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Interfacial strength and deformation mechanism of SiC–Al composite micro-pillars

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ABSTRACT

A comprehensive understanding of the interfacial properties in particle-reinforced metal matrix composites (PRMMCs) requires an accurate determination of the cohesive strength of the interface. In this study, micro-pillars containing a slanted SiC/Al interface were fabricated, and were tested by uniaxial compression. The interfacial shear strength was found to be 133 ± 26 MPa, consistent with values predicted by numerical simulations. The stress–strain response of the composite pillars was characterized by shorter strain bursts and more significant strain hardening, as compared with their monolithic Al counterparts. These observations were interpreted by grain fragmentation and dislocation pile-up at the SiC/Al interface upon deformation.

Particle-reinforced metal matrix composites (PRMMCs) are ideally suited for many structural and functional applications because of their high specific strength and stiffness, isotropic properties and relatively simple processing as compared with monolithic materials and conventional fiber-reinforced composites [1]. Among the parameters that may affect the mechanical performance of these composites, the structure and properties of the particle matrix interfaces play a critical role. A comprehensive understanding of the interfacial properties requires an accurate determination of the cohesive strength of the interface, which is found to correlate with the interfacial structures [2], such as the crystallinity of the reinforcement and the matrix, and the orientation relationships at the interface [3]. Furthermore, it was found that the interfacial bond strength is correlated with the structure of misfit dislocations at the interface, and is also affected by the segregation of other elements [4,5]. However, in the case of PRMMCs, such an interfacial structure-strength correlation was sought, but not obtained, owing to the difficulty in direct measurement of the interfacial strength caused by the irregular shapes of the particle reinforcements [6] and the complex interfacial structures [3]. Therefore, one has to simplify the shape of the reinforcement particles by assuming them to be either spheres [7,8], or thin plates [9], and estimate the interfacial strength by fitting experimental test data with the curve predicted by analytical models.

Recent development of mechanical characterization on small volume materials provides new tools for probing interfacial properties and deformation mechanisms in boundary-containing systems that were once unattainable by conventional experimental techniques [10]. For example, using focused ion beam (FIB), Ng and Ngan [11] and Kunz et al. [12] fabricated pure Al micro-pillars containing a single grain boundary, and uniaxial compression tests and subsequent post-mortem microstructural analysis on these pillars revealed that the grain boundary may act as either a dislocation sink or a dislocation barrier, depending on the particular type and structure of the boundary. A relevant study demonstrates frictional sliding along the grain boundary when the boundary plane is inclined at 24° to the loading direction of the pillar, and the critical resolved shear stress for sliding initiation was estimated [13]. This technique was also adopted for nanolaminated structures, such as Cu–Nb [14] and amorphous-ZrCu/crystalline-Zr multilayers [15], where compression tests on micro-pillars milled from the multi-layered structure were used to assess interfacial properties and their effect on the deformation behavior of the laminate. Using a FIB less fabrication route, Guo & Greer [16] and Landau et al. [17] studied the compressive and tensile responses of ~100 nm-diameter bi-crystal nano-pillars containing a single Cu/Fe interface, and evaluated the structural and property evolution of the pillars upon ion irradiation. It was found that for both as-fabricated and irradiated pillars, the bi-crystals would subject to brittle fracture at the Cu/Fe interface, and the interfacial strength was then estimated.

The above-mentioned studies provide important foundation for understanding the deformation mechanism in interface-containing

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materials systems. However, in these studies, the interface was mostly formed between similar materials, i.e., between two grains of the same materials but with different crystallographic orientations, or between two metal–metal thin films. Studying the properties of interfaces formed between two dissimilar materials such as a metal/ceramic interface in an MMC using micro-/nano-mechanical characterization is relatively more difficult, as the contrasting properties of the two constituent phases would make the fabrication and testing challenging. Recently, Singh et al. [9] and Lotfian et al. [18] fabricated and tested polycrystalline Al/amorphous SiC micro-pillars milled from sputter-deposited multi-layered thin films. However, the sputtered composite films have distinct microstructures from bulk SiC particle (SiC_p)-reinforced Al matrix composites, so their results may have limited implications for practical applications of MMCs. In this study, bi-layer SiC/Al micro-pillars were fabricated in a way such that the resulting interface structure resembles that in an actual SiC_p/Al composite produced by the conventional stir casting procedure. These pillars contain a single 45°-slanted interface relative to the pillar axis, so upon uniaxial compression loading, the interface was subject to the maximum resolved shear stress, and thus the shear strength of the interface can be evaluated. SiC–Al was selected as our model system, since the SiC_p-reinforced Al matrix composites are important engineering composites widely used in structural and thermal management applications due to their high specific stiffness and strength, good thermal conductivity, and light weight. To simplify the problem and avoid the complexities associated with the shape and crystallinity of the particle reinforcement, single crystal SiC wafers that have the same hexagonal crystal structure as the SiC_p reinforcement were used as the starting material.

330 μm-thick, double-side polished 4 H-SiC (0001) wafer pieces were cleaned by acetone, isopropanol alcohol (IPA), and then blow-dried, before being dipped into molten Al (99.99% in purity) at 700 °C for 4 s. Manual stirring was applied during the SiC immersion to ensure uniform deposition of Al onto the SiC substrate. The dipping/lifting-out procedure was repeated for a few times, to achieve an Al total thickness of at least 1 mm on SiC. Subsequently, the bi-layer structure was mounted on a 45°-wedge, from which ~1 μm-diameter, ~4 μm-high composite pillars (height ratio between the SiC and Al parts was kept at ~1:1) containing a single, 45° inclined SiC/Al interface relative to the pillar axis (Fig. 1a) were milled out using FIB (FEI Scios). Meanwhile, monolithic Al and SiC pillars of a similar size and the same crystallographic orientation were also fabricated from the bulk part of the bi-layer sample to serve as benchmarks for analyzing the combined behavior of the composite pillars. The vertical taper of all pillars was controlled within 3° to ensure reliable interpretation of the mechanical test data [19,20]. Uniaxial compression tests were conducted using an Agilent G200 Nanoindenter equipped with a 15 μm-diameter flat punch diamond tip, under the displacement-controlled mode, and at a nominally constant strain rate of 0.001 s^{-1}. Continuous stiffness measurement (CSM) with 2 nm oscillation magnitude and 45 Hz oscillation frequency was deployed to monitor the change in contact stiffness during compression. For the composite pillars, since the shear strength of interface was initially unknown, multi-steps of compression with small strain increments were carried out. The diameter for area calculations was taken from the top cross-section of the pillars. Analysis of the compression data followed the methodology developed by Greer et al. [21]. At least five pillars were tested for each sample set to get the statistics. The morphology of pre- and post-compression pillars were studied by SEM, and the SiC/Al interfacial structures before and after the mechanical test were characterized by site-specific TEM analysis (JEOL, 2100F).

Fig. 1a demonstrates an as-fabricated SiC–Al micro-pillar, where small cracks are shown to exist at the interface, which are likely to be caused by the accelerated etching in that region during pillar fabrication by FIB. As a result of the 45° inclination of the interface, the height of the Al part is characterized by a shorter (L_0) and a longer (L_1) sides. Dark field (DF) TEM image of a typical as-fabricated SiC/Al bi-layer lamella structure taken under two beam condition from the [011] zone axis and (111) diffraction vector of Al is illustrated in Fig. 1b. The Al layer in the TEM sample contained predominantly a single grain, indicating that the grain size of Al adjacent to the interface is at least a couple of micrometers. The dislocation density on the Al side adjacent to interface was estimated to be ~3×10^{15} m^{-2} measured by a line-intercept method [22]. As no deformation processing steps were used in the fabrication, it is unlikely that such a high dislocation density was caused by statistically-stored dislocations (SSDs) accumulated during plastic deformation. Instead, it is likely the result of geometrically necessary dislocations (GNDs) that primarily arise from the mismatch in the coefficients of thermal expansion (CTE) between the reinforcement and the metal matrix [6], and that were formed during cooling from high temperature (700 °C). These dislocations would often lead to a "dislocation punched zone" in the metal matrix near the reinforcement-matrix interface in PRMMCs, the size of which often ranges from a few tens of nanometers to several micrometers [3,6]. Although no particular orientation relationships were found at the interface, the out-of-plane orientation of Al, i.e., the orientation in parallel with the interface normal, is close to its <111> direction. This is consistent with the orientation relationship found at the SiC/Al interface of SiCp-6061 Al composites ($(0001)_{SiC}//\{111\}_{Al}$; $\langle 2110\rangle_{SiC}//\langle 110\rangle_{Al}$) [23]. A magnified view of the interface structure (Fig. 1c) reveals a ~15 nm thick, distinct interfacial layer, indicating good adhesion between SiC and Al. The formation of aluminum carbide (Al_4C_3) was not detected at the interface, and the interfacial layer was determined to be amorphous Al_2O_3 (detailed

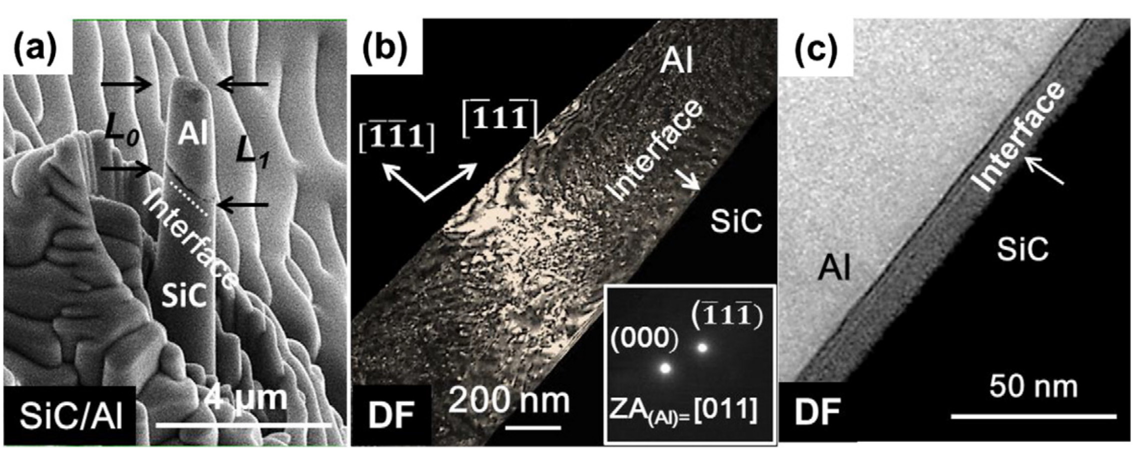

Fig. 1. (a) SEM image of ~1 μm-diameter SiC–Al composite pillar (height ratio between the SiC and Al parts is ~1:1). The Al part is characterized by a shorter (L_0) and a longer (L_1) sides. (b) DF TEM image of a typical as-fabricated SiC–Al bi-layer lamella structure taken under two beam conditions (from the [011] zone axis and (111) diffraction vector of Al). (c) A magnified rendition of the interfacial structure under TEM.