Measuring Grain Size in Heavily Twinned Materials

Introduction

In automated measurements of grain size in heavily twinned materials it is important to exclude twin boundaries in order to accurately characterize the grain size. While OIM is an ideal tool for identifying grain boundaries based on misorientation, misorientation is only part of what makes a boundary a twin.

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

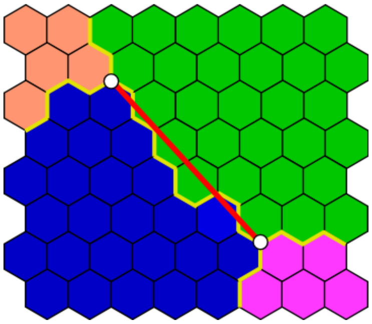
Figure 1 – Schematic of a reconstructed boundary in OIM.

The first criterion of the misorientation across twin boundary being of 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 facility to link these segments together to reconstruct the boundaries as straight lines as shown in Figure 1. 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.

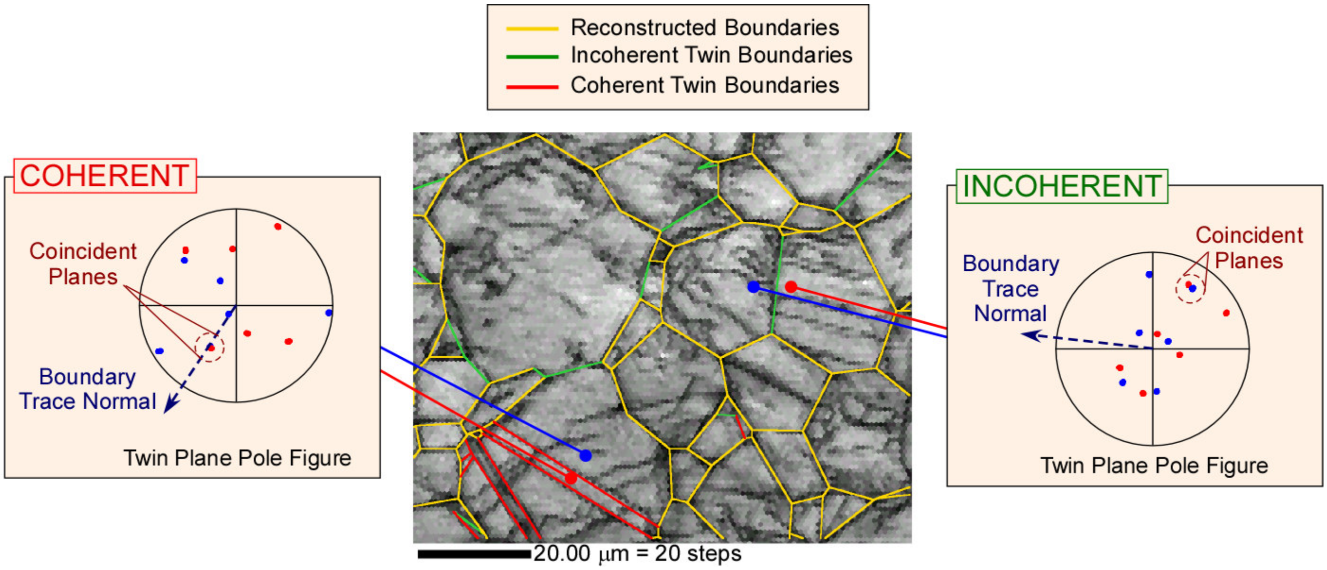

Figure 2 – A coherent and non-coherent twin in deformed zirconium.

By examining the three dimensional character of many twin boundaries, Randle 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 too 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 show 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 shows the result of applying the twin coherency test to the data and the corresponding grain size distribution.

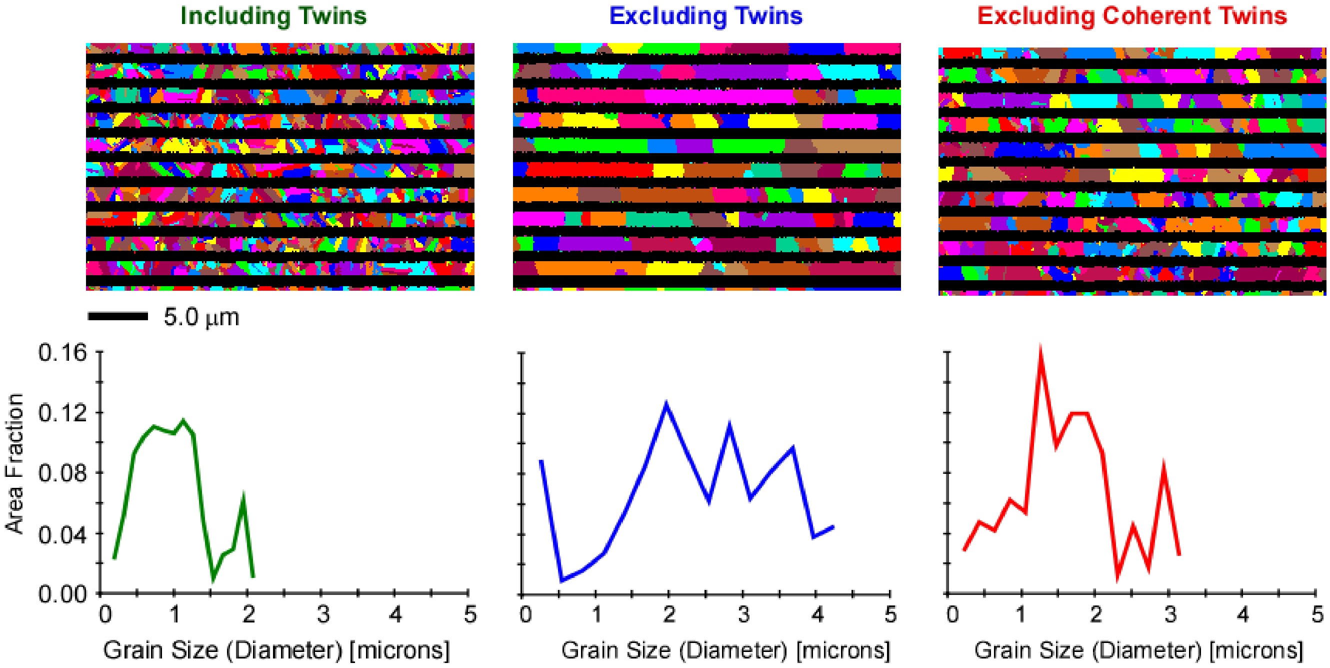

Figure 3 – Copper damascene test structures: effect of excluding twins from the grain-forming algorithm in OIM.

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 tecnhique 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.

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