Acta Materialia 114 (2016) 25–32

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Anisotropy, size, and aspect ratio effects on micropillar compression of Al–SiC nanolaminate composites

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ARTICLE INFO

Article history:
Received 23 February 2016
Received in revised form
9 May 2016
Accepted 10 May 2016

Keywords:
Multilayer
Nanocomposite
Orientation dependence
Finite elements
Weibull analysis

ABSTRACT

Metal-ceramic nanolaminate composites show promise as high strength and toughness materials. Micropillar compression was used to characterize the mechanical behavior of Al–SiC multilayers in different orientations including loading at 0°, 45° and 90° with respect to the direction of the layers. The 0° orientation showed the highest strength while the 45° orientation showed the lowest strength. Each orientation showed unique deformation behavior. Effects of pillar size and aspect ratio were also studied. Higher compressive strengths were observed in smaller pillars for all orientations. This effect was shown to be due to a lower probability of flaws using Weibull statistics. Additionally, changes in the aspect ratio was shown to have no significant effect on the behavior except an increase in the strain to failure in the 0° orientation. Finite element analysis (FEA) was used to simulate and understand the effect of these parameters on the deformation behavior.

1. Introduction

Laminate composite materials have long been used to obtain material properties which are a combination of the properties of their constituents. Reduction of the dimensions of the individual constituents of these laminates to the nanoscale (≤100 nm layer thickness) has allowed these materials to exhibit unique properties which are superior to that of their macroscale counterparts. These nanolaminate composites show promise in applications requiring properties such as high strength, toughness, and wear resistance [1–6], as well as biocompatibility [7], and certain optical properties [8]. Additionally, like macro-scale composites, the properties of these materials can be tailored by varying composition and layer thicknesses to obtain optimum properties.

Metal-ceramic nanolaminates were chosen as the material of interest in this study. From a practical perspective, these materials exhibit a combination of high toughness and strength which is attractive for many applications [9,10]. Scientifically, these materials provide an ideal environment to study plasticity under extreme degrees of constraint due to the large elastic and strength mismatch between the layers. However due to the limited volume of material which is able to be deposited, the mechanical characterization methods available are limited to micro scale techniques.

The orientation dependence of macro-scale laminated composites has been studied previously, with the majority of the studies focusing on the orientations implications on fracture toughness [11–13]. Research performed by Roy et al. [12] is very analogous to the present study, where the compressive behavior of bulk aluminum - aluminum oxide laminar composites (20–220 μm individual lamina thickness) was determined as a function of laminate orientation. This work showed that due to the varying load transfer to the reinforcement, as the angle between the lamina and the loading axis increases there is a steep decrease in strength from 0° to 30°, a minimum at 45°, and finally a slight increase at 90°. In addition, the strain the material is able to accommodate varies in the opposite manner, increasing as the load becomes more misaligned with the reinforcement phase.

Pillar compression is becoming an increasingly popular micro-scale mechanical testing technique [6,14–19]. This technique

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http://dx.doi.org/10.1016/j.actamat.2016.05.018
1359-6454/

utilizes a flat punch along with the high load and displacement resolution of instrumented indentation in order to carry out extremely sensitive compression experiments. Pillar compression provides an approximately uniaxial and uniform stress state which also allows the stress strain behavior to be determined. The uniaxial stress state is particularly critical in characterizing the anisotropy, as shown by our previous attempts to characterize this dependence using nanoindentation [20]. The results of the experimental Berkovich indentations and finite element modeling from that study showed that, due to the complex stress state caused by the indenter geometry, the modulus determined through indentation shows a much weaker dependence on orientation than what is predicted through the classical laminate theory. Spherical nanoindentation has also been used to determine the stress strain response and elastic anisotropy in other materials [21], however micropillar compression has the advantage of a clearly defined stress state which is more appropriate for this study.

In order to determine the anisotropy in the deformation behavior of these materials, three orientations, with the loading axis forming 0°, 90°, and 45° with respect to the layer direction, were characterized using pillar compression. Pillars compressed perpendicular to the nanolaminate surface (90°) subject the layers to a nearly isostress condition, while pillars compressed in the transverse direction (0°) load the layers in a nearly isostrain condition. Finally, pillars oriented at 45° with respect to the laminate surface generate the largest amount of shear stresses parallel to the layers.

The size effect phenomenon in pillar compression, where the flow stress of the materials increases as the size of the pillars is reduced, has been documented in a number of cases in single phase materials at small scale lengths [14,15,17,22]. Having microstructural features smaller than the pillar size precludes one of the most common mechanism the size effect is attributed to, namely dislocation starvation. Therefore, this effect requires further investigation in nanostructured materials where the testing geometry is still much larger than the structural features.

This work aims to characterize two aspects of nanolaminate behavior which have not been addressed previously. First, as nearly all previous experimental efforts have been focused only on loading normal to the layers, there is a need to characterize the properties of these composites in other orientations to determine if the classical laminate behavior is still applicable at the nanometer length scale. Second, it is necessary to determine both if and why geometric factors, namely the size and aspect ratio of these pillars, have an effect on the measured response. In order to achieve these aims, the quantitative measurement of the material response gained through pillar compression will be paired with both post deformation imaging and finite element modeling in order to provide a comprehensive understanding of the deformation mechanisms dictating the behavior.

2. Materials and experimental procedure

The metal-ceramic nanolaminate composites were fabricated using an automated magnetron sputtering procedure, the details of which are given elsewhere [23,24]. The samples used in this study consisted of alternating layers of Al and SiC, each having an individual layer thickness of approximately 50 nm. These layers were deposited sequentially until a total multilayer thickness of approximately 15 μm was obtained (~300 individual layers). The 0° sample was able to be used for pillar compression without further preparation. The 90° and 45° samples, however, required mounting in epoxy and polishing in order to expose the edge to be tested, shown schematically in Fig. 1.

Pillar fabrication, post-mortem imaging and cross-sectioning were performed using a dual beam FIB operated at 30 keV ion beam accelerating voltage, which provides a high milling rate, and 5 keV electron beam accelerating voltage, which provides high imaging resolution. Pillar fabrication was performed using an annular milling procedure, which enables a higher throughput of pillars compared to lathe milling [6], but results in approximately 2° of taper, as shown in Fig. 1. For each orientation, pillars were milled with nominal dimensions of 2 × 4 μm, 2 × 6 μm, and 1 × 2 μm (diameter by height) with a 20 μm surrounding trench to allow clearance for the indenter. These pillar sizes were chosen in order to study the size effect as well as the effect of aspect ratio. Various ion beam currents were used depending on the material removal rate and precision needed, but final polishing of the surfaces was always carried out using ion currents below 50 pA (at 30 keV).

Pillar compression was carried out using a commercial nanoindenter (Nanoindenter XP-II, Agilent) equipped with a diamond flat punch. Samples were mounted to aluminum stubs for testing using a mounting adhesive. Tests were performed using constant displacement rates of 5 nm/s, 10 nm/s, and 15 nm/s for 1 × 2 μm, 2 × 4 μm, and 2 × 6 μm pillars, respectively, yielding an initial strain rate of $2.5 \pm 0.2 \times 10^{-3}$ s^{-1} (mean ± standard deviation) for all experiments. The drift rate for all tests was held below 0.05 nm/s. The stress and strain was calculated based off of the initial pillar dimensions, yielding engineering stress and strain.

The deformation of the micropillars was simulated by finite element modeling (FEM) using the commercial software Abaqus (Abaqus, v. 6.12, Dassault Systems Simulia Corp., Providence, R.I.). The simulations were performed in 2D plane strain conditions. The models consisted of a rigid flat punch, micropillars with layers oriented at 0°, 45°, and 90° with respect to the micropillar axis and base material. Three different pillar sizes were modeled to account for size and aspect ratio effects, with similar dimensions to the pillars tested experimentally. To match more precisely with experimental conditions, a 2° taper was also included in the pillar models. In each case, the effect of layer waviness was accounted for by comparing the results of the simulations performed with micropillars containing flat layers, to those containing undulated layers. The undulated layers were modeled by imposing a standard sinusoidal waveform with a wavelength of 0.5 μm and amplitudes of 15 and 45 nm. The 45 nm amplitude is close to the amplitude observed experimentally, although there is considerable variability in the actual microstructure. All the pillar models were meshed by 4-node bilinear plane strain quadrilateral meshes (CPE4) with approximately 30,000 elements (after performing a mesh convergence study). Constraint boundary conditions were imposed at the bottom of the base material, while the rest of the surfaces were set free.

For all of the models, the Al and SiC layers were modeled as elastic perfectly plastic materials, with no strain hardening, due to the small layer thickness, which precludes any dislocation storage. The Young's modulus of Al and SiC were taken as 70 GPa and 300 GPa, and the corresponding Poisson's ratios were 0.34 and 0.14, respectively. The yield stress of Al was 935 MPa [25], and the apparent yield stress of SiC was chosen as 7 GPa (a large value estimated from nanoindentation results of 1 μm thick monolithic SiC films). The base material was modeled as a purely elastic material, with the elastic modulus and Poisson's ratio estimated as an average value of the isostrain and isostress composite moduli between Al and SiC. The Al–SiC interfaces were considered perfectly bonded in all cases.

3. Results and discussion

3.1. Effect of layer orientation on deformation morphology

Engineering stress-strain curves obtained from the pillar