THERMAL RESIDUAL STRESS IN Al_2O_3/SiC_{nano} CERAMIC COMPOSITES MEASURED BY NANOINDENTER*

Zhong LING

LNM, Institute of Mechanics, Chinese Academy of Sciences, Beijing 100080, P.R China

Summary Thermal residual stresses in Al_2O_3 ceramics containing SiC_{nano} particles were measured by nanoindentation instrument. Pure Al_2O_3 ceramics was selected as a reference without thermal residual stress. Considered the contact stiffness would vary due to local heterogeneities of microstructure in current composites, data on the surface sensitivity of a series of samples probed at different peak depths were collected. The experimental results on the residual stress of two composites were obtained in local region probed by indenter tip. Also the influence of volume fraction of SiC particles to magnitude of thermal residual stress and the external work and deformation energy during indentation under a fixed applied load, 250mN, were investigated.

Key words: nano-ceramic composite, thermal residual stress, nanoindentaion experiment

INTRODUCTION

Nano-ceramic matrix composites are ceramics containing nanosized-ceramic particles of 1-5% volume fractions. The nanosize-particles dispersed in the grains of matrix would lead to a change in the fracture modes, from predominantly inter-granular to predominantly trans-granular fractures. This would also lead to enhanced fracture toughness of the composite. Among the toughening mechanisms of such particulate ceramic composites, a primary one is residual stress (strain) in the matrix grains due to a mismatch between the coefficients of thermal expansion (CTEs) of the ceramic matrix and nano-sized particles. Obviously, the estimation of thermal residual stress is an important issue in these nano- ceramic composites.

In the past decades, a number of experimental work had been reported on the averaged residual stresses in matrix composites determined by X-ray and neutron diffraction [1]. More recently, however, with the rapid development of nano-instruments, the nanoindentation technique has been applied to probe quantitatively the residual stresses at a very tiny volume [2,3].

Motivated by the above research and the need to better understand how the thermal residual stress quantitatively distributed in the ceramic composites, we preformed nanoindentation experiments for typical nano-ceramic composites, Al_2O_3/SiC_{nano}. The thermal residual stress developed in the matrix of the composites was also probed.

MATERIALS AND EXPERIMENTAL RESULTS

Materials and experiments

The testing materials for the present study were sintered aluminum and its composites containing SiC particles (Al_2O_3/SiC). The volume fractions of SiC particles were 5% and 10%. The SiC particle size was ~60-100 nm (d_{SiC} = 60-100 nm). Because the SiC particles existed as a second phase in the current composite, the microstructure of the composite have involved a number of scales over the entire indentation process. The distance between two nearest SiC particles, δ_{SiC}, is an intrinsic scale of particulate reinforced composite, which is related with particle size and its volume fraction. Microstructure scales in current materials were list in Table I. Nanoindentation tests were carried out under fixed applied indentation loads, 3-250mN, with a constant load rate over load, $\dot{F}/F = 2$. The typical loading curves of current materials are presented in Figure 1.

Table I. Microstructure coefficients in current materials

Al_2O_3/SiC_p	Ratio	Size	δ_{SiC}
Al_2O_3	balance	~5μm	—
Al_2O_3/SiC_p	f_v=0.05	60-100nm	145-242nm
Al_2O_3/SiC_p	f_v=0.10	60-100nm	88-147nm

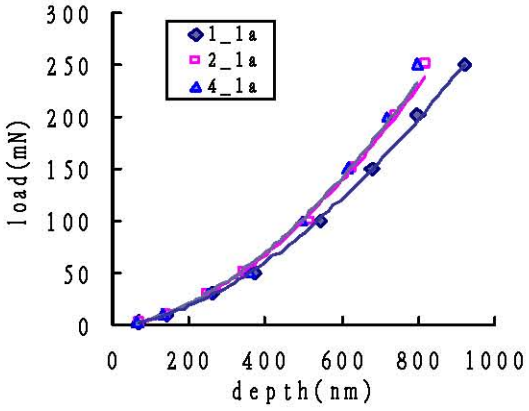

Figure 1. Loading-depth curves for three materials, 1-1:Al_2O_3; 2-1: Al_2O_3/SiC(5%); 4-1: Al_2O_3/SiC(10%)

Experimental results and analysis

Usually, the recorded indentation curves could actually be evaluated with respect to their curvatures and preliminarily pooled as apparently 捞oft" and 揾arder" locations on the microstructure of the contact surface. Also the contact stiffness, S, would vary due to local heterogeneities of the material. Considering the matrix and SiC particles in the current composites, data on the contact stiffness of a series of samples probed at different peak depths were collected and analyzed. Figure 2 showed relations between contact stiffness and depth for three test materials. At

depth less 200nm, contact stiffness of three materials does not change significantly (Figure 2). Notice the distance between two nearest SiC particles, δ_{SiC}, not over 200nm for Al_2O_3/SiC (10%), and δ_{SiC}~150-240nm for Al_2O_3/SiC (5%). So present samples were almost all initially local to aluminum phase between two nearest SiC particles of the current materials. In the other word, information on thermal residual stress in matrix of current composites could be probed by indenter tip at depth<200nm.

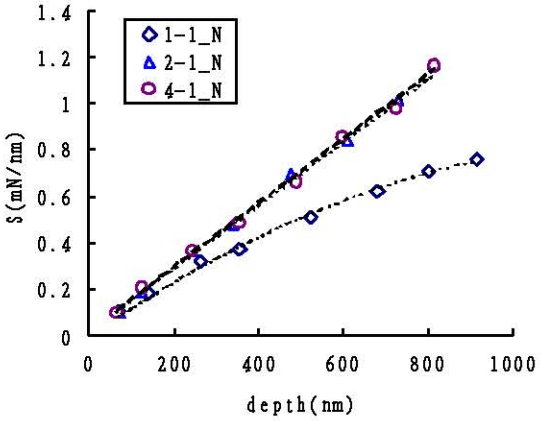

Figure 2 Contact stiffness obtained at different peak load of current materials:
◇Al_2O_3,△- Al_2O_3/SiC(5%),○- Al_2O_3/SiC(10%)

According to the method described in [3], the residual stress state in the local region could be estimated in the loading curves. As a reference material, Al_2O_3 should be in a non-residual stress state compared with both composites during loading. For another two composites, compressive residual stress seems to have been probed. In Figure 1, at fixed applied indentation loads, 3-250mN, indentation depths of two composites were smaller than that of Al_2O_3. This should be a contribution of compressive residual stress in matrix of two composites. The compressive residual stress would make the effective indentation loads smaller and consequently arise apparently the indentation stiffness. Obviously, magnitudes of the residual stress varied slightly with volume fractions of SiC particles in the composites.

Additionally, external work done by the load, elastic (recoverable) and plastic (permanent) deformation energy during indentation were investigated. Let $\eta_e=W_e/W_t$ and $\eta_p=W_p/W_t$ as normalized elastic and plastic deformation energy, respectively. Where W_t is external work, W_e elastic deformation energy and W_p plastic deformation energy. Experimental results presented that, under maximum peak load, 250mN, $\eta_{e1}=W_{e1}/W_{t1}$, normalized elastic deformation energy of Al_2O_3 material is greater than that of both composites. And the normalized elastic deformation energy of Al_2O_3/SiC(5%) is greater than that of Al_2O_3/SiC(10%). That is, $\eta_{e1}>\eta_{e2}=W_{e2}/W_{t2}>\eta_{e4}=W_{e4}/W_{t4}$. The normalized plastic deformation energy of these materials, however is, another way around, $\eta_{p1}<\eta_{p2}<\eta_{p4}$. As stated above, compressive residual stress developed by SiC particles in composites raised apparently the indentation stiffness therefore the percent of recoverable deformation energy in total work would be lower under a fixed applied load. As for the permanent deformation energy of these materials, it is probably related to other factors, such as plastic deformation in the matrix, surface roughness and interface between particles and the matrix as well.

CONCLUSSIONS

Considering microstructure scales in current composites, data on the contact stiffness of a series of samples probed at different peak depths were collected. At indentation depth less 200nm, recorded contact stiffness of three materials did not change significantly. So present samples were almost all initially local to aluminum phase between two nearest SiC particles of the current materials.

Compressive residual stresses in two composites were obtained from loading-depth curves referenced to that of matrix material, Al_2O_3; The compressive residual stress would make the effective indentation loads smaller and consequently arise apparently the indentation stiffness and their magnitudes depended on volume fractions of SiC particles of the composite;

Under the fixed applied indentation load, 250mN, the normalized elastic deformation energy, η_e, of three materials followed as $\eta_{e1}>\eta_{e2}>\eta_{e4}$; but normalized plastic deformation energy of these materials followed as $\eta_{p1}<\eta_{p2}<\eta_{p4}$.

References

[1] I. Levin, et al, Restidual Stresses in Alumina-SiC Nanocomposites, *Acta Metall. Mater*. **42:**1147-1154, 1993

[2] W.C. Oliver and G.M.Pharr, An Improved Technique for Determining Hardness and Elastic Modulus using Load and Displacement Sensing Indentation, *J.Mater. Res*. **7:**1564-1583, 1992

[3] S.Suresh and A.E. Giannakopoulos, A New Method for Estimating Residual Stress by Instrumented Sharp Indentation, *Acta. Mater*. **46:**5755-5767, 1998

*This work is financially supported by National Natural Science Foundation of China (Grant No.50172053) and the Chinese Academy of Sciences (Grant No. KJCX-SW-L2)