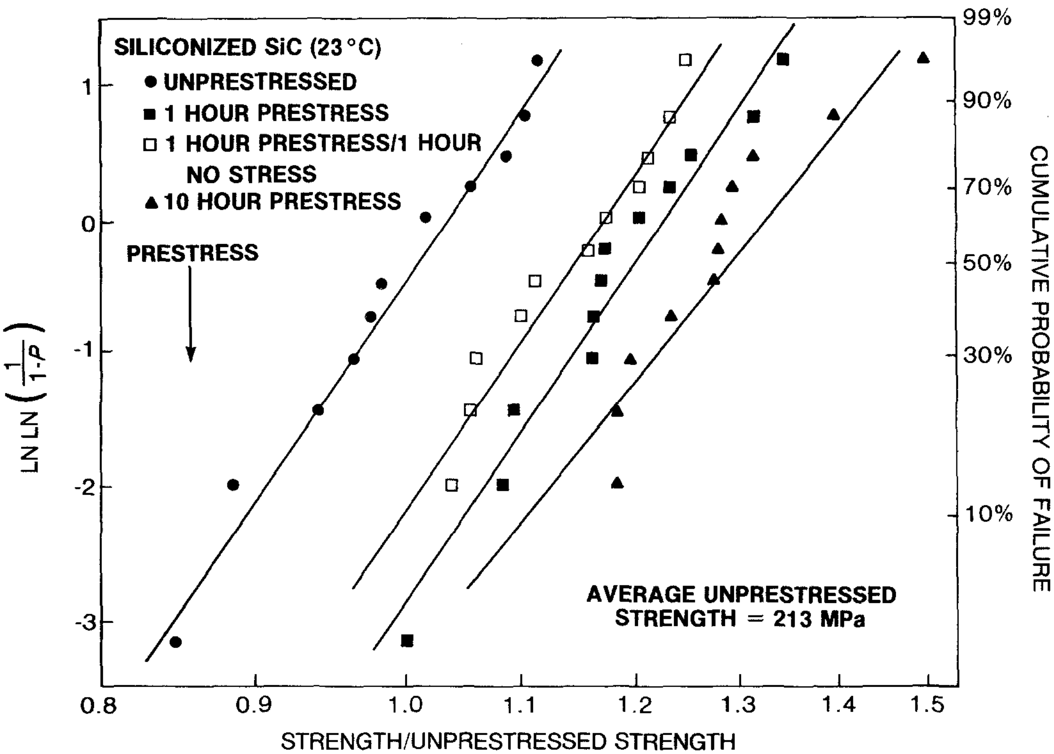

Figure 2 Strengthening by 1200° C prestressing.

simple graphical method requires only a rearrangement of Equations 1 and 2

$$\ln \ln \left(\frac{1}{1-P}\right) = \ln \sigma + \text{constant}, \qquad (3)$$

and a least squares estimation of the slope, m, of the ordered strength measurements. The probability was calculated as $(n-0.5)/N$ where n is the ordering number and N is the total number of specimens. Through computer generation of a large amount of artificial data, this relationship has been shown to converge rapidly to the real m value with increasing number of specimens [5].

The Weibull distribution of fracture strengths for the unprestressed group of specimens and for three groups of specimens with different prestress/proof test conditions is shown in Fig. 2. For the groups of specimens where one or more specimens failed during the prestress/proof test, the strengths of the failed specimens are unknown and are thus not plotted. It is assumed, however, that these specimens are the weakest and thus have the lowest rank in the strength distribution. Under these prestress conditions, the strength is apparently increased by crack blunting or stress relaxation at the crack tip induced by high-temperature time-dependent deformation at the crack tip.

Two groups of 12 specimens were tested at 1200° C following prestressing and proof testing (time-to-failure ≈ 3 sec). Both groups were prestressed at 1200° C and at 184 MPa for 1 h followed by 1 additional hour at 1200° C with no load. The average strength at 1200° C was higher than the room-temperature strength for the same prestress condition but the Weibull modulus was lower (Table I).

The second group of specimens was subjected to a room temperature proof test of 248 MPa prior to the 1200° C strength test. Three specimens failed during the prestress and 4 more specimens failed during the proof test. The room-temperature strength of the proof test failures was well predicted from previous results for the same prestress condition (1 h prestress and 1 h no stress). Also, the 5 survivors of the prestress and proof test that were then tested at 1200° C were consistent with the other group tested at 1200° C (Fig. 3).

It should be noted that for 72 specimens that were prestressed at 1200° C, two failed upon applying the proof test and 5 failed during the prestress with times-to-failure of about 1, 2, 5, 13 and 40 min. The fact that about 10% of these specimens failed during the prestress is not surprising.

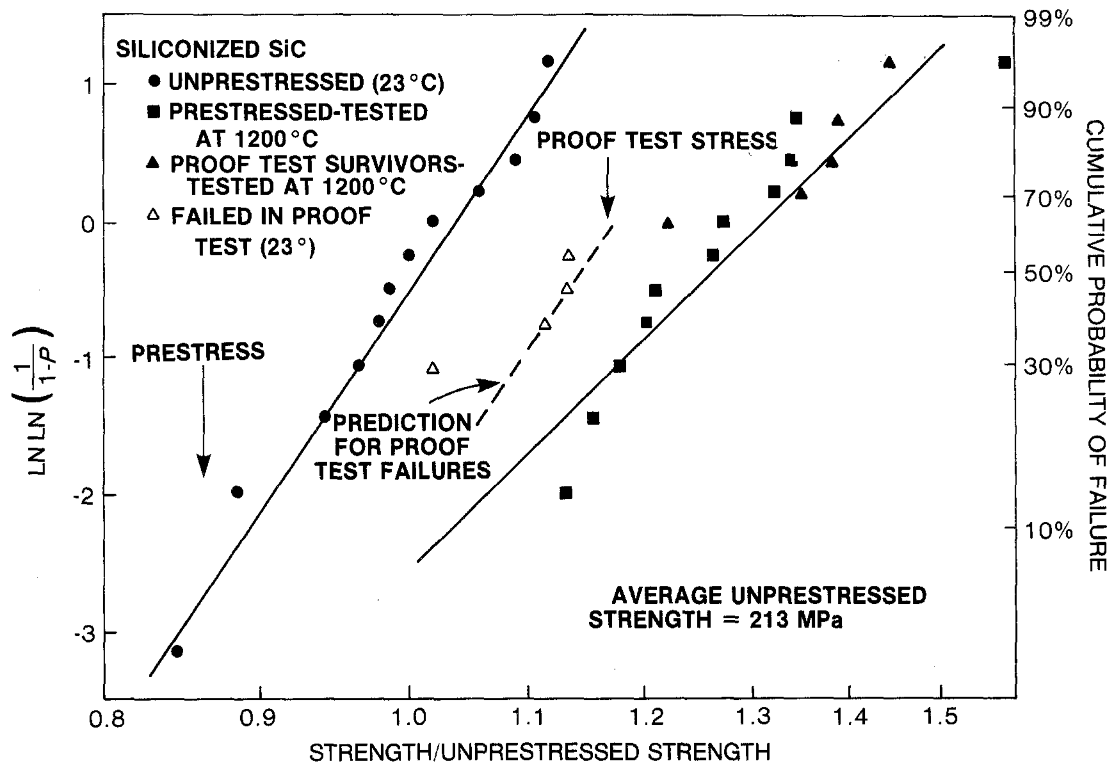

Figure 3 Strength at 1200° C following 1200° C strengthening (1 h prestress/1 h no stress).

The material exhibits a very small amount of strength degradation due to subcritical crack growth at 1200° C [1]. Considering this degradation along with the scatter in strength measurements, it is expected that the average time-to-failure for the prestress of 184 MPa is about 30 000 h [1]. This extremely long average time-to-failure and the large scatter in times-to-failure is the result of a small amount of strength degradation and considerable scatter in the material strength (initial defect size). Materials with a large amount of strength degradation have much less scatter in their times-to-failure [6].

Two groups of 12 specimens with bonded siliconized SiC butt joints were tested (Table I). The average strength of the unprestressed group was slightly less than the average strength for the specimens without a joint and there was more scatter (Fig. 4). The second group of specimens was prestressed to the same stress that was used for the unbonded specimens, i.e., 184 MPa. A similar strengthening was demonstrated for the 1 h prestress (Fig. 4).

To provide a more complete interpretation of the strengthening mechanism, fracture mechanics tests were performed by using the indentation technique for introducing defects [6]. This technique involves using a Knoop microhardness indentation to produce a surface crack. Strength measurements can then be made and if the artificial surface crack controls the fracture process, the fracture toughness can be calculated by knowing the size of the crack. It would be of interest to demonstrate the strengthening process with specimens with artificial cracks. A series of tests were performed and the results are summarized in Table II. For indentation loads of 3.5 and 5.0 kg on a ground surface of a bend specimen, the strength is controlled by the indentation crack reducing the strength and decreasing the scatter (Table II). However, with a 1200° C, 1 h anneal, the strength returns to nearly the same value as for specimens without indentation cracks, since most of the failures in the indented specimens do not occur at the indentation crack (Table II). It is postulated that annealing reduces the residual stresses produced by the microhardness indentation [7]. The natural defects are on the average larger than the indentation cracks and the strength is nearly unaffected by the indentation cracks. Thus,

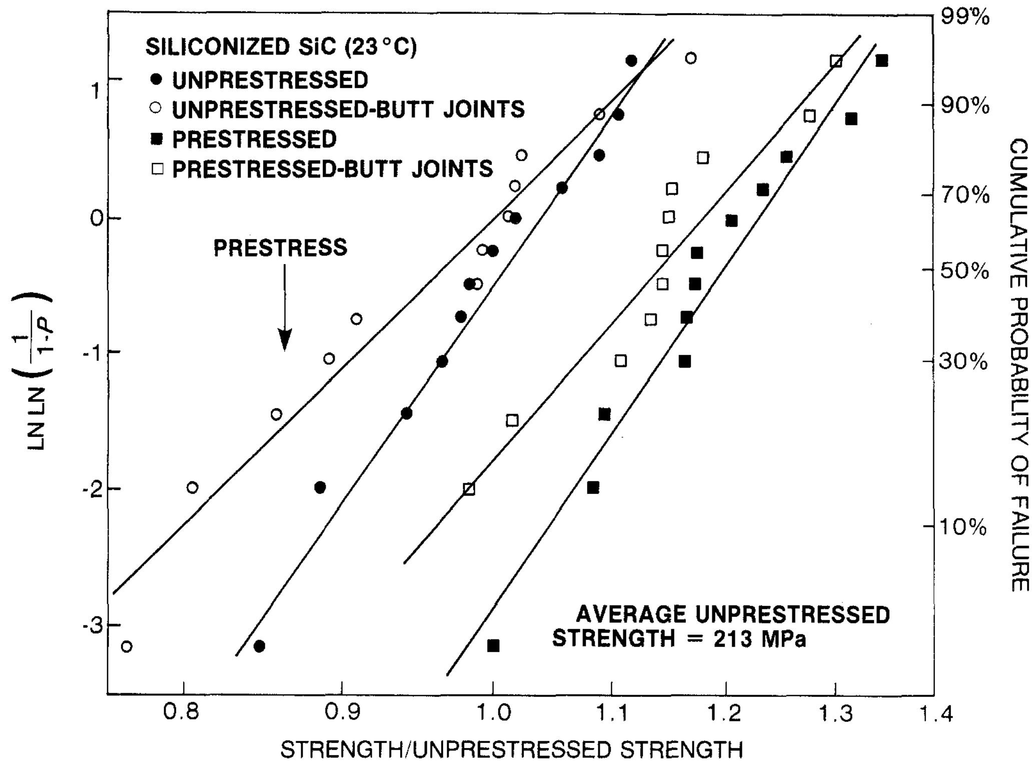

Figure 4 Strengthening of joints by prestressing 1 h at 1200° C.

it was not possible to evaluate the strengthening mechanism in this material with indentation surface flaws.

The strengthening mechanism and the loss of strength observed for stress-free annealing of prestressed materials is not well understood. A plausible mechanism involves consideration of time-dependent deformation at the tip of the crack that controls the fracture strength. Relaxation of stresses at the crack tip during periods of high temperature, constant stress loading, would produce compressive stresses at the crack tip upon unloading and, thus, a strengthening. In addition, these compressive stresses at the crack tip would relax with high-temperature, stress-free annealing and result in a loss of the strengthening. This mechanism is consistent with the experimental results discussed in this paper.

4. Conclusions

Significant strengthening in siliconized SiC can be produced by a prestress/proof test. A 1200° C, 10 h prestress at 86% of the room-temperature strength results in more than a 25% increase in the room-temperature strength. A small reduction in strengthening was observed for prestresses followed by periods with no stress at 1200° C. The Weibull distribution can be used to treat the strength

TABLE II Strength of specimens with cracks produced by Knoop indentation

Total number of specimens	Number failed at indent crack	Indentation load (kg)	Fracture stress (MPa)	Weibull modulus
6	–	0	293	11.6
5	5	3.5	170	15.4
6	6	5.0	160	19.0
6	2	3.5	266	5.4*
6†	2	3.5	292	6.9*

*These samples had a 1200° C/1 h anneal.
†Tested over 22.9 mm (0.9 in) span. All other tests with a 12.7 mm span.

populations including those that are truncated by the prestress/proof test or additional proof testing. A similar strengthening effect occurs for bonded siliconized SiC butt joints. This information is essential in defining proof testing and strengthening conditions for siliconized SiC and in predicting lifetimes of siliconized SiC structures.

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