Application Note

Non-destructive 3D Studies of Annealing Phenomena with LabDCT

Annealing - Nucleation, Recrystallization and Grain Growth

The mechanical properties of materials, such as hardness, strength, ductility, fracture toughness etc. are strongly dependent on the thermomechanical processing. During recrystallization of the deformed materials, new almost perfect nuclei form and grow by boundary migration until the deformed matrix is replaced by recrystallized grains. With further annealing, grain growth can occur, with some grains growing at the expense of others. Annealing treatment normally leads to significant modifications of the polycrystalline structure such as grain size distribution, grain orientation as well as texture, and is therefore critical for designing the microstructure in order to achieve optimal performance of the materials.

Characterization of the dynamic structural evolution during annealing is often a challenge for traditional 2D approaches such as scanning electron microscopy and electron backscattered diffraction. The examination of microstructure from sectioned sample surfaces may suffer from free surface effects as well as introduce biased analysis due to lack of evidence from the bulk of the sample.

LabDCT has the natural advantages for characterization of the microstructure subject to annealing treatments. Examination of samples in 3D enables unbiased analysis of the grain structure, particularly for polycrystalline samples with structural heterogeneities. As a non-destructive imaging technique, LabDCT provides the accessibility for studies of temporal variations in grain structure through 4D (x, y, z, time) experiments. Direct interpretation is therefore possible as the grain structure evolution is followed in a corresponding manner.

Fig. 1 presents two diffraction contrast projections from a partly recrystallized aluminum sample (left) and a fully annealed pure iron sample (right). Distinct, bright line shaped spots from recrystallized grains can be observed.

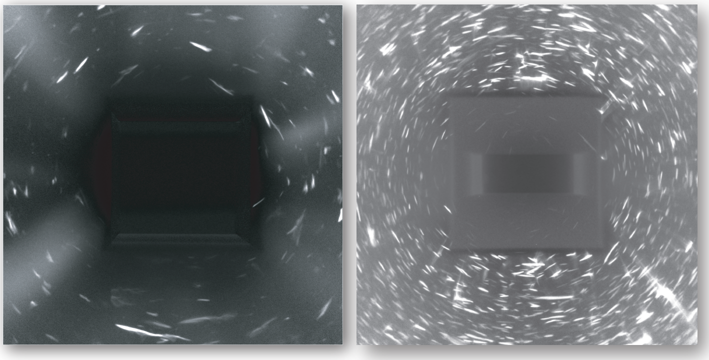

Fig. 1 *Left: a diffraction contrast projection from a partly recrystallized Al sample. The sharp, line-shaped spots originate from the recrystallized grains. The broad, banded diffraction signals are from the recovered matrix. Right: a diffraction contrast projection-from a fully recrystallized pure iron sample.*

Recrystallization Study with LabDCT

For recrystallization studies, the key questions to answer are where do the nuclei form and what are their crystallographic orientations. Local strutural heterogeneities such as the various types of dislocation strutures and secondary phase particles generate a matrix with non-homogeneous distribution of stored energy, making it difficult to understand the preferential nucleation sites and how the boundaries of recrystallized grains migrate.

With the reconstructed 3D grain map, it is now possible to make direct correlation between the recrystallized grains and other strutural features such as secondary phase particles. Tracking grain boundary migration during recrystallization is also enabled with 4D experiments.

Fig. 2 shows a reconstructed 3D grain map of a partly recrystallized aluminum alloy sample. It is revealed that the preferential nucleation sites in this sample are along the original grain boundaries.

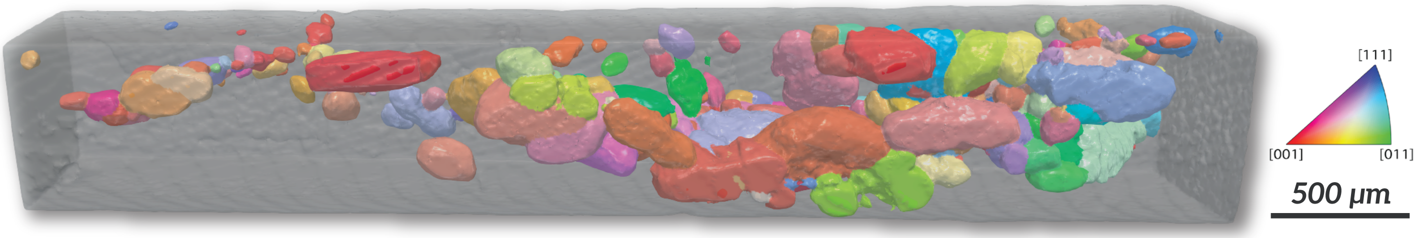

Fig. 2 *Reconstructed 3D grain map showing the recrystallized grains in a partly recrystallized aluminum sample.*

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Grain Growth Study with LabDCT

The kinectics of grain growth has been a main focus for many decades, exploring factors such as the grain topology and grain boundary properties. While considerable efforts have been made to model the grain growth process, there have been considerably fewer efforts to provide the experimental verification of these models.

With LabDCT, the 3D grain structure can be mapped out at several growth stages of an annealing process. A direct correlation between the grain boundary character and growth offers a wealth of 4D information that can be used to both understand and predict grain growth.

Fig. 3
3D grain map of an Armco iron sample at three annealing steps:

t_0 - initial state

t_1 - 8 hrs at 880°C

t_2 - 16 hrs at 880°C

More than 1200 grains and 8000 grain boundaries were mapped out for analysis. A grain that grows in an abnormal manner was observed after the first annealing treatment (shown in pink close to the top).

The significant grain statistics made available by LabDCT is large enough to capture rare events such as abnormal grain growth (as shown in ***Fig. 3***) as well as the presence and annihilation of grains with only three faces.

In the 3D grain map from the initial state a grain with three faces was observed to neighbour an abnormal grain, while it had disappeared after the first annealing treatment. With the grain boundary information readily accessible from the 3D grain map, a closer look at the boundary between the two grains at the initial state (***Fig. 4***) reveals that they share a low-angle grain boundary with a high positive curvature towards the three face grain.

It has long been known that low-angle grain boundaries are predicted to have low mobilies, while the driving forces of high-curvature grain boundaries tend to be high; hence, theoretically predicting the growth or shrinkage of this particular grain pair upon annealing requires the correct relationship between two opposing effects. The local experimental evidence clearly shows that the grain boundary in question does move in order for the abnormal grain to swallow up the small grain with only three faces.

It has been demonstrated that the essential information for studies of grain growth, such as grain shapes, sizes, crystallographic orientations, and boundary properties, can be obtained non-destructively at multiple temporal states using LabDCT. The approach provides access to the neceesary 4D experimental evidence that is neither accessible via 2D techniques such as EBSD nor via destructive 3D techniques such as serial sectioning.

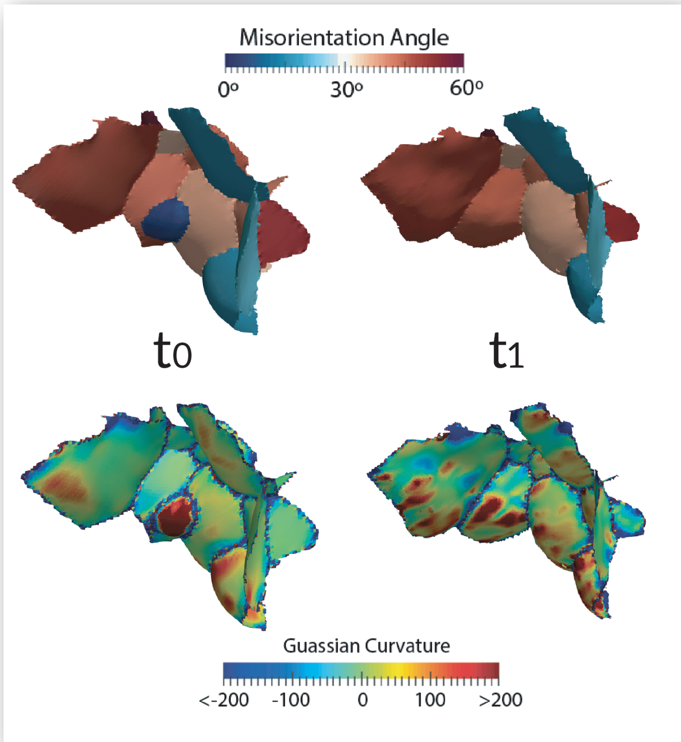

Fig. 4 *Grain boundaries of an abnormal grain with several neighboring grains at the initial state and after the first annealing step, colored according to misorientation angle and curvature.*

References

1. J. Oddershede, et al, (2019), IMMI, vol. 8, pp. 217-225.
2. J. Sun, et al, (2017), IOP MSE, vol. 219, 012039.

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