MD Technical Review Letter -

Diffraction Contrast Tomography
Latest Development and Possible Applications on Diamonds

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Diffraction contrast tomography is a new emerging technology moving from synchrotron to laboratory. It combines traditional X-ray computed tomography and X-ray diffraction, providing three-dimensional structural and crystallographic information over a sample non-destructively. Experiments have been done to reveal the crystallographic information of diamonds. It has great potential in the diamond industry for possible applications on the inspection of rough diamonds and clarity checking for polished diamonds.

Introduction

Crystallographic information is important for a material. It can tell how the crystals orientate in the material, the strain in the material, etc. There has been a lot of effort working on techniques retrieving crystallographic information, such as X-ray diffraction (XRD) and electron backscatter diffraction (EBSD). Although these methods are now easy to access and apply, they have certain limitations.

XRD is very common in laboratories nowadays as a useful technique to measure crystallographic information. The advantage of XRD is the non-destructive nature of X-ray to non-biological materials. There are some advance in measurement strategies, like two-dimensional data collection [1]. XRD can also retrieve two-dimensional crystallographic information by sample mapping. However, the applicable sample depth is limited by the attenuation of X-ray beam so the applicability of XRD is also limited by the sample nature, geometry, thickness, surface flatness, etc.

On the other hand, EBSD can give detailed crystallographic information. Implemented in scanning electron microscope (SEM), EBSD detects electron backscattering pattern for the crystallographic information [2]. With electron scanning through the surface, two-dimensional crystallographic information can be retrieved. EBSD can be advanced into three-dimension (3D EBSD) by the combination of focused ion beam (FIB) [3]. 3D EBSD is now the gold standard of three-dimensional crystallography. However, 3D EBSD needs serial sectioning of sample by FIB, which is a destructive testing method, prohibiting its application on time-evolution studies and precious samples.

Developing from synchrotron, Diffraction Contrast Tomography (DCT) [4] [5] now can provide an alternative way to existing techniques to retrieve three-dimensional crystallographic information non-destructively.

Principle

DCT is a novel technique combining X-ray computed tomography (CT) and XRD [6] [7]. CT is a well-developed technology in both medical and material sciences. By using penetrating X-ray irradiation, the structural information can be retrieved from absorption contrast [8]. This is particular important in non-destructive testing of materials. DCT can retrieve crystallographic information like XRD and determine microstructure non-destructively like CT [4] [5].

The fundamental equation of DCT is the same as XRD, which is the Bragg law [1] [9]

$$2d \sin\theta = n\lambda$$

where λ is the wavelength, d is the distance between each adjacent crystal planes, θ is the Bragg angle at which one observes a diffraction peak, and n is the order of reflection. The experimental setup of DCT is similar to CT. X-ray is irradiated on the sample, which is rotating between the source and X-ray detector. When the sample is irradiated by X-ray, photons will interact with the sample material. Materials with higher density will give a lower transmission of X-ray and vice versa. Therefore, when the transmitted X-ray beam reach the detector, absorption contrast images can be formed.

At the same time, photons satisfying the Bragg law at the crystal grains will be diffracted away from the central main beam. To capture these diffracted photons, an aperture is added to allow central X-ray beam illuminating the sample only. There will be no X-ray illuminating the outer part of the detector so the diffracted photons can be captured by the detector. These diffracted photons appear as diffraction spots. By conservation of energy, the crystalline grains satisfying the Bragg law will have less photons reaching the detector, leaving extinction spots with additional diffraction contrast.

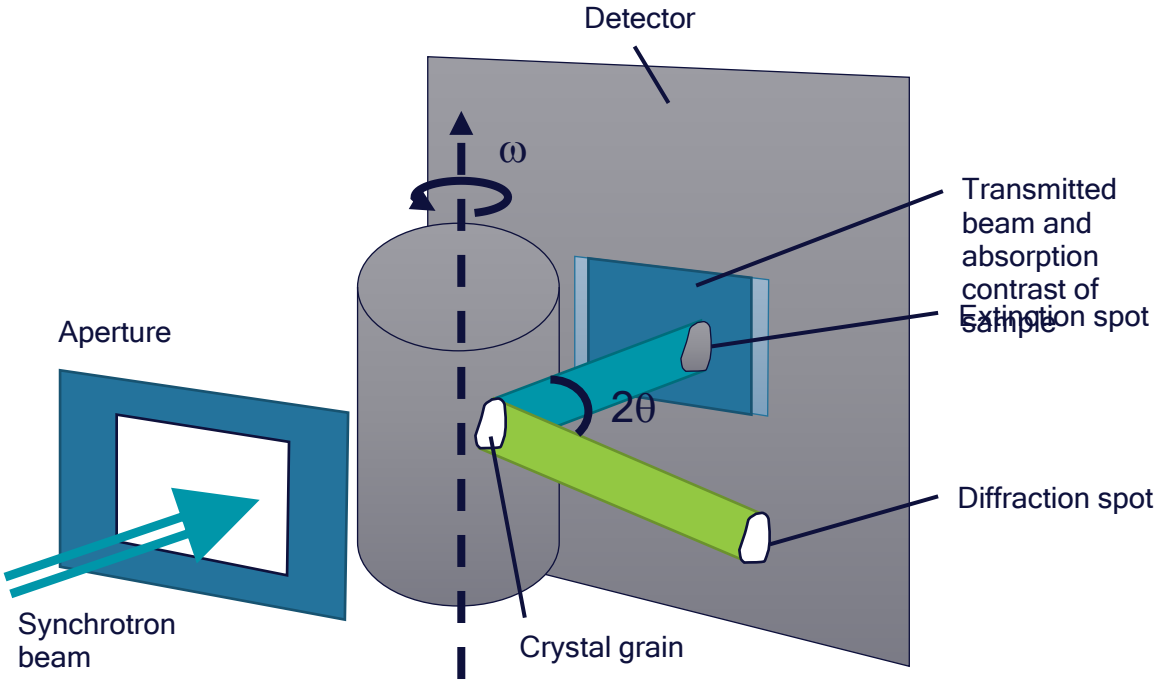

Figure 1 Schematic diagram of DCT with synchrontron X-ray source.

The diffraction contrast of the extinction spots can give the structural information of the crystal grains satisfying the Bragg law. On the other hand, the position of the diffraction spots can give the 2θ information. Hence, the orientations of the crystal grain can also be obtained. By correlating the diffraction spots and extinction spots, the three-dimensional structural and crystallographic information can be obtained non-destructively.

Latest Development

DCT was first developed in synchrotron facilities, in which monochromatic X-ray beam is used. Recently, the technology has been brought to laboratory (LabDCT) [10]. As in synchrotron, the experimental setup is similar to CT with addition of aperture. The main difference is the nature of X-ray source. In laboratory, there is no well collimated and monochromatized parallel X-ray beam. Instead, the traditional method, using accelerated electrons to hit metal target, is used to produce polychromatic X-ray cone beam.

Because of the continuous energy spectrum of polychromatic X-ray, LabDCT has diffraction spots moving in a wide range of angles instead of particular angle during sample rotation. In other words, the dispersion of λ gives a wide range of θ in the Bragg law. On the other hand, because of the energy dependence of intensity as a result of Bremsstrahlung and characteristic radiations, the intensities of diffraction spots also change with the Bragg angle θ.

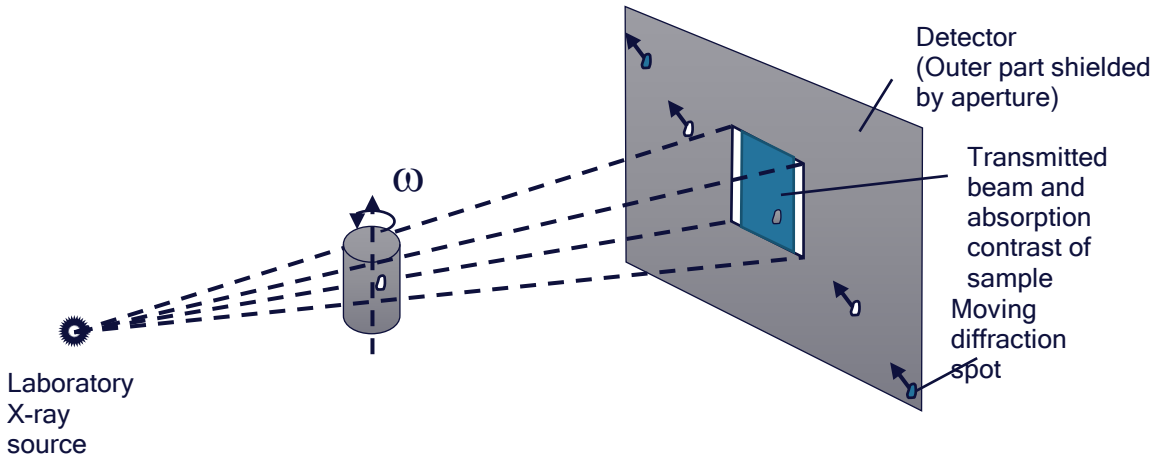

Figure 2 Schematic diagram of DCT in laboratory X-ray source.

LabDCT has now been implemented in commercial X-ray instrument [11] [12]. The instrument uses polychromatic X-ray cone beam with energy up to 160 keV and is able to obtain three-dimensional crystallographic information over sample with volumes up to 8 mm^3 [12]. Moreover, because of the non-destructive nature of LabDCT, the instrument is able to conduct time-evolving “4D” experiments, such as material change under heat or stress over time.

Possible Applications on Diamonds

Diamond is the single crystal of carbon with face-centered cubic (fcc) structure. The lattice constant is around 3.567 Å at 300 K [13]. Therefore, diamond can give sharp diffraction spots when illuminated with X-ray. Fig. 3 shows the diffraction spots of 3 diamonds contained in a bottle of charcoal, an amorphous form of carbon. Only diamond single crystals give distinct diffraction spots.

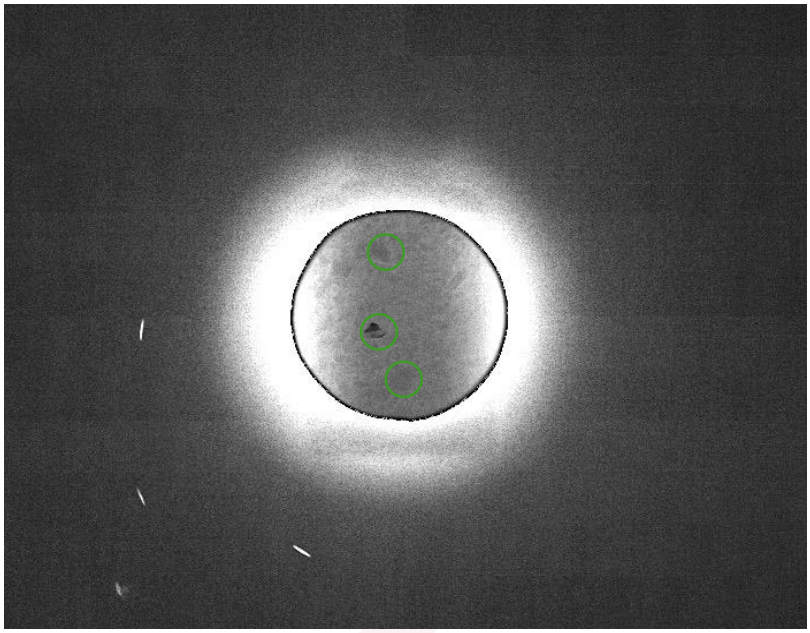

Figure 3 Diffraction spots of diamonds of 3 diamonds contained in a bottle of charcoal. The circles drawn indicate the position of the diamonds on the absorption contrast image inside the aperture. One of the diamond was stained with silver paint for ease of reconition.

The different diffraction behaviors of diamond crystals and amorphous carbon can provide a useful tool in inspecting rough diamonds. Rough diamond is the uncut and unpolished raw

diamond mined directly from the ore. Rough diamonds are not transparent on the surface and look dull as in the right image of Fig. 4. Therefore, whether a rough diamond contains single crystal diamond inside is a question. Traditional X-ray CT can give a distribution of impurity of the rough diamond but no information on crystallinity. DCT can detect the presence of single crystals by sharp diffraction spots. This can assist the diamond industry to select high quality rough diamonds.

In Fig. 4, the left image is the DCT image of a rough diamond measured in Master Dynamic Ltd. Only very fine and random distribution of diffraction spots can be seen. This indicates that the rough diamond contains only polycrystalline diamond. The brighter rod shape spot at the lower right corner indicates that there is a small region with better crystallinity but still not a sharp diffraction spot indicating the presence of a single crystal.

Figure 4 Left: DCT of a rough diamond. Right: Appearance of the rough diamond.

On the other hand, although polished diamond is the single crystal of carbon, most of the diamond is not perfect. These imperfections, if can be visible under 10X microscope, are called inclusions. Some of the inclusions are related to imperfection of crystallinity. For example, tiny crystals can be present inside the diamond, resulting in pin points, needles or clouds. There can also be twinning, distortion or irregularities of crystal growth, which can result in grain center, internal graining or twinning wisp. [14] All these kinds of inclusions are crystal related so they can also be detected by DCT in principle.

Conclusion

DCT is a new emerging technology moving from synchrotron to laboratory. It combines traditional X-ray CT and XRD, providing a three-dimensional crystallographic information over a sample non-destructively. It has great potential in the diamond industry for possible applications on the inspection of rough diamonds and clarity checking for polished diamonds.

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