

## Application Note #1505

# Nanoindentation of Duplex Stainless Steel with e-Flash<sup>FS</sup> and Hysitron PI 88 SEM PicoIndenter

Duplex 2205 is a two-phase, ferritic ( $\alpha$ ) and austenitic ( $\gamma$ ) stainless steel alloyed with 22% Cr, 3% Mo, and 5 to 6% Ni. It is characterized by good fatigue strength, outstanding resistance to stress corrosion cracking, and general corrosion in severe environments. Duplex steel offers very high yield strength compared to the standard austenitic stainless steel. The main application for duplex steel is chemical processing, transport, storage, pressure vessels, tanks, oil field piping, and heat exchangers. When these steels are exposed to high temperatures, deleterious intermetallic phases like Chi and Sigma can form leading to a decrease in quality of mechanical and corrosion properties. This application note discusses research performed to confirm the presence of Chi and Sigma phases as well as their distribution in the Ferrite/Austenite matrix of an annealed duplex steel using electron backscatter diffraction (EBSD). The mechanical properties of individual Ferrite/Austenite phases were then measured via in-situ nanoindentation with the Hysitron<sup>®</sup> PI 88 SEM PicoIndenter<sup>®</sup>.

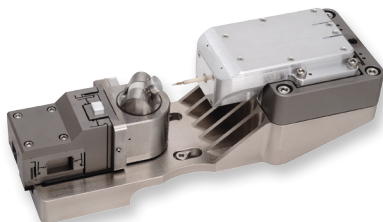


Figure 1. Hysitron PI 88 SEM PicoIndenter.

### Hysitron PI 88 SEM PicoIndenter

The Hysitron PI 88 SEM PicoIndenter is a depth-sensing nanomechanical test instrument that is specifically designed to leverage the advanced imaging capabilities of modern scanning electron microscopes (SEM, FIB/SEM). When equipped with the optional tilt and rotation stages, the Hysitron PI 88 features flexible sample positioning with five degrees of freedom (X, Y, Z, tilt, rotation) giving the user the ability to align the sample with an ion beam for sample preparation or detectors such as EBSD, EDS, or WDS to obtain a deeper understanding of a material's mechanical response.

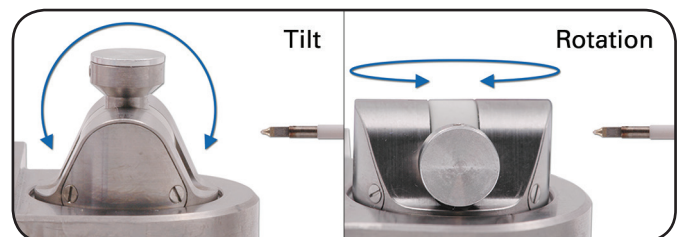


Figure 2. A schematic of the optional rotation and tilt stages for the Hysitron PI 88 SEM PicoIndenter.

## e-Flash<sup>FS</sup> EBSD Detector™

The EBSD results shown in this document were acquired with the new e-Flash<sup>FS</sup> high-speed detector. Its excellent speed and signal sensitivity combined with innovative features like the ARGUS FSE/BSE imaging system and

in-situ tilting make e-Flash<sup>FS</sup> the perfect choice for in-situ experiments and a great complement to high-resolution mechanical testing such as nanoindentation.

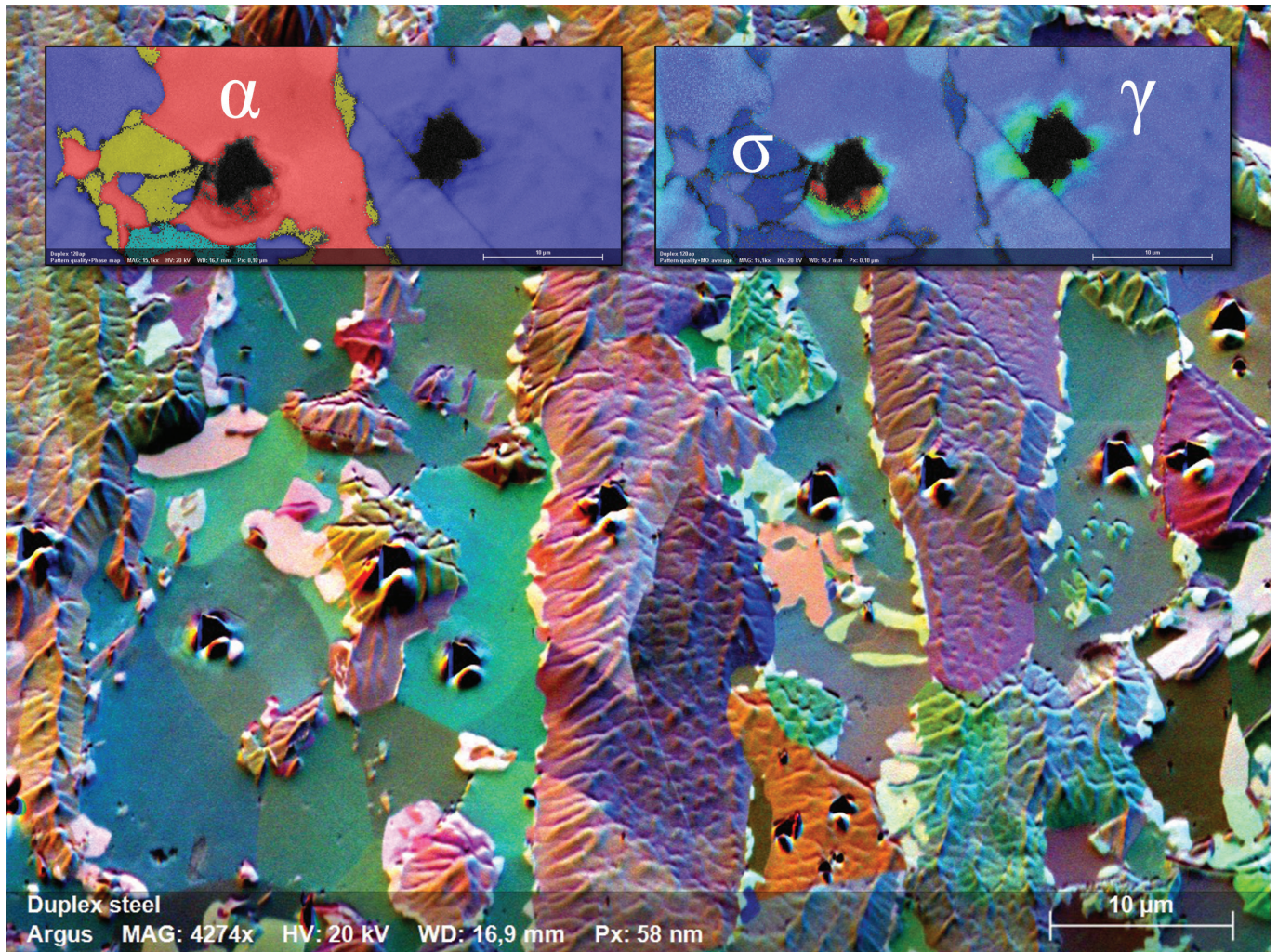


Figure 3. Color coded ARGUS FSE image showing orientation contrast in one of the indented areas; EBSD phase map (top left) with Ferrite (red), Austenite (blue), Sigma (yellow) and Chi (aqua) phases; grain orientation spread (GOS) map (top-right) indicating that the plastic strain field developed by the indent does not cross from the Ferrite grain into the much harder Sigma phase grain.

## Experimental Procedure

A polished sample of duplex steel was attached to a microscopy stub with conductive adhesive, and mechanically secured in the staging of the Hysitron PI 88. Using the optional tilt and rotation capabilities of the Hysitron PI 88, the sample was aligned with the  $e^-$  Flash detector for grain and phase mapping using ESPRIT 2.1 software. After mapping, the sample was re-oriented with

the indentation probe, and load-controlled nanoindentation tests were conducted to peak loads of 1, 5, and 20 mN. After mechanical tests, the sample was again aligned with the EBSD detector to map the regions where the indentations were performed (see FSE image in Figure 3 and EBSD results in Figure 4).

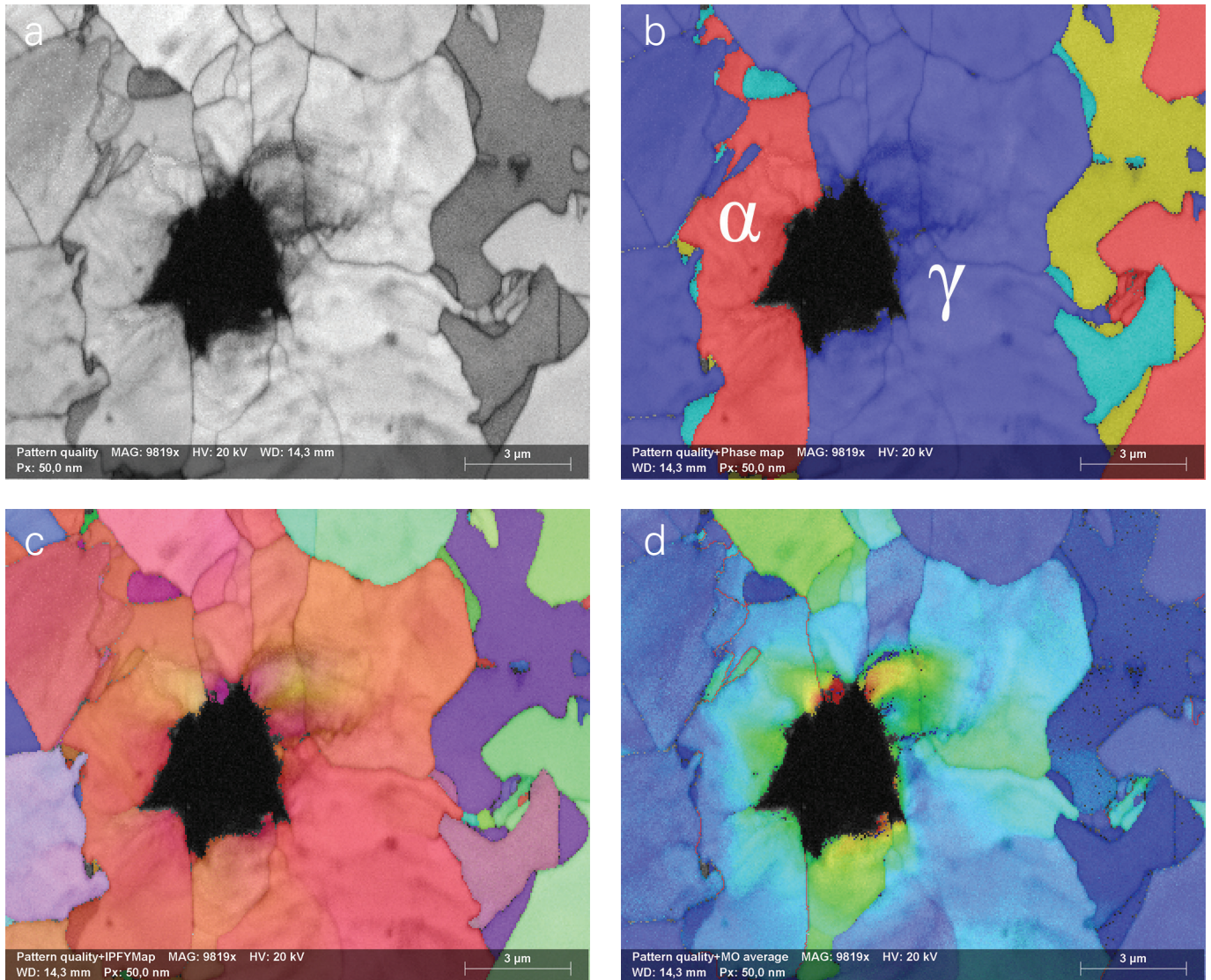


Figure 4. (a) Pattern quality map, (b) phase map, (c) orientation (IPFy) map, and (d) GOS map. Colors indicate orientation spread in degrees from 0° (blue) up to 15° (red) EBSD results.

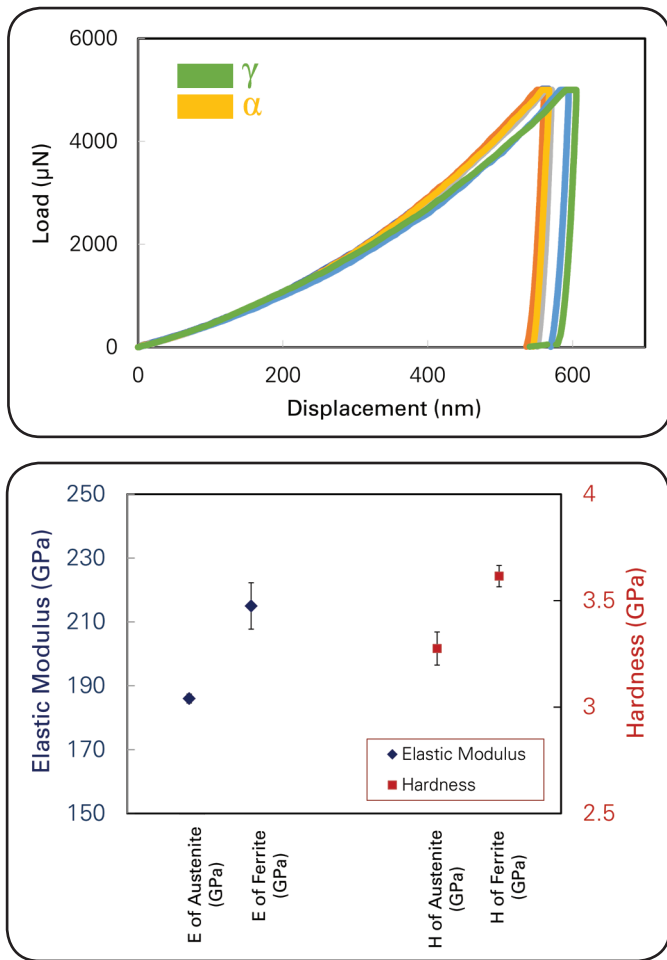


Figure 5. Typical P-h curves from austenitic ( $\gamma$ ) and ferritic ( $\alpha$ ) phases (top), elastic modulus and hardness variation in ferrite and austenite (bottom).

## EBSD-Enhanced Nanoindentation Results

Figure 4 shows the EBSD results acquired from an area containing an indent made at the boundary between a Ferrite ( $\alpha$ ) grain and an Austenite ( $\gamma$ ) grain. Results indicate that the amount of plastic strain field developed by the indent is much larger in the Austenite, i.e., the softer phase.

The local elastic and plastic properties of the phase domains were determined from load-displacement curves by analyzing 5 to 8 indentations from each phase. The elastic moduli ( $E$ ) were measured to be  $215 \pm 7.2$  GPa and  $186 \pm 1.4$  GPa for the ferritic and austenitic phases, respectively. The hardness values ( $H$ ) show a similar trend with values of  $3.6 \pm 0.05$  GPa for ferrite and  $3.2 \pm 0.07$  GPa for austenite. Although the actual numbers vary slightly with the results reports by Gadelrab et al., Campos et al., and Guo et al., the relative difference in the mechanical properties of the two phases agrees well with the trends reported in the previous studies. Also, the results confirm the expected enhancement provided by solid solution hardening of Ni and Mo in the ferrite phase.

Using the Hysitron PI 88 equipped with tilt and rotation stages in conjunction with QUANTAX EBSD system enables a more robust characterization of metallic materials by combining high-resolution phase and grain orientation mapping capabilities with targeted nanomechanical property measurements. This combination could also be used to extend the scope of research related to other advanced textured, anisotropic, or multi-phase materials.

## References

1. K. Gadelrab, G. Li, M. Chiesa, and T. Souier, *J. Mater. Res.*, 27 (2012), 1573.
2. M. Campos, A. Bautista, D. Caceres, J. Abenojar, J.M. Torralba, *J. of the European Ceramic Society* 23 (2003) 2813.
3. L. Q. Guo, M. C. Lin, L. J. Qiao, A. A. Volinsky, *Applied Surface Sci.*, 287 (2013) 499.