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# High-temperature slow crack growth of SiC–$Mo_5(Si,Al)_3C$ composites determined by constant-stress-rate testing

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**Abstract**

A novel SiC–$Mo_5(Si,Al)_3C$ composite was fabricated through a melt-infiltration process, and the slow-crack-growth (SCG) behavior of the composite was characterized over four stressing rates ranging from 0.01 to 20 MPa/s both at 1200 and 1300°C in 1 atm argon, using the constant-stress-rate (''dynamic fatigue'') test. The composite shows a high resistance to the SCG at 1200°C, where the slow-crack-growth parameter $n$ was determined to be 86. At 1300°C, however, no significant SCG behavior was observed for $n$ higher than 500. 

*Keywords:* Composites; Crack growth; Mechanical properties; Melt infiltration; SiC–$Mo_5(Si,Al)_3C$

## 1. Introduction

SiC-based ceramics are candidate materials for high-temperature structural applications in heat engines and heat recovery systems.[1,2] One of the major limitations of these materials in these high-temperature applications is delayed failure, where the slow crack growth (SCG) of inherent flaws can occur and cause catastrophic failure even when the applied load is much lower than the fracture strength.[3] Therefore, it is important to evaluate the slow-crack-growth behavior when new ceramic composites are developed. In previous reports,[4,5] novel SiC–$Mo_5Si_3C$ and SiC–$Mo_5(Si,Al)_3C$ composites were developed through a melt-infiltration process. The composites exhibited superior high-temperature mechanical properties where the fracture toughness at 1400°C is more than doubled compared with the value at room temperature. The purpose of the present study is to evaluate the high-temperature slow-crack-growth behavior of the SiC–$Mo_5(Si,Al)_3C$ composite.

The slow crack growth behavior of ceramics could be characterized through direct measurement techniques like the double-torsion method[2,6] and double cantilever method[7] where the crack velocity ($v$) is recorded as a function of the stress intensity factor ($K_I$) or through indirect measurement techniques like the constant-stress-rate (''dynamic fatigue'')[8,9] and the constant-stress (''static fatigue'') tests,[10] from which the crack growth parameters ($n$ and $A$) could be determined. The direct measurement techniques demonstrate the advantage of allowing the determination of the precise shape of the $v$–$K_I$ curves. However, these techniques are relatively complex to carry out. Moreover, the slow-crack-growth behavior on a large artificial crack (generally longer than 1 mm) that is introduced in these tests may not be relevant for predicting the fatigue behavior of natural flaws.[11,12] Some discrepancies between the results from specimens with macro-cracks and from specimens with natural flaws have already been reported.[13,14] In the case of the indirect measurement techniques, the SCG behavior is normally characterized using bending bars with natural flaws or with the indentation-induced cracks. The crack growth parameters are then determined through fitting the experimental data to the presupposed mode. The major advantage of these techniques lies in the fact that they are easy to perform. In the present study, the SCG behavior of the SiC–$Mo_5(Si,Al)_3C$ composite is studied using bending bars with natural flaws via the constant-stress-rate technique.

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## 2. Experimental procedures

The SiC–$Mo_5(Si,Al)_3C$ composite was fabricated through a melt infiltration process. Details of the composite preparation have been previously reported,[5,15] and therefore they are only briefly summarized here. Namely, SiC preforms with dimensions of 40×16×6 mm were prepared from the raw SiC powder (α, circa 3.2 μm, purity 99%, Showa Denko, Japan) through a cold isostatic pressing (CIP) process under 300 MPa. A powder mixture of the $Mo_{10}Si_5AlC_2$ composition, which was prepared from the raw powders of $MoSi_2$ (circa 2.93 μm, Japan New Metals), SiC (the same as above), Mo (circa 1.3 μm, purity 99.94%, Japan New Metals) and Al (circa 1.3 μm, purity 99.94%, Japan New Metals), served as the infiltrant. The infiltration was performed under 1 atm argon in an induction furnace. Spontaneous infiltration could be achieved above 1950°C, and several minutes were found to be sufficient for a complete infiltration. The composite was typically infiltrated at 1950°C for 20 min. The infiltrated composites are relatively dense, but the density could only reach 95% of the theoretical value. The remaining porosity is attributed to the uninfiltrated areas up to 20 μm and closed pores in the SiC matrix that could not be reached during the infiltration process. The volumetric shrinkage during solidification may also contribute to the porosity. Further improvement in product density is very difficult. The typical microstructure of the composite is shown in Fig. 1. The composite is only composed of SiC and $Mo_5(Si,Al)_3C$ phases as confirmed by XRD and SEM/EDS investigations.

The high-temperature slow-crack-growth behavior was determined in flexure using constant-stress-rate ("dynamic fatigue") testing. The specimens were cut from the infiltrated samples and ground to the nominal dimensions of 12 × 3 × 1.5 mm. The fracture strength was tested using a three-point bending fixture with an outer span of 8 mm under 1 atm argon. Four stress rates ranging from 0.01 to 20 MPa/s were employed to study the slow-crack-growth behavior of the composites. A total of five to 10 specimens was tested at each stress rate.

## 3. Results and discussion

Fig. 2 shows the fracture strength of the composite as a function of temperature determined under the stress rate of 2 MPa/s. The symbols represent the mean values obtained from 10 tests, while the total range of the individual strength data is given by the scatter bars. The room-temperature fracture strength of the composite was 350±42 MPa. As demonstrated by the figure, a significant increase in fracture strength was observed between 1300 and 1500°C, reaching the highest value of 590±68 MPa at 1400°C. This strength increase has been tentatively attributed to the plastic deformation of the infiltrated phases, which act as ductile toughening inclusions at elevated temperatures.[5]

The dependence of fracture strength on stress rate at 1200°C is illustrated in Fig. 3, where both average strength (circle) and Weibull median strength (square) are plotted in the figure. This figure shows a decrease of strength with decreasing stress rate. The load deflection curves were monitored and the specimens were inspected for permanent deflection after the tests to check whether specimens underwent creep deformation during fracture. The composite did not reveal any appreciable creep deformation under the stress rates investigated at 1200°C. Also, creep-induced micro- or macro-cracks were not observed in the tensile surfaces of the specimens tested even at the lowest stress rate. This indicates that the failure was governed by one mechanism: slow crack growth. The parameter of the slow-crack-growth (crack velocity exponent $n$) was determined using the

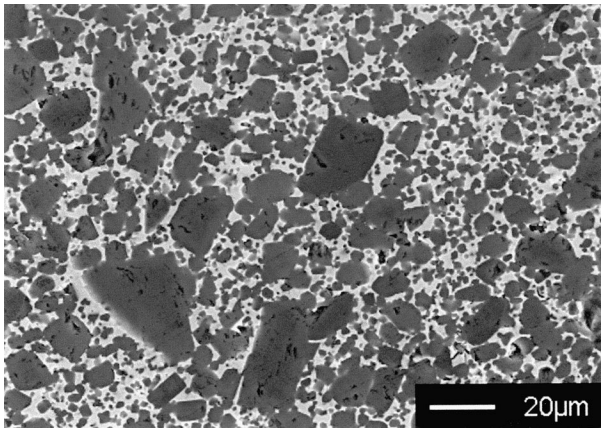


Fig. 1. Typical microstructure of the SiC–$Mo_5(Si,Al)_3C$ composites infiltrated at 1950°C for 20 min under 1 atm argon. The dark phases are SiC, and the bright phases are $Mo_5(Si,Al)_3C$.

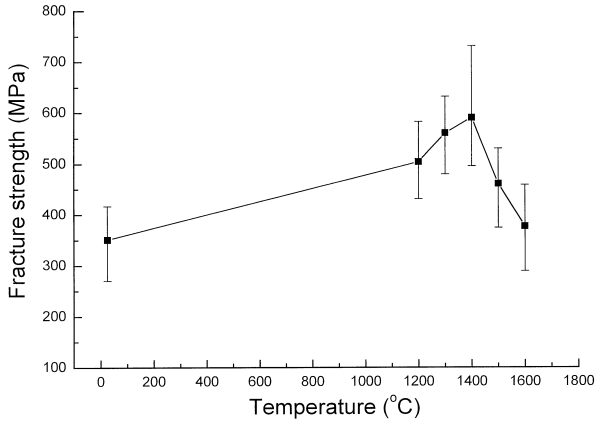


Fig. 2. Fracture strength of the SiC–$Mo_5(Si,Al)_3C$ composite as a function of temperature determined by a three-point bending fixture under a stress rate of 2 MPa/s.

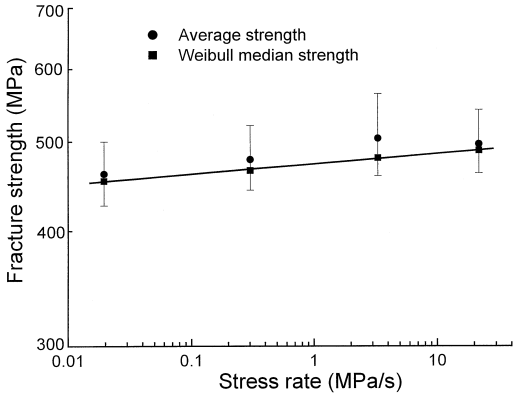


Fig. 3. Fracture strength data as a function of stress rate for the SiC–$Mo_5(Si,Al)_3C$ composite at 1200°C in 1 atm argon.

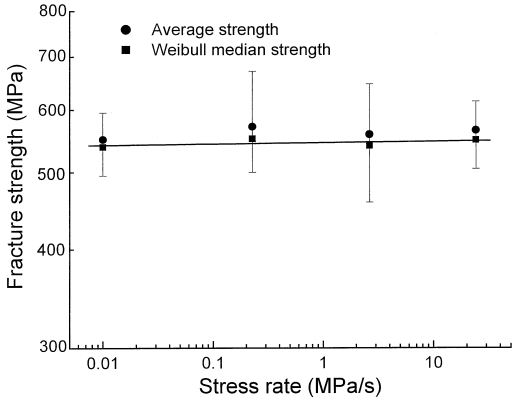


Fig. 4. Fracture strength data as a function of stress rate for the SiC–$Mo_5(Si,Al)_3C$ composite at 1300°C in 1 atm argon.

well-known relation between dynamic bending strength $\sigma_f$, inert strength $\sigma_i$, and the stress rate $\dot{\sigma}$:[16]

$$\sigma_f^{n+1} = B\sigma_i^{n-2}\dot{\sigma}(n+1) \tag{1}$$

$$B = \frac{2}{AY^2(n-2)K_{IC}^{n-2}} \tag{2}$$

where $K_{IC}$ is the mode I fracture toughness of the material, and the parameters $Y$, $A$ and $n$ are correlated by the power law for the crack growth in the range of the linear-elastic fracture mechanics as:

$$v = \frac{da}{dt} = AK_I^n \tag{3}$$

and the stress intensity factor $K_I$ is defined by:

$$K_I = Y\sigma\sqrt{a} \tag{4}$$

where $\sigma$ denotes the applied stress and $v$, $a$, $t$ are the crack velocity, crack size and time, respectively. The crack velocity exponent n was determined to be 86 using the Weibull median strength data in Fig. 3, demonstrating a high resistance to slow crack growth of the composite. Basically, the parameter $A$ in Eq. (3) could also be determined using the "dynamic fatigue" tests, however, the evaluation of A requires the inert strength $\sigma_i$ that could only be correctly measured under the stress rate of $\sim 10^4$ MPa/s.[3] The determination of inert strength is not attainable in the present study due to the limitation of the testing equipment.

Fig. 4 shows the slow crack growth behavior of the composite at 1300°C in 1 atm Ar. The decrease in strength with decreasing stressing rate was not significant at this temperature. The fracture strength was determined to be 560 MPa under the stressing rate of 20 MPa/s, which corresponds to a failure time of less than 40 s, while the strength was recorded as 550 MPa at the slowest stressing rate of 0.01 MPa/s that results in a total time-to-failure of ~15 h. The crack velocity exponent $n$ calculated from the Weibull median strength data in Fig. 4 is greater than 500, indicating that the SCG is not significant at 1300°C. It should be noted that the composite underwent significant creep deformation under the slowest stress rate of 0.01 MPa/s at 1300°C, but the creep deformation was negligible at the other stress rates. The slow-crack-growth behavior of the SiC based composites has been extensively studied in the past two decades.[2,3,8–10] It was generally found that the SCG behavior of these composites is sensitive to microstructure, composition, testing temperature and even small amounts of impurity. It was also proposed that a plastic flow mechanism through grain boundary sliding or separation was responsible for the SCG in some cases.[2,3,17] The present composites revealed a distinct strength and toughness increase above ~1300°C,[5] which is tentatively attributed to the plastic deformation of the intermetallic phases. This correlates well with the SCG behavior of the composite at 1200 and 1300°C. However, further study is definitely necessary to elucidate detailed SCG mechanism for the present composite.

## 4. Conclusions

The slow-crack-growth behavior of a novel SiC–$Mo_5(Si,Al)_3C$ composite was studied at 1200 and 1300°C in 1 atm argon over four stress rates ranging from 0.01 to 20 MPa/s. The composite shows a high SCG resistance at 1200°C with the crack velocity exponent of 86, while no significant SCG behavior was observed at 1300°C. The mechanism for the slow-crack-growth behavior of the composite has not yet been clarified.

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