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Micromechanical and *in situ* shear testing of Al–SiC nanolaminate composites in a transmission electron microscope (TEM)

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ABSTRACT

Nanolaminate composites show promise as high strength and toughness materials. However, due to the limited volume of these materials, micron scale mechanical testing methods must be used to determine the properties of these films. To this end, a novel approach combining a double notch shear testing geometry and compression with a flat punch in a nanoindenter was developed to determine the mechanical properties of these films under shear loading. To further elucidate the failure mechanisms under shear loading, *in situ* TEM experiments were performed using a double notch geometry cut into the TEM foil. Aluminum layer thicknesses of 50 nm and 100 nm were used to show the effect of constraint on the deformation. Higher shear strength was observed in the 50 nm sample (690 ± 54 MPa) compared to the 100 nm sample (423 ± 28.7 MPa). Additionally, failure occurred close to the Al–SiC interface in the 50 nm sample as opposed to failure within the Al layer in the 100 nm sample.

1. Introduction

Nanolaminate materials consist of alternating layers of dissimilar materials having an individual layer thickness on the order of tens of nanometers. Nanolaminates show promise in applications requiring excellent mechanical attributes including high strength, toughness, and wear properties [1–4], as well as biocompatibility [5] and optical properties [6]. Additionally, the properties of these films can be tailored by changing the layer thicknesses to obtain optimum properties.

Quantifying the shear strength of the interfaces in nanolaminates is a challenge. It is not practical to make bulk samples due to the size limitations of the sputtering fabrication process. With focused ion beam (FIB) milling, however, precise geometries on the micrometer scale can be machined. This capability is now being exploited for mechanical testing purposes, the most common of these techniques being micropillar compression [4,7–11]. Other types of mechanical tests, although less common, have been implemented on the micrometer scale including tensile loading [12], cantilever beam [12,13], and fracture toughness measurement geometries [14].

A few other methods to quantify the shear properties on the microscale have been attempted, although there are experimental issues intrinsic to these techniques. Previous work by Li et al. [15] on metal–metal nanolaminate composites utilized pillars milled with interfaces inclined 45° to resolve the maximum amount of shear stress on the interface. The drawback of this geometry is that in addition to the shear stress resolved on the interface, there is also a large normal stress component superimposed on the shear stress. The *in situ* characterization in that work utilized pillars which were inclined relative to the flat punch. While this does generate a shear stress, there is also a large bending moment on the pillars. The use of these methods is limited to interfaces with low shear strengths relative to the normal strength so that the normal and bending stresses have a minimal effect. Other studies by Pfetzing-Micklich et al.[16] and Heyer et al. [17] also made shear strength measurements using a FIB milled geometry having a large beam supported on either side by a thinned region that will shear when a load is applied to the center of the beam. Although this geometry allows for a fairly homogeneous shear stress state, alignment of the indenter is critical. Any deviation of the indenter from the center of the beam would induce a bending stress and/or an unequal load distribution between the two gauge sections. Additionally, this type of geometry is not ideal for *in situ* TEM studies because both gauge sections cannot be easily viewed simultaneously.

The double notched interlaminar shear test involves a specimen notched on opposite sides, and loaded in compression, such that the longitudinal plane between the notches is subjected to pure shear [18–20]. This test has been used extensively in bulk fiber reinforced composites, where shear failure between plies occurred consistently [18,19].

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In this paper we report on a new micromechanical compressive double-notch shear technique for accurately quantifying shear strengths of nanolaminates. This work is centered on characterizing the interfacial shear strength in Al–SiC nanolaminates. With the double notch geometry under a compressive load, a shear stress develops between the two notches in plane with the layers of the composite. To elucidate the failure mechanisms, double notch samples were also made on TEM foils and fractured *in situ*. This characterization technique allowed for the crack path to be observed in relation to the layers and individual grains.

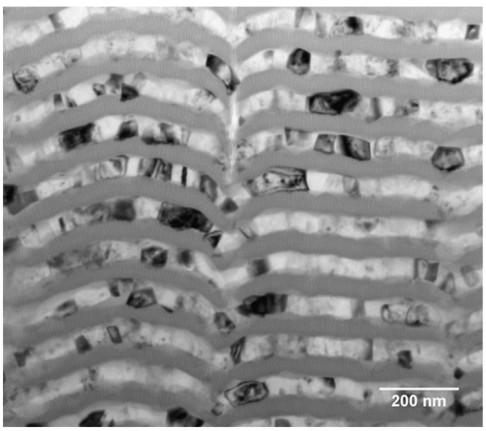

Fig. 1. TEM micrograph of layered nanostructure of these composites. Uniform gray layers are amorphous SiC and layers with varying contrast are crystalline Al.

2. Materials and experimental procedure

The Al–SiC nanolaminate samples (Fig. 1) were fabricated using magnetron sputtering on a Si (100) substrate. The targets used were pure Al (99.99%) and SiC (99.5%) (Kurt J. Lesker, Clarion, PA). Prior to multilayer deposition, the Al and SiC targets were sputtered for 10 min at 40 and 95 W, respectively, to remove any contamination and oxides. The deposition was carried out at 95 and 215 W for Al and SiC respectively to achieve a deposition rate of 7.5 nm/min. An automated routine was used to deposit alternating layers until the total multilayer thickness was approximately 15 μm. Two types of samples with different individual layer thicknesses were used in this study. The first sample consisted of 50 nm Al layers and 50 nm SiC layers, whereas the second consisted of 100 nm Al layers and 50 nm SiC layers.

Shear testing of the interface required that the films be oriented parallel to the loading axis. Therefore, the samples were mounted in epoxy edge on. Mechanical polishing to a final polish of 0.05 μm colloidal silica was then carried out on 2 faces to expose a 90° corner. The amount of material that needs to be removed using the FIB is dependent on the rounding at the edge. This rounding was kept to a manageable level by carefully hand polishing using SiC abrasive paper and only using the colloidal silica slurry the minimum amount of time to obtain a smooth surface finish.

Fabrication of the double notch shear pillars (Fig. 2) was performed using a dual beam SEM-FIB (FEI Nova 200). Milling from two orthogonal directions was required to fabricate double notch pillars, necessitating the polished and square corner mentioned above. An ion beam current of 20 nA was used to quickly

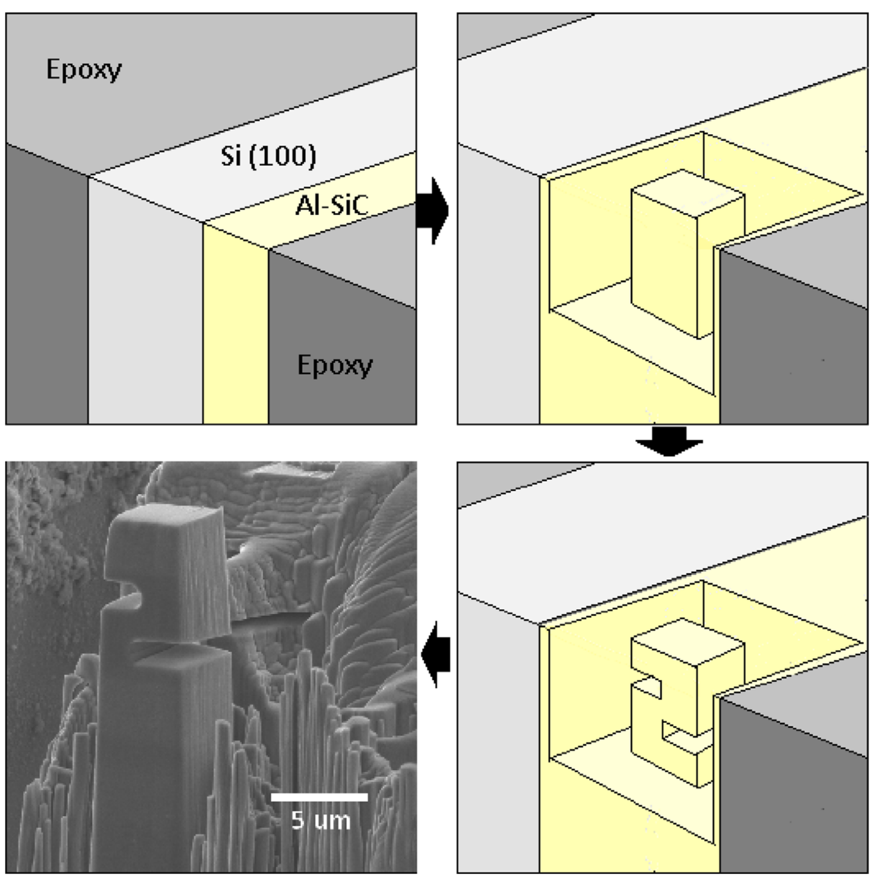

Fig. 2. An outline of the milling procedure for double notch pillars. A corner is milled from the top to create a square pillar with access to the side. The notches and top are then milled from the front.