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Micropillar compression of Al/SiC nanolaminates

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Abstract

Al/SiC nanolaminates possess an excellent combination of mechanical strength and flexibility. While nanoindentation provides a reasonable estimate of the mechanical properties such as Young's modulus and hardness of these materials, the stress state under nanoindentation is extremely complex. Micropillar compression has become an attractive method of studying the mechanical properties of materials at small length scales in a nominally homogeneous stress state. In this work, micropillars of Al/SiC nanolaminate were fabricated using focused ion beam milling. Compression testing was carried out using a flat-end nanoindenter head. The actual displacement of the pillar during micropillar compression was deconvoluted by subtracting the "extraneous" displacements of the system. Fractographic analysis showed that Al squeezes out between the SiC layers and that a mutual constraint is observed between the hard and soft layers. Numerical finite element modeling was also employed to provide physical insight into the deformation features of the multilayered pillar structure and agreed well with the experimental observations.

Keywords: Nanoindentation; Micropillar compression; Nanolaminate; Focused ion beam

1. Introduction

Multilayered materials at the nanoscale exhibit unique electrical [1–3], magnetic [4], optical [5,6] and mechanical properties [7–9]. Metal–ceramic systems, in particular, can be tailored to obtain a combination of high strength, hardness and toughness [8–14]. Nanoindentation has been used extensively to probe the modulus and hardness of homogeneous bulk materials and thin films. In multilayered materials, however, it has been shown that a highly complex and inhomogeneous stress state is developed under the indenter [11,15]. Furthermore, the multilayered structure results in complex plasticity, which can take place even during unloading [15,16].

A more straightforward way of obtaining nominally uniaxial stress–strain response at small volumes is by microcompression of pillars [17]. Pillars in the micrometer range, or smaller, can be fabricated by the focused ion beam (FIB) technique [17–21]. These pillars are then compressed using a flat punch in a nanoindenter, or by scanning electron microscopy (SEM). This method has recently been used to study the mechanical response of metallic single crystals [22,23], metallic alloys [24] and metallic nanolaminates [25].

This paper reports on the microcompression behavior of model Al/SiC nanolaminates. The pillars were fabricated by FIB and tested in compression using a nanoindenter. The effect of pillar taper was studied. The evolution of damage was quantified by interrupted experiments and cross-sectioning of deformed pillars, also with the FIB. The finite element method (FEM) was used to model the deformation behavior of the pillars numerically and to provide a mechanistic understanding of deformation.

2. Materials and experimental procedure

Al/SiC multilayer samples were synthesized by physical vapor deposition, using magnetron sputtering. Details of the sputtering method are discussed elsewhere [11–13].

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The samples were grown on a Si(1 1 1) substrate and the individual layer thickness of Al and SiC were targeted to be ~50 nm. A total of 40 (20 layers of Al and SiC each) layers were deposited. The cross-section of the as-deposited microstructure, as well as that of the damaged pillars was studied using FIB. Ten measurements of layer thickness were taken for each layer in the sample to obtain statistics of the layer thickness.

The micropillars were fabricated using FIB. Two types of samples were fabricated: (a) pillars using an annular milling approach which resulted in a slight taper (~4°); and (b) pillars using a "lathe milling" procedure similar to that described by Uchic and Dimiduk [17], which resulted in pillars with negligible taper (<1°). The latter pillars are termed "taper-free". The tapered pillars were milled in two steps. A relatively large current (7 nA) was used to mill a pillar with a relatively large diameter (4 μm). This was followed by a second milling operation at a much lower current of 50 pA, which resulted in a final pillar diameter of ~2 μm. Straight pillars (with minimal taper) were fabricated by initially milling a pillar of ~3 μm, using the annular milling approach described above. Next, the sample surface was tilted so as to make a small angle with the initial sample surface (~28°). In this manner, the ion beam impinged the sample tangent to the surface. The sample was rotated in 5° intervals, and the milling operation was repeated. The milling current in this step was ~50 pA. A thin layer of Pt was deposited in both processes to protect the sample surface from ion beam damage. To align the pillars during its rotation for lathe operation, a small central hole was drilled in the top Pt layer. The hole served as a marker for centering the pillar after each incremental rotation. Ion beam damage from Ga^+ ion implantation can take place, although the depth of damage has been estimated to be no more than 60 nm at 30 kV beam under normal incidence [26,27]. For the pillars studied here, this is a very small fraction of the total pillar diameter (~2 μm).

One of the major issues affecting microcompression data is the misalignment of the sample relative to the punch. It has been shown that even small degrees of misalignment can have a significant effect on the measured stiffness of the pillar [28]. In the present experiments, a novel nanoindentation *in situ* method was used to measure the misalignment of the sample. A sharp-tip nanoindenter was used as a high resolution displacement gauge to determine the surface profile of the sample. The indenter tip is brought in contact with the surface at various locations on the sample. The surface is "found" by the indenter, as measured by an extremely small contact stiffness (140 mN). This *z* displacement, measured across the surface, can be used to compute and, if necessary, correct the sample inclination. Using this approach, the sample inclination in the present experiments was measured to be <0.3°.

Compression experiments were carried out using a nanoindenter (MTS XP, Agilent Systems, Chandler, AZ). A Berkovich (three-sided pyramid) diamond indenter with a flat triangular cross-section with a 10 μm side was used. The largest diameter circular cross-section that would fit in this triangle had a radius of ~6 μm (three times as large as the pillar diameter). The experiments were carried out in a continuous stiffness measurement (CSM) mode [29], enabling continuous measurement of contact stiffness instantaneously as a function of depth. The pillars were compressed to varying depths to study the progression of damage with increasing load. Fractographic analysis after deformation was carried out by dual-beam FIB, and imaging was conducted by SEM.

3. Results and discussion

3.1. Micropillar compression experiments

The cross-section of the microstructure of the as-processed nanolaminate obtained by FIB is shown in Fig. 1. A total of 40 alternating layers of Al and SiC were grown on a Si(1 1 1) substrate. The individual layer thickness for Al was 58 ± 2 nm, while that for SiC was 73 ± 1 nm. This corresponds to a volume fraction of SiC of ~56%. A small degree of roughness is associated with the individual layers and is due to the columnar grain structure of the Al layers. Fig. 2 shows the two types of pillars that were fabricated (with taper and without taper). The average taper in the tapered pillar was ~4°.

The total compliance measured during micropillar compression is a function of several factors, as shown in Fig. 3. These include a short Si post under the nanolaminate, the Si base and a small contribution from the diamond indenter. Thus, the total compliance of the system is the sum of several compliances and can be written as follows:

$$C_{\text{measured}} = C_{\text{p}} + C_{\text{Si-post}} + C_{\text{Si-base}} + C_{\text{Di}} + C_{\text{Pt}}$$

where C_{p} is the compliance of the deformed pillar, C_{Pt} is the deformation of the thin layer of Pt, $C_{\text{Si-post}}$ is the compliance due to deformation of the Si post, $C_{\text{Si-base}}$ is the compliance due to the "punching effect" on the Si base (i.e. the Sneddon's correction [30] described below), and

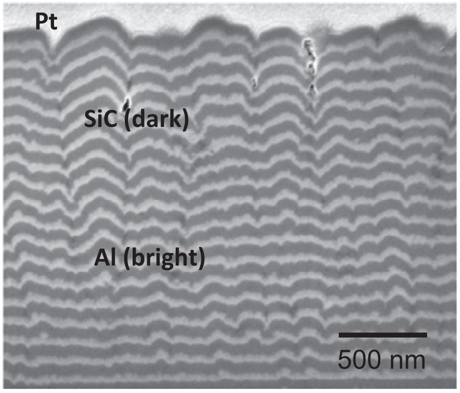

Fig. 1. Microstructure of Al/SiC multilayer used in the study. A total of 40 alternating layers of Al and SiC were grown on a Si(1 1 1) substrate. The layer thicknesses are homogeneous and show little variability.