In situ nanomechanical testing of Al-SiC nanolaminates using 3D X-ray Microscopy

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Summary: Laboratory based nanoscale x-ray tomography enables three dimensional imaging of materials. Due to the non-destructive nature of this imaging modality, it is possible to conduct real time investigations of deformation under load at resolutions of up to 50 nm. Here we present an example of nano-mechanical testing carried out on a micropillar sample extracted from a nano-laminate composite material and highlight the key advantages of the technique.

1. CONTEXT

Metal-ceramic nano-laminate composites show promise as high strength and high toughness materials [1,2]. 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 composites show promise in applications requiring properties such as high strength, toughness, and wear resistance, as well as biocompatibility, and certain optical properties. Additionally, like macroscale composites, the properties of these materials can be tailored by varying composition and layer thicknesses to obtain optimum properties. However, due to the limited volume of these materials, microscopic mechanical testing methods must be used to determine the properties of these films. Due to the small size scales, investigating the underlying failure mechanisms is challenging. Micropillar compression testing is an effective means of characterizing the mechanical behaviour of Al-SiC multilayers. *In situ* compression of the micro-pillars while imaging in the Scanning Electron Microscope (SEM) is a commonly used technique that provides direct visualization of the failure of the samples under load. However, SEM observations are restricted to the surface and do not reveal the full extent of the sub-surface cracks or the underlying defects in the material being tested. Laboratory based nanoscale x-ray tomography enables non-destructive 3D imaging of samples with resolutions down to 50 nm. Using a specially designed nanomechanical testing stage, *in situ* compression tests can be performed to observe the failure mechanisms in the micropillars in three-dimensions. This technique further allows assessing the uniformity of layers and detecting the presence of processing-induced defects in the samples. Analysis of the reconstructed x-ray tomography data collected over the course of an entire compression experiment provides valuable insights into how these nanolaminates fail under load.

2. EXPERIMENTAL METHOD

The metal-ceramic nanolaminate composites consisting of alternating layers of Al and SiC, each having an individual layer thickness of approximately 100 nm were fabricated using an automated magnetron sputtering procedure. Layers were deposited sequentially until a total multilayer thickness of approximately 15 µm was obtained (~300 individual layers). Pillar fabrication was performed using a dual beam FIB operated at 30 keV ion beam accelerating voltage using an annular milling procedure. Pillar compression was performed on an *in situ* load stage operated in a laboratory nano-scale X-ray imaging microscope (ZEISS Xradia 810 Ultra). Since the x-ray absorption contrast between aluminium and SiC is low, the microscope was operated in a Zernike phase contrast mode which enhances interfaces and cracks due to phase differences. The load stage was operated in displacement controlled mode and 3D tomographs were acquired at three incremental loadsteps. The x-ray

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projection data was reconstructed using filtered back projection method and used to investigate defects and analyse deformation initiated by the applied load.

3. RESULTS

We present in this study, the results from an *in situ* compression experiment conducted on a nanolaminate micropillar with layers parallel to the top surface. From the reconstructed tomography data we were able to discern individual aluminium and SiC layers and also identify voids that were distributed throughout the volume of the pillar. The data also reveals the 3D nature of the failure under compressive loading. Using volumetric quantification of segmented data, the porosity was analysed and visualized. Important quantitative parameters such as pore volume, pore diameter and size distribution were also determined. It was seen that the voids were connected and formed channels which acted as crack initiation sites triggering failure in those regions.

References

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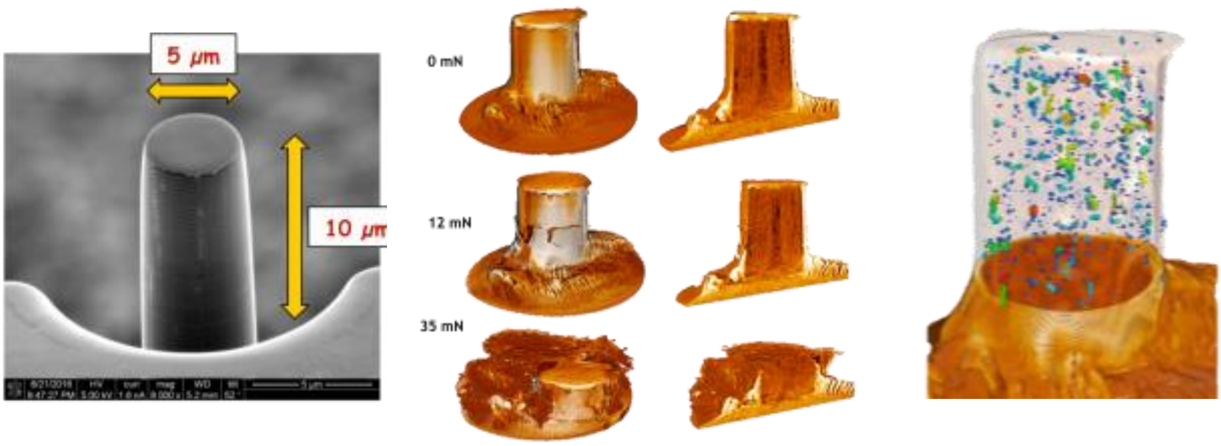

(a) (b) (c)

Figure 1: (a) SEM image of the Al-SiC nanolaminate micropillar prepared using focused ion beam milling. 100nm thick layers can be observed. (b) 3D visualization of micropillar compression at different applied loads. (c) Pore size distribution within the micropillar rendered in 3D.