Residual Stresses on Particle-Reinforced Ceramic Matrix Composites Using Synchrotron Radiation

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Introduction

The addition of second-phase particles into ceramic matrix has been used to toughen silicon nitride (Si_3N_4) ceramics. Residual stresses can be induced in particle-reinforced ceramic matrix composites (CMCs) by a significant mismatch in the coefficient of thermal expansion between the matrix and the particles during processing.[1] It is known that the magnitude and distribution of residual stress have an important influence on the mechanical properties of composites. Therefore, in order to select reliable materials and components with considerably improved material properties and performance under severe operating conditions, it is necessary to know where the benefits of residual stresses may occur and control them successfully.

Materials and Methods

A Si_3N_4 baseline composition, containing 3 wt% Al_2O_3 and 9 wt% Y_2O_3, and Si_3N_4-TiN composites, containing 5 wt% TiN as a particulate addition, were investigated. The baseline Si_3N_4 and Si_3N_4-TiN composites were processed by turbomilling, pressure casting, and isopressing. They were then continuously sintered to full density, under a pressureless, flowing nitrogen atmosphere. The samples, machined into rectangular bars of 3 × 4 × 45 mm^3, were subjected to quenching in room-temperature water from 1000ºC, 1100ºC, and 1200ºC. Flexural strength was measured by a four-point bending test with the inner and outer spans of 20 mm and 40 mm, respectively. Fracture toughness (K_{IC}) was determined by a modified indentation technique with a 5-kg load using Vickers indentation.[2]

To measure the triaxial residual stresses in the ceramic samples with the 2θ-$\sin^2\psi$ method, synchrotron x-ray diffraction in the angle dispersive configuration (ADXD) was performed at the 12-BM beamline of the Basic Energy Sciences Synchrotron Radiation Center (BESSRC) at the Advanced Photon Source (APS). The x-ray beam produced by a bending magnet was monochromated to the energy of 8.856 keV, using a Si(111) crystal and was collimated to the cross-section of 1 × 2 mm^2. The diffractometer was step-scanned over the 2θ range 101º to 105º with a 0.01º 2θ-step interval at each different tilt angle. The diffraction peaks of the Si_3N_4-TiN composite for the (441) and (531) planes were collected using a scintillation counter with a Ge(111) crystal analyzer.

Results

Figure 1 shows the diffraction peak positions for the (441) reflection from the Si_3N_4-TiN composites as a function of $\sin^2\psi$. The thermally shocked sample from 1000ºC exhibited the largest positive slope, which indicated the development of compressive residual stress, due to the thermal treatment. In all cases, the variation of 2θ with $\sin^2\psi$ could be considered to be linear, within experimental error, with no ψ splitting.

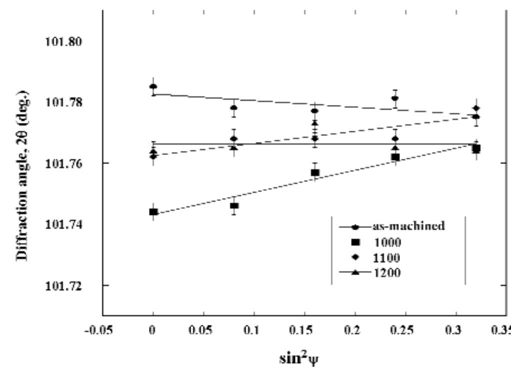

FIG. 1. Relationship between the centroid peak positions, corresponding to the Si_3N_4-TiN (441) plane, and different tilted angles. Lines represent the linear curve fits.

By using the diffraction angle, $2\theta_0$, measured from the powdered sample and the values of slope and intercept obtained from Figure 1, both the transverse (σ_{11}) and longitudinal (σ_{33}) residual stresses for the near-surface region were determined from the following equation:[3]

$$2\theta = 2\theta_0 - 2\tan\theta_0\left(\frac{1+\nu}{E}\right)(\sigma_{11} - \sigma_{33})\sin^2\psi + 2\tan\theta_0\frac{\nu}{E}\left(2\sigma_{11} - \frac{\sigma_{33}}{\nu}\right)$$

The mechanical values of Poisson's ratio ν, and Young's modulus, E, used in these calculations were 0.27 and 313.22 GPa, respectively.[4] The calculated average values of σ_{11} and σ_{33}, obtained from Si_3N_4-TiN (441) and (531) planes, are summarized in Table I.

Table I. Average values of residual stress in the Si_3N_4-TiN composite

Thermal Shocking Temperature (ºC)	$\langle\sigma_{11}\rangle$ (MPa)	$\langle\sigma_{33}\rangle$ (MPa)
As-machined	-35.73	-59.69
1000	-232.65	-75.21
1100	-141.54	-74.49
1200	-100.59	-54.65

The residual stresses on Si_3N_4-TiN composite, induced by thermal shocking, were found to be compression in both directions. The residual stresses in the direction parallel to the surface were much higher than in the perpendicular direction. With increasing thermal shocking temperatures, the residual compressive stresses in both directions were found to decrease.

Discussion

The measurements of residual stress using x-ray diffraction methods have been mostly conducted without considering the presence of σ_{33} according to the mechanical equilibrium conditions in material. However, it has been shown by Ruppersberg and Tanaka that assuming σ_{33} to be zero in the bulk of the materials leads to inconsistent results in the calculation of the stress on the surface of material.[5,6] If the penetration depth of the x-ray is larger or at least comparable to the grain size and if the materials is polyphase, as in the Si_3N_4-TiN composites, the finite values of σ_{33} do exist, along with the stresses in the direction parallel to the surface (σ_{11}). The use of Eq. (1) has made it possible to analyze the residual stress in the particle-reinforced ceramic composites.

The results from Table 1 show the critical shocking temperature would be about 1000ºC. This would be the temperature expected to develop the most surface compression in the Si_3N_4-TiN composites. This developed compressive residual stress could play an important role in preventing the initiation and propagation of cracks, resulting in the improvement of both flexural strength and fracture toughness. Therefore, the highest strength and toughness values are expected in the sample that was thermally shocked from 1000ºC. The effect of thermal shocking temperatures on the mechanical properties of the materials was consistent with the residual stress results obtained from synchrotron x-ray diffraction. The compressive residual stress of the Si_3N_4-TiN composites was much higher than that of the baseline Si_3N_4. As a consequence of these high compressive residual stresses, both the fracture toughness and flexural strength of the Si_3N_4-TiN composites were improved.

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