EBSD

Identification of complex phases in strontium modified silicon alloys

Application Note # EBSD-05

Introduction

Duplex 2205 is a two-phase, ferritic (α) and austenitic (γ) stainless steel alloyed with 22% Cr, 3% Mo, and 5–6% 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 applications for duplex steel are chemical processing, transport, storage, pressure vessels, tanks, oil field piping, and heat exchangers.

When duplex 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.

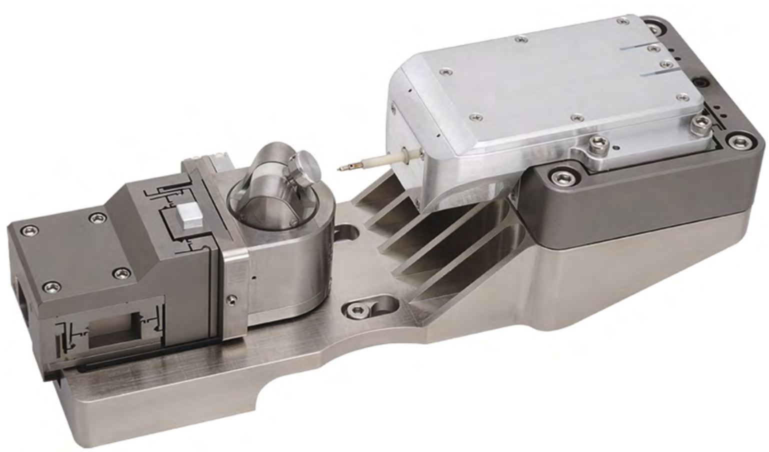
The present work aims to confirm the presence of chi and sigma phases as well as their distribution in the ferrite/ austenite matrix of an annealed duplex steel using EBSD. The mechanical properties of individual ferrite/austenite phases were measured via in-situ nanoindentation with Bruker's Hysitron® PI 88 SEM PicoIndenter®.

Instruments and experimental procedure

Hysitron PI 88 SEM PicoIndenter

The Hysitron PI 88 SEM PicoIndenter, shown in Figure 1, 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).

Figure 1
Bruker's Hysitron PI 88 SEM PicoIndenter



When equipped with the optional tilt and rotation stages, the PI 88 SEM PicoIndenter 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 with detectors such as EBSD, EDS, or WDS to obtain a deeper understanding of the material's mechanical response.

e^{-}Flash^{FS} EBSD detector

The EBSD results shown in this application note were acquired with the new e^{-}Flash^{FS} high speed EBSD detector. Its excellent speed and signal sensitivity combined with innovative features like the ARGUSTM FSE/BSE imaging system and in-situ tilting make the e^{-}Flash^{FS} the perfect choice for in-situ experiments and a great complement to high resolution mechanical testing such as nanoindentation.

Sample

The investigated sample is a two-phase ferritic (α) and austenitic (γ) stainless steel (Duplex 2205). Prior to the experiment the sample was annealed for 8 hours at ~850°C to induce the formation of deleterious intermetallic phases like sigma and chi. The polished duplex steel sample was attached to a pin stub with silver paint which was afterwards mechanically secured to the stage of the PI 88 SEM PicoIndenter.

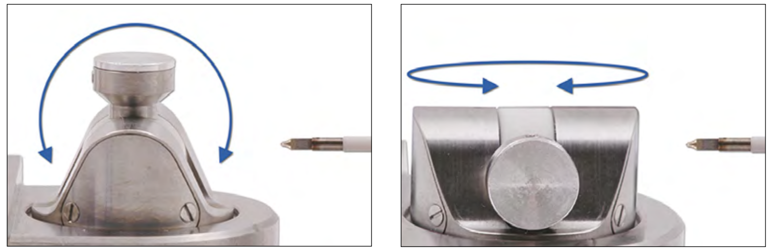


Figure 2
Schematic representation of a) the tilt stage and b) the rotation stage, available accessories for the Hysitron PI 88 SEM PicoIndenter.

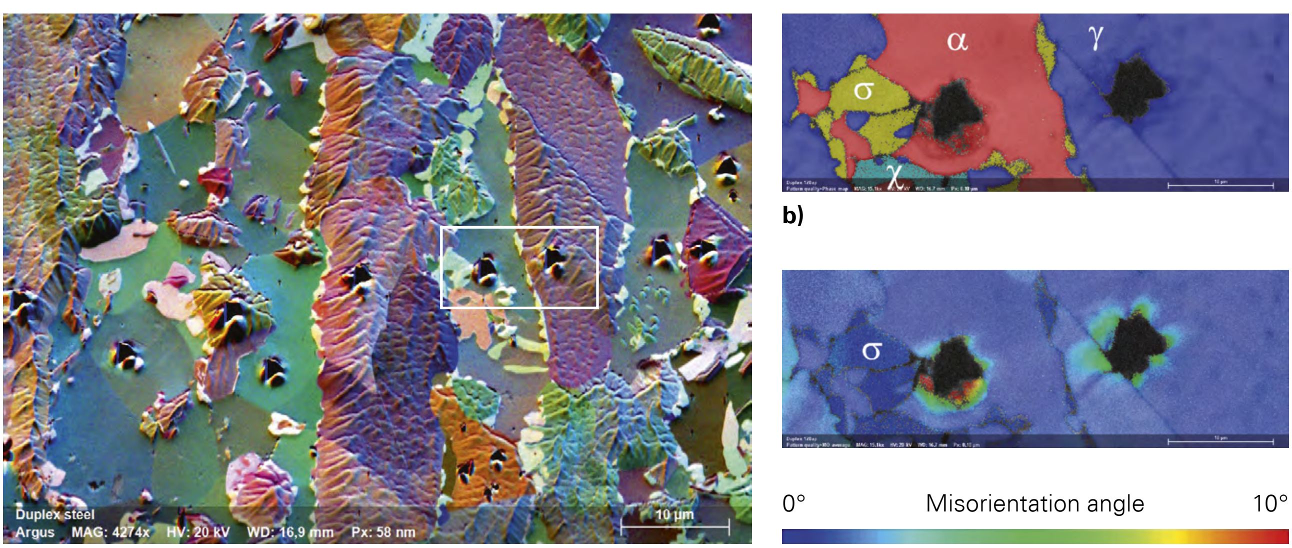
Using the optional tilt and rotation capabilities of the PI 88 SEM PicoIndenter, the sample was aligned with the e^{-}Flash^{FS} detector for grain and phase mapping using the ESPRIT 2.1 software.

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 e^{-}Flash^{FS} EBSD detector to map the regions where the indentations were performed.

EBSD Results

Figure 3a displays the ARGUS™ FSE image showing the orientation contrast in one of the indented areas. Figures 3b and 3c show the phase map and the grain orientation spread (GOS) map of the area highlighted by the white rectangle in Figure 3a. The GOS map indicates

Figure 3
a) Color coded ARGUS™ FSE image showing the orientation contrast in one of the indented areas; b) EBSD phase map with the phases ferrite (α, red), austenite (γ, blue), sigma (σ, yellow), and chi (χ, aqua); c) grain orientation spread (GOS) map with legend.



that the plastic strain field developed by the indent does not cross from the ferrite grain into the much harder sigma phase grain.

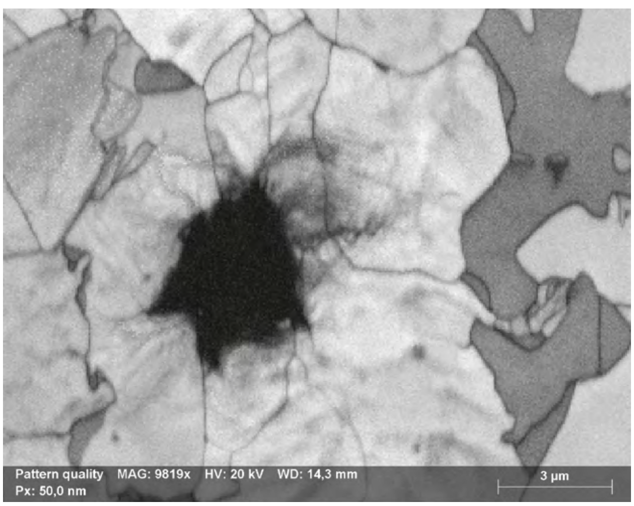
Figure 4 shows the EBSD results acquired from an area containing an indent made at the boundary between a ferrite (α) grain and an austenite (γ) grain. The results indicate that the amount of plastic deformation induced by the indent is much larger in the austenite, i.e. the softer phase.

EBSD-Enhanced Nanoindentation Results

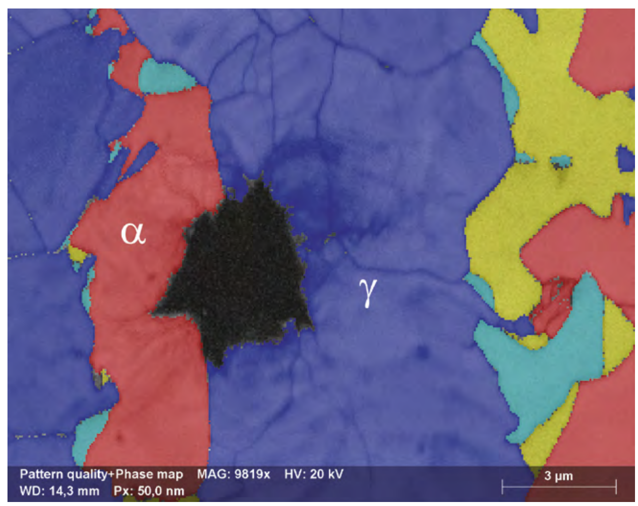
The local elastic and plastic properties of the phase domains were determined from load-displacement curves by analyzing five to eight indentations from each phase (Figure 5a). The elastic moduli (E) were measured to be 215 ± 7.2 GPa and 186 ± 1.4 GPa for the ferritic and austenitic phases, respectively (Figure 5b).

The hardness values (H) show a similar trend with values of 3.6 ± 0.05 GPa for ferrite and 3.2 ± 0.07 GPa for austenite (Figure 5b). Although the actual numbers vary slightly from the results reported 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.

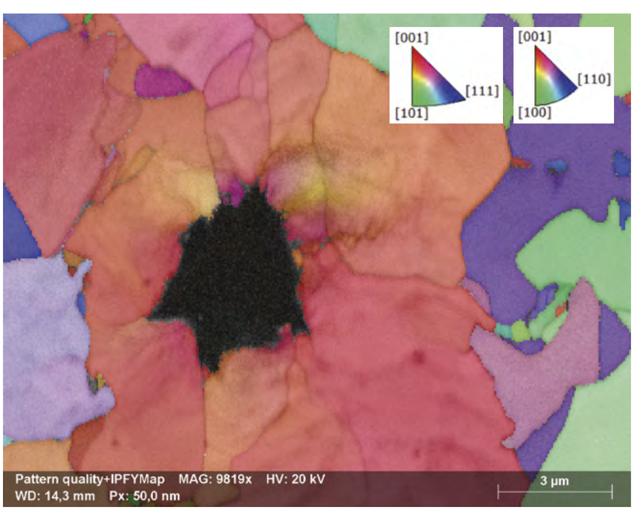
Also, the results confirm the expected enhancement provided by solid solution hardening of Ni and Mo in the ferrite phase.



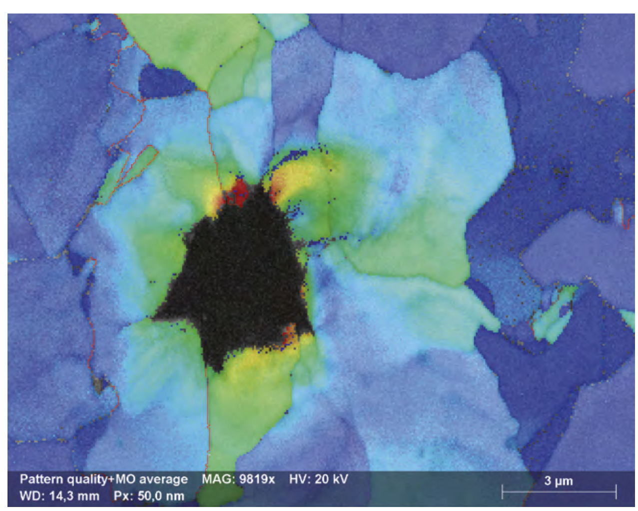
a)



b)



c)



d)

Figure 4

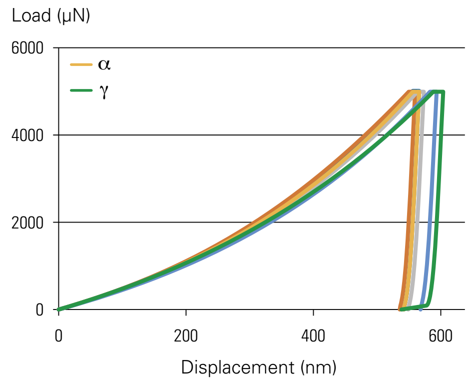
a) Pattern quality map;
b) phase map;
c) orientation map (IPFy),
d) GOS map.
The colors in d) indicate the orientation spread in degrees from 0° (blue) up to 15° (red)."

Conclusion

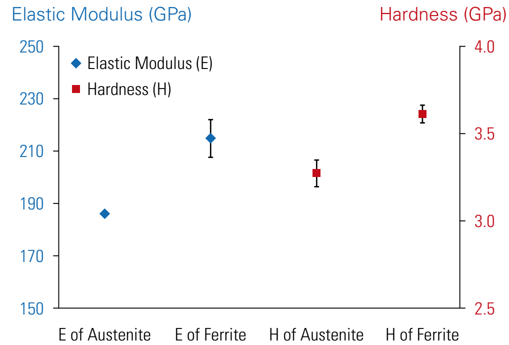
Using Bruker's Hysitron PI 88 SEM PicoIndenter equipped with tilt and rotation stages in conjunction with the QUANTAX EBSD system enables a more robust characterization of metallic materials by combining high resolution phase and grain orientation mapping capabilities with targeted nanomechanical property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

The extension to other applications includes but is not limited to the study of:

- deformation mechanisms in metallic materials by 3D EBSD/EDS analysis of the deformed volume underneath the indent
- residual strain in a cantilever bent using the picoindenter
- in-situ tensile testing of electron transparent samples with the Push-to-Pull and OPTIMUS TKD options.



a)



b)

Figure 5

a) Typical p-h curves from ferritic (α) and austenitic (γ) phases, b) elastic modulus and hardness variation in ferrite and austenite.

References

[1] K. Gadelrap, G. Li, M. Chiesa, T. Souier, Journal of Material Research, Vol. 27, 2012, p. 1573.

[2] M. Campos, A. Bautista, D. Caceres, J. Abenojar, J.M. Torralba, Journal of European Ceramic Society, Vol. 23, 2003, p. 2813

[3] L.Q. Guo, M.C. Lin, L.J. Qiao, A.A. Volinsky, Applied Surface Science, Vol. 287, 2013, p. 499.

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