Keysight Technologies
Rapid Hardness of Nano-Structured Metals

Application Note

Introduction

Nano-structured metals are highly valued for their strength. Nano-structured metals are metals which have been manufactured to have grains (i.e. coherent crystals) that are on the scale of 1–100nm. Such materials manifest great strength, because dislocations (i.e. crystalline imperfections) tend to exhaust themselves at grain boundaries due to incoherence.

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The phenomenon of plastic deformation due to dislocation motion can be understood with a simple analogy: the movement of a floor rug. A floor rug can be moved easily by introducing a kink at one end of the rug, and then pushing that kink to the other end of the rug. When the kink reaches the other end of the rug, it disappears, and the net effect of the motion of the kink is that the entire rug has been moved. Now let us suppose that we place two rugs end-to-end and again introduce a kink at one end of the rug and translate it to the other end. When the kink reaches the end of the first rug, it does not translate to the second rug due to incoherence between the two rugs. In this way, the second rug remains unaffected by the deformation and net motion of the first rug. Crystalline materials deform in a similar way. Flaws (dislocations) which arise in one part of the grain are translated through the grain causing net deformation, but these flaws tend to exhaust themselves at the grain boundaries. They do not perpetuate into adjacent grains due to incoherence. This intragranular behavior is the cause of the high strength observed in nano-structured metals. Smaller grains inhibit the perpetuation of dislocations in the material.

Further, because the scale of the deformation is generally large compared to the grain size, the strength of a nano-structured metal is independent of the scale of deformation. (This is in contrast to metals which have large grains, wherein the strength varies inversely with the scale of deformation.) Thus, not only are nano-structured metals strong, but the strength is uniform and independent of the scale of deformation.

There are many ways to make nano-structured metals. Small grains can result from quenching, extreme deformation, or geometrical boundaries which naturally limit grain size. In this work, grain size was constrained by depositing two metals (copper and nickel) in alternating layers so that grains could not grow larger than the individual layers. Various samples were made by varying the thickness of the individual layers in order to investigate the relationship between hardness and layer thickness.

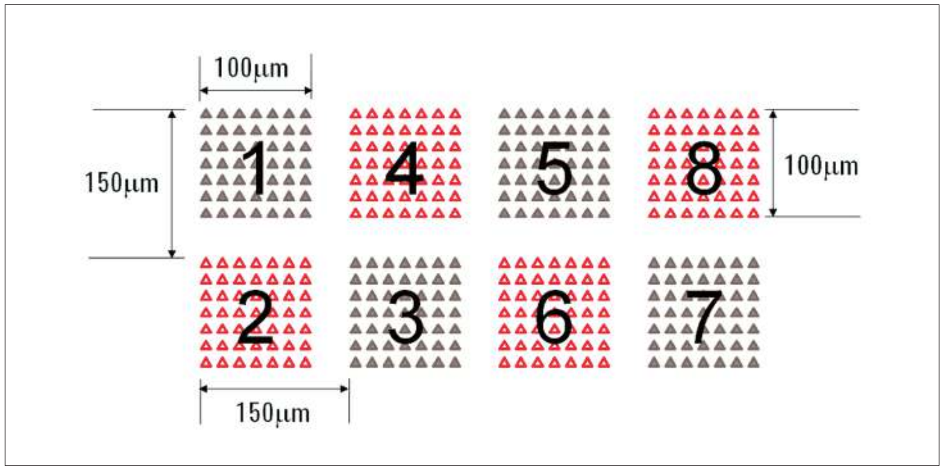

Figure 1. Schematic of indentation testing performed on each material. Observation set 1 is shown in gray; observation set 2 is shown in red. Bold numbers correspond to the order in which each array was performed. Observation sets were deliberately interlaced in order to randomize the influence of uncontrolled variables such as testing temperature, wafer location, and surface elevation.

Experimental Method

Samples

Four materials were tested in this work. All four were Cu/Ni multilayers with the independent variable being the thickness of the individual layers. The films were built upon a substrate of (111) silicon, and the individual copper and nickel layers perpetuated this (111) texture. The tested materials were:

- 1nm individual layers; 820nm total thickness.
- 5nm individual layers; 1200nm total thickness.
- 50nm individual layers; 1800nm total thickness.
- 100nm individual layers; 1800nm total thickness.

Equipment and Test Method

All testing was performed with a Keysight Technologies, Inc. G200 NanoIndenter having Express Test[1], NanoVision, and a DCM II fitted with a Berkovich indenter. The test method “Express Test to a Displacement” was employed, with the depth limit being 10% of the total film thickness. For example, the depth limit for the 1nm multilayer was 82nm.

Procedure

Figure 1 illustrates the testing procedure for each of the four samples. Two observation sets were acquired for each of the four materials, with each observation set comprising four 7x7 arrays of indents. The two observation sets were interlaced in order to randomize the influence of uncontrolled variables such as temperature, wafer location, or surface elevation. The total testing time for each sample (2 observation sets, each comprising 196 indents) was about 22minutes[2]. Testing was complete for all four samples in about 90 minutes.

1. Keysight’s Express Test option for G200 NanoIndenters implements traditional indentation testing in a revolutionary way in order to achieve unprecedented testing speeds. Express Test performs one complete indentation cycle per second, including approach, contact detection, load, unload, and movement to the next indentation site. Fifty indentations can be performed at fifty different sites in less than 50 seconds.
2. The total testing time is greater than 1 indent per second, because the head is repositioned for each array, and this takes about 2 minutes.

Material	Total Thickness nm	Indent Depth nm	Set	H(s) GPa	E(s) GPa
1nm alternating Cu/Ni layers	820	82	1	4.91 (0.21)	182.1 (9.5)
			2	4.90 (0.21)	181.0 (9.8)
5nm alternating Cu/Ni layers	1200	120	1	5.43 (0.20)	174.7 (5.6)
			2	5.41 (0.20)	174.8 (5.9)
50nm alternating Cu/Ni layers	1800	180	1	4.03 (0.16)	174.6 (6.5)
			2	4.04 (0.16)	175.2 (6.5)
100nm alternating Cu/Ni layers	1800	180	1	3.71 (0.13)	183.2 (9.1)
			2	3.72 (0.13)	183.1 (7.0)

Table 1. Summary of results.

Results and Discussion

Table 1 summarizes the measured properties (Young's modulus and hardness), and the hardness results are plotted in Figure 2. With the exception of the 1nm multilayer, the hardness decreases with increasing layer thickness as expected. The 5nm multilayer has the greatest hardness (5.42+0.2GPa), which is 46% greater than the hardness of the 100nm multilayer. For the 1nm multilayer, it is possible that the individual layers are so thin that some coherence exists between them – more refined microscopy could shed light on
this result.

Whereas hardness quantifies resistance to plastic deformation, Young's modulus quantifies elasticity. Varying grain size generally does not give rise to mechanisms for varying elasticity, and so it is not surprising that the measured Young's modulus is quite consistent among these four materials.

Comparing results from each observation set reveals the extraordinary repeatability of these measurements. Average results from each observation set differ by no more than 0.6%. Standard deviations are less than 5% of the mean and are also extraordinarily repeatable.

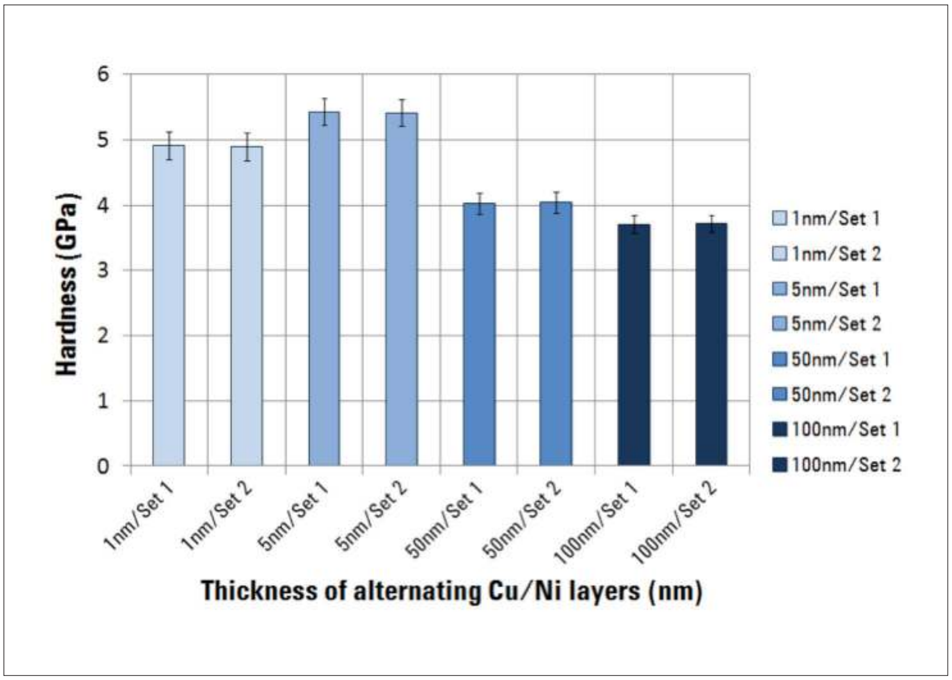

Figure 2. Hardness of various copper-nickel multilayers as a function of individual-layer thickness. Each bar represents 196 individual indentations. Total testing time was 90 minutes.

Conclusions

An experimental method is presented for rapidly and rigorously measuring the mechanical properties of nano-structured metals. The method is applied to the characterization of copper-nickel multilayers wherein individual layers vary in thickness between 1nm and 100nm. As expected, layer thickness has little influence on the Young's modulus but strongly influences the hardness. The 5nm-multilayer had the highest hardness of 5.42+0.20GPa, which was almost 50% greater than the hardness of the 100nm-multilayer. Extraordinary repeatability is demonstrated by comparing results from two interlaced observation sets for each material.

Nano Mechanical Systems from Keysight Technologies

Keysight Technologies, the premier measurement company, offers high-precision, modular nano-measurement solutions for research, industry, and education. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Keysight's leading-edge R&D laboratories ensure the continued, timely introduction and optimization of innovative, easy-to-use nanomechanical system technologies.

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