Measuring Grain Size in Heavily Twinned Materials

Introduction

In twinned materials, it is important to exclude twin boundaries when characterizing the grain size. However, differentiating twin boundaries from regular high-angle boundaries is not always easy to do using traditional metallographic approaches, particularly in heavily twinned materials. Electron Backscatter Diffraction (EBSD) is very good at identifying twin boundaries based on misorientation. However, misorientation is only one part of the criteria needed for positive twin identification. Orientation Imaging Microscopy (OIM™) goes beyond the misorientation criterion to provide a more sophisticated analysis of the potential twin boundaries to greatly reduce the number of false positives.

Incoherent vs. Coherent Twin Boundary Criteria

For a boundary to be considered a twin boundary, the misorientation across the grain boundary must be very near the twin misorientation relationship. For example, the primary recrystallization twin in face-centered-cubic materials can be described as a 60° rotation about a <111> crystal axis. Thus, a boundary segment which has a misorientation of 60.7° about a <10 10 11> axis could be considered a twin. However, for a boundary segment to be considered a coherent twin it must satisfy an additional requirement. The boundary plane must coincide with the twinning plane. For the example already given, this means that one of the {111} planes of the crystals on either side of the grain boundary must be aligned (within a given tolerance) with the grain boundary plane. Twins that only satisfy the first requirement are sometimes termed incoherent twins; whereas twins which satisfy both requirements are termed coherent twins.

Coherent Twins in OIM™

The first criterion of the misorientation across a twin boundary in determining a specific type has been implemented within OIM™ since the very first version. Since OIM™ scans are inherently two dimensional, it is not possible to determine whether a given boundary satisfies the second criterion of the twin planes being aligned with the boundary planes without performing serial sectioning or some other three-dimensional sampling technique. However, assessing whether the trace of the boundary plane is aligned with the trace of the twinning planes provides a partial confirmation. Boundary segments in OIM™ follow the path of the scanning grid. However, OIM™ has the ability to link these segments together to reconstruct the boundaries as straight lines as shown in Figure 1.

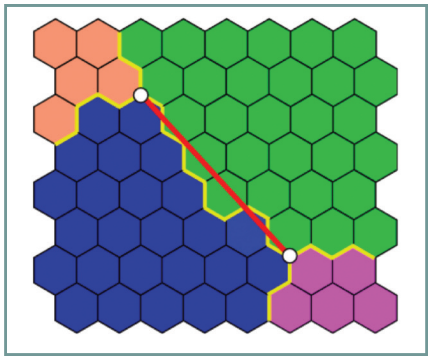

Figure 1. Schematic of a reconstructed boundary in OIM™ .

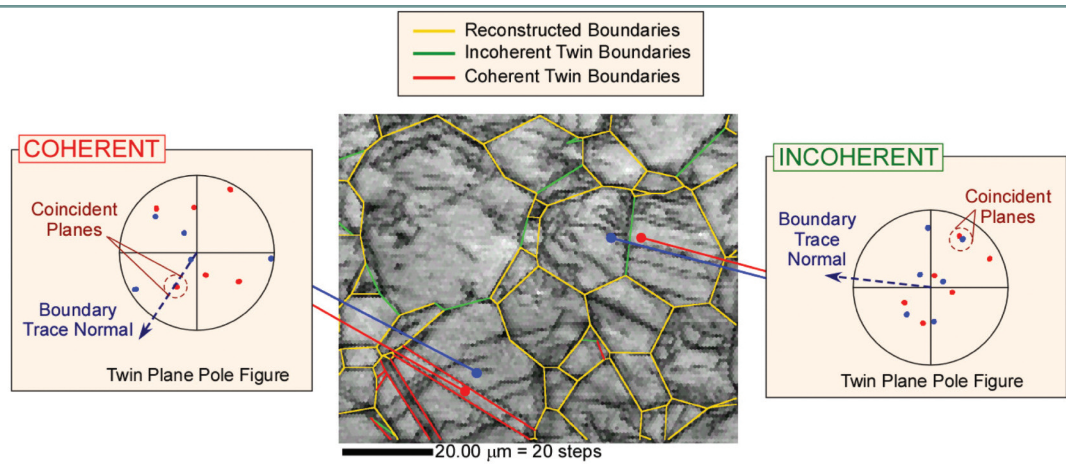

Figure 2. A coherent and non-coherent twin in deformed zirconium.

These reconstructed boundaries define the boundary traces. For a given reconstructed boundary, the first step is to see if it meets misorientation criterion. If it does, then the second step is to see if the twin planes on either side of the boundary are aligned with the boundary trace. This is illustrated in the pole figures shown in Figure 2.

Both of the boundaries highlighted satisfy the misorientation criterion. However, only one of the grains satisfies the second criterion where the twinning planes of the crystal lattices on either side of the boundaries are aligned with normal of the boundary trace, as illustrated in the two pole figures in Figure 2. Using this methodology, OIM™ can automatically identify which boundaries are incoherent twins or coherent twins.

By examining the three dimensional character of many twin boundaries, Randle (2001) has shown that when the traces are aligned the boundary and twinning planes are also aligned 90% of the time.

Grain Size

One important application of the advanced twin characterization in OIM™ is in the estimation of grain size in materials with significant twin populations. The example below shows copper damascene test structures, which have been analyzed using OIM™ . These maps depict the identified copper grains as raw data on the left. The center map shows the result of applying the OIM™ standard twin-finding algorithm, in which the number of grains has been dramatically reduced, as well as the shift in grain size distribution. The right hand map displays the result of applying the twin coherency test to the data and the corresponding grain size distribution.

Conclusions

Improved measurements of grain size can be achieved in heavily twinned materials using OIM™ due to its ability to accurately distinguish twins from regular high angle grain boundaries. The technique works very well in fully recrystallized poly crystals. In deformed materials, distinguishing coherent twins from incoherent twins becomes more difficult as the boundaries tend to be curved. It should also be noted that twin boundaries often have special properties relative to other boundaries. This makes OIM™ an excellent tool for materials research problems where grain boundaries play an important role. Examples would include any problems where intergranular degradation occurs, such as in stress corrosion cracking or void formation at grain boundaries during electromigration or creep.

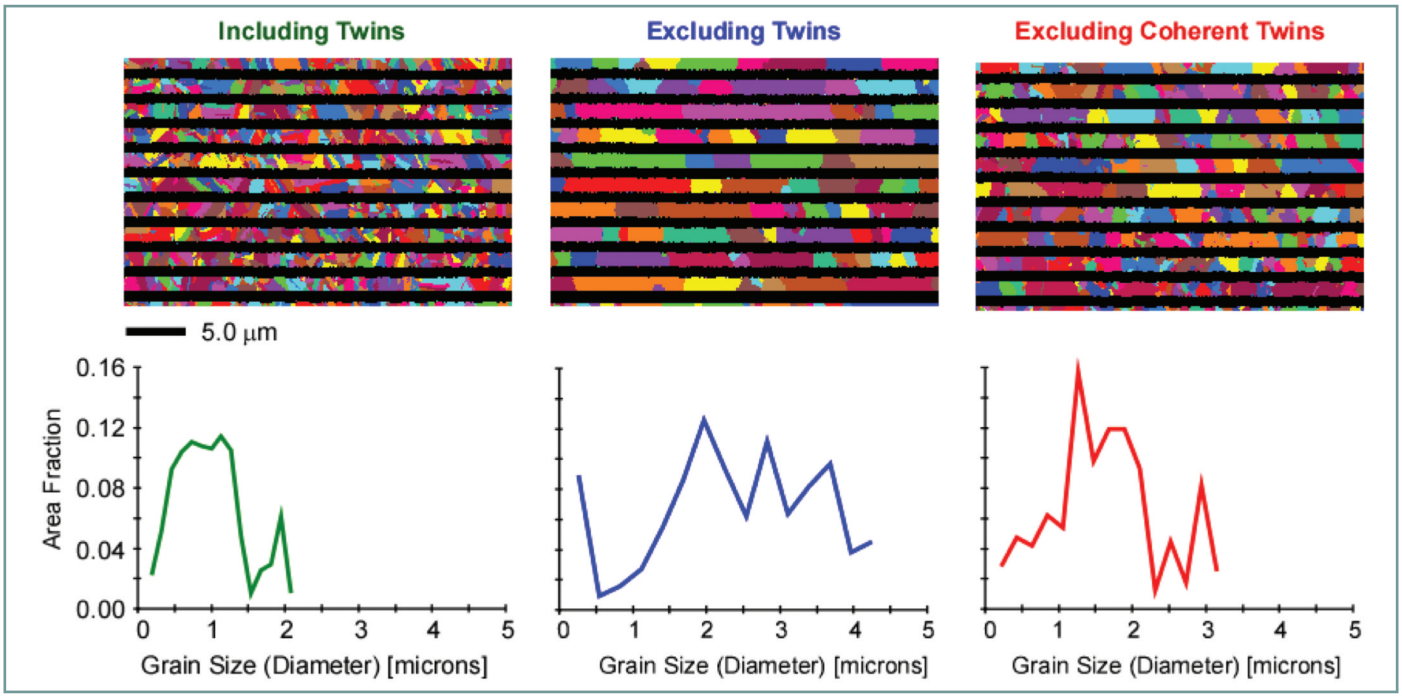

Figure 3. Copper damascene test structures: effect of excluding twins from the grain-forming algorithm in OIM™ .

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