

Nanoindentation of Nanoscale Ti/Au Multilayer Composites

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Introduction

Nanoscale metallic multilayer (NMM) composites display mechanical properties which are significantly superior to those of metallic thin films composed of a single metal, including higher yield strength, higher ductility, and better resistance to irradiation damage, among others. Due to their desirable properties, NMM composites have many potential applications as nanoscale engineering materials, including applications in the semiconductor industry and as thin protective coatings. In this study, nanoindentation was performed on Ti/Au NMM composites to determine hardness and elastic modulus values.

Experimental Method

Alternating layers of Ti and Au were sputtered onto both glass and SiO₂ substrates. Individual layers had a thickness of 20 nm, and total multilayers thicknesses of 400 nm and 1 micron were tested. A Hysitron Nanoindenter was used to perform the testing, and a diamond Berkovich indenter tip was used to make the indents, which was calibrated on SiO₂ with a known elastic modulus of 73 GPa. A depth-controlled load function with several partial unloading segments was used to obtain hardness and modulus data at depths ranging from 10 to 180 nm. Nanoindentation was also performed on the bare glass and SiO₂ substrates to determine the mechanical properties of the substrates.

Results

Figure 1 contains representative load-depth curves for all the samples tested in this study.

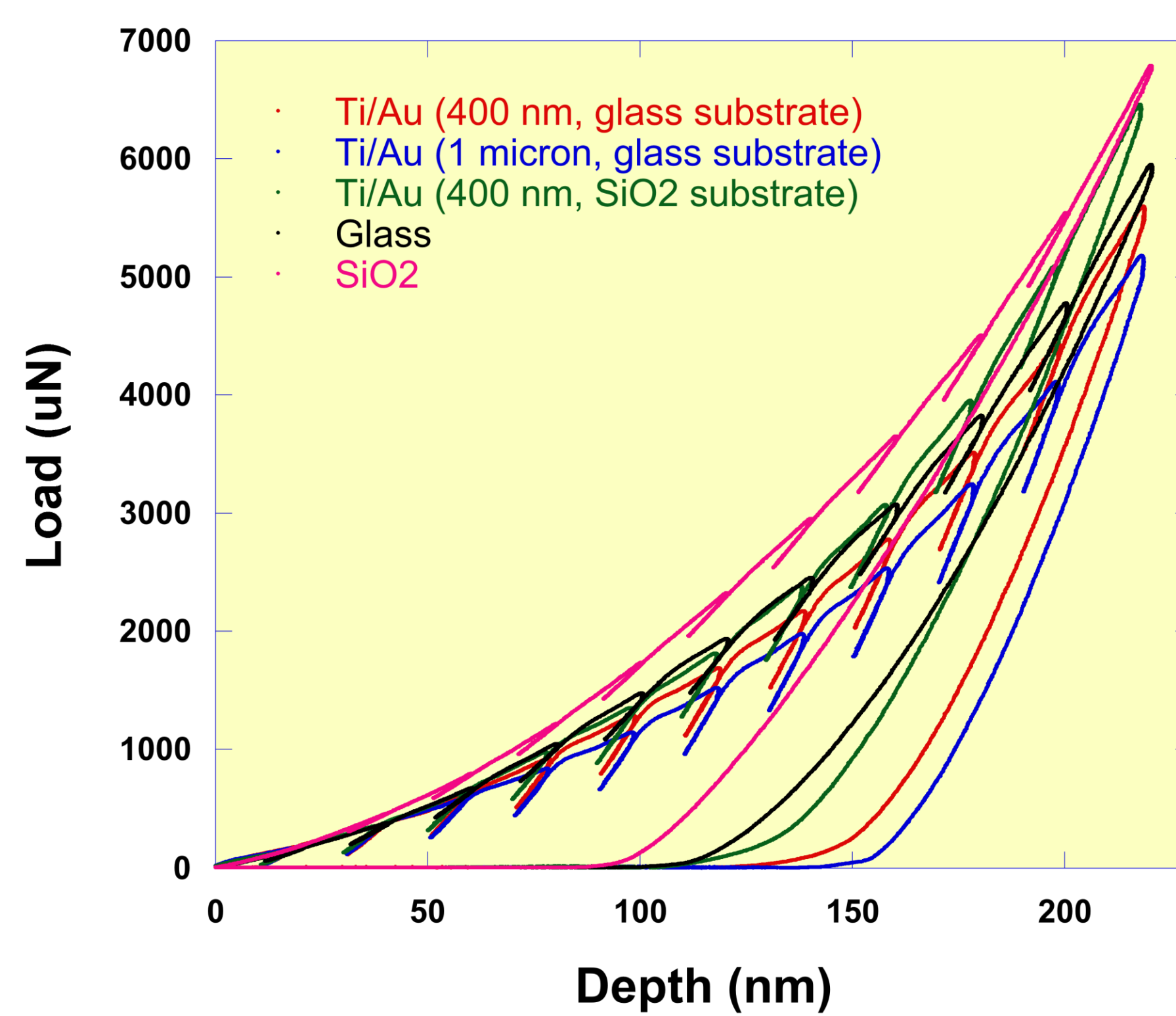


Figure 1. Representative load-depth curves for the three Ti/Au multilayer samples tested, as well as for the bare glass and SiO₂ substrates.

Figure 2 displays the measured hardness (as measured at each partial unloading segment of the load-depth curve) for the three Ti/Au samples, as well as the hardness values for the bare glass and SiO₂ substrates, as a function of indentation depth.

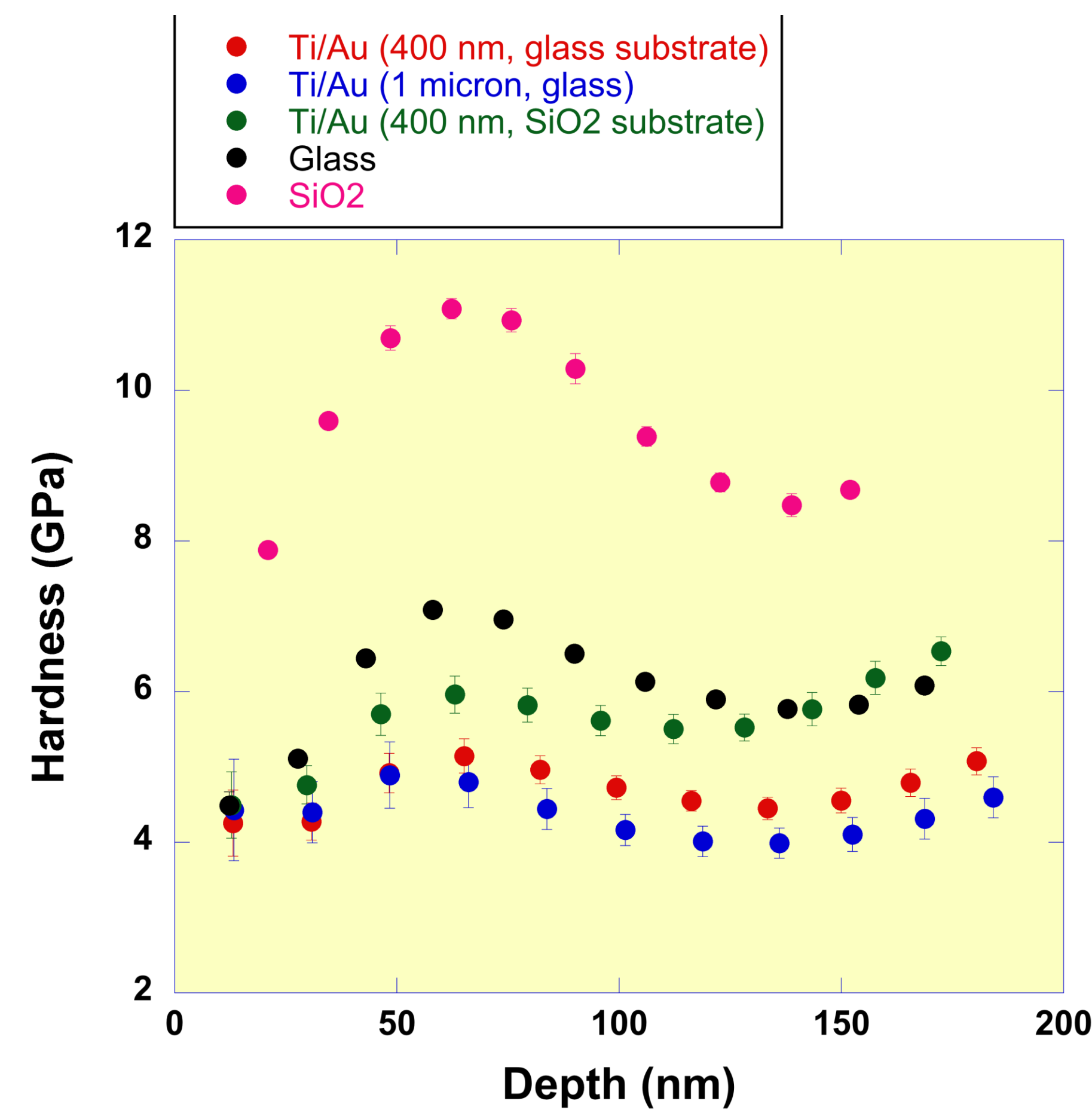


Figure 2. Hardness vs. indentation depth for the three Ti/Au multilayer samples tested, as well as for the bare glass and SiO₂ substrates. Error bars indicate one standard deviation.

Figure 3 displays the measured modulus of elasticity of the five samples as a function of indentation depth.

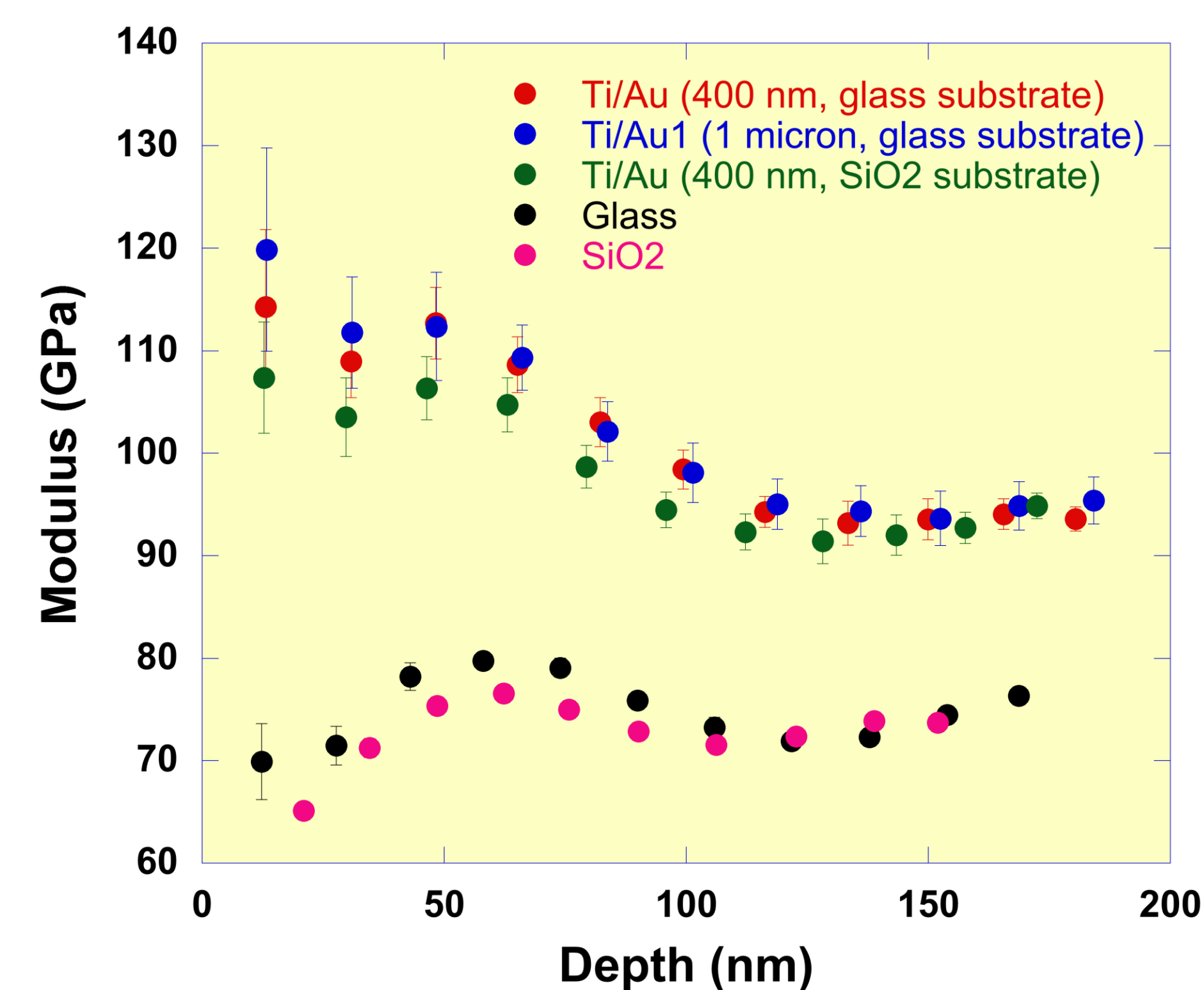


Figure 3. Elastic modulus vs. indentation depth for the three Ti/Au multilayer samples tested, as well as for the bare glass and SiO₂ substrates. Error bars indicate one standard deviation.

Discussion

As can be seen in Figure 1, the load-depth curves for the three Ti/Au samples tested all appear quite similar to each other, as well as to the load-depth curves obtained from indenting the bare glass and SiO₂ substrates. This is not surprising considering the measured hardness and elastic modulus of all three Ti/Au multilayer samples were found to be roughly equivalent, regardless of substrate or overall film thickness. The notable exception is the measured hardness of the 400 nm thick Ti/Au multilayer film on the SiO₂ substrate, which increases with indentation depth more than the Ti/Au multilayer samples with glass substrates. This difference can be attributed to substrate effects.

Interestingly, the glass and SiO₂ substrates possessed nearly identical values of elastic modulus, which was about 73 GPa, as can be seen in Figure 3. This equivalence in the elastic moduli values of the two different substrates makes it difficult to assess the relative magnitude of substrate effects on the mechanical properties of the NMM composites based on differences in the measured elastic modulus – as can be seen in Figure 3, the elastic modulus values for all three Ti/Au multilayer samples are nearly identical at approximately 110 GPa.

Another interesting feature of the data is the almost complete lack of dependence of the mechanical properties of the multilayer film on the total film thickness. For example, in Figure 2, the measured hardness values of the two Ti/Au multilayer films mounted on glass are nearly identical and show nearly identical dependence on indentation depth, although one film has a total thickness of 1 micron, and the other has a total thickness of only 400 nm. This result implies that the effect of the substrate on the measured mechanical properties of the film is not highly dependent on how deep the indentations are made, or how near the indenter tip comes to the underlying substrate during indentation.

A final observation is the apparent lack of strain hardening behavior in the Ti/Au multilayer composites. Strain hardening is expected in this system for several reasons: the incoherent interfaces between the face-centered cubic Au nanolayers and the hexagonal Ti nanolayers should provide a strong barrier to dislocation propagation between layers, and as dislocations become trapped in a layer, they should provide resistance to dislocation propagation within that layer, effectively hardening the material. The fact that this behavior was not observed is puzzling. This could be due to a variety of reasons, but before a mechanistic explanation is proposed, the results of this study should be confirmed by recalibrating the tip used to make the indents and re-analyzing the load-depth data using a new tip area function. During the course of this study, the tip was recalibrated several times, but results were often unsatisfactory and resulted in modulus values for glass and SiO₂ which depended strongly on indentation depth, which is known to be incorrect. In short, further work is needed to confirm these results.

Conclusions

Nanoindentation was used to measure the mechanical properties of nanoscale Ti/Au multilayer composites. Alternating 20 nm thick layers of Au and Ti were sputtered onto glass and SiO₂ substrates to make the samples for this study. These Ti/Au multilayers were found to have hardness and modulus values of approximately 4.3 GPa and 110 GPa, respectively. Very little if any strain hardening behavior was observed in these materials, and the effect of the substrate on the measured mechanical properties did not display a strong dependence on the relative indentation depth.

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