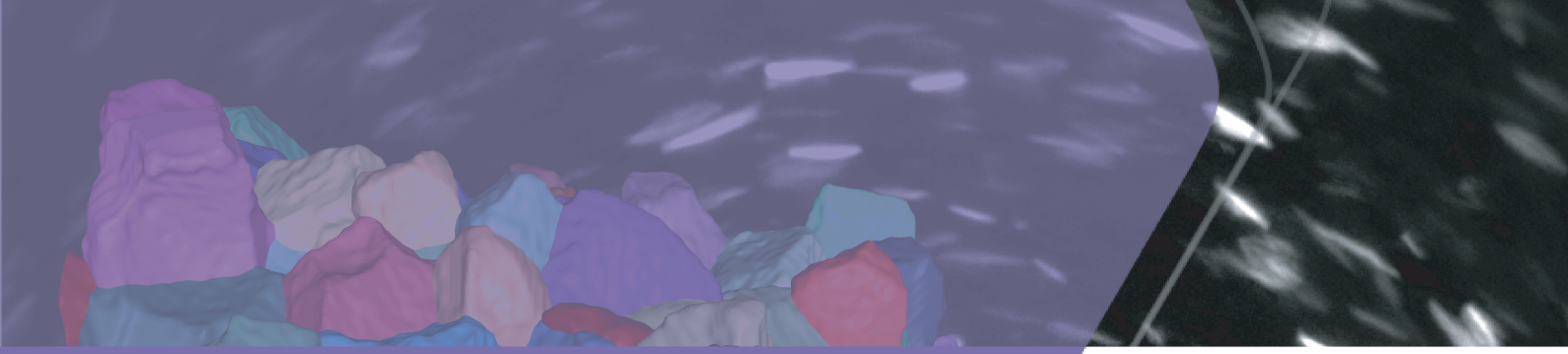

Application Note

A Recrystallization Study of Severely Cold-rolled Pure Iron during Annealing using Laboratory Diffraction Contrast Tomography

Recrystallization of Cold-rolled Iron

The mechanism of recrystallization texture development of cold-rolled steel is largely dependent on the material chemical composition, cold rolling reduction, and annealing treatment conditions. Cold-rolled steel exhibits its unique evolution of preferred orientation of grains (texture) during annealing, which has been explained by two mechanisms: *oriented nucleation* and/or *selective grain growth*. Understanding the dominating mechanism under various annealing parameters is important in order to control the final texture.

The cold rolling process introduces non-homogeneous distribution of various types of dislocation structures, which subsequently leads to anistropic annealing responses across the processed materials. The structural heterogeneity is essentially a 3D characteristic, which poses a challenge for microstructure examination on the sectioned sample surface using electron microscopy (EM) techniques such as scanning electron microscopy (SEM) or electron backscattered diffraction (EBSD). While the 2D EM characterization can be extended to 3D through destructive serial sectioning, its application to samples with a large-scale structural anisotropy is often expensive and time-consuming,

3D Grain Mapping with LabDCT

LabDCT, measuring both the grain shape and crystallographic orientation for a statistically significant number of grains, is a powerful approach to collect the necessary information for understanding the annealing responses in cold-rolled steel and correlating the present texture components to the 3D rolling geometry.

Fig. 1 shows the collected diffraction contrast diffraction projections for 99.2% cold-rolled steel samples annealed at 500, 600 and 700°C. The 'banded' distribution of the diffraction spots visible especially at 500 and 600°C originates from the sample texture with many grains of similar crystallographic orientation.

Fig. 2 presents the 3D grain maps for the annealed samples reconstructed using GrainMapper3D™, comprising 1400, 1200 and 800 grains at 500, 600 and 700°C, respectively.

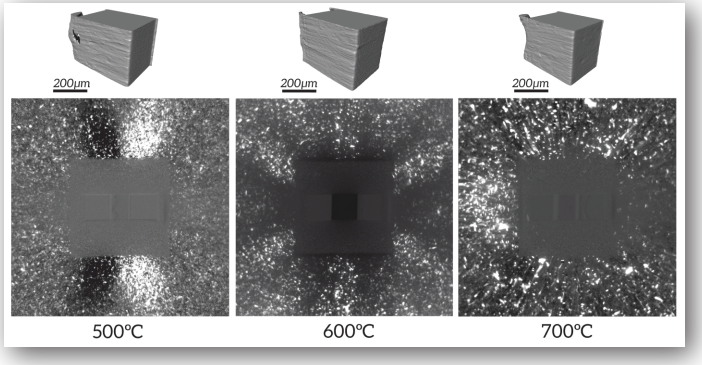

Fig. 1 *Example diffraction contrast projections collected from the samples annealed at* 500, 600 and 700°C. The scanned volumes rendered from absorption contrast tomography are on the top.

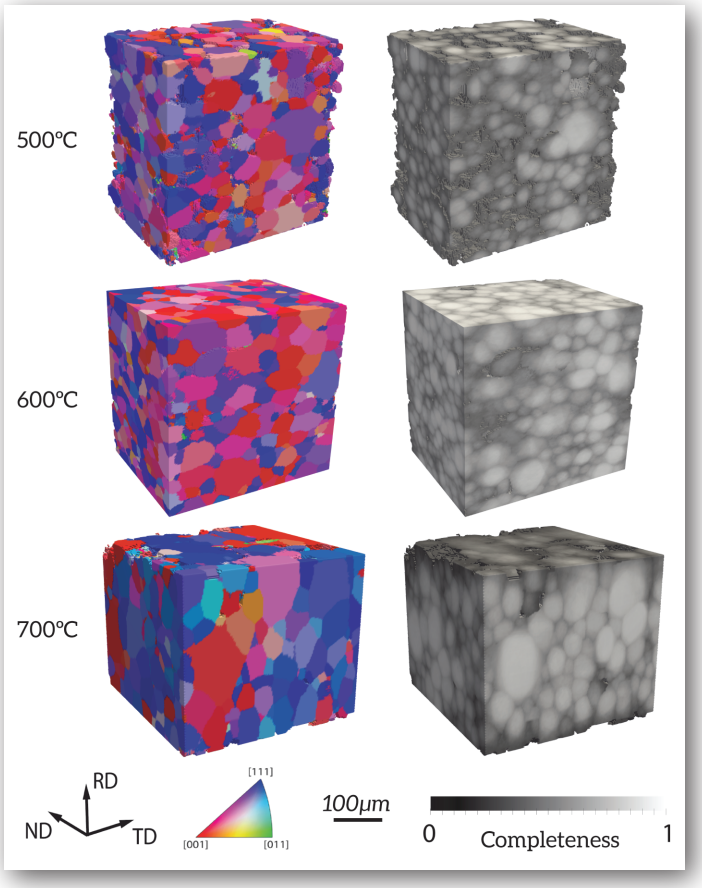

Fig. 2 *Reconstructed 3D grain maps at the various annealing temperatures colored by IPF along ND (left) and the reconstruction completeness.*

xnovotech

Texture and Grain Size Analysis

The reconstructed 3D grain map provides comprehensive information on grain size, shape and crystallographic orientation, enabling the evolution of texture and grain size as a function of annealing temperature to be derived, as presented in ***Fig. 3***.

The histogram of grain size represented as equivalent sphere diameter (ESD) distribution exhibits a clear shift towards larger ESDs for increasing annealing temperatures.

The orientation distribution (ODF) of all the grains in the reconstructed 3D grain maps show that the recrystallized grains were formed with textural components of {100}, {211}, {111}, and {411}, and the strong **α**-fiber deformation texture was found to change to the {100}<012> component during annealing in the range from 500 to 700°C.

Detailed investigation of the 3D grain map from the sample annealed at 500°C has shown that recrystallized grains with sizes of ~20-100 µm formed rather homogeneously within the sample, indicating a random recrystallization progress. Grains near the {100}<012> direction are observed to grow larger than others with increasing annealing temperature, which indicates that selective grain growth is the dominant mechanism for texture development.

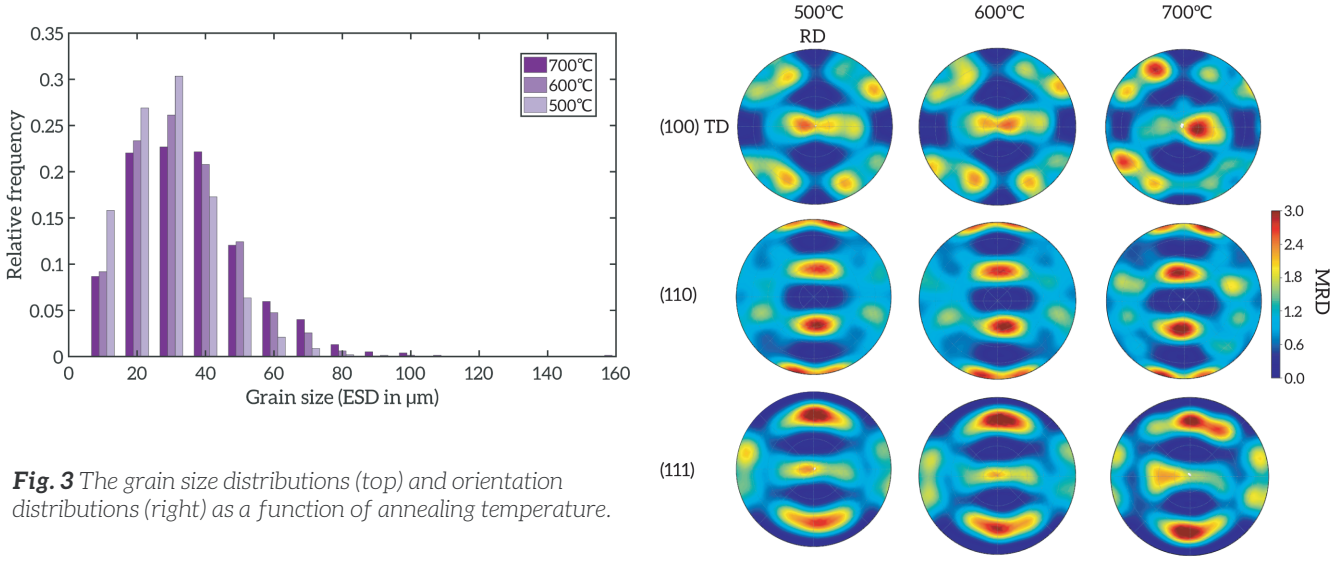

Fig. 3 *The grain size distributions (top) and orientation distributions (right) as a function of annealing temperature.*

Anisotropic Annealing Microstructure

By characterizing the microstructure at various positions along the rolling normal direction (ND), it is observed that for all annealing temperatures, the size of recrystallized grains in the center region is 20–30% larger than those near the rolling surface, as shown in ***Fig. 4***. The strain gradient introduced during cold rolling results in higher stored energy closer to rolling surfaces, which then generates a larger number of nucleation sites when the sample is annealed. Impingement of grains during growth results in smaller grains near the rolling surfaces compared to the central region of the sample.

Strutural heterogeneity can lead to biased analysis in case the region of interest selected for characterization is insufficiently representative. As shown from the current analysis, the 3D grain map from LabDCT offers a straightforward approach to capture the large-scale anisotropy present in polycrystalline materials.

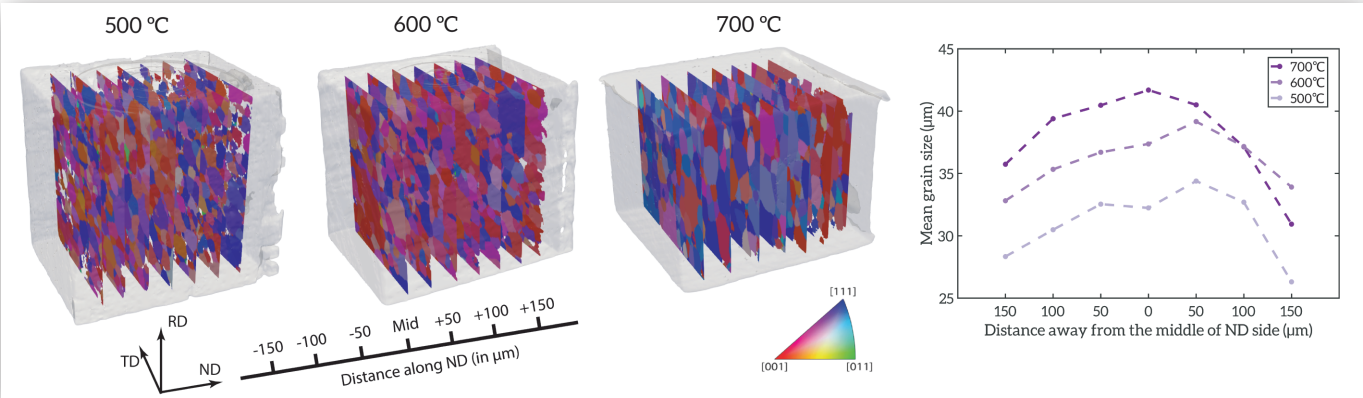

Fig. 4 *RD-TD slices at a 50 µm interval along ND (right) and the measured mean grain size of each slice plotted as a function of distance away from the center along ND (right).*

Reference

J. Sun, et al. (2019), ISIJ, under review.

Xnovo Technology ApS
Theilgaards Alle 9, 1.th
DK-4600 Køge
info@xnovotech.com
www.xnovotech.com

xnovotech