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Direct Experimental Measurement of Grain Boundary's Five-Parameters and Solute Segregations at Atomic Level

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Grain boundaries (GBs) are one of the most important defects in polycrystalline materials, as they substantially influence the properties and performance of materials for a wide range of engineering applications. The recent emergence of nanocrystalline and ultrafine-grained materials, which often exhibit exceptional properties, further emphasizes the important role that GBs play in materials. Obtaining interfacial parameters for GBs is an essential microscopic task for materials design.

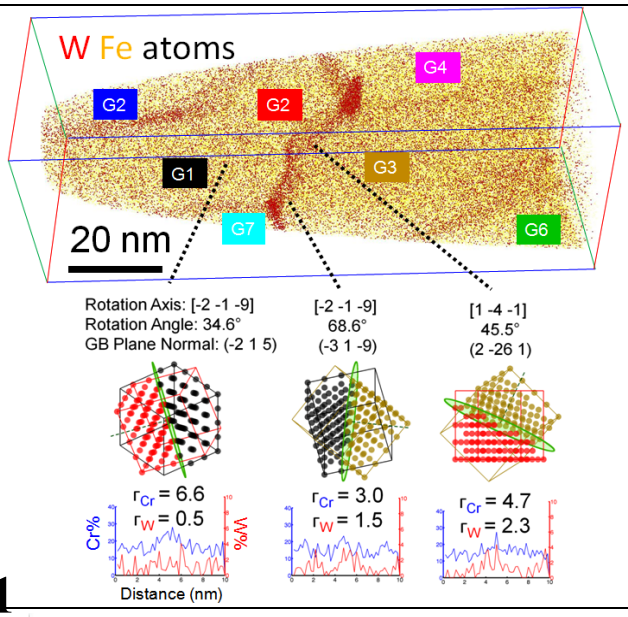
By definition, a GB is simply an interface between two adjacent grains. But in real terms, GBs require five macroscopic parameters to fully define their geometry, which describe the orientation relationship between the two grains as well as the boundary plane defining the interface. As the GB plane is only a few atomic layers thick, imaging and measuring these parameters is a microscopic challenge. This research illustrates a way to obtain the key structural information by atom probe tomography (APT) using a nanostructured ferritic alloy (NFA), which has nanometer-scale grain size (20-200 nm) and exhibits exceptional creep performance largely contributed by GBs.

The atom map, Fig. 1, indicates the three-dimensional (3D) solute distribution from seven ferrite grains located within the same volume. The grain orientations were derived using a 3D Hough transformation method.[1] By selecting a small region around each GB, the five–parameters defining the GB misorientation and interface plane normal were obtained and are shown schematically in Fig. 1. Solute segregations of Cr and W, in terms of solute excess values, were derived from acquiring one-dimensional concentration profiles along the normal direction of the GB plane (Fig. 1). Based on these data, systematic investigations of GBs from different NFA heat-treated conditions were conducted.

The solute excesses of the two main segregants, Cr and W, are shown in Fig. 2. Increases in the Cr and W excesses can be distinguished between the 1 h and 24 h 400 °C conditions; however, trends in the solute excesses in the 500 °C series are difficult to ascertain. As the solute excess is largely related to GB energy, which is related to GB misorientation, the GB's five-parameter data is required to quantitatively determine the solute excess. Therefore, in order to investigate the relationship between solute excess and GB misorientation, a more comprehensive representation is presented in Fig. 3. Each GB is represented by a circle, in which the diameter graphically reflects its solute excess. The center of the circle on the stereographic projection denotes the rotation axis of the misorientation. The lower and upper occupation percentage of the circle reflects the degree of misorientation angle and GB plane normal (with respect to the rotation plane). Possible degree of fit value (Σ) from a certain rotation angle and given a measured rotation axis is also shown, which may imply the level of GB energy of a certain misorientation type. In this way, the relationship between the experimentally measured five-macroscopic-parameters and solute excesses at the same GBs can be determined.

In addition to the visible differences in the solute excesses for GBs between 1 h and 24 h at 400 °C, the rotation axes of GB misorientation show that the misorientation type slowly develops from high-energy to lower energy GBs. The rotation axes of most misorientations collected from the NFA heat-treated

24 h at 500 °C are aligned along zone lines, which reflects not only the transformation of chemical potential but also stabilization of preferential misorientation types and energy minimization of GBs.[2]



1. L. Yao *et al.*, *Ultramicroscopy* **111**, 458 (2010).
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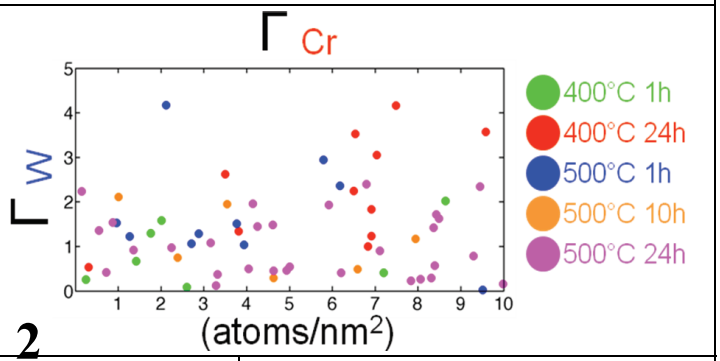

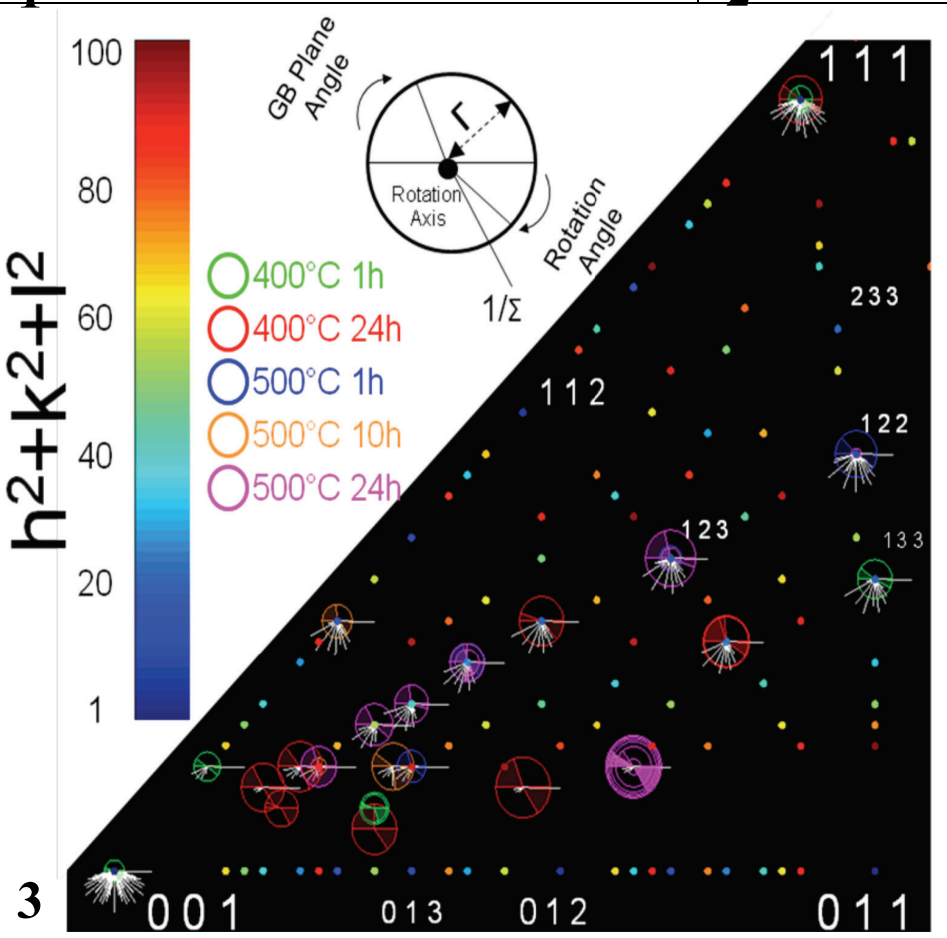

Figure 1. Three-dimensional atom map of Fe and W atoms, with labels for each individual grain, enlarged schematic views around boundaries defined by five macroscopic parameters, and solute excess of Cr and W derived from one-dimensional composition profiles across the boundaries.

Figure 2. The evolution of Cr and W solute excess on grain boundaries under different heat treatments.

Figure 3. Stereographic-based plot of five-parameters and solute excess information for GBs from different heat-treated samples shown in Fig. 1. The center of circle denotes rotation axis on the stereographic map. Lower and upper occupation percentages of the circles represent rotation angle and the angle between rotation plane and GB plane, respectively. The radius of the circles reflect solute excess amount of Cr plus W. Coincident site lattice value Σ from a possible angle is shown by white lines given a measured rotation axis.

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