Brief Report

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The Debye–Scherrer technique – rapid detection for applications

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Abstract: The Debye–Scherrer (DS) technique is a common technique for determining spacings in atomic layers by X-ray diffraction. The spacings of the atomic layers are proportional to the macroscopic stress in the radiated area. When a crystalline powder is irradiated with X-rays, the diffracted X-rays produce a ring that photographic films detect. Afterward, the film is developed to reveal the rings. Earlier, this procedure took lots of time. With upcoming multiple wavelength anomalous dispersion-detectors, a very sensitive area detector, DS rings can be detected and analyzed in minutes. This allows for the rapid determination of residual stresses prior to inspection in a production environment. Additional information from the intensity distribution within the ring can be obtained. A description of the new technique is given.

Keywords: Debye–Scherrer, MAD-detector, residual stress, intensity distribution, X-ray

1 Introduction

In the years 1916–1917, Peter Debye and Paul Scherrer developed a method to determine the atomic structure of crystal powders that was later called the Debye–Scherrer (DS) method [1]. It has been one of the standard methods for more than 100 years up to today. For a long time, photo plates were used to record the rings for investigations. It was very time-consuming, and only geometrical information about the DS ring could be obtained. With the upcoming new detectors, called multiple wavelength anomalous dispersion (MAD), new opportunities will appear, and fast analysis can be done. After explaining the basics, the determination of residual stresses is described as one possibility also usable in production lines.

2 Basics

2.1 DS method

The DS method is based on Bragg's law [2]. When monochromatic X-rays are shot at a specific angle θ (between X-rays and the surface of the solid), high intensity scattered X-rays are obtained because they interfere constructively. Knowing the wavelength λ of the X-ray and measuring θ, the lattice distance d of the atoms can be calculated.

Because powders are used as polycrystalline materials, different orientations of the atomic layers exist, and all scattered X-rays create a ring known as the DS ring. Reflection at different atomic layers produces rings with different diameters and can be detected simultaneously. In ref. [2], a detailed explanation is given.

Because of backscattering, a second ring appears in the direction of the incident beam that fulfills the same properties as the other ring.

2.2 MAD-detector

This further type of detector described is called today the MAD-detector. In Figure 1, such a detector is shown. It is a plate with a layer of photostimulable phosphor ($BaFBrEu^{2+}$). This layer is sensitive to X-rays with an energy between 8 and 17 keV [3].

After exposure with X-rays, electrons are captured at a higher energy level. After exposing the layer to a Helium–Neon laser light (λ = 633 nm), photons with a wavelength of λ = 390 nm are emitted. The number of photons is proportional to the incident X-ray intensity. The photons are detected by a photomultiplier and the

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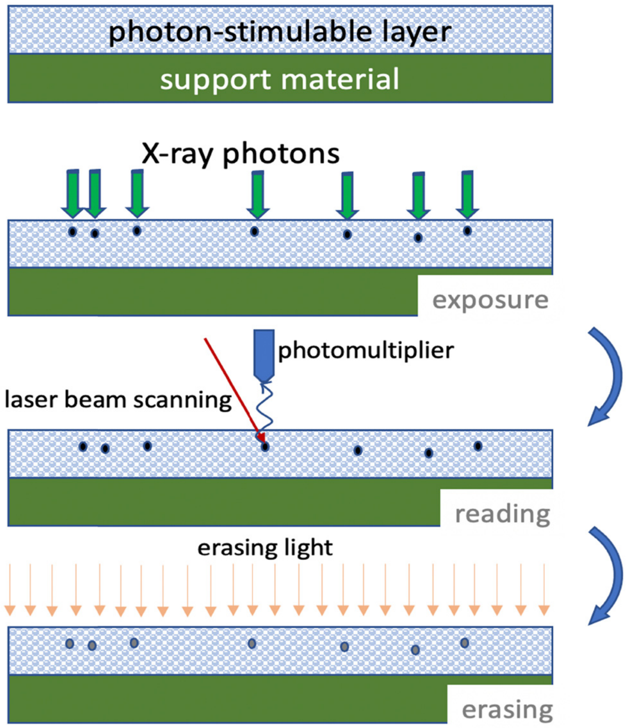

Figure 1: Working sequence of an MAD-detector (adapted from ref. [3]).

signals from the multiplier are further processed. The resolution is determined by the laser beam and not by the photomultiplier. The last step is erasing the plate with light of high brightness at a wavelength greater than 633 nm. This sequence is shown in Figure 1.

The whole process of reading the information from the detector lasts about 15 s. The erasing takes only some seconds.

MAD-detectors with diameters more than 300 mm are now available. A dynamic range of up to 10^5 can be reached, which causes a wide range of materials to be measured [4].

3 Example for application

The combination of the DS method and this new kind of detector opens a new field of application. If the spacing of the atomic layers is a little bit different, e.g., because of an external force, the reflection angle θ must change a little bit, respectively, the DS ring moves a little bit on the plate (Figure 2). The atomic layers must be a little bit closer together by compressive stress (force).

A solid or component contains not only external forces but also internal stresses. The presence of internal stresses has different origins (*e.g.*, different temperature

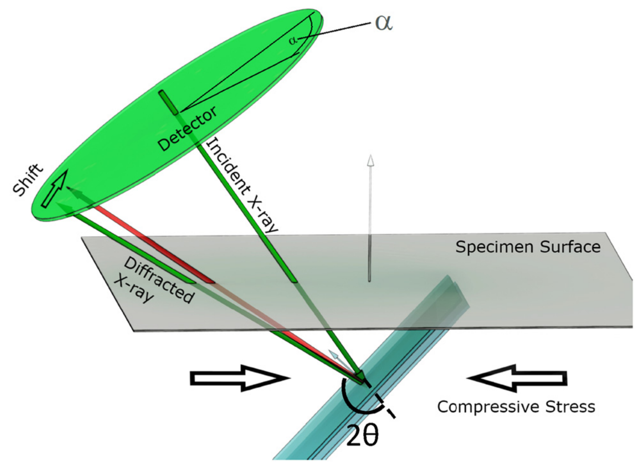

Figure 2: Shift of the DS ring by compressing the specimen (α azimuth angle on the detector).

distribution by cooling down a component or mechanical work at the surface). This fact induces residual stress in the component.

Tensile residual stress extends the spacing of the atomic layers proportional to the elastic macroscopic strain. This fact causes cracks and the final failure of the component.

The amount of local shift of the ring is proportional to $\cos\alpha$ (azimuth angle of the ring, Figure 2), respectively, to the normal stress at this radiated spot [5]. A DS ring also obtains a deformation of the round shape that is proportional to $\sin\alpha$, respectively, shear stress [6]. A complete ring is not necessary for analysis.

Taking the intensity distribution perpendicular to the ring (like a Gauss curve), the full width of half maximum gives a relative number of the dislocations in the material [7].

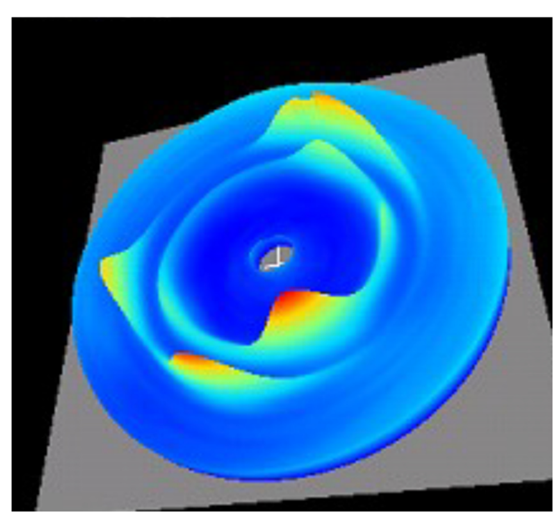

Figure 3: Different intensities of DS rings recorded with an MAD-detector (80 mm diameter of the sensitive plate).

If the intensity along the ring (Figure 3) differs much, then it is an indication of coursed grain or texture in the material.

This is the fourth piece of information obtained from the ring.

By changing the angle θ and the distance of the detector to the specimen, only one ring appears in many cases, making automatic analysis quick and reliable.

Figure 3 is from the X-ray diffractometer Pulstec μ-X360s that uses backscattering. In the middle of the detector plate is the aperture of the X-ray beam. The radiated area on the specimen has diameters between 400 and 4,000 μm and an expose time between maximum 4 min and 5 s for ferrite steel depending on the aperture used. The parameter is calculated in less than 30 s. It can also be used for portable applications.

For industrial quality assurance, a diffractometer has been developed with fixed settings and higher intensity. In this case, the whole period from exposing, reading the detector, and analyzing is reduced to 10 s.

4 Conclusion

Analyzing whole DS rings with MAD-detectors gives a good opportunity to obtain numerical values of normal and shear stresses, a relative number of dislocation density, and an indication for coursed grain in minutes.

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