Nanoindentation of a Multiphase Composite with NanoVision

Application Note

Introduction

Modern materials engineers 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 extremely important to be able to accurately locate and test a phase. Using the NanoVision imaging option for the G200 Nano Indenter, 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, Cr_3Si, 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 topological 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 0.5 nm.

The Agilent G200 Nano Indenter has a standard XP head and an optional high-resolution DCM 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 was used for both image generation and indentation

Agilent Technologies

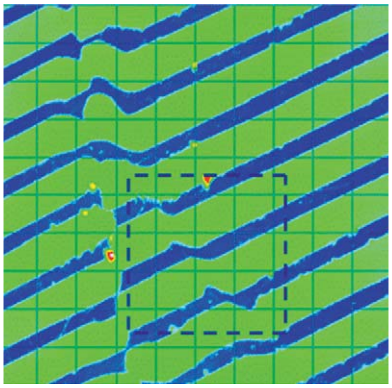

Figure 1. A 50 x 50 µm scan of the multiphase material.

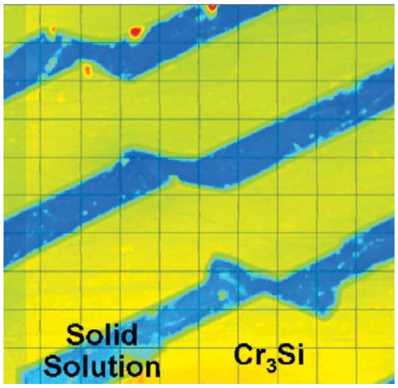

Figure 2. A 20 x 20 µm scan of the multiphase material.

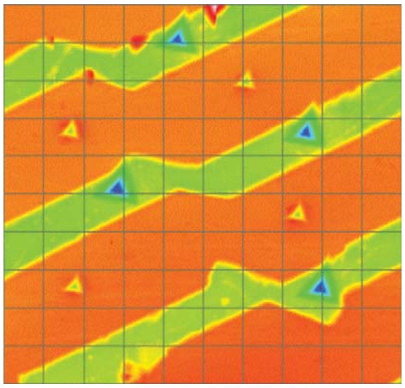

Figure 3. A 20 x 20 µm scan of the multiphase material after indentation.

The image in Figure 1 is a 50 µm x 50 µm 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 Option (CSM). With this option, elastic modulus and hardness are measured as a continuous function of penetration. (Without this 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 four indents were 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. The very high resolution and accuracy of indent placement that NanoVision provides is clear in this image.

Discussions

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 75 GPa 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 clearly show the repeatability of the instrument.

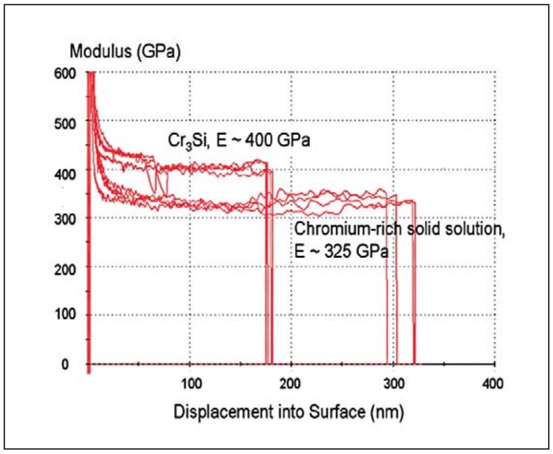

Figure 4. The distinct modulus curves for the two different phases of the material.

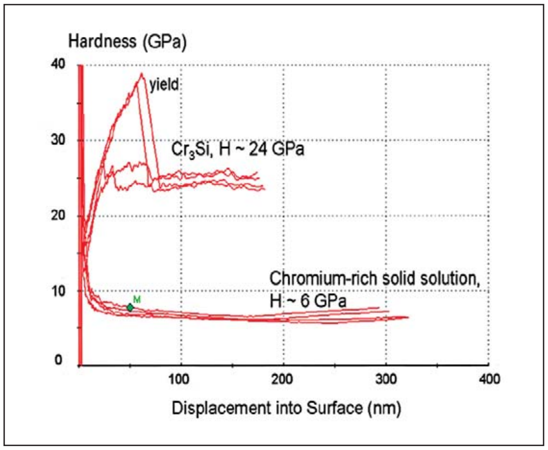

Figure 5. The distinct hardness curves for the two different phases of the material.

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

Without the NanoVision option and its closed-loop position control, the different phases of the material could not have been distinguished with such clarity and the probe could not have been placed as 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 is powered by electromagnetic actuation to achieve unparalleled dynamic range in force and displacement. The instrument's unique design avoids lateral displacement artifacts, while software compensates fully for any drift in force. The DCM II offers impressive performance and advantages including 3x higher loading capability (30 mN max load), easy tip exchange for quick removal and installation of application-specific tips, and a full 70 µm range of indenter travel. Using the G200, 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 films; composite materials, fibers, and polymers; metals and ceramics; and biomaterials and biology.

References

1. Bei H, George EP, Kenik EA, and Pharr GM, (2003) "Directional solidification and microstructures of near-eutectic Cr-Cr_3Si alloys", Acta Materialia, 51, 6241-6252.
2. Bei H, George EP, and Pharr GM, manuscript in preparation.

Acknowledgements

Agilent Technologies would like to thank Professor Pharr of the University of Tennessee and ORNL and Hongbin Bei of ORNL for the sample.

Nano Mechanical Systems from Agilent Technologies

Agilent 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. Agilent's leading-edge R&D laboratories ensure the continued, timely introduction and optimization of innovative, easy-to-use nanomechanical system technologies.

www.agilent.com/find/nanoindenter

Americas	
Canada	(877) 894 4414
Latin America	305 269 7500
United States	(800) 829 4444

Asia Pacific	
Australia	1 800 629 485
China	800 810 0189
Hong Kong	800 938 693
India	1 800 112 929
Japan	0120 (421) 345
Korea	080 769 0800
Malaysia	1 800 888 848
Singapore	1 800 375 8100
Taiwan	0800 047 866
Thailand	1 800 226 008

Europe & Middle East	
Austria	43 (0) 1 360 277 1571
Belgium	32 (0) 2 404 93 40
Denmark	45 70 13 15 15
Finland	358 (0) 10 855 2100
France	0825 010 700* *0.125 €/minute
Germany	49 (0) 7031 464 6333
Ireland	1890 924 204
Israel	972-3-9288-504/544
Italy	39 02 92 60 8484
Netherlands	31 (0) 20 547 2111
Spain	34 (91) 631 3300
Sweden	0200-88 22 55
Switzerland	0800 80 53 53
United Kingdom	44 (0) 118 9276201

Other European Countries:
www.agilent.com/find/contactus

Product specifications and descriptions in this document subject to change without notice.

© Agilent Technologies, Inc. 2009

Printed in USA, November 11, 2009
5990-5022EN

Agilent Technologies