WORK-OF-FRACTURE OF SiC-WHISKER REINFORCED Al_2O_3 COMPOSITE AT ELEVATED TEMPERATURES

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ABSTRACT

The work-of-fracture of a monolithic Al_2O_3 and a SiC whisker reinforced Al_2O_3 composite was evaluated from room temperature to 1270°C at which both the materials maintained elasticity from a macroscopic view point. The work-of-fracture of the composite increased with elevating temperature, while that of the Al_2O_3 showed only a slight increment in this range of temperature. The increment of the work-of-fracture was reversibly proportional to the 2nd power of ΔT which was temperature difference from 1350°C. This is the reason a compressive residual stress perpendicular to a whisker/matrix interface decreased with elevating temperature, and then the stress relaxation enhanced the toughening of the composite caused by the pulling-out of the SiC whiskers.

Keywords: whisker, composite, work-of-fracture, high temperature, stress relaxation

1 INTRODUCTION

Ceramic whiskers are applied to make structural ceramic composites, because the composites show high fracture toughness comparing with monolithic ceramics /1,2/. Hence, many researchers have investigated the fracture toughness including *R*-curve behavior and the work-of-fracture of the composite at room temperature /3-11/. In contrast, the fracture resistance of the composite at elevated temperatures was barely reported in spite of the importance of engineering application /11-14/. In the case of SiC whisker reinforced Al_2O_3 composites, a high residual stress occurs due to the mismatch of thermal expansion coefficients between the SiC whiskers and the Al_2O_3 matrix /15/. It was reported that a compressive residual stress perpendicular to the whisker/matrix interface of the SiC whisker/Al_2O_3 composite reached approximately 1GPa at room temperature considering the thermal expansion mismatch and the whisker geometry /16-19/. Thus, the increase of fracture resistance with elevating temperature is expected in the SiC whisker/Al_2O_3 composite, because;

(1) It is considered that the pulling-out of whiskers markedly contributed to the toughening of the composite /20/.

(2) Not all SiC whiskers are pulled-out in a process zone wake at room temperature, and some or most of them are broken at the onset of pulling-out by the high radial compressive stress /20/,

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(3) The high compressive residual stress is relaxed in proportion to the increase of temperature[16].

Although *R*-curve behavior at high temperatures begins to be studied recently /21/, it is experimentally difficult to measure the length of a crack at high temperature, which is indispensable for the accurate evaluation of the *R*-curve. Work-of-fracture can be evaluated with a chevron notched specimen even at elevated temperature without experimental difficulties. In this study, the work-of-fracture of a monolithic Al_2O_3 and the SiC whisker/Al_2O_3 composite was evaluated by the 3-point bending of a chevron notched specimen as a function of temperature. Comparing the increase of the work-of-fracture and the relaxation of the residual stress, the contribution of whisker pull-out to the toughening of the composite was discussed.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

The monolithic Al_2O_3 was fabricated by the hot-pressing of pure and fine Al_2O_3 powder (Taimicron TM-100, Taimei Chemicals Co. LTD.) at 1500°C under 33MPa for 1 hour in an Ar gas atmosphere. Two sorts of the SiC whisker reinforced Al_2O_3 composites were made by hot-pressing the Al_2O_3 powder mixed with two kinds of SiC whiskers (TWS100 and TWS400, Tokai Carbon Co.LTD.) at 1750°C under 33MPa for 1 hour in an Ar gas atmosphere. The average diameters of the SiC whiskers were 0.4μm for TWS100 and 1.1μm for TWS400, respectively. According to ESCA analysis, SiO_2 was hardly detected on the surface of the SiC whiskers. The whiskers were de-agglomerated by ultra-sonic vibration for 10 minutes and were homogeneously dispersed in the Al_2O_3 powder by tumbling mixing for 24 hours with n-butyl alcohol solvent. The volume fraction of the SiC whiskers in the composite was adjusted to be 20%. Bodies of the monolithic Al_2O_3 and the composite being dense enough were successfully obtained by this series of the fabrication processes.

2.2 Evaluation of work-of-fracture

The work-of-fracture of the Al_2O_3 and the composite was evaluated by 3-point bending (speed of cross-head motion is controlled to be 0.005mm/min, span is equal to 30mm) with a chevron notched specimen (4mm in width and 3mm in thickness) in which a crack propagated in a stable manner until its separation into two pieces /22/. It has been reported that whiskers lie vertically to the hot-pressing direction at two-dimensional random /2,23/. Then, the chevron notches with the tip angle of 90° were machined at the center of all the specimens (as shown in Fig.1) parallel to the

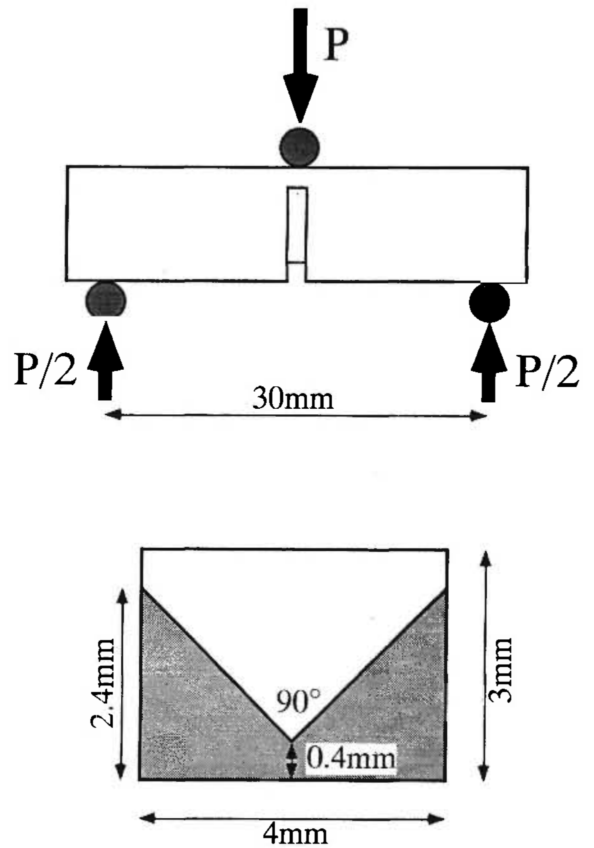


Fig. 1: Schematic illustrations of 3-point bending with a chevron notched specimen.

hot-pressing direction so that the crack could propagate perpendicularly to the plane with whisker orientation. The compliance of a testing system, consisting of a testing machine, a load cell, a bending apparatus, a push rod, etc. must be high to realize the stable fracture /22/. Thus, the bending apparatus, a setting apparatus, the push rod and a fixed rod were made of normal pressure sintered SiC (Hexoloy, Hitachi Chemicals Co. LTD.) as

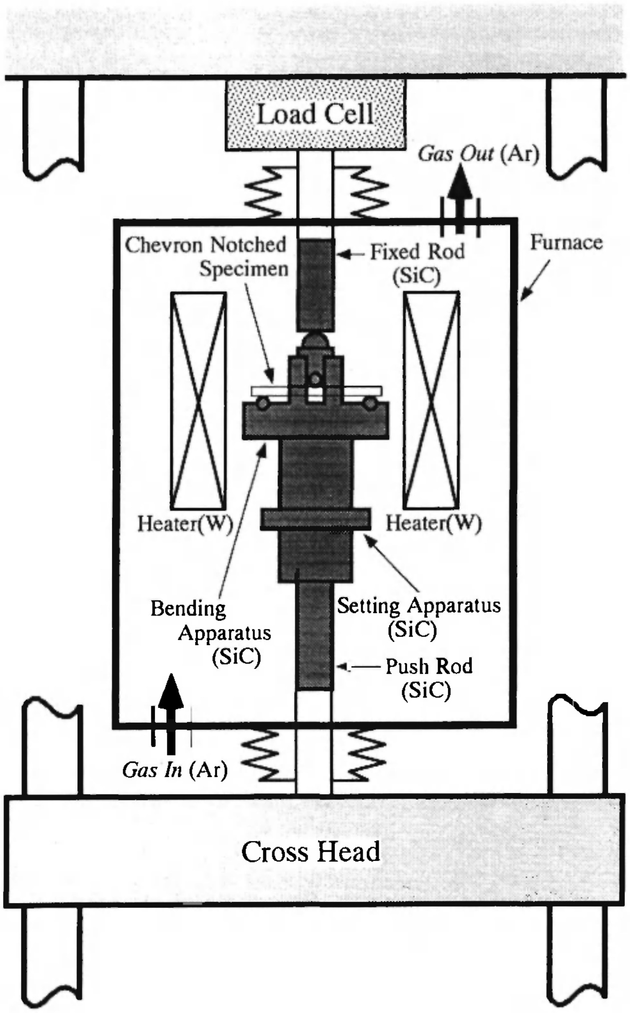


Fig. 2: Schematic illustration of a testing system which consists of testing machine, furnace, push rod, bending apparatus, etc.

shown in Fig.2, and then the compliance of the testing system became sufficiently high to be about 2.7×10^{-7} m/N at room temperature. The work-of-fracture was evaluated using the following equation /24/ from room temperature to about 1270°C in an Ar gas flow atmosphere,

$$\Gamma = \frac{U}{2A} \tag{1}$$

where Γ was the work-of-fracture, U was the total work done by the testing machine for the separation of the specimen in two pieces, and A was the projective cross section area of the unnotched ligament of the specimen. After the bending test, fractured surface of the Al_2O_3 and the composite was observed by SEM.

3. RESULTS AND DISCUSSION

The relationship between the load and the displacement of the chevron notched specimen during the 3-point bending test is shown for the monolithic Al_2O_3 and the SiC whisker reinforced Al_2O_3 composite in Figs.3 and 4, respectively. The load increased proportionally to the increase of the displacement until the crack began to propagate, and the slope in the diagram did not markedly change from room temperature to about 1270°C. From the macroscopic point of view, the effect of plastic deformation is negligible on irreversible energy consumption which is independent of crack propagation. Then, the linear fracture mechanics can be applied to analyze the work-of-fracture in this range of temperature. The work-of-fracture of the Al_2O_3 and the composite is shown as a function of temperature in Fig.5. The work-of-fracture of the composite increased with elevating temperature, while that of the Al_2O_3 showed

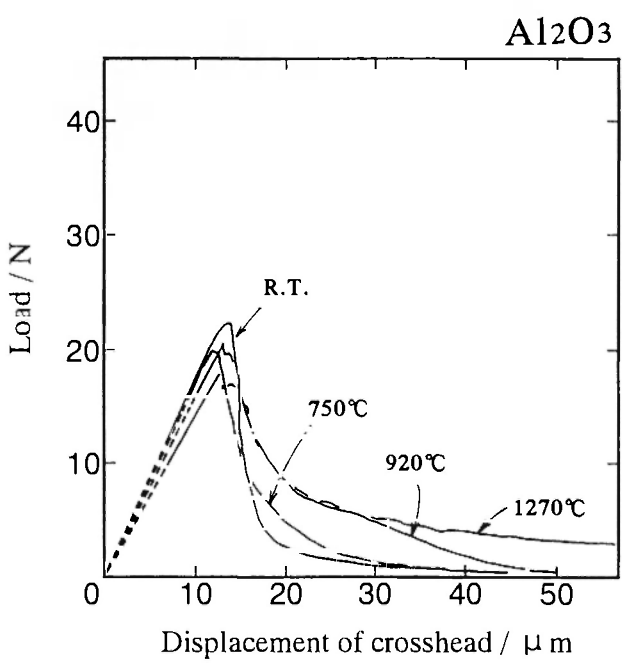


Fig: 3. Load vs. displacement diagram for the monolithic Al_2O_3 from room temperature to about 1270°C.

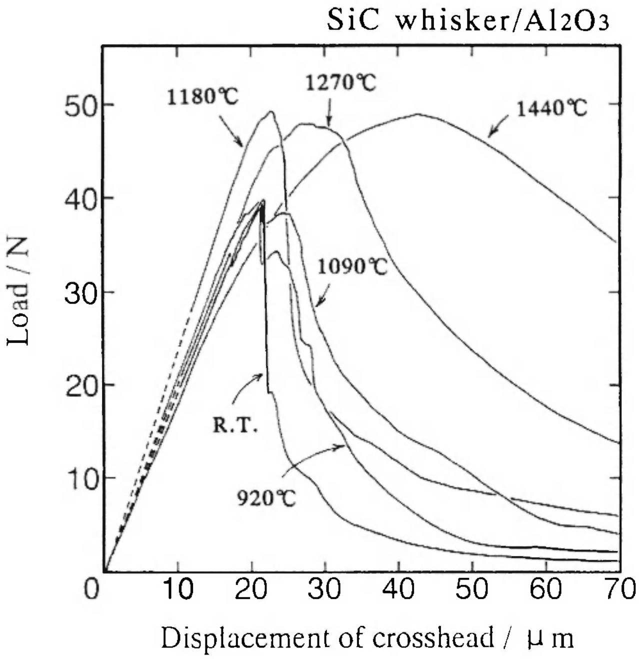


Fig. 4: Load vs. displacement diagram for the SiC whisker/Al_2O_3 composite from room temperature to about 1270°C.

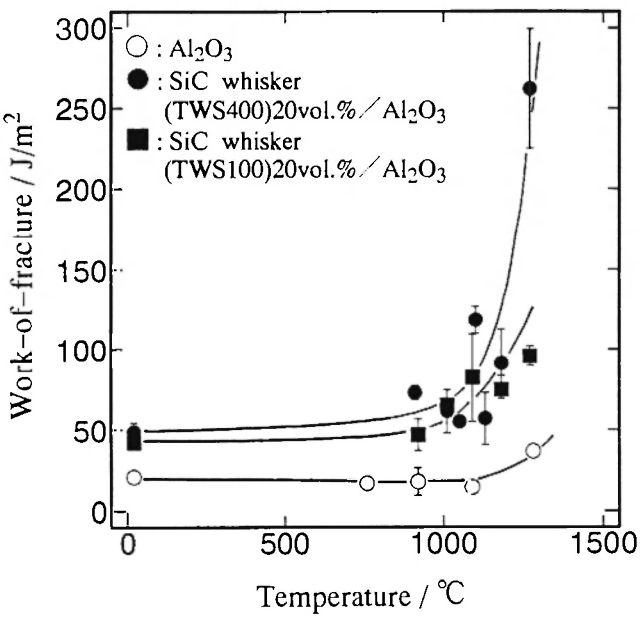


Fig. 5: Work-of-fracture of the monolithic Al_2O_3 and the SiC whisker/Al_2O_3 composites as a function of testing temperature.

only slight increase in this range of temperature. Moreover, the thick whiskers increased the work-of-fracture of the composite with elevating temperature more steeply than the thin whiskers. The calculation of a simple pull-out bridging model /20/ is applied to explain the experimental results. The increment of work-of-fracture ΔWOF caused by the pulling-out of whiskers is approximately expressed using the pull-out bridging model as follows,

when $\frac{l_w}{r_w} < \frac{\sigma_F}{\tau_i}$,

$$\text{then } \Delta WOF = \frac{1}{2^5} \psi V_f \tau_i \frac{l_w^2}{r_w} \tag{2}$$

when $\frac{l_w}{r_w} \geq \frac{\sigma_F}{\tau_i}$,

$$\text{then } \Delta WOF = \frac{1}{2^5} \psi V_f \frac{\sigma_F^3}{\tau_i^2} \frac{r_w^2}{l_w} \tag{3}$$

where l_w is the average length of the whiskers, r_w is the average radius of the whiskers, σ_F is the tensile strength of the whiskers and τ_i is the interfacial shear stress. ψ is the constant concerning the effective orientation of the whiskers for pulling-out, and ψ of approximately 0.3 - 0.4 should be adapted for the composite /25/. Considering the high compressive residual stress perpendicular to the whisker/matrix interface at room temperature /15/, the interfacial shear stress is considered to be high and the Eq.(3) should be adopted for the SiC whisker/Al_2O_3 composite in this range of temperature /20/. The interfacial shear stress seems to decrease with elevating temperature because of the relaxation of the compressive stress normal to the interface. Then, the interfacial shear stress is expressed like a frictional stress as follows, to simplify the calculation,

$$\tau_i = \mu \sigma_{rr} \tag{4}$$

where μ is the coefficient of friction at the interface and σ_{rr} is the compressive residual stress perpendicular to the interface. Using the neutron diffraction and theoretical consideration, Majumdar *et al.* /16/ confirmed that the compressive residual stress perpendicular to the interface in the SiC whisker/Al_2O_3 composite linearly decreased from about 1GPa at 0°C to

0Pa at 1350°C. Hence, the compressive stress normal to the interface is approximately expressed as follows,

$$\sigma_r = \frac{\Delta T}{1350} \times 10^9 \text{ Pa} \tag{5}$$

where ΔT is the difference between testing temperature and 1350°C. The following equation is derived by the combination of the Eqs.(3), (4) and (5),

$$\Delta WOF = 5.7\times10^{-14}\psi V_f \sigma_F{}^3 \frac{r_w{}^2}{l_w}\mu^{-2}\,\Delta T^{-2} \tag{6}$$

Therefore, the proportional relationship between ΔWOF and ΔT^{-2} is theoretically expected. The difference of work-of-fracture between the Al_2O_3 and the composite is plotted as a function of ΔT^{-2} as shown in Fig.6. The linear relationship in the ΔWOF vs. ΔT^{-2} diagram is observed with rough approximation and gave suitability of the model calculation As shown in Fig.7, fractured surface of the Al_2O_3 and the composite was observed by SEM. The pulled-out whiskers from the fractured surface were frequently found on the cross section of the composite fractured at high temperature. It is considered that the number of the whiskers which contribute to the pulling-out from a crack face increases by the relaxation of the compressive residual stress perpendicular to the interface, and the pulling-out of the whiskers increases excess irreversible energy loss during crack propagation. This is the reason the work-of-fracture of the composite typically increased with elevating temperature. Considering the Eq.(6), it is theoretically considered that the slope of the linear relationship between ΔWOF and ΔT^2 is proportional to $\frac{r_w^2}{l_w}$. Thus, the work-of-fracture of the composite with the thick whiskers increased more steeply with elevating temperature than that of the composite with the thin whiskers. The ratio of the slopes between two kinds of the composite is theoretically estimated to be about 1:5 using Eq.(6) with the following data: thin whiskers (TWS100) were 0.20μm in radius and 30μm in length, and thick whiskers (TWS400) were 0.55μm in radius and 45μm in length. However, the ratio of the slopes in the ΔWOF vs. ΔT^{-2} diagram was indeed observed to be approximately 1:9. This variance in the slope ratio between the theoretically expected value and the experimentally obtained value is caused by the assumption involved in the model calculation, in which the differences of the tensile strength and the coefficient of interfacial friction in the two kinds of whiskers are not considered. Concerning the tensile strength of whiskers, it is natural that thin whiskers are stronger than thick whiskers due to the decrease of effective volume. Thus, it is unreasonable to explain the variance in the ratio with the differences of the tensile strength in the two kinds of whiskers. On the other hand, by SEM observation, it is found that the surface of the thin whisker is rougher than that of the thick whiskers as shown in Fig.8. Thereby, the slope of the composite with thin whiskers in the ΔWOF vs. ΔT^{-2} diagram seems to be lower beyond expectation because of a high coefficient of friction at the rough interface. Then, the variance in the ratio was reasonably explained with the differences of the coefficient of interfacial friction in the two kinds of the composite.

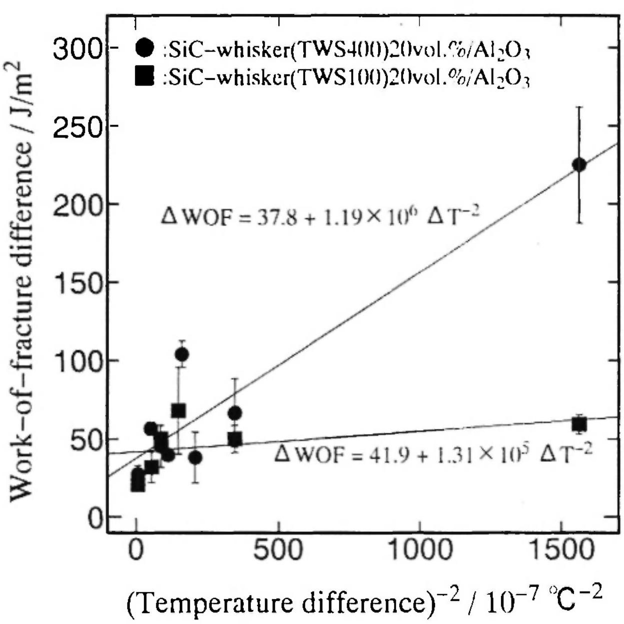


Fig. 6: Work-of-fracture difference between the monolithic Al_2O_3 and the SiC whisker/Al_2O_3 composites as a function of the -2nd power of ΔT which is temperature difference from 1350°C.

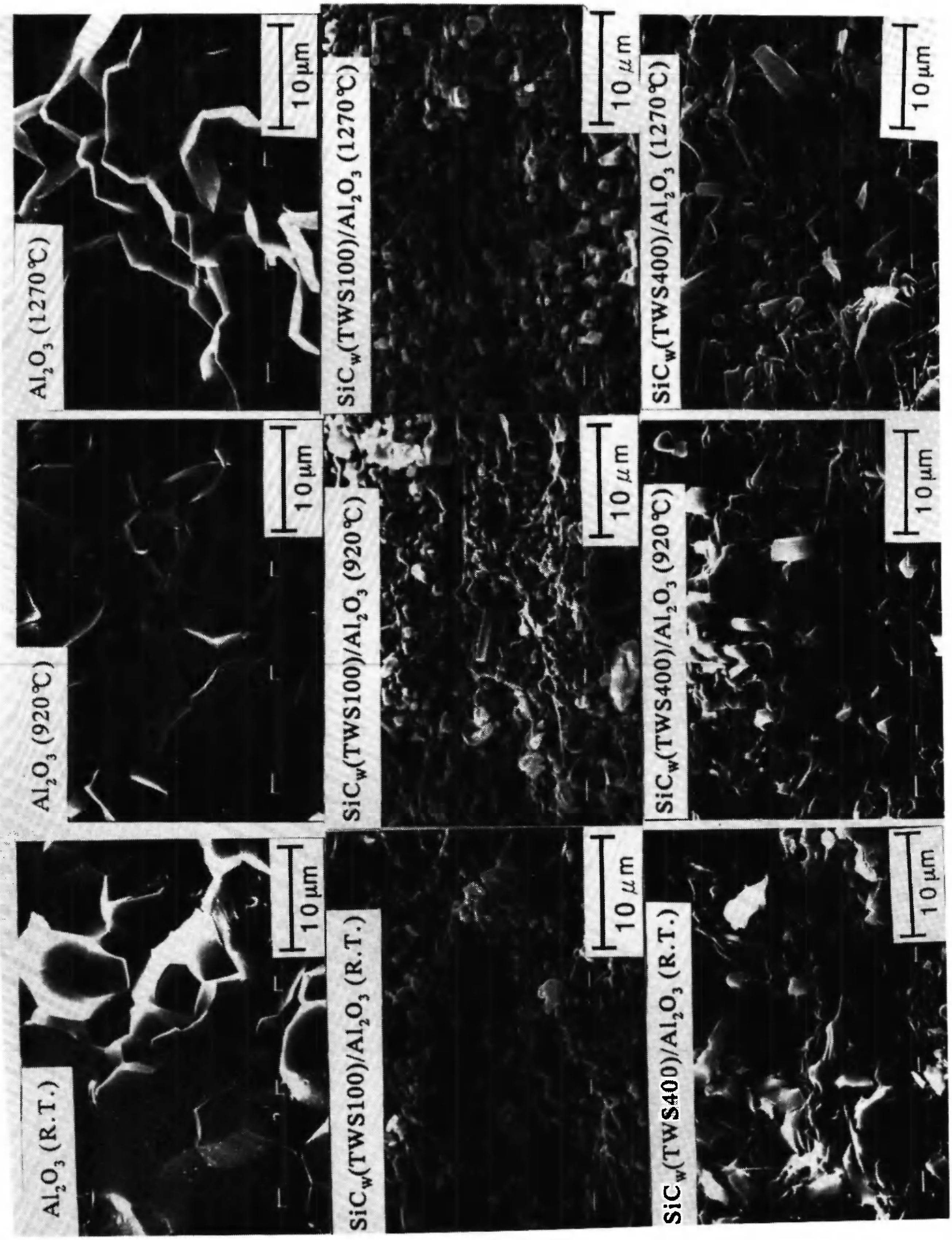
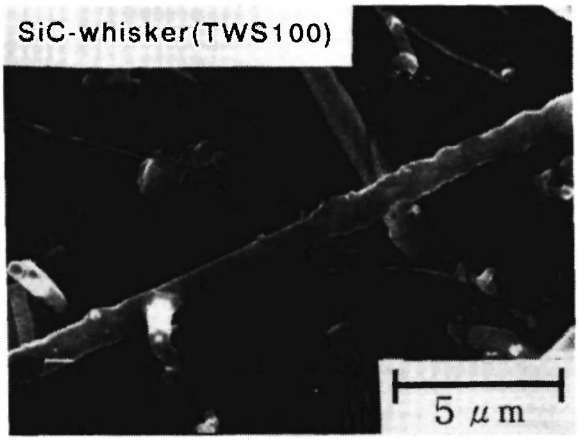


Fig. 7: SEM micrographs of fractured surfaces of the monolithic Al_2O_3 and the SiC whisker/Al_2O_3 composites.



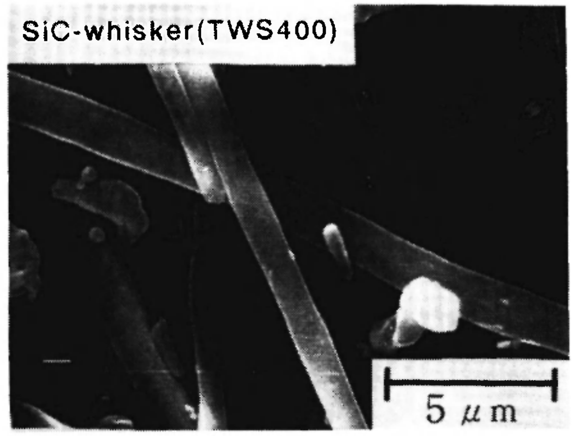


Fig. 8: SEM micrographs of SiC whiskers.

4 CONCLUSIONS

The work-of-fracture of the monolithic Al_2O_3 and the SiC whisker reinforced Al_2O_3 composite was evaluated from room temperature to 1270°C. In this range of temperature, both the materials maintained elasticity from a macroscopic viewpoint. The work- of-fracture of the composite increased with elevating temperature, while that of the Al_2O_3 showed only slight increase in this range of temperature. The increment of the work-of-fracture was reversibly proportional to the 2nd power of ΔT which was temperature difference from 1350°C. This is the reason a compressive residual stress perpendicular to a whisker/matrix interface decreased with elevating temperature, and then the stress relaxation enhanced the toughening of the composite caused by the pulling-out of the SiC whiskers. Comparing the work-of-fracture between two kinds of composite, whisker geometry and roughness at the whisker/matrix interface affect toughening at high temperature. It was considered that the increment of the work-of-fracture of the SiC whisker/Al_2O_3 composite from room temperature to 1270°C increased with decreasing the coefficient of interfacial friction.

ACKNOWLEDGMENTS

The authors thank Mr. M. Imai of the Research Laboratory for Nuclear Reactors, Tokyo Institute of Technology for his experimental help with the evaluation of work-of-fracture. This research work was supported by the Ministry of Education, Science, Sports and Culture of Japan under a Grant-in-Aid for Encouragement of Young Scientists (No.07855078), Scientific Research (A) (No.08555158) and COE Research entitled "Study on Drastic Toughening Improvement of High Performance Brittle Materials".

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