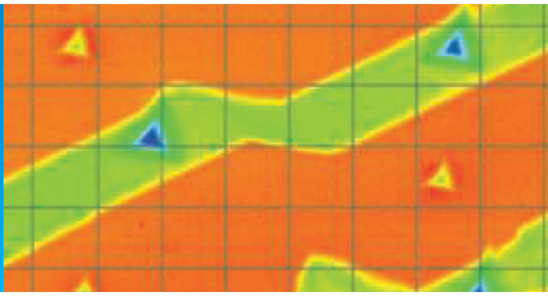
Application Note

Nanoindentation of a Multiphase Composite with NanoVision



Introduction

Modern materials engineers can create materials with very fine microstructures. In the case of multiphase composites, the mechanical properties of each phase will affect the overall performance of the material. Therefore, it is important to be able to accurately locate and test each phase. Using the NanoVision imaging option for the Nano Indenter® G200 system, a multiphase material has been tested to determine the hardness and modulus of each phase present.

Sample Preparation and Imaging

The sample, obtained from Oak Ridge National Laboratory (ORNL), is a lamellar eutectic alloy. The sample was directionally solidified to achieve an elegant microstructure. The primary phase is chromium silicide, Cr3Si, and the secondary phase is a chromium-rich solid solution.[1]

NanoVision generates surface images by rastering the sample beneath the indenter tip while applying a small, constant force to the surface. Because the indenter is constrained to apply a constant force to the surface, it follows the surface profile as the sample moves underneath it. These profile data are then assembled to generate a topographic image of the surface. The image can be leveled and otherwise manipulated using sophisticated image-analysis tools in order to reveal and emphasize subtle surface features. The X-Y translation system that accomplishes the rastering uses piezo-actuation with closed-loop control to achieve a positioning resolution of 0.5nm.

The Nano Indenter G200 instrument has a standard XP head and an optional high-resolution DCM 2 (Dynamic Contact Module) head. (The “head” is the sub-system that imposes force and measures displacement normal to the surface.) Either head can be used with the NanoVision option, but the DCM allows for faster scanning because it has a smaller moving mass. In this work, a DCM head fitted with a Berkovich indenter tip was used for both image generation and indentation.

The image in Figure 1 is a 50µm x 50µm scan of the multiphase composite. The boxed area in Figure 1 was rescanned to produce a more detailed image of the phases, as seen in Figure 2. From Figure 2, four different locations in each phase were selected to determine mechanical properties via nanoindentation.

Indentation

Indentation experiments were performed using the Continuous Stiffness Measurement option (CSM). With this option, Young’s (elastic) modulus and hardness are measured as a continuous function of penetration. Without the CSM option, measurements of elastic modulus and hardness can only be achieved at the maximum penetration depth. The first four indents were made in the relatively wide chromium silicide phase of the material — two indents in each of two bands. The second set of four indents was made in the chromium-rich solid solution phase. Force was applied using a constant strain rate of 0.2/sec to a maximum load of approximately 18mN.

Figure 3 shows the surface of the sample, with the same dimensions as Figure 2, after the eight indents have been made.

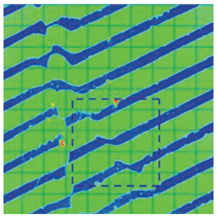


Figure 1. A 50µm x 50µm scan of the multiphase material.

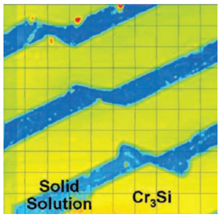


Figure 2. A 20µm x 20µm scan of the multiphase material.

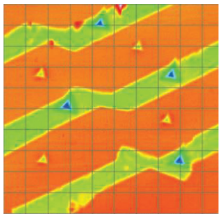


Figure 3. A 20µm x 20µm scan of the multiphase material after indentation.

From the modulus and hardness curves as a function of indentation depth (Figures 4 and 5, respectively), the mechanical properties of the two materials are shown to be very distinct. The modulus of the chromium silicide phase is approximately 75GPa higher than the modulus of the solid solution phase. Similarly, the hardness of the chromium silicide phase is approximately four times higher than the modulus of the solid solution. The differing hardness values can also be seen in Figure 3: the depth of the indents in the chromium silicide is shallower due to the higher hardness of that phase. The results demonstrate the repeatability of the instrument.

A point of interest is the sharp slope of the hardness curve followed by a drastic drop and then leveling of the hardness for two of the indents in the chromium silicide phase. Both of these indents were made in the same band. The sudden drop in hardness may be associated with initial yielding of the material. Such behavior may be explained by a lack of mobile dislocations in the vicinity of the indenter as the initial load is applied. As the indenter proceeds into the material, the volume of affected material grows and eventually incorporates mobile dislocations. Plasticity ensues, and the strength drops.[2]

Conclusion

The NanoVision option and its closed-loop position control allowed the different phases of the material to be distinguished with clarity and the probe to be placed accurately. Since the two phases were made obvious from the image captured by the scan, they were able to be probed effectively, and repeatably, to determine the mechanical properties of the distinct phases.

Technology and Applications

The Nano Indenter G200 system is powered by electromagnetic actuation to achieve high dynamic range in force and displacement. The instrument's design avoids lateral displacement artifacts, while software fully compensates for any drift in force. The DCM II offers 30mN maximum loading capability, easy tip exchange for quick removal and installation of application-specific tips, and a full 70µm range of indenter travel. Using the Nano Indenter G200 system, researchers can measure Young's modulus and hardness in compliance with ISO 14577. Deformation can be measured over six orders of magnitude (from nanometers to millimeters). Applications include semiconductor, thin films, and MEMs (wafer applications); hard coatings and DLC (diamond-like carbon) films; composite materials, fibers and polymers; metals and ceramics; and biomaterials.

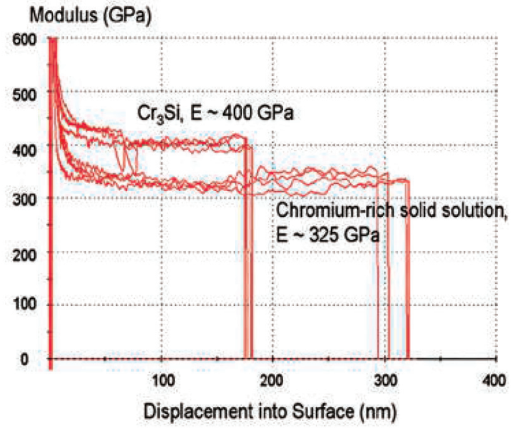


Figure 4. The distinct modulus curves for the two different phases of the material.

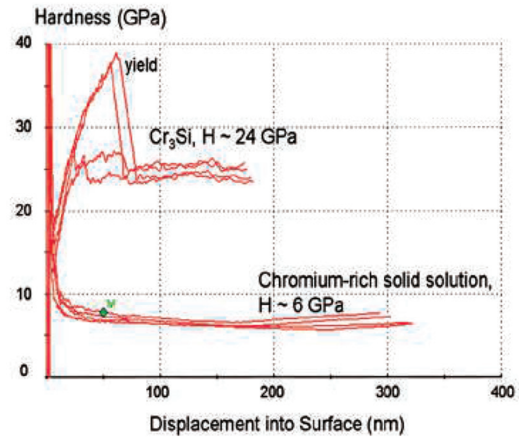


Figure 5. The distinct hardness curves for the two different phases of the material.

References

1. Bei H., George E.P., Kenik E.A., and Pharr G.M., (2003) "Directional solidification and microstructures of near-eutectic Cr-Cr3Si alloys", Acta Materialia, 51, 6241–6252.

2. Bei H., George E.P., and Pharr G.M., manuscript in preparation.

Acknowledgements

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KLA SUPPORT

Maintaining system productivity is an integral part of KLA's yield optimization solution. Efforts in this area include system maintenance, global supply chain management, cost reduction and obsolescence mitigation, system relocation, performance and productivity enhancements, and certified tool resale.

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