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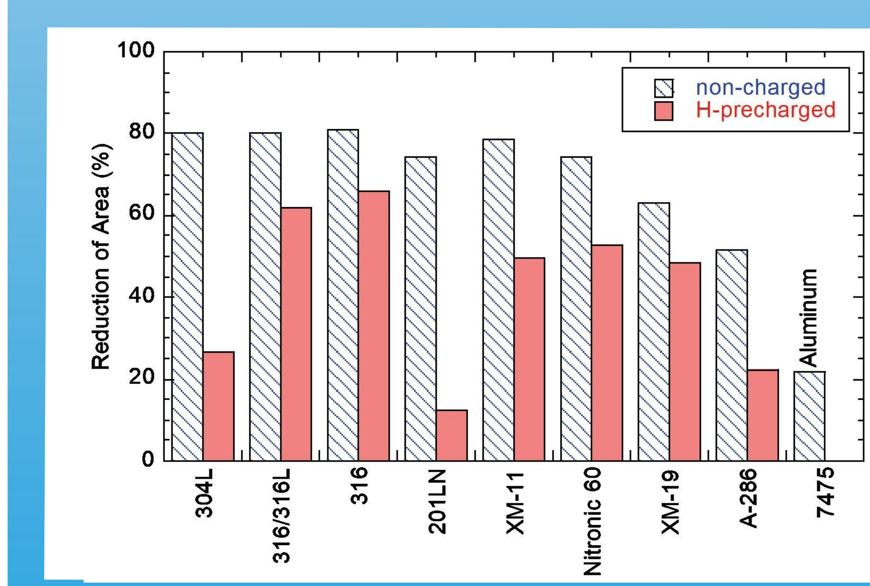
Ion-Beam Induced Structural Transformations in Austenitic Stainless Steels

Joshua D. Sugar[1], Doug Medlin[1], and Suzy M. Vitale[2]

[1]Sandia National Laboratories, Livermore, CA, 94550
[2]Carnegie Institution for Science, Washington D.C., 20015

Introduction

Austenitic stainless steels are an important structural material used in harsh environments such as high-pressure hydrogen. These stainless steels are less susceptible to embrittlement in hydrogen than other materials, which makes them important for green energy hydrogen-fuel based applications. Often, electron microscopy characterization is used to understand the interactions between hydrogen atoms and the microstructural features/defects present in these stainless steels. In particular, transmission electron microscopy (TEM) is important because it allows the direct observations of defects, but artifact-free samples are needed if the microstructural data is to be interpreted correctly. The focused ion beam-scanning electron microscope (FIB/SEM) is a common tool used to prepare microscopic samples because it allows site specificity and has a history of making samples appropriate for atomic resolution analysis. However, it has been shown that the incident ion beam can cause undesired phase transformations in these steels that alters the microstructure and obscures the appropriate interpretation of the data.



Thermal precharging: 300°C 140 MPa H2

The plot at the left shows the effect of hydrogen pre-charging on the ductility of several steel alloys. Some are more susceptible than others to embrittlement. Microstructural investigation of deformed microstructures informs the mechanisms of strengthening and embrittlement that can ultimately lead to reliable, high performing materials.

Plot reproduced from San Marchi, C, & Somerday, BP. "Comparison of Stainless Steels for High-Pressure Hydrogen Service." Proceedings of the ASME 2014 Pressure Vessels and Piping Conference. Volume 6B: Materials and Fabrication. Anaheim, California, USA. July 20–24, 2014. V06BT06A023. ASME. https://doi.org/10.1115/PVP2014-28811

Question

What parameters can we use in the FIB to minimize the formation of the BCC/BCT artifact phase and make high-fidelity samples?

Challenge

It has been documented in the literature that when energetic ion beams interact with these stainless steels, they can cause the formation of BCC/BCT phases from the FCC parent phase.

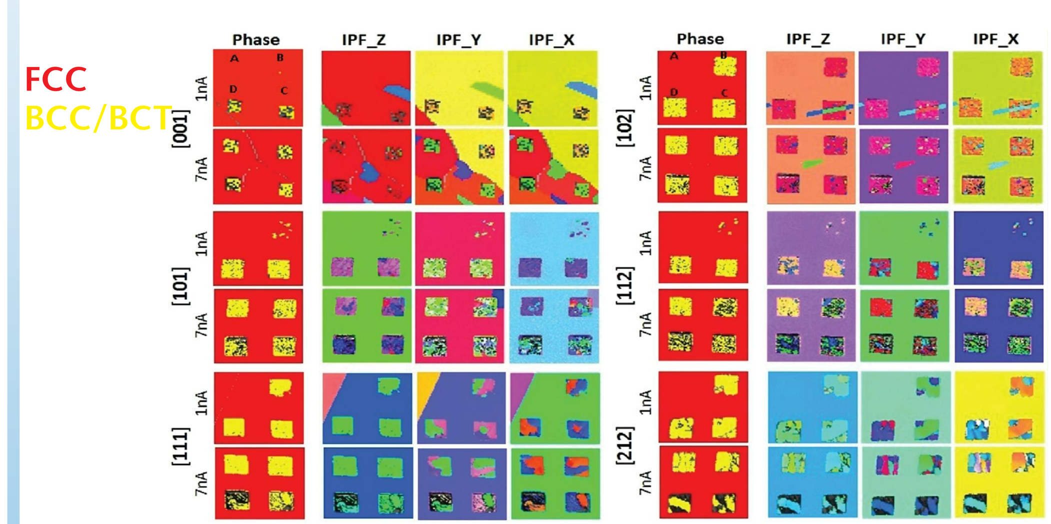


Figure from R.P. Babu, et al., Acta Materialia 120 (2016) showing orientation effects on the formation of the BCC phase (yellow) from the parent FCC phase (red) with 90° incident 30 kV Ga+ ions for 10, 30, 60, and 120 s (a, b, c, d, respectively).

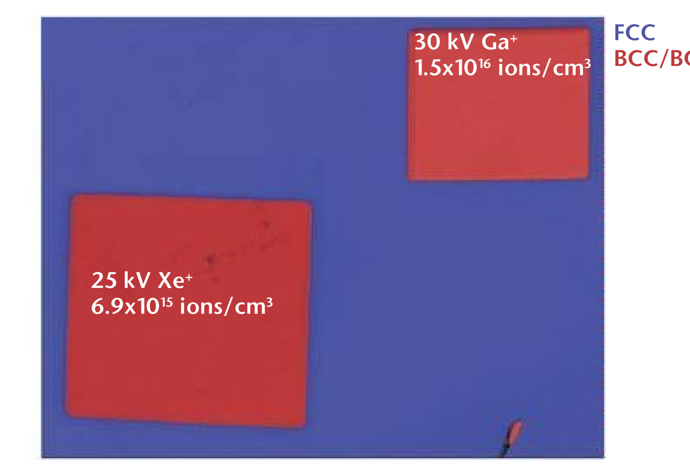
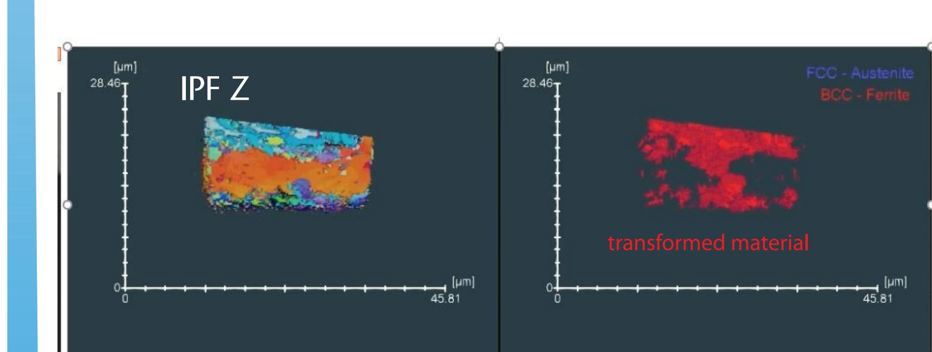
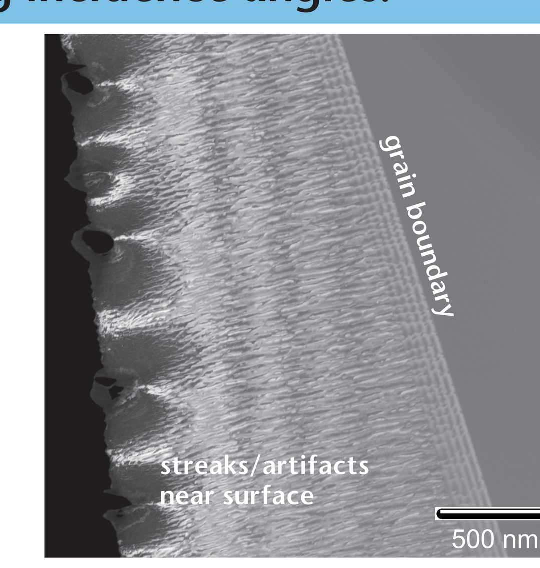


Figure from J.R. Michael, et al., Microsc Microanal 28 (2022) showing that both Ga and Xe ions can cause the FCC to BCC/BCT transformation to occur such that the effect is not solely chemical and due to Ga concentrations.

When performing techniques such as 3D EBSD or TEM sample preparation, this artifact can be particularly bad because of the large ion doses required to complete these specialized tasks, even at the typical glancing incidence angles.



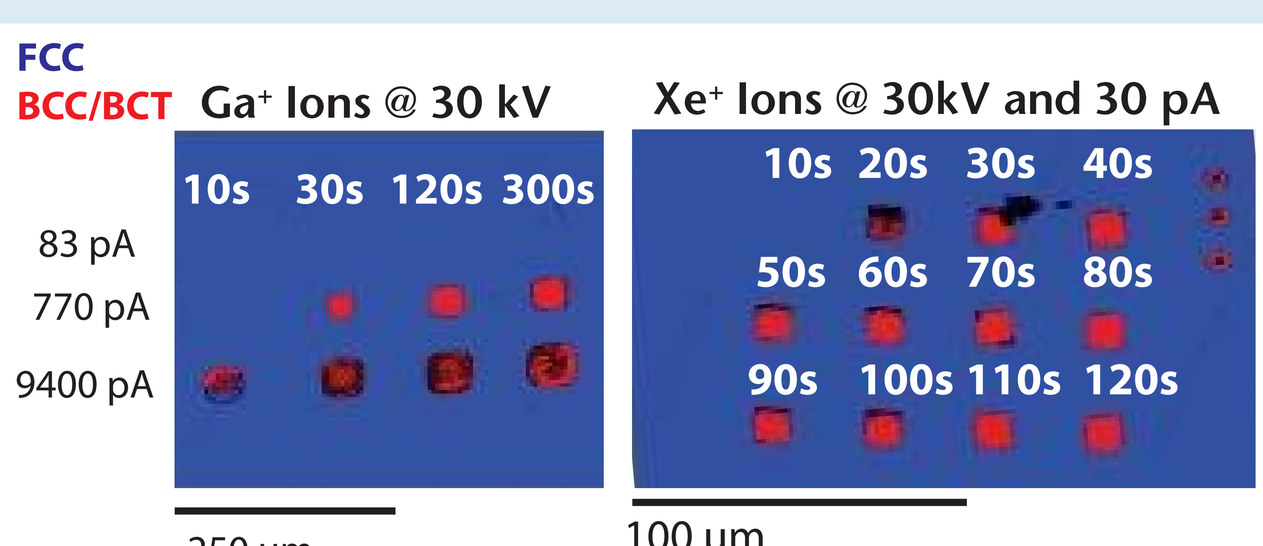
This 3D EBSD example shows a volume of material where a large amount has transformed near the edges of the analysis volume. The long milling times at glancing angle for each 3D slice of material are detrimental to preserving the native microstructure.



This diffraction contrast STEM image was taken with a 111 systematic row excited and shows streaks in the image. These streaks are likely thin regions at the surface of the lamella that have transformed from FCC to BCC/BCT.

Incident Ion Specie

Consistent with previous results[1], we find that this FCC to BCC/BCT transition happens regardless of whether Ga^+ or Xe^+ ions are used.



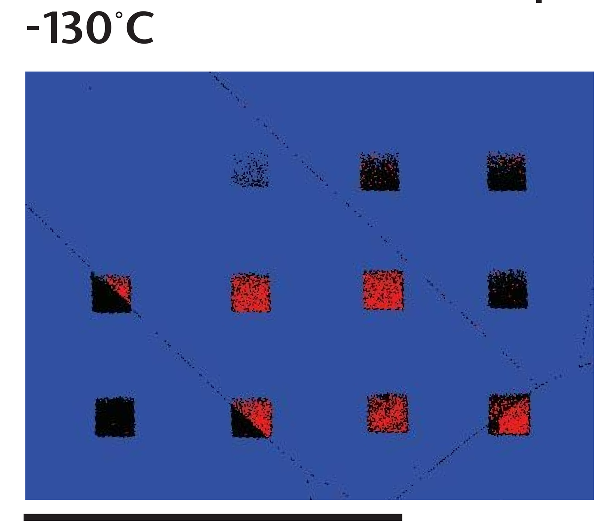
It is clear that the transformation mechanism is not due to a chemical effect of Ga doping. The transformation is sensitive to dose, and at low currents and low exposure times, the transformation does not occur. However, at any appreciable dose needed to remove material for TEM sample prep or 3D slice and view experiments, it is likely a transformation will occur. Therefore, simply using lower currents is not a feasible way to prevent the unwanted transformation.

[1] J.R. Michael, et al., Microsc Microanal 28 (2022),

Temperature

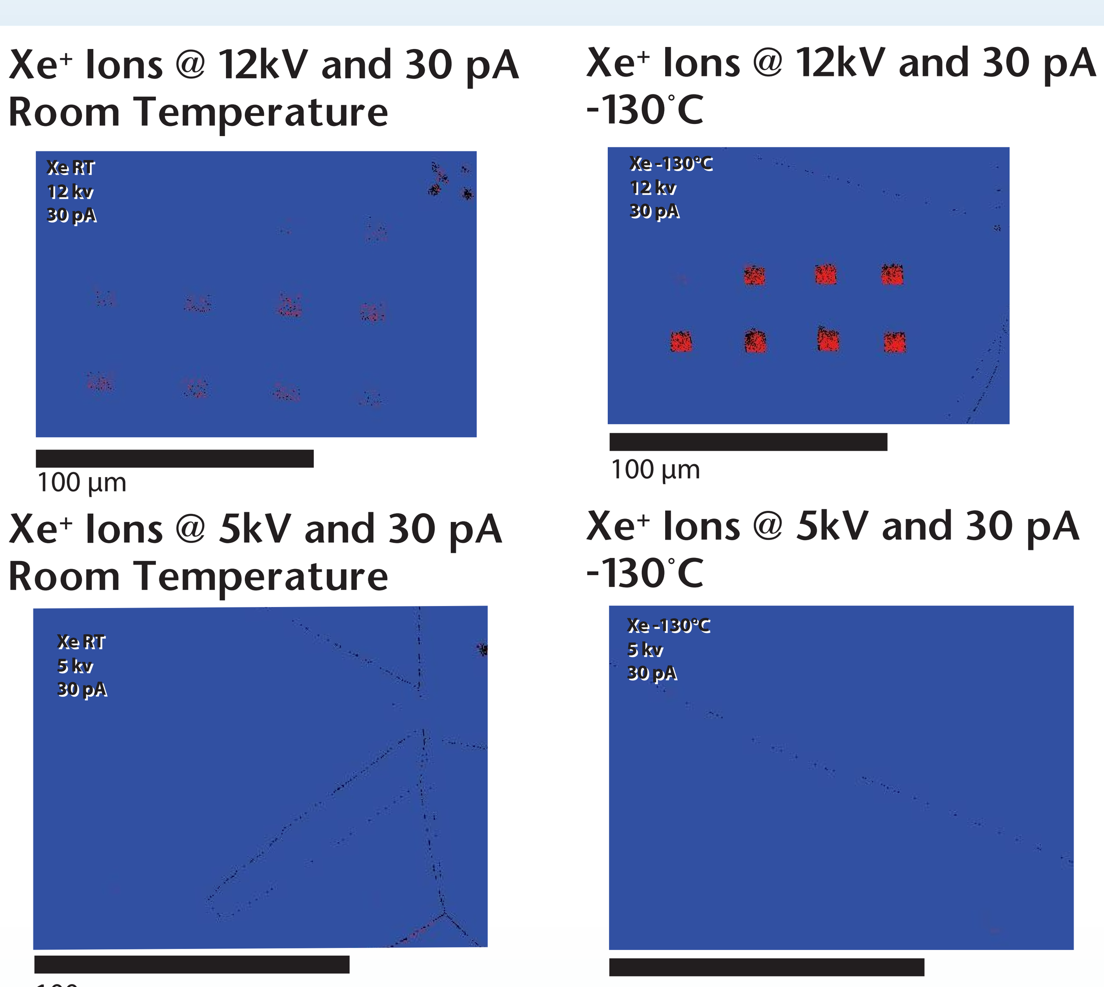
Experiments were also performed at cryogenic temperatures to investigate if the transformation could be inhibited at cold temperatures.

Xe^+ Ions @ 30kV and 30 pA -130°C



The results indicate that even at cryogenic temperatures (-130°C), the transformation occurs. There does not appear to be any reduction in the amount of transformed material at cryogenic temperatures.

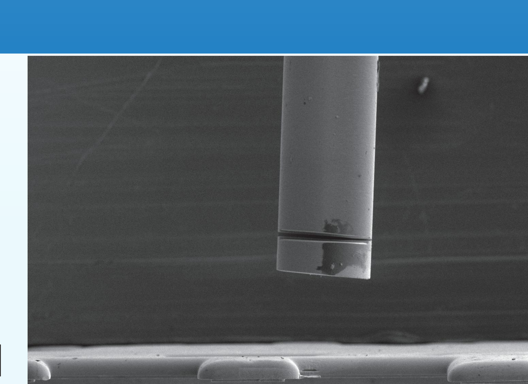
Accelerating Voltage



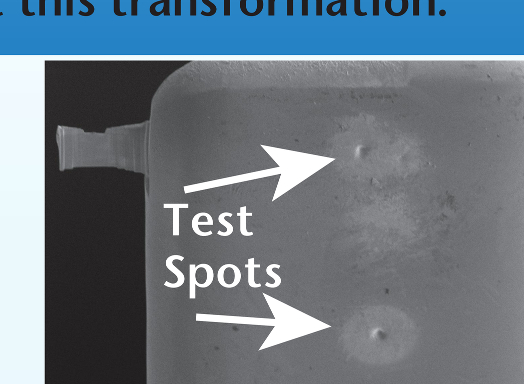
In addition to temperature, lower accelerating voltages were investigated, and they showed that less transformation does occur. At 12 kV, the cryogenic temperature seems to have exacerbated the transformation. Lower V_{ac} seem to be the most promising way to prevent this transformation.

Conclusions and Future Opportunities

The most promising method of reducing the FCC to BCC/BCT transformation appears to be using a lower accelerating voltage of incident ions. It may be possible to use low energy gas beams ionized by incident electrons to clean the surface of samples in situ in the FIB and prevent artifacts and unwanted transformed material. Removal of the damaged/transformed surface region with these low energy beams can reduce artifacts and result in pristine unaltered samples.



The image at the left shows the configuration of an Ar GIS in a Ga FIB for low energy cleaning of a TEM lamella. At the right, test spots are shown on the Cu grid that appear brighter than the surrounding material because the surface is cleaned with low energy Ar^+ ions.



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