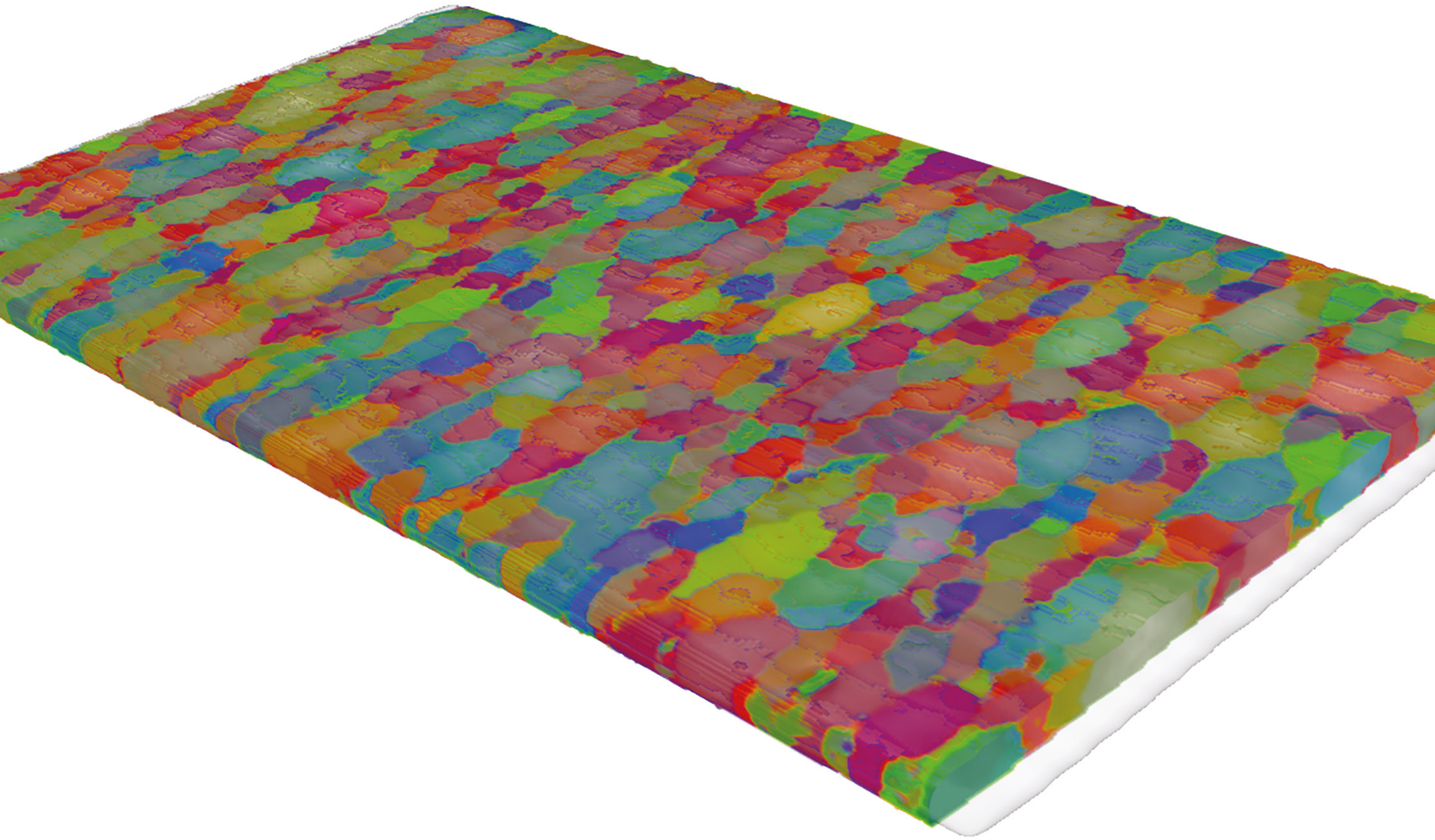
Application Note



Evaluating 3D Grain Structure in Aluminum Foil with LabDCT

ZEISS Xradia 520 Versa with LabDCT

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Date: October 2017

Diffraction contrast tomography (DCT) is a nondestructive characterization technique that utilizes a series of X-ray diffraction patterns to map the 3D grain structure of crystalline materials. Originally developed at the European Synchrotron Radiation Facility (ESRF), the capability has recently been implemented for the first time on a laboratory system. LabDCT on ZEISS Xradia 520 Versa has been applied to many bulk crystalline samples; in the present study, the technique was applied to a thin foil sample of aluminum. Promising results indicate the ability to non-destructively quantify, map, and visualize numerous small grains in 3D in such a material.

Introduction

To date, most studies utilizing diffraction contrast tomography, either at the synchrotron or in the lab, have focused on relatively low-aspect-ratio samples, typically cylindrical or rectangular pillars. In the present study, we tested the feasibility of performing lab-based nondestructive 3D grain mapping on a high-aspect-ratio structure: aluminum foil. Like other metals, the aluminum foil contains a crystalline microstructure, which in this case will also be constrained by the thin, nearly 2D geometry of the object itself. As a representative of thin metal foils in general, the applications of aluminum range from insulation materials to electronic mobile devices, the mechanical and electrical properties and performance of which depend on the foil's discrete crystalline structure, orientation, and texture.

We performed the experiment using LabDCT (laboratory diffraction contrast tomography), which is an optional module for the ZEISS Xradia 520 Versa X-ray microscope, enabling the user to nondestructively investigate and map the grain structure of crystalline materials in 3D. The technique is based on recent developments at synchrotron facilities now being incorporated by ZEISS into a laboratory instrument. The laboratory integration is particularly unique, enabling broad accessibility and usability of the characterization methodology. This technique is based on illuminating a sample with polychromatic X-rays in a cone-beam geometry and collecting a series of diffraction patterns in a Laue focusing condition over a range of projection angles of the sample, as shown in Figure 1.

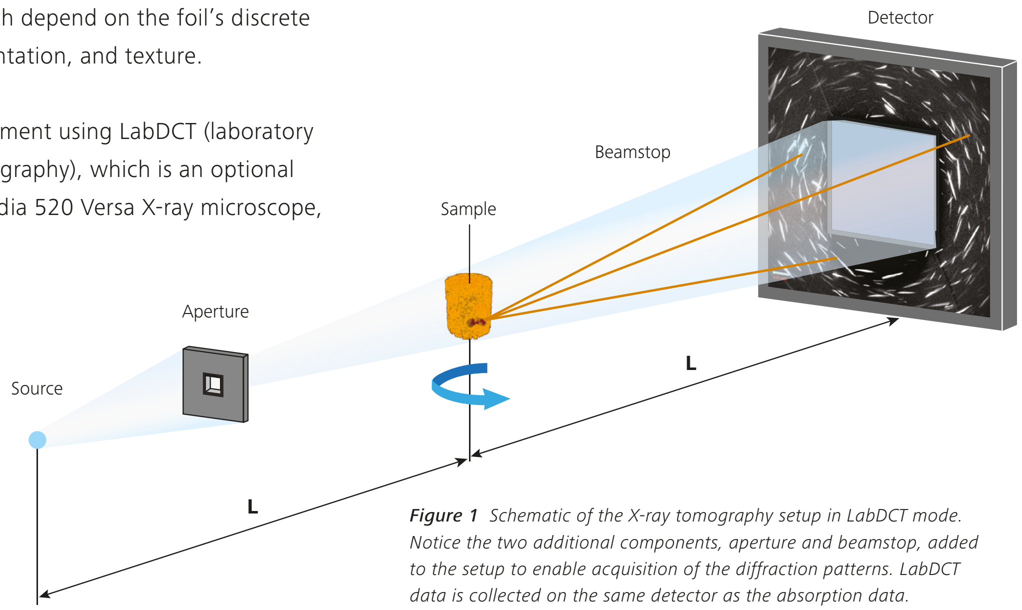

Figure 1 Schematic of the X-ray tomography setup in LabDCT mode. Notice the two additional components, aperture and beamstop, added to the setup to enable acquisition of the diffraction patterns. LabDCT data is collected on the same detector as the absorption data.

Such diffraction patterns contain information about the location, crystallographic orientation, and size of grains within the sample and can subsequently be computationally reconstructed to recover the 3D crystalline information. This data can then be correlated to the results of standard absorption-contrast tomography, or even other imaging modalities such as light or electron microscopy, allowing crystallographic information to be tied to features of interest located by the X-ray microscope – cracks, welds, pores, flaws, etc. [1,2]

Experimental Procedure

A small section (1 mm x 2 mm) of 17 µm-thick aluminum foil was cut using a razor blade and glued to the tip of a pin. No additional sample preparation or surface treatments were necessary.

The sample was then mounted in a ZEISS Xradia 520 Versa X-ray microscope equipped with the LabDCT module. An aperture was inserted between the source and sample to restrict the illumination to the sample. A rapid absorption contrast tomograph was first collected (1s exposure, 720 projections), to capture the 3D geometric structure using traditional X-ray imaging techniques. A beamstop was then inserted between the sample and detector to block the primary transmitted beam's illumination of the detector, isolating the X-ray diffraction pattern.

Diffraction contrast tomography was then performed at 50 kV, collecting 181 patterns over the full 360 degree sample rotation range. An example of one such diffraction pattern is shown in Figure 2.

The LabDCT workflow consists of data acquisition and data processing steps, which are shown in the schematic in Figure 3. LabDCT reconstruction of the data was performed using the incorporated GrainMapper3D (Xnovo Technology ApS, Galoche, Koge, Denmark) software. The software consists of a workflow-based procedure and is designed with emphasis on the ease-of-use.

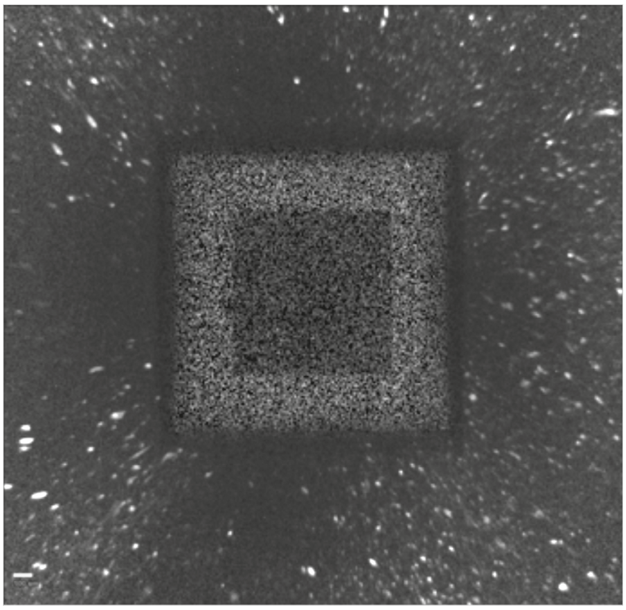

Figure 2 *Diffraction patterns seen in an image frame collected as part of the LabDCT scan. The spots on the detector are reflections from several grains. The central square portion of the detector corresponds to the detector area behind the beam stop.*

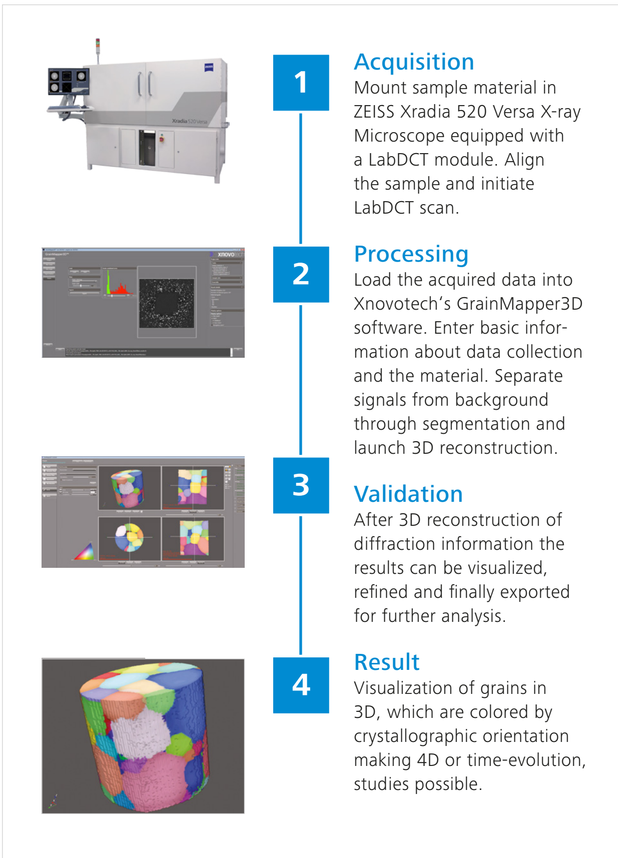

Figure 3 *LabDCT acquisition and grain reconstruction workflow*

A screenshot of the user interface of GrainMapper3D is shown in Figure 4. Data from both the absorption contrast tomography and diffraction contrast tomography, along with crystal space group and lattice parameters for aluminum, were input. The software then reconstructed the grain information contained in the diffraction patterns to provide location, orientation, and size of the numerous grains. Results were immediately displayed in the built-in visualizer of GrainMapper3D.

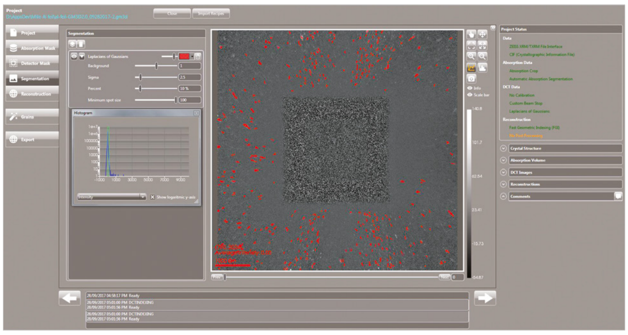

Figure 4 Screenshot of the guided user interface of the 3D grain reconstruction software – GrainMapper3D software.

Results

Reconstructed data was used to generate 3D grain maps. In Figure 5, the grain map is by crystal orientation (right). Grain ID assignments can be evaluated by the completeness map. The completeness map is the difference between the expected reflections and the reflections measured on the detector. The sample is roughly 1x2 mm in size. This provides statistics on larger volumes, at faster acquisition times, to supplement other analyses like EBSD or synchrotron methods. From the plots, it is apparent that the grain distribution is relatively uniform and shows no strong texture or anisotropy in 2D as can sometimes be found in rolled metal sheets depending on the fabrication process. (The final steps of aluminum foil production typically consist of cold rolling followed by an annealing step that would lead to a relatively homogeous microstructure.)

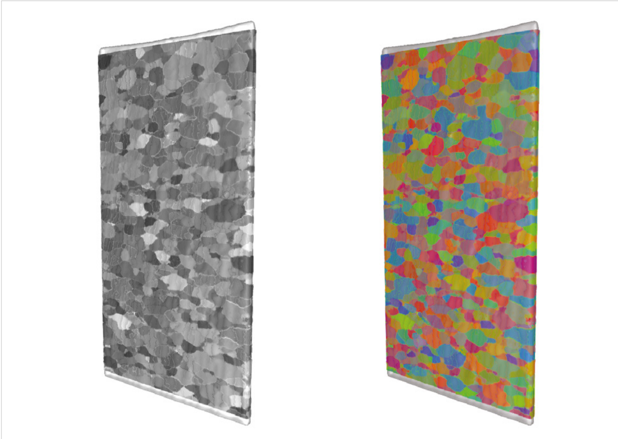

Figure 5 Reconstructed grains in the aluminum sample (left) completeness map and (right) 3D grain map. Sample size is 1x2 mm.

The grain size results were used to plot a distribution of grain equivalent diameter, as shown in Figure 6. Most grains were found to be in the 30-80 µm range, with a few larger grains in the 100-120 µm range. Note: these values are larger than the thickness of the foil (17 µm) due to larger dimensions of the grains in the planar direction.

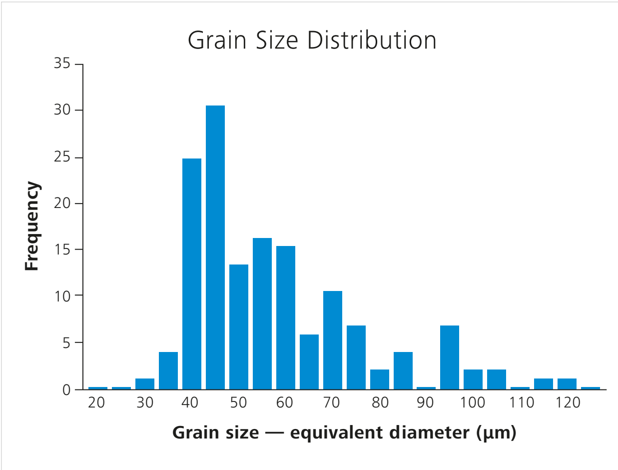

Figure 6 Histogram showing the grain size distribution in the aluminum foil sample calculated from the LabDCT data.

It is noteworthy that the grain structure of this sample was reconstructed with high fidelity, considering its small thickness. With LabDCT data from “traditional” samples with cylindrical or pillar geometry, best detection limits with regard to grain size have typically been demonstrated on the order of 30 or 40 µm diameter. In this sample, many grains were analyzed despite the fact that their dimension in one direction was limited to less than 17 µm. This is because the sampled volume is significantly smaller than most bulk volumes imaged using LabDCT, allowing even the weak diffracted signals from the small grains to emerge from the sample. Moreover, significantly decreasing the sample volume reduces the number of overlapping diffraction spots on the detector, thereby making them easier to analyze and subsequently reconstruct smaller individual grains.

Conclusions

In this work, LabDCT was applied to nondestructively investigate the grain structure in a thin foil sample of aluminum without any significant sample preparation. This sample is representative of thin metal foils used in electronic devices or other lightweight metals applications.The grains were successfully reconstructed, showing that the foil has a uniform distribution of grain sizes, mostly with equivalent diameters of several tens of microns without any noticeable texture. The results confirm the feasibility for LabDCT analysis of small grains within thin, planar samples. Possible future extensions of this study could include the 4D evaluation of the grain structure under extended treatment (such as heat) or external load (cracking under tension).

References:

[1] S. A. McDonald, et al., Non-destructive mapping of grain orientations in 3D by laboratory X-ray microscopy, Scientific Reports, 5 (2015) 14665.

[2] C. Holzner, et al., Diffraction Contrast Tomography in the Laboratory – Applications and Future Directions, Microscopy Today, July 2016, p. 34-42.

[3] Carl Zeiss X-ray Microscopy, Unlocking Crystallographic Information from Laboratory X-ray Microscopy, Technical Note, (Pleasanton, CA), 2015.

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[5] S. A. McDonald, et al., Microstructural evolution during sintering of copper particles studied by laboratory diffraction contrast tomography (LabDCT), Scientific Reports, 7 (2017) 5251.

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