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A nanoindentation analysis of the effects of microstructure on elastic properties of Al_2O_3/SiC composites

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Abstract

Nanoindentation tests were carried out to investigate certain elastic properties of Al_2O_3/SiC_p composites at microscopic scales (nm up to μm) and under ultra-low loads from 3 mN to 250 mN, with special attention paid to effects caused by SiC particles and pores. The measured Young's modulus depends on the volume fraction of SiC particles and on the composite porosity and it can compare with that of alumina. The Young's modulus exhibits large scatters at small penetrations, but it tends to be constant with lesser dispersion as the indentation depth increases. Further analysis indicated that the scatter results from specific microstructural heterogeneities. The measured Young's moduli are in agreement with predictions, provided the actual role of the microstructure is taken into account.

Keywords: A. Ceramic–matrix composites (CMCs); B. Microstructures; C. Elastic properties; Nanoindentation test

1. Introduction

The nanoindentation technique has been developed over the last few decades as an effective tool for probing mechanical properties of materials at very small scales (nm–μm). Its principal advantage is to continuously monitor with high precision and accuracy both the load (P) and displacement (h) of an indenter during loading and unloading in the micro-Newton and nanometer ranges [1–3]. Based on the analysis of the loading–unloading data derived from indentation tests, elastic moduli (E) and hardnesses (H) can be determined. Nanoindentation has been applied to a variety of solids including metals, ceramics, almost all homogenous and monolithic [1–7]. Taking advantage of the sensitivity of the indenter tip to microstructural features at the submicron-nanometer scale, nanoindentation investigations on heterogeneous materials or multiphase composites have also been reported [8–13]. The indenter tip can accurately probe very small volumes, even in anisotropic materials or multiphase composites. For instances, Fan et al. [9] could quantitatively evaluate the local anisotropy of human bones. Ling [10] applied this technique to probing the elastic properties of different phases of a ceramic composite, with the tip indenting the different phases selectively. A limitation takes place though when heterogeneities scale with the size of the nanoindent. This was illustrated by Hu and Lawn [11] who studied the indentation stress–strain behaviour of bilayer composites, in which case the microstructure scales with the layer thickness. In the same vein, Jung et al. [12] evaluated the mechanical properties of composite bilayers by nanoindentation. In polycrystalline Al_2O_3, Gong et al. [13] remarked that when the maximum indentation depth compares with the grain size of the tested material, the mechanical properties probed by nanoindentation reflect local rather than bulk properties. In this context, it is essential to understand further how the nanoindentation can help probing the effects of heterogeneities on the mechanical properties of multiphase materials.

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In this work, alumina and multiphase ceramic composites, Al_2O_3/SiC, were studied. In the composites, SiC particles are embedded within alumina grains engendering heterogeneous microstructures at the nm-scale and up to the μm-scale, which makes the composite fully appropriate to pursue the above-mentioned goals. The microstructure heterogeneity is at the origin of the fracture mode transforming from predominantly intergranular in alumina to predominantly transgranular in the composite [14,15].

2. Experimental procedure

2.1. Materials and test conditions

Alumina and its composites, α-Al_2O_3/SiC with 5 and 10% volume fraction of SiC particles used in this study were provided by the Institute of Ceramics, Chinese Academy of Sciences. A solid alumina sample was fabricated from as-received powders by sintering at 1550 °C for 30 min under 30 MPa. For the composites, the SiC powder with a mean particle size of about 60–100 nm, was first dispersed and then mixed with Al_2O_3 powder. The mixture was dried, sieved, calcined, and then sintered at 1700 °C for 30 min at 30 MPa. The density was determined from separate measurement of the mass and volume of each disc. For each sample, the morphology of the microstructure was characterized on freshly fractured surfaces in a high-resolution scanning electron microscope (SEM Sirion400NC).

Fig. 1 presents the fracture surfaces of both alumina and its composite containing 5 vol.% SiC particles. The size of the alumina grains is about 2 μm in the single-phase alumina samples (Fig. 1a) and about 5 μm in the composite (Fig. 1b). Both grain sizes being larger than the depths of nanoindentation, it is reasonable to assume that the elastic properties probed away from grain boundaries and SiC particles should scale with those of single crystal alumina (sapphire). The mean size of SiC particles in the composites is about 100 nm, close to the starting sizes of the powders. It can also be noticed from Fig. 1 that the fracture mode is almost intergranular for alumina (Fig. 1a) and dominantly transgranular for the composite, a difference which can therefore be ascribed to the SiC particles (Fig. 1b). The specimen size was $15 \times 3 \times 3$ mm with flat-machined parallel surfaces. Each test surface was finely diamond-polished to scratch-free mirror-like, suitable for indentation tests.

The mean distance between two nearest-neighbouring particles, δ_{SiC}, can be expressed as [16]

$$\delta_{SiC} = \frac{d_s}{2}\left(\sqrt{\frac{2\pi}{3f_v}} - \sqrt{\frac{8}{3}}\right) \quad (1)$$

where, d_s is the particle size and f_v is the volume fraction of the particles. δ_{SiC} is about 240 nm for Al_2O_3/SiC_p(5%) and 150 nm for Al_2O_3/SiC_p(10%). SiC particles distributed along the alumina grain boundaries are not taken into account. Before nanoindentation, the surface roughness, R_a, amounted to 8.4, 15.9 and 9.4 nm for Al_2O_3, Al_2O_3/SiC_p(5%) and Al_2O_3/SiC_p(10%), respectively. Except for shallow indentation depth, surface roughness is, however, not thought to have influenced the present results since most of the various length scales characterizing the microstructure together with the depth of nanoindentation were large enough compared to the roughness of each sample. Table 1 gives the main properties and the typical length scales of the tested materials. The reference Young's moduli of alumina Al_2O_3 and SiC were taken from the NIST database [17]. The densities of both tested composites are lower than that of alumina (Table 1), revealing the presence of pores.

The tests were performed at room temperature using a nanoindentation device (CSEM Instrument) equipped with a Berkovich tip. The device was fully calibrated with the Oliver–Pharr (O–P) method [2]. Considering the heterogeneity of the tested materials, smaller increments were used at lower load levels. The peak load levels were preset at 3, 5, 10, 15, 20, 25, 30 mN and then from 50 to 250 mN with an increment of 50 mN. The maximum peak load was less than the fracture threshold of alumina [8,18]. In each test,

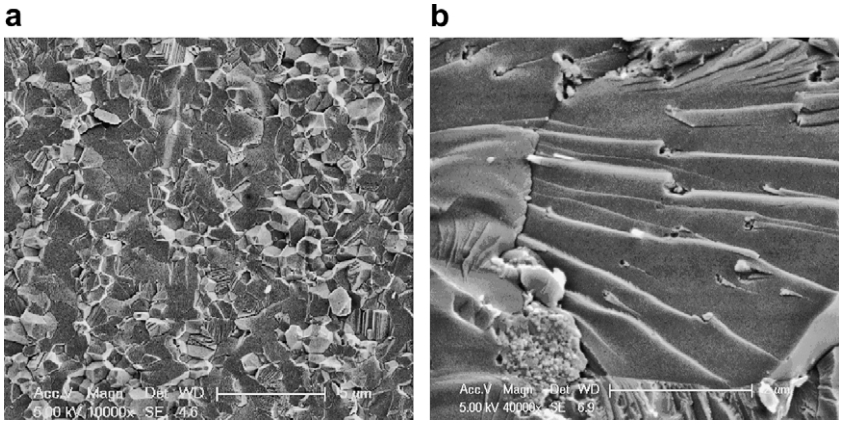

Fig. 1. Micrographs of the tested materials: (a) intergranular fracture in the pure alumina and (b) transgranular fracture in composite matrix grains, α-Al_2O_3/SiC(5%), where small white SiC particles dispersed in the alumina grains.