Prediction of fracture strength in Al_2O_3/SiC_p ceramic matrix nanocomposites

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Abstract

Based primarily on a recent publication [S.M. Choi, H. Awaji, Sci. Tech. Adv. Mater. 6 (2005) 2–10.], where the dislocations around the nano-sized particles in the intra-granular type of ceramic matrix nanocomposites (CMNCs) were modeled, dislocation activities in Al_2O_3/SiC_p CMNCs were discussed in relation to the processing conditions. The dislocations around the nano-sized particles, caused by the thermal mismatch between the ceramic matrix and nano-sized particles, were assumed to hold out the effect of Orowan-like strengthening, although the conventional Orowan loops induced by the movement of dislocations were unlikely in the ceramic matrix at room temperature. A model involving the yield strength of metal matrix nanocomposites (MMNCs), where the Orowan strengthening effect was taken into consideration, was thus modified and extended to predict the fracture strength of the intra-granular type of CMNCs without and with annealing. On the basis of the characteristics of dislocations in the CMNCs, the load-bearing effect and Orowan-like strengthening were considered before annealing, while the load-bearing effect and enhanced dislocation density strengthening were taken into account after annealing. The model prediction was found to be in agreement with the experimental data of Al_2O_3/SiC_p nanocomposites reported in the literature.

Keywords: Ceramic matrix nanocomposites; Fracture strength; Dislocation activities; Strengthening mechanisms

1. Introduction

A model for predicting the yield strength of particulate-reinforced metal matrix nanocomposites (MMNCs) has recently been developed, on the basis of the load-bearing effect, the enhanced dislocation density due to the residual plastic strain caused by the difference in the coefficients of thermal expansion between the matrix and particles, and Orowan strengthening effect [1]. The equation can be expressed as follows:

\[ \sigma_{yc} = \sigma_{ym}(1 + f_l)(1 + f_d)(1 + f_{Orowan}), \] (1)

\[ f_l = 0.5 V_p, \] (1a)

\[ f_d = \frac{1.25G_m b}{\sigma_{ym}} \sqrt{\frac{12(T_{\text{process}} - T_{\text{test}})(\alpha_m - \alpha_p)V_p}{b d_p (1 - V_p)}}, \] (1b)

\[ f_{Orowan} = \frac{0.13G_m b}{\sigma_{ym} d_p \left( \frac{1}{2\pi} \right)^{1/3}} \ln \frac{d_p}{2b}, \] (1c)

where \( \sigma_{yc} \) is the yield strength of particulate-reinforced MMNCs, \( \sigma_{ym} \) is the yield strength of the monolithic matrix under the same processing conditions as those of MMNCs, \( f_l \) is the improvement factor due to the load-bearing effect, \( f_d \) is the improvement factor associated with the enhanced dislocation density in the matrix induced by the thermal mismatch between the matrix and the reinforcement particles, \( f_{Orowan} \) is the improvement factor due to the Orowan strengthening effect, \( G_m \) is the shear modulus of the matrix, \( b \) is the Burgers vector of dislocations in the matrix, \( T_{\text{process}} \) is the processing temperature, \( T_{\text{test}} \) is the test temperature, \( \alpha_m \) is the coefficient of thermal expansion.

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of the matrix, \(x_p\) is the coefficient of thermal expansion of the reinforcement phase, \(V_p\) is the volume fraction of reinforcement nanoparticles, \(d_p\) is the particle size. The relative contribution of the strengthening factors was also analyzed. While the Orowan strengthening effect might be small in the micro-sized particulate-reinforced composites, it played an important role in the nano-sized particulate-reinforced composites [2]. The prediction based on the proposed model was found to be in good agreement with the experimental data [1,2]. The aim of the present investigation was to explore the possibility of extending the proposed model to predict the fracture strength of ceramic matrix nanocomposites (CMNCs).

2. Dislocations in CMNCs

Considerable interest in developing CMNCs, such as Al\(_2\)O\(_3\)/SiC\(_p\) nanocomposites, has been stimulated since 1991 [3]. The basic characteristics of CMNCs observed by many researchers have been summarized in Refs. [4,5], including improvements in the strength and fracture toughness due to the change of fracture mode from intergranular cracking in the monolithic alumina to transgranular cracking in nanocomposites. Other aspects involving the mechanical properties of CMNCs include the improvement in the creep and wear resistance. To explain these characteristics, several models involving the strengthening mechanisms of CMNCs have been proposed, including a reduction in the critical flaw size (C-mechanism), an increase in the fracture toughness (K-mechanism), and more complex mechanisms that are based on grain boundary strengthening and internal stresses [6,7]. The C-mechanism is based on the fact that the matrix becomes smaller following the addition of nano-sized particles. The grain size refinement results in a smaller critical flaw size and higher strength in terms of the Hall–Petch relationship. The K-mechanism is associated with K-curve behavior, crack deflection, and crack bridging during crack extension. Nano-sized particles present within matrix grains strengthen the grain boundaries due to compressive radial stresses, where the alumina matrix has a larger thermal expansion coefficient than that of silicon carbide particles [5,8]. Others include flaw-healing mechanisms [9,10].

However, it has been pointed out that the observed mechanical characteristics in CMNCs cannot be fully explained by these mechanisms [5]. The mechanisms based on dislocation activities have recently been explored by several investigators [4,5,11,12]. While dislocations may seldom be present in ceramics, e.g., alumina, it has been demonstrated that under a certain stress state they are generated in the monolith [8,12,13] and plentifully in Al\(_2\)O\(_3\)/SiC\(_p\) nanocomposites [3,11,12,14,15]. Strengthening of metallic alloys via dislocation–dislocation or dislocation–particle interactions is well documented. In comparison with metallic materials, ceramics do not yield much before failure. However, since some plastic yielding (dislocation generation) is observed during their deformation, any factor that constrains the occurrence of the limited yielding in ceramic materials is likely to raise their strength [11]. Then the role of dislocations in the alumina system, particularly in the annealed state where the annealing conditions promote dislocation activities, is worthwhile to be explored [11].

The dislocations or dislocation networks in the alumina/silicon carbide nanocomposites have been observed by means of TEM [3,5,11,12,15,16]. Similar to the MMNCs, the creation of dislocations in CMNCs is also considered to be due to the highly localized residual stresses in the matrix grains, generated by the mismatch of thermal expansion coefficients between the matrix and the dispersed particles during the cooling process after sintering [5]. A theoretical analysis indicates that the residual stresses are large enough to create dislocations around the dispersed particles, and only the nano-sized particles could create dislocations [4]. The dislocations generated release the tensile residual stresses existing in the matrix and thus improve the strength of sintered CMNCs. If a high-temperature annealing process is subsequently applied following sintering, the dislocations around the nanoparticles can disperse into matrix grains, which is considered to become nano-crack nuclei in a frontal process zone (FPZ) ahead of the propagating crack tip at room temperature, leading to the toughening and strengthening CMNCs [4,5]. It should be noted that the FPZ in Refs. [4,5] indeed means the plastic zone in front of the crack tip in fracture mechanics, which has been directly observed around Vickers indents using TEM to be about 10 μm for Al\(_2\)O\(_3\)/SiC\(_p\) nanocomposites and slightly smaller for alumina [12]. The crack tip blunting or plastic deformation in ceramics, which has been established earlier [8,13], is further corroborated via a direct comparison between the observed critical flaw sizes and those predicted using Griffith's model (where an atomically sharp crack tip is considered) and Orowan's model (where a blunt crack tip is considered) [12]. Furthermore, the dislocation-based strengthening mechanism was reported to be more consistent with experimental observations than the flaw healing mechanism [11]. As well, two types of dislocations have been specified, i.e., pre-existing dislocations that may not blunt the crack tip, and newly created dislocations resulting from the fracture process [12,13]. From these results reported in the literature, it may be necessary to consider the dislocation-based strengthening mechanism in CMNCs. The present study represents an attempt of predicting the fracture strength of CMNCs on the basis of the dislocation-based strengthening mechanism.

3. Fracture strength of CMNCs

3.1. CMNCs without annealing

As mentioned above, in this case the dislocations generated during the synthesizing or sintering process could only be present around nanoparticles [5]. The pre-existing
dislocations induced due to the difference of coefficients of thermal expansion between the matrix and reinforcement nanoparticles would be difficult to move at room temperature. While these dislocations are sessile, they would restrict the movement of newly created dislocations resulting from the fracture process when the crack tip approaches this area. The pre-existing dislocations have indeed been considered to form a loop-like configuration around the nanoparticles, as schematically illustrated in [5]. Such a dislocation configuration led to an assumption that the Orowan-type strengthening effect might be operative, since it is analogous to the Orowan loops left around the nanoparticles in MMNCs or precipitates in metallic alloys (e.g., aluminum alloys and superalloys) after the dislocations have by-passed the nanoparticles or precipitates on the slip plane. Hence, in this case, the strengthening caused by Orowan-like loops and load-bearing effect is considered to estimate the fracture strength of CMNCs. By modifying Eq. (1), the fracture strength of CMNCs without annealing becomes

$$\sigma_{fc} = \sigma_{fm}(1 + f_i)(1 + f_{Orowan}),$$

(2)

$$f_{Orowan} = \frac{0.13G_mb}{\sigma_{fm}d_p}\left(\frac{1}{\sqrt{\pi}d_p}\right)^{1/3} \ln \frac{d_p}{2b},$$

(2a)

where $\sigma_{fc}$ is the fracture strength of particulate-reinforced intra-granular type CMNCs, $\sigma_{fm}$ is the fracture strength of the monolithic matrix under the same processing conditions as those of CMNCs, $f_i$ is expressed by Eq. (1a), $f_{Orowan}$ has the same meaning as specified above, but is related to the fracture strength of the ceramic matrix (instead of the yield strength).

3.2. CMNCs with annealing

In the case of CMNCs with annealing after sintering, the dislocations generated around the nanoparticles can move and disperse into the matrix, due to holding at a high temperature with sufficient energy activation, as reported in Ref. [5]. Like MMNCs, the dispersed dislocations in the matrix would play a role in resisting the crack extension, thus enhancing the fracture strength of CMNCs [4,5]. Again, it is difficult for the dispersed dislocations in the ceramic matrix to move at room temperature. One explanation has been given as follows [4,5]. Since the dispersed sessile dislocations would serve as origins of stress concentrations, and create nano-cracks in the plastic zone ahead of a propagating crack tip at room temperature, the highly stressed state in the plastic zone would be released by the nano-crack nucleation, enhancing the fracture strength and toughness [4,5]. In this case, Orowan-like loops are no longer present around the nanoparticles. Thus, there would be no contributions from the Orowan-type strengthening effect. Then the load-bearing effect and the dispersed dislocations after annealing would contribute to the fracture strength of CMNCs ($\sigma_{fc}$), i.e.,

$$\sigma_{fc} = \sigma_{fm}(1 + f_i)(1 + f_d),$$

(3)

$$f_d = \frac{1.25G_mb}{\sigma_{fm}} \sqrt{\frac{12(T_{process} - T_{test})(a_m - a_p)V_p}{b_b(d_p(1 - V_p))},}$$

(3a)

where $f_i$ is also expressed by Eq.(1a), $f_d$ has the same meaning as specified above, but is related to the fracture strength of the ceramic matrix (instead of the yield strength). All other parameters are the same as those stated above.

3.3. Burgers vector

It is seen from Eqs. (2a) and (3a) that the Burgers vector of dislocations in the matrix is involved. The formation and motion of dislocations generally require more energy in ceramics than in metals. Due to the complex ceramic structures, which often have large unit cells, and typically strong and localized bonding between ions, dislocations in ceramics are relatively harder to create and more difficult to move in the form of perfect dislocations [17]. To facilitate the dislocation movement, a further reduction in the total dislocation energy and in the energy necessary to move the dislocation through the lattice is needed. This could be achieved by splitting the total dislocation into four partials separated by areas of stacking faults. In particular, at elevated temperatures and lower strain rates, the $\text{Al}^{3+}$ and $\text{O}^{2-}$ motion in the dislocation core can be synchronized and the stress required for the plastic deformation becomes lower [18]. A dissociation of $\frac{1}{2}<10\bar{1}0>$ partial into quarter partials, with the introduction of a fault in the oxygen ion lattice, was proposed by Kronberg according to the following reaction [19,20]:

$$\frac{1}{2}<10\bar{1}0> \rightarrow \frac{1}{4}<2\bar{1}0> + \frac{1}{4}<11\bar{2}0>.$$

(4)

This reaction has been observed during the dissociation of the prism plane dislocations [20–22] and within dislocation networks produced by crack healing [20,23]. In the following section, the Burgers vector in alumina-based nanocomposites is considered to be $\frac{1}{2}<11\bar{2}0>$ type, with a magnitude of $0.4759/3 \approx 0.16 \text{ nm}$ [18,20].

4. Verification and discussion

A number of experimental data reported by different investigators using varying processing conditions are used to test the above model. Fig. 1 shows a comparison of the model prediction with the experimental data for SiC particulate-reinforced alumina nanocomposites [9]. In this figure, the following data for Al$_2$O$_3$/SiC$_p$ nanocomposites are used [9]: $\sigma_{fm} = 560 \text{ MPa}$, $E_m = 393 \text{ GPa}$, $\nu = 0.25$, $G_m = E_m/[2(1 + \nu)] = 157.2 \text{ GPa}$; $b = 0.16 \text{ nm}$ [20–23]; $a_m = 8.8 \times 10^{-6} (\text{ C}^{-1})$, $a_p = 4.7 \times 10^{-6} (\text{ C}^{-1})$ [4]; $T_{process} = 1775 ^\circ \text{ C}$, $T_{test} = 20 ^\circ \text{C}$, and $d_p = 150 \text{ nm}$. A fairly good agreement can be seen in both cases without and with
annealing. Fig. 2 shows another comparison, where the following data for Al₂O₃/SiCₚ nanocomposites reported by Wu et al. [10,11] are used: $\sigma_{fm} = 295 \text{ MPa}$ [10,11], $T_{process} = 1650^\circ \text{C}$, $T_{test} = 20^\circ \text{C}$, $d_p = 90 \text{ nm}$, and other parameters remain the same as given above. It appears reasonable that the experimental result could be lower than the model prediction after annealing, since all the dislocations were assumed to punch out and disperse into the matrix to enhance the strength, as indicated by Eq. (3a). On the other hand, the strengthening effect of the dislocations around the nanoparticles before annealing could be somewhat stronger or weaker than the assumption of only Orowan strengthening effect, depending on the dislocation generation caused by the thermal mismatch between the matrix and nanoparticles. Then the experimental data could be slightly higher or lower than the model predictions based on Eq. (2), which can be seen in Figs. 1 and 2. Another reason why the experimental value after annealing is slightly lower than that predicted via Eq. (3) would be the existence of porosity in the test specimens, as indicated by the lower density in the Al₂O₃/5% vol. SiC nanocomposites, where $\rho_{\text{theoretical}} = 3.951 \text{ g/cm}^3$, while $\rho_{\text{experimental}} = 3.89 \pm 0.02 \text{ g/cm}^3$ [24]. The lower the density, the higher the porosity, and the lower the fracture strength [25]. One more reason could be associated with the coarsening of the matrix and nanoparticles caused by annealing. The coarser the matrix grains and the reinforcement particles, the lower the fracture strength.

Fig. 3 shows a comparison between the experimental data and model prediction with different nanoparticle sizes. The following data given by Wu et al. [10] and Carroll et al. [24] are used: $\sigma_{fm} = 491 \text{ MPa}$, $E_m = 396 \text{ GPa}$, $v = 0.25$, $G_m = E_m/[2(1+v)] = 158.4 \text{ GPa}$, $b = 0.16 \text{ nm}$ [20–23], $\alpha_m = 8.8 \times 10^{-6} \text{ C}^{-1}$, $\alpha_p = 4.7 \times 10^{-6} \text{ C}^{-1}$ [4], $T_{process} = 1700^\circ \text{C}$, $T_{test} = 20^\circ \text{C}$, $V_p = 0.05$, and $d_p = 50, 55, 90$ and $115 \text{ nm}$. Reasonably good agreement is also seen from Fig. 3. In addition, it is noticed from the experimental results in Ref. [24] that the fracture toughness $K_{IC}$ values of nanocomposites are also higher than those of the
monolithic alumina, and the value of $K_{IC}$ increases with decrease in size of SiC nanoparticles as well.

Good agreement can be further seen in Fig. 4 with different volume fractions of SiC nanoparticles, where the following data reported recently by Sun et al. [26] are used: $\sigma_{fm} = 344$ MPa, $E_m = 380$ GPa, $v = 0.25$, $G_m = E_m/[2(1 + v)] = 152$ GPa, $b = 0.16$ nm [20–23], $x_m = 8.6 \times 10^{-6} \cdot ^\circ C^{-1}$, $x_p = 4.5 \times 10^{-6} \cdot ^\circ C^{-1}$, $T_{process} = 1600 ^\circ C$, $T_{test} = 20 ^\circ C$, $d_p = 100$ nm, and $V_p = 0.02$, 0.03, 0.04 and 0.05. It is seen from both the model prediction and experimental results that the fracture strength increases with increasing volume fraction.

From Eq. (3a), it is noticed that if $x_p \approx x_m$, $f_d \approx 0$. It implies that there is no enhanced dislocation strengthening effect in this case. Consequently, this analytical model prediction is also in agreement with the results presented by Walker et al. [27] and Derby [28], who reported that there exists no (or very weak) strengthening in Al$_2$O$_3$/TiN nanocomposites because of $x_{Al_2O_3} \approx x_{TiN}$.

It should be pointed out that while the above analysis exhibits a fairly good agreement with the experimental results reported in the literature [9–11,24,26], the dislocation activities in the CMNCs are different from those in the precipitation-hardened metallic materials. For example, unlike aluminum alloys and nickel-based superalloys, the dislocations in ceramics can hardly move on the slip plane and be stopped by the barrier to form the conventional Orowan loops at room temperature. However, the dislocations generated surrounding the nanoparticles are present in CMNCs [5], which are assumed to be analogous to the Orowan loops in the metallic materials. While one could understand the effect of Owowan-type strengthening in such a way that the formation mechanism of Orowan-like loops around the barrier is different in metals and in CMNCs, i.e., via the movement and interaction of dislocations with the nano-sized precipitate particles in the former case, and the thermal mismatch between the ceramic matrix and nano-sized particles in the latter case, more experimental and theoretical/modeling studies are needed to further understand the dislocation activities in different types of CMNCs.

## 5. Conclusions

In light of the dislocation activities and fabricating conditions, a simple model was applied to predict the fracture strength of intra-granular type of ceramic matrix nanocomposites. Before annealing the load-bearing effect and Owowan-like strengthening were considered, while the load-bearing effect and enhanced dislocation density strengthening were taken into account after annealing. The model prediction was found to be in agreement with the experimental data for SiC particulate-reinforced alumina nanocomposites fabricated with varying processing parameters in both cases without and with annealing.

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