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PLASTIC DEFORMATION OF ALUMINA REINFORCED WITH SiC WHISKERS

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INTRODUCTION

Addition of SiC whiskers to polycrystalline Al_2O_3 , has been shown to improve mechanical properties of the matrix,¹ including creep resistance.^{2,3} Recent work^{4,5} has shown that the observed reinforcement was due to the pinning of grain boundaries by the whiskers, and therefore, to a partial or complete inhibition of grain-boundary sliding (GBS), the principal plastic deformation mechanism for fine-grained Al_2O_3 at low stresses. The SiC whiskers exhibited no plastic deformation whatsoever at any temperature.

The precise creep mechanisms active for the composites with increasing whisker content is still a matter of discussion. In a previous publication,⁵ a study of creep in compression was made on $\text{SiC}_w/\text{Al}_2\text{O}_3$ samples, obtained from different manufacturers, which contained a wide range of whisker concentrations. In that work, it was concluded that for partial volumes of whiskers, V_w , larger than 15%, the whiskers effectively impede grain-boundary sliding, causing a change in the deformation mechanism from GBS to pure diffusional creep (PD), without significant changes in diffusional kinetics. However, for stresses over a critical stress σ_c , which was lower for composites containing larger amounts of glassy phase, the whiskers enhanced cavitation resulting in severe damage formation.

An important finding was that the creep rates exhibited by samples containing more than 15 vol%, despite having very different nominal matrix grain sizes were nearly equal. The ratio of the creep rates of the composites to that of the matrix, was larger than calculated from creep equations for GBS and PD. Under constant temperature and diffusion mechanisms, grain size, d , is the only significant materials parameter:

$$\dot{\epsilon} \propto d^{-p} \sigma^n \quad (1)$$

where $\dot{\epsilon}$ is the strain rate, d is the grain size, and σ is the applied stress, with p being 2 or 3, and n the stress exponent.⁶ In light of Equation 1, the extra creep reinforcement might be understood if the composite behaved as if it had larger grain size than the nominal reported value. A tentative explanation was that the matrix grain size in the creep equation should be replaced by another microstructural parameter, called effective grain size, d_{eff} , that was proposed to be related to the available space for the matrix grains between the whiskers.

In the present paper, creep data are re-analyzed to clarify the evolution of the plastic deformation mechanisms and to extend the understanding of the physical meaning of the effective grain size concept. A model is proposed to relate d_{eff} to the space between whiskers.

MATERIALS, CREEP BEHAVIOR, AND CREEP PARAMETERS

Compressive creep experiments were conducted in an inert atmosphere on samples fabricated at Argonne National Laboratory, ANL, and Oak Ridge National Laboratory, ORNL. Composite materials contained up to 30 vol% chemically etched whiskers, and no sintering additives, thus clean grain boundaries have been documented. The whiskers had typical lengths of 10 μm and diameters of approximately 1 μm . Further data on processing techniques and microstructural development can be found elsewhere.^{4,7}

Throughout this paper, the samples are referred to by the manufacturer, ANL or ORNL, followed by a number that represents the whisker volume percent.

Figure 1 compares creep rates obtained for composites with those of polycrystalline Al_2O_3 from the same source. Initially, these data have been analyzed with respect to a classical creep equation:

$$\dot{\epsilon} = A\sigma^n \quad (2)$$

where A depends principally on temperature and grain size.⁶

Table 1 contains the n and A values determined by linear regression. Pure alumina samples yield constant n values of 1.3 over the whole stress range. The stress exponent for ANL5 was 1.8 over the entire stress range. However, the data of Figure 1a could be fit equally as well by $n = 2.6$ for lower stresses and 1.3 at high stresses. ANL15, ORNL20, and ANL30 composites exhibited a stress exponent that changed from $n \approx 1.0$ to $n > 2$, depending mainly on stress. An estimate of the stress at which the change occurred (σ_c , critical stress) is also included in Table 1.

Microstructural evidence showed that the $n > 3$ regime found in samples with more than 15 vol% of whiskers corresponded to accelerated creep as a result of damage formation: the whiskers act as stress concentration sites that promote the formation of cavities. Lower n -regimes were governed by a series of steady states, for which classical creep models were applicable. An evolution from GBS to PD with increasing whisker content was proposed. For lower whisker content samples (ANL5), some GBS is possible. The low stress regime for ANL15, ORNL20 and ANL30 is purely diffusional, because whiskers effectively pin grain boundaries. These conclusions were also supported by microstructural observations.

Finally, it was found that diffusional processes controlling plastic deformation remained essentially unchanged with the addition of SiC whiskers. For pure Al_2O_3 , grain boundary diffusion of the specie Al^{3+} is creep-rate controlling, implying a dependence of d^{-3} with the grain size.⁸

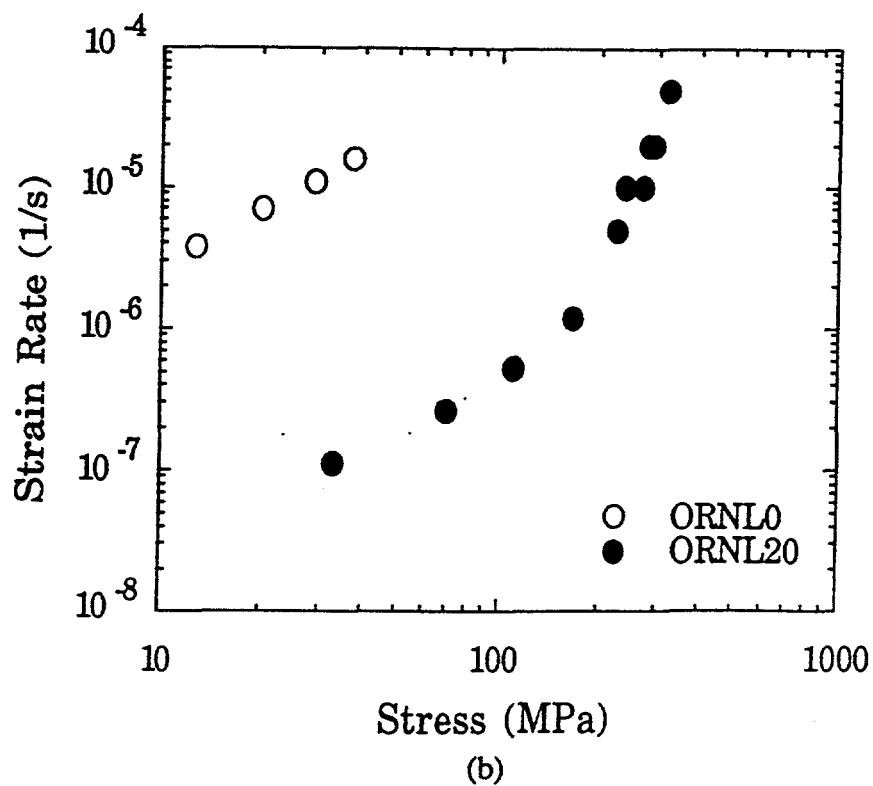
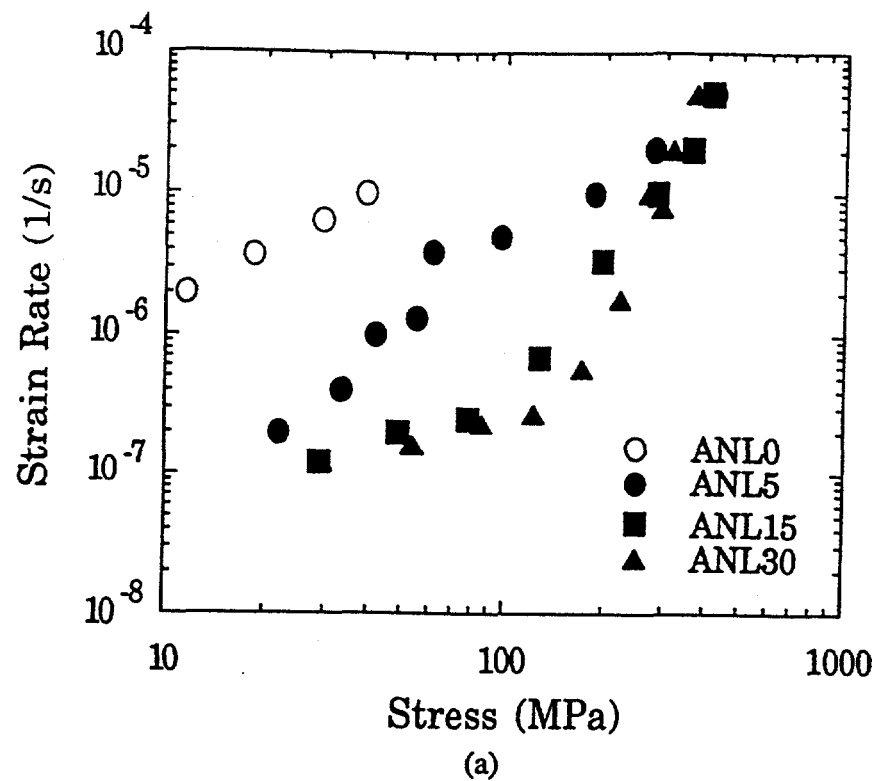


Figure 1. Strain rate vs. stress plots of compressive creep results obtained in (a) ANL and (b) ORNL matrixes and composites at 1400°C in argon. Significant creep rate reduction in composite is observed.

Table 1: Creep Parameters.

Samples	d (μm)	n_1	A_1	Transition σ (MPa)	n_2	A_2
ANL0	1.8	1.3	7.9×10^{-8}			
ANL5	2.8	2.6	4.5×10^{-11}	60	1.3	1.4×10^{-8}
ANL15	3.3	0.9	1.0×10^{-8}	100	3.4	3.9×10^{-14}
ANL30	1.5	0.9	5.0×10^{-9}	170	5.9	4.0×10^{-20}
ORNLO	1.5	1.3	1.3×10^{-7}			
ORNL20	2.0	1.3	1.3×10^{-9}	160	5.4	1.0×10^{-18}

ANALYSIS OF CREEP RESULTS BY CLASSICAL EQUATIONS

Creep of Al_2O_3

Stress exponents ranging between 1 and 2 have been reported for monolithic Al_2O_3 . These values depend on grain size, impurity content, and test conditions.⁶ In our experiments, n was equal to 1.3 for both ANL and ORNL samples. GBS accommodated by diffusional transport of matter was considered to be the dominant mechanism of plastic deformation.⁸

Ashby and Verrall⁹ established a model for steady-state creep based on diffusion-controlled grain-boundary sliding:

$$\dot{\epsilon}_{\text{GBS}} = \frac{98(\sigma - \sigma_t)\Omega D^{\text{eff}}}{kTd^2} \quad (3)$$

where σ_t is a threshold stress, Ω is the atomic volume, D^{eff} is the effective diffusion coefficient, and k and T have their usual meanings. When the creep-rate-controlling transport takes place through the grain boundaries, the effective diffusion coefficient depends on d^{-1} , so the strain rate is proportional to d^{-3} , as stated before. The difference between the experimental stress exponents from the predicted $n = 1$ for diffusional flow is ascribed to the fact that the threshold stress was not taken into account. In fact, an alternative explanation of a stress dependent n is that the threshold stress is altered by the addition of whiskers. Instead of Equation 2, we consider the dependence:

$$d_m^3 \dot{\epsilon} = B_m (\sigma - \sigma_{t(m)})^n \quad (4)$$

where B is a grain-size-independent parameter, and the subscript m refers to parameters of the matrix. If we assume $n = 1$, it is possible to estimate the threshold stress, by plotting $d^3 \dot{\epsilon}$ vs. σ , to determine B_m and $\sigma_{t(m)}$ (Figure 2), together for ANL and ORNL pure Al_2O_3 matrixes. When normalized by grain size, the agreement between the of creep rates of both samples is excellent. Regression analysis resulted in:

$$B_m = (1.66 \pm 0.11) \times 10^{-6} \mu\text{m}^3 \text{MPa}^{-1} \text{s}^{-1}$$

$$\sigma_{t(m)} = 5.3 \pm 2.5 \text{ MPa.}$$

Creep of Composites

Addition of small concentrations of whiskers to the matrix could initially change the threshold stress because the whiskers pin the grain boundaries. When tested at high enough stress, $n = 1.3$ for ANL5, the same value that was found for the pure matrix. In that range, the increment in the threshold stress due to the whiskers can be neglected in comparison with the applied stress, thus explaining the same exponents. At low stresses, σ_i is significant compared with the applied stress, and the value $n = 2.6$ is a mathematical artifact.

Using Equation 4, we can calculate B and σ_i for the composite, denoted with the subscript c (Figure 2):

$$B_c = (1.65 \pm 0.30) \times 10^{-6} \mu\text{m}^3 \text{MPa}^{-1} \text{s}^{-1}$$

$$\sigma_{i(c)} = 27.0 \pm 20.0 \text{ MPa.}$$

The agreement found in B values is very good, confirming that the diffusional kinetics are unchanged. The only effect of the whiskers is the increase in the nominal threshold stress, recognizing the large scatter.

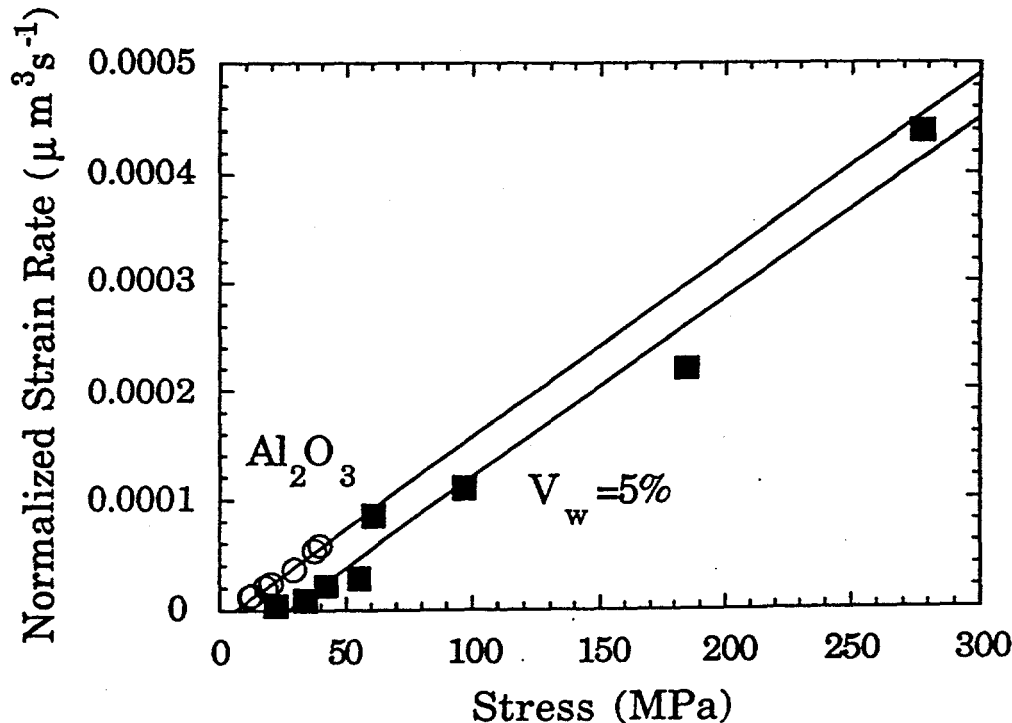


Figure 2. Normalized strain rate ($d^3 \dot{\epsilon}$) vs. stress linear plots to estimate threshold stresses in monolithic Al_2O_3 and in 5 vol.% composite.

With 15 vol% or more of SiC whiskers, the evolution of the creep mechanism from GBS to PD is complete. The whisker concentration is large enough to establish a threshold stress that is higher than the critical value σ_c . For $\sigma < \sigma_i$ the creep controlling mechanism is purely diffusional. The composite creep results should then follow the equation:¹⁰

$$\dot{\epsilon}_{PD} = \frac{14 \sigma \Omega D^{eff}}{k T d^2} \quad (5)$$

In Table 1, the low-stress regime of ANL15, ORNL20 and ANL30 are characterized by $n \approx 1$.

ANALYSIS OF CREEP-RATE REDUCTION

Theory

When GBS controls creep, there can be some contribution of PD to the strain rate. On the contrary, when GBS is impossible, PD controls alone. Consequently, creep of Al_2O_3 is governed mainly by Equation 3, whereas creep of composites with more than 15 vol% principally follows Equation 5. Those are extreme situations of GBS and PD, respectively. In samples with intermediate loading of whiskers, the creep equation is a combination of Equations 3 and 5, because the effective stress in GBS is reduced by σ_i , making both contributions to the strain rate comparable.

We define the grain-size-normalized creep-rate reduction (*creep reinforcement*), R , as:

$$R = \frac{d_m^p \dot{\epsilon}_m}{d_c^p \dot{\epsilon}_c} \quad (6)$$

with $p = 3$ in our study. The theoretical *creep reinforcement*, denoted by t , due only to the progressive change of creep mechanism, from Equations 3, 5 and 6:

$$R_t (V_w \approx 5\%) = \frac{d_m^3 (\dot{\epsilon}_{m(GBS)} + \dot{\epsilon}_{m(PD)})}{d_c^3 (\dot{\epsilon}_{c(GBS)} + \dot{\epsilon}_{c(PD)})} = \frac{1.14\sigma - \sigma_{t(m)}}{1.14\sigma - \sigma_{t(c)}} \quad (7)$$

for samples with low whisker content, and:

$$R_t (V_w \geq 15\%) = \frac{d_m^3 (\dot{\epsilon}_{m(GBS)} + \dot{\epsilon}_{m(PD)})}{d_c^3 \dot{\epsilon}_{c(PD)}} = 7(1.14 - \frac{\sigma_{t(m)}}{\sigma}) \quad (8)$$

for composites with higher whisker content. Figure 3 is a plot of Equations 7 and 8. From Equation 7, and for stresses over σ_i , R_t falls rapidly to values close to 1. In samples with more than 15 vol% of whiskers (Equation 8) the *creep reinforcement* grows to values close to 8 for stresses that are high enough such that the threshold stress of the matrix can be neglected.

Experiments

To determine experimentally the creep reinforcement factor R_e for ANL5, we have plotted normalized strain rates versus stress for the Al_2O_3 samples and for ANL5 (Figure 4). At high enough stress, in the range for which σ_i is negligible for both materials, creep rates fall on the extrapolation, resulting in $R \approx 1$, equal to theoretical calculations for $\sigma > 100$ MPa (Figure 3).

The R_e factors for ORNL20, ANL15, and ANL30 composites, calculated for $\sigma \approx 40$ MPa through Equation 6, from the data plotted in Figure 1, and the grain sizes in Table 1, are contained in Table 2.

Substantial differences from the theoretical factor $\cong 7$ are observed between R_e and R_t for $V_w \geq 15\%$. Additionally, the absolute creep rates of each of the three samples are very similar, despite differences in nominal grain size and whisker content.

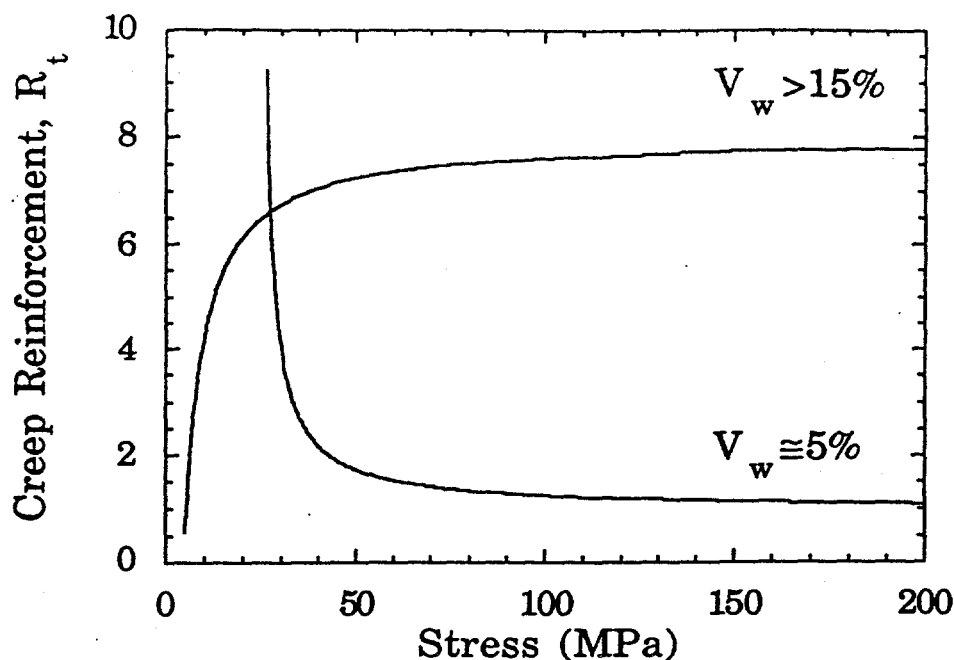


Figure 3. Theoretical *creep reinforcement* calculated through Equations 7 and 8

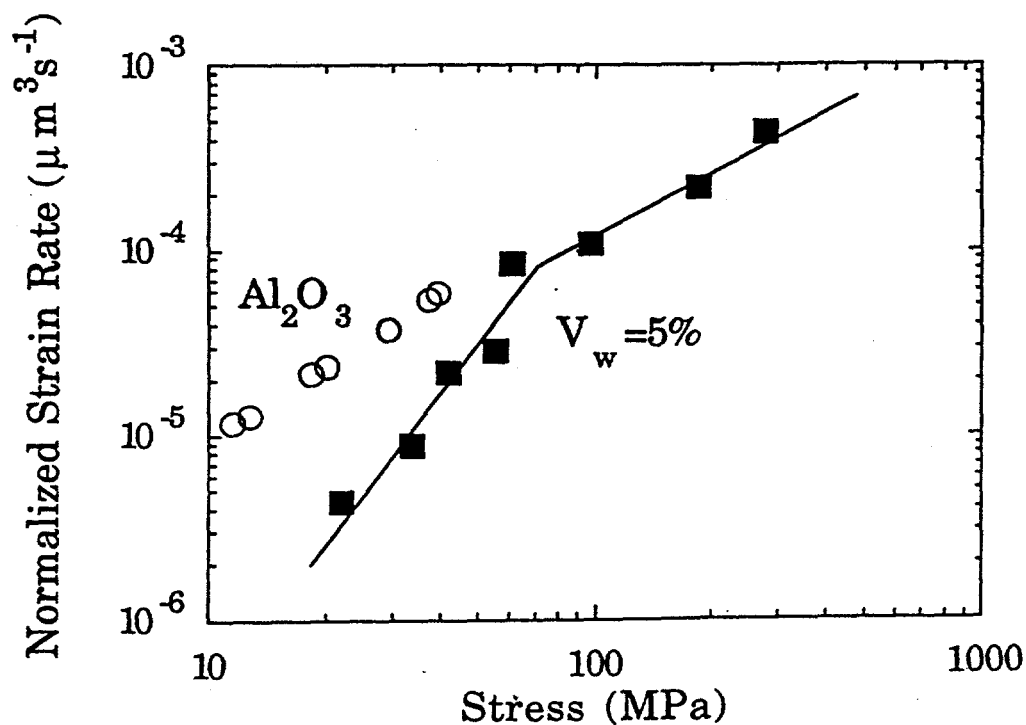


Figure 4. Normalized strain rate ($d^3 \dot{\epsilon}$) vs. stress plots of creep results of Al_2O_3 and 5 vol% whisker composite. The high stress region, for which σ_t can be neglected for both materials falls in a perfect extrapolation.

Table 2. Experimental Creep Reinforcement Factors R_e for Composites with $V_w \geq 15\%$

SAMPLE:	ANL15	ORNL20	ANL30
R_e :	10	60	140

EFFECTIVE GRAIN SIZE

At a fixed temperature, with constant diffusional kinetics, grain size is the only significant materials parameter. However, in light of the above discussion with regard to R and normalized $\dot{\epsilon}$, the nominal grain size is not the important materials parameter. We can define an effective grain size, d_{eff} ,

$$d_{eff} = d_c \left(\frac{R_e}{R_t} \right)^{1/3} \quad (9)$$

Measured effective grain sizes ($d_{meas.}$), calculated from Tables 1 and 2 are listed in Table 3.

Table 3. Effective Grain Size Values for Composites with $V_w \geq 15\%$

SAMPLE:	ANL15	ORNL20	ANL30
$d_{meas.}(\mu m)$	3.7	4.0	4.0

Several authors have claimed that when the amount of whiskers added to a matrix is large enough, the whiskers form a network that can control some mechanical properties.¹¹ Anelastic creep recovery behavior in an Al_2O_3/SiC composite has been explained using this concept.¹² One tentative explanation of the physical meaning of d_{eff} correlates it with the dimensions of the network of stiff whiskers inside of which deformation of the matrix takes place.

A two-dimensional model can be easily developed to estimate the section of the free space, d' , into the square cells of an homogeneous distribution of cylindrical reinforcements of radius r , in a concentration that is a partial volume fraction V_w . In Figure 5, the reinforcements are located at the corners of cells of size ℓ , that are smaller as whisker content increases. In this model, d' is the diameter of a circle with an area that is proportional to the space available between the whiskers:

$$\pi \left(\frac{d'}{2} \right)^2 = \lambda^2 (\ell^2 - \pi r^2) \quad (10)$$

Additionally, the partial volume fraction of whiskers is:

$$V_w = \frac{\pi r^2}{\ell^2} \quad (11)$$

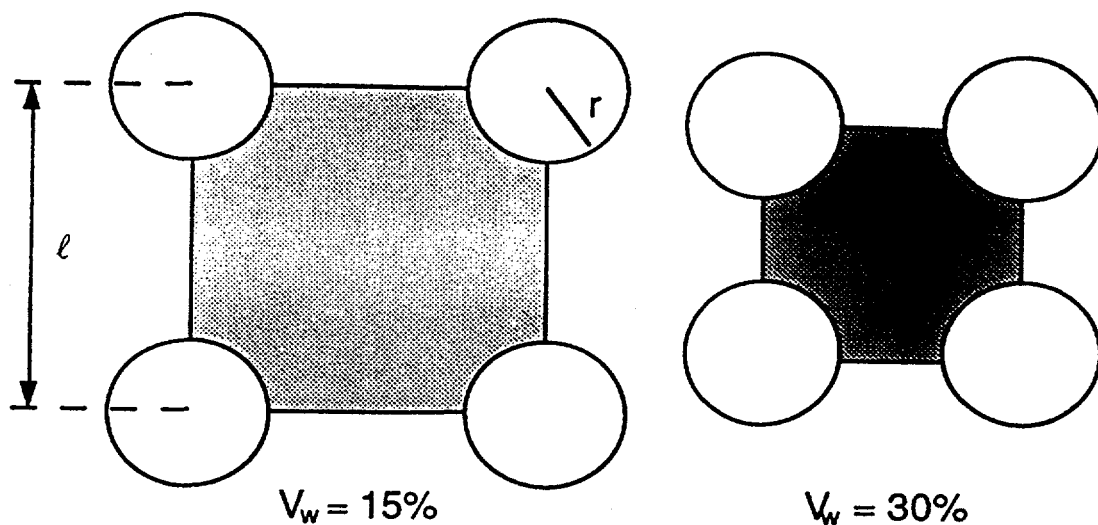


Figure 5. Distribution of different partial volumes of whiskers of radius r in cells of size ℓ , following the model proposed.

From Equations 10 and 11, the analytical form of d' is:

$$\frac{d'}{r} = 2\lambda \sqrt{\frac{1 - V_w}{V_w}} = \alpha \sqrt{\frac{V_m}{V_w}} \quad (12)$$

where V_m is the partial volume of the matrix. The function is represented in figure 6, for several values of the parameter α . If the equivalent circle area is equal to the $\ell^2 - \pi r^2$, then $\alpha = 2$. The values in Table 2 for d_{eff} of the composites are plotted in Figure 6 for different diameters of the whiskers.

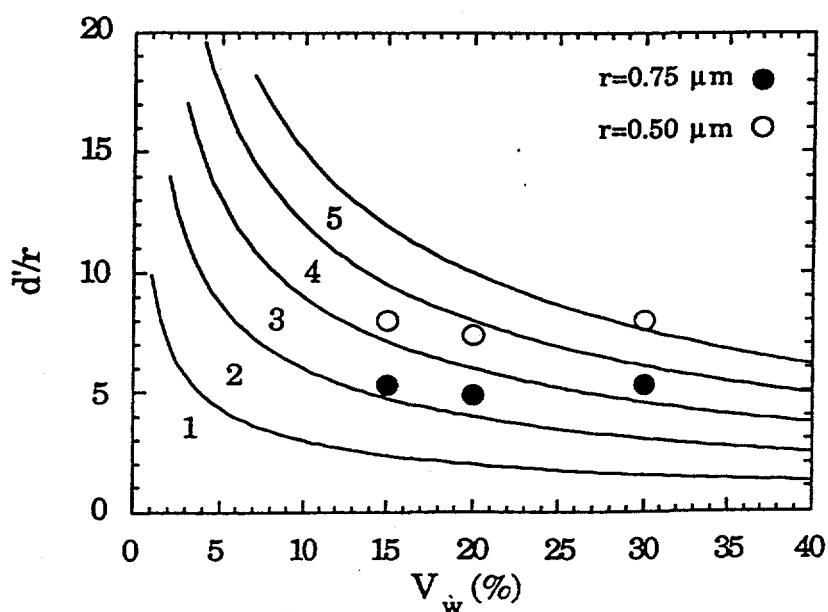


Figure 6. Plot of Equation 12, for different values of α (numbers). Circles represent d_{eff} values for ANL15, ORNL20 and ANL30, relative to whisker radius of 0.75 and 0.50 μm .

For ANL15, $\alpha \approx 2$ for $r = 0.75 \mu\text{m}$. For a given r , the value of α increases as the amount of whiskers increases. Such behavior can be a consequence of the difficulty of obtaining good dispersions when the whiskers loading is high. The whisker radius is an important parameter. As r decreases, α increases.

CONCLUSIONS

Addition of small amounts of stiff reinforcement (SiC whiskers) to a polycrystalline Al_2O_3 matrix partially inhibits grain boundary sliding because of an increase in threshold stress. When the concentration of whiskers is high enough, a pure diffusional mechanism takes over the control of plastic deformation of the composites. For higher whisker loadings, the materials creep properties depend on a microstructural feature different from the nominal grain size. A tentative correlation of this effective microstructural parameter with the spacing between the whiskers was established through a model.

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