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Abstract

Diffraction contrast tomography (DCT) is a non-destructive way of imaging microstructures of polycrystalline materials such as metals or crystalline organics. It is a useful technique to map 3D grain structures as well as providing crystallographic information such as crystal orientation, grain shape, and strain. Understanding the internal microstructure of a material is important in understanding the bulk material properties. This report gives a general overview of the similar techniques, DCT data acquisition, and analysis processes. Following the short literature review, potential work and research at Los Alamos National Laboratory (LANL) is discussed.

Introduction

X-ray computed tomography (CT) is a widely used technique in many industries. -The medical industry uses X-ray CT for diagnostic, treatment, planning, or screening purposes by providing cross-sectional images of the body, such as for identifying osteoporosis.[1] -The largest and most broad use is in materials science characterization. -The technique has been used in research in materials sciences[2], nuclear sciences, chemistry, biology, toxicology, and various engineering fields from identifying soil particulate[3] to observing strength of asphalt mixtures.[4]

Polycrystalline materials are solids composed of an aggregate of crystals, known as grains, of varying size and orientation. Most inorganic solids and engineering materials, such as metals, ceramic, and rock, are polycrystalline[5], however, the material properties of these solids are poorly understood at the internal microstructure level. Much of the current, available technology allows for understanding the polycrystalline structure as a whole, or amorphous properties, but the orientation, shape, and size of individual grains is fundamental in understanding the material behavior.[6] To understand the bulk material property, it is necessary to map grain shape, position, strain, and orientation. It is also important that this be done non-destructively so further characterization can be combined with other techniques, or used in further processing. Current X-ray absorption methods of tomography, show no contrast between grains of the same phase and no orientation or strain information of crystals.

Monochromatic beam X-ray diffraction techniques, such as three-dimensional X-ray diffraction microscopy (3DXRD) and diffraction contrast tomography (DCT) are imaging techniques that provide high resolution, three dimensional, grain maps of polycrystalline materials. It allows for the mapping of individual grain shape and crystal orientation using a nondestructive method. The DCT procedure, a variant of 3DXRD, aims at simultaneous reconstruction of the microstructure of the material but still reflects similarities to conventional X-ray tomography. Using diffraction of synchrotron radiation, information on grain shape, orientation and average elastic strain tensor components can be easily found in a nondestructive manner.

DCT is primarily used for materials science characterization such as metal and alloy analysis, observing grain growth or crystallization, or semiconductor analysis. This literature review will focus primarily on the DCT methodology, data acquisition, and data analysis. The final section will provide insight into the current and potential applications at Los Alamos National Laboratory.

Current State of the Technology

Data Acquisition

DCT uses standard laboratory instrumentation with the addition of a beam stop, aperture, and source & detector positioning that allow for diffraction conditions. A millimeter sized sample is placed in a beam of parallel, monochromatic synchrotron radiation large enough to illuminate the whole field of interest. The beam is controlled through an aperture, then illuminates the sample as it rotates.[7] Throughout the 360° rotation, grains from the sample pass through alignments, diffracting the incident beam onto a high resolution, 2-D detector.[8] The detector collects a continuous series of these diffraction projections while simultaneously collecting absorption information to later be used in reconstruction. The diffraction spots spread over a series of consecutive images and need to be summed together in later data analysis. A basic schematic of this technique is shown in *Figure 1*. In general, the parameters of this setup are short wavelength and low divergence. The x-ray energies used are relatively high (>20 kVp).[6a, 9]

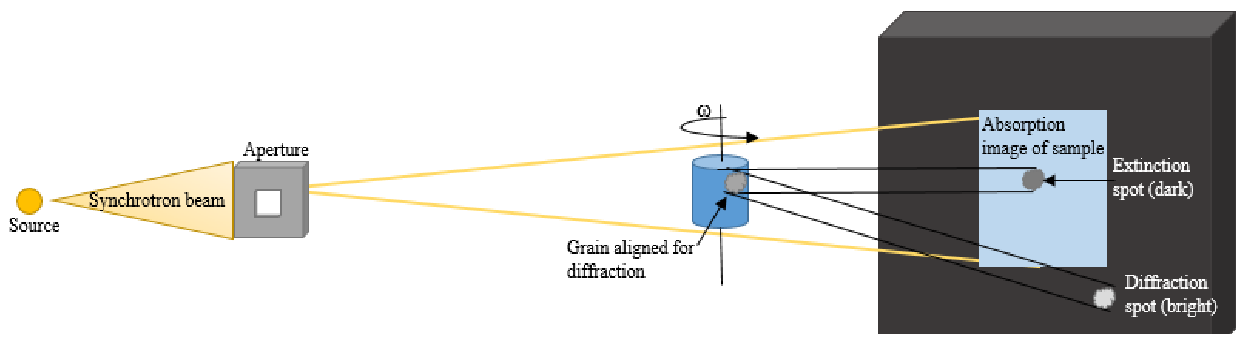

Figure 1. A schematic view of the DCT setup.

Data Analysis

Information about the diffraction spots such as position, intensity, and area, is gathered after segmentation through thresholding techniques and are then stored in a database. The diffraction pattern and spots before processing is shown in *Figure 2*. Due to axial symmetry, a grain which diffracts at ω also diffracts at 180°+ω and can be labelled as a Friedel pair, or diffraction pair. Friedel pairs are key to the accuracy of the data processing as each crystal plane of a grain can give rise to a maximum of four diffraction spots, or two pairs. [6a, 9a, 9b, 9d]

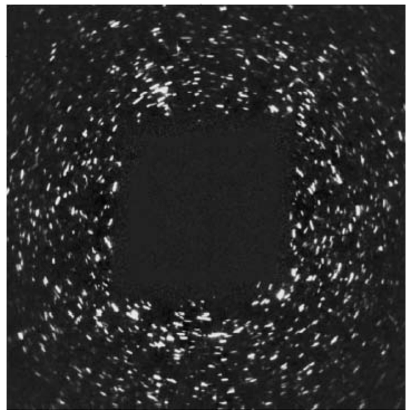

Figure 2. Zeiss et. al. 2D diffraction pattern from a Ti alloy sample.[4]

Through spatial and crystallographic criteria and a search algorithm, diffraction spots can be sorted and pairs will be automatically detected. When these diffraction pairs are detected, the diffraction angles describing the geometry can be calculated using the center of mass of the two spo~~r~~ts. [9c]

An indexing procedure was developed to further analysis the diffraction pairs and gain information on the grain orientation and position. An algorithm, consisting of three main processes: building grains, merging grains, and extending grains, is applied in a loop pattern to the diffraction pairs with decreasing tolerance. After the final iteration, statistics of diffraction spot properties and discrepancies of location and orientation are collected and stored. [9a, 9d]

The 3D structure is reconstructed using more iterative algorithms, or algebraic reconstruction techniques (ART), that are commonly used in other computer tomography techniques. The algorithm uses the diffraction spots, information found in indexing, and a limited number of grain projections to reconstruct each grain individually. The individual grains are then assembled into a 3D sample volume and voxels of one grain are compared with neighboring grains' voxels in a post-processing step.[6a, 9a, 9d]

Optional post-processing steps are available to ensure all voxels are assigned to a grain, and only one grain. The absorption contrast images that were collected are used to create a filtered backprojection reconstruction that can be superimposed onto the 3D grain map creating your final reconstruction image.[9a] An example of a reconstructed image is shown in *Figure 3*.

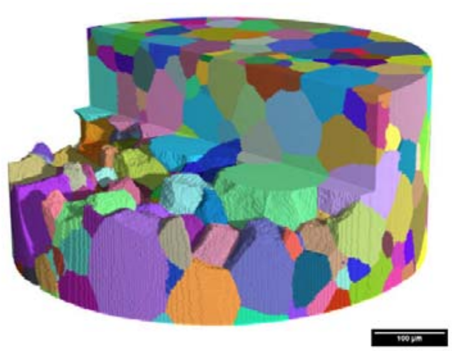

Figure 3. DCT reconstruction of a Ti alloy sample from King et. al.[9a]

Sample Information

Various polycrystalline materials can be used with this technique such as metals and alloys, foams, energetic materials, semiconductor and component parts, and polymer composites. However, it is important to note the atomic number will affect beam transmission through the sample thus sample dimension must be taken into careful consideration. Typical sample dimensions are summarized in *Table 1*.[10]

Parameter	Typical Range
Grain Size	20-200 micrometers
Sample Diameter	20-200 mm
Axis-Detector Distance	3-10 mm

Table 1. Typical parameter ranges for LabDCT samples.

Potential at Los Alamos National Laboratory

The current tomography techniques readily available to MST-7 at Los Alamos National Laboratory are micro-CT and nano-CT. The primary technique is the micro-CT which has used for the analysis of voids, in situ deformation, structures, and mechanical performance in materials such as polyurethane foams, silicone foams[11], 3D printed metals[12], damaged metals[13], high explosives[14], carbon-fiber filled polymer composites, and targets.[4, 8]

Improving the lab's imaging capabilities would still allow for all the above analysis, but would additionally provide insight into the crystallographic properties and grain growth of lab-wide materials with in situ heating. The in situ heating would have potential to examine thermal expansion, and mechanical properties near transition temperature of polymers.

It would also allow for further improvement in additive manufacturing for both metals and polymeric materials by collecting images during a print process. This would provide insight into the various printing parameters, and how they affect each material's microstructure. Additive manufacturing can produce complex, anisotropic 3D structures that rely heavily on their microstructure.[15] It is imperative to fully understand these resultant microstructures as a result of processing conditions to determine optimal processing parameters, especially for materials designed for high-stress environments.

Previous research has been conducted by *Proudhon et. al.* on crack fatigue in Al and Ti alloys using DCT[16]. *Nakai et. al.* also explored fatigue by looking at the misorientation of grains after radiation to stainless steel[17]. Time dependent studies have also been performed to observe the effect of precipitation on Al alloys.[18] 3D shape and orientation of grains from DCT in metals and alloys could be further explored at the lab in terms of crack growth modeling and material corrosion-fatigue.[19]

Void analysis plays a huge part of the productivity of the current micro-CT system. In addition to the standard void analysis, DCT has been recently used to explore damage that can occur around a void dependent on elastic and plastic properties of HE crystals. The crystal orientation affects not only these properties, but also the damage mechanism of the material, which can be observed via DCT.

The research that has been conducted with DCT thus far has shown its ability to help solve problems and enhance research in the materials science world. This new technique is still growing and expanding its capabilities in the way polycrystalline materials are studied, and would greatly benefit the research and development at many departments across Los Alamos National Laboratory.

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