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Using XeF2 FIB imaging to contrast and quantify precipitation in metal.

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Background incl. aims

Precipitation within a material is known to affect material properties, from mechanical to chemical [1]. Depending on the type, location, orientation, and distribution of precipitates, these properties can vary significantly [2]. In this study we utilize the XeF_2 gas injection system (GIS), a common feature on dualbeam microscopes, in combination with FIB imaging to drastically enhance the contrast of precipitates and therefore make image processing easier. By combining the FIB-XeF_2 technique with low magnification and high resolution, it allows the extraction of large amounts of statistical data using software such as Dragonfly [Computer Software] 2022.2.

Methods

The FIB-XeF_2 contrast mapping procedure and micrograph capture were collected using a ThermoFisher Scios 2 dual-beam FIB system, which contains a gallium ion source. A full procedure can be found in previous work [3]. We have completed this technique using three different materials, all of which had different initial conditions and reasons for evaluating. First, an experimental Fe9Cr1Mo steel exposed to high-temperature and-pressure CO_2 environments, second, a 316H steel that had undergone creep testing, and third, an Alloy 690 nickel-based alloy that has undergone a surface mechanical process.

First, the surface was initially exposed to a high current FIB beam (30kV:50nA) to remove the surface oxide layer and expose hard precipitates in the matrix. The GIS needle was inserted and XeF_2 gas was flowed across the area of interest. Once the GIS needle was retracted, the area of interest was then exposed to a much lower FIB current of 30kV:1nA. Under the influence of this FIB beam, the XeF_2 gas binds preferentially to the metallic matrix and less to the precipitates, resulting in an enhanced contrast effect. This imaging was continued until the maximum contrast between the precipitates and the metal matrix was obtained, following which a high-resolution image was taken. In this study, the final images were taken at a resolution of 6144 x 4096, integrated 4 times with a dwell time of 1 µs. This produced an image of significant quality for quantitative analysis without altering the contrast too much. The precipitates were also individually characterised with Transmission Electron Microscopy (TEM) diffraction, Energy-dispersive X-ray spectroscopy (EDS), and Transmission Kikuchi diffraction (TKD) to identify their crystal structure on elemental composition and diffraction pattern. Once a characteristic of a particular morphology was identified using TEM / EDX / TKD, precipitates of similar contrast and morphology in the FIB-XeF_2 images were labelled as this precipitate identity.

Results

FIB-XeF_2 images were processed using image recognition in Dragonfly [computer software] 2022.2. The features within each image were segmented according to greyscale contrast using an Otsu threshold selection method [4]. This allowed the separation of precipitate from bulk, voids, and subsequently segmented different precipitates into separate virtual objects. The graphic shows an example of this approach – top left shows the raw FIB-XeF_2 image of a region of Fe9Cr1Mo steel, showing a lighter metal matrix and several precipitates with different greyscale contrasts and morphologies. The subsequent subfigures show in pink, blue, and yellow the segmentation of these features into three distinct populations based on greyscale contrast and morphology that were identified using TEM diffraction as $M_{23}C_6$, M_7C_3, and M_2C respectively. These segmented features are spatially located datasets, that can subsequently be parsed for the following statistical information: area fraction as a function of distance, distribution, size, orientation, and type. This enables quantification of microstructure over large areas with much greater confidence.

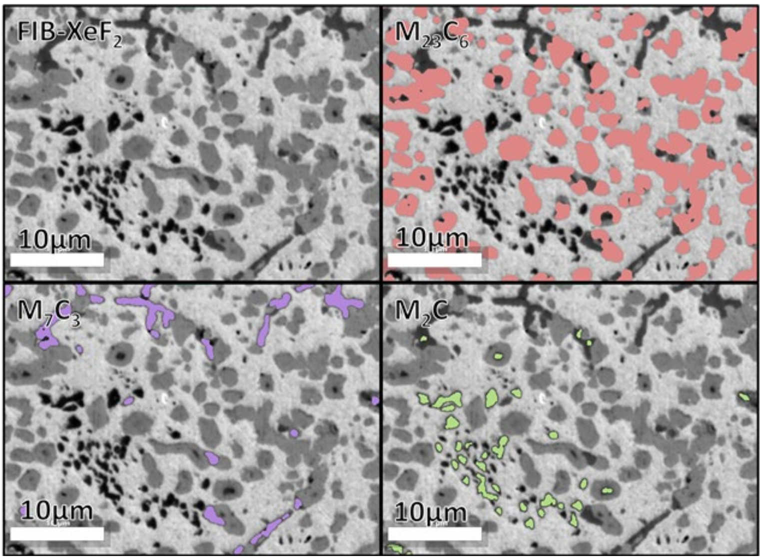
For 316H, precipitate locations were combined with grain boundary locations and compared with voids within the sample to obtain directional correlation information across the sample. Complete results for 316H steel can be found in previous work [5].

For the Alloy 690 sample, FIB-XeF_2 images provided clear microstructural information, such as precipitate pocketing along grain boundaries that was missed in other techniques.

Conclusion

Precipitation is an important factor when considering the chemical and mechanical behaviour of a material, especially for a material exposed to extreme environmental conditions. By re-utilising the XeF_2 gas module most dual beam FIB instruments have, it is possible to bring out the contrast of different precipitates against the bulk in such a way that image processing software can easily extract statistical information. This data will lead to a greater understanding of the behaviour of the material.

Graphic:



Keywords:

XeF_2, FIB, Contrast, Dragonfly

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