METHOD AND APPARATUS FOR DIFFRACTION MEASUREMENT USING A SCANNING X-RAY SOURCE

BACKGROUND OF THE INVENTION

Previous to this invention, X-ray diffraction (XRD) measurements were made in a slow and tedious manner. FIG. **1** shows a schematic of the prior art, a typical X-ray diffraction apparatus. It comprises an X-ray source A, a collimator B, a specimen C, and an x-ray detector D. In the prior art the collimator is attached directly adjacent to the x-ray source. The collimator B changes the wide-angle output of the X-ray source A into a narrow beam. The x-ray beam E is aimed towards the specimen. The beam of X-rays E interacts with the specimen C. Some of the X-rays are diffracted by the specimen and are redirected towards the detector D. The X rays pass through the aperture F prior to entering the detector D. The characteristics of the diffracted X-rays are subject to mathematical description.

The Bragg Equation

The Bragg Equation:

$$n\lambda=2d(\sin\theta) \quad (1)$$

describes X-ray diffraction. Lambda, X, is the wavelength of the diffracted X rays in Angstroms. Theta, θ, is ½ of the diffraction angle 2θ, which is the angle between the incident and diffracted X rays. The crystalline lattice plane spacing is "d", in Angstroms. For non-crystalline materials "d" is the interatomic spacing. The order of diffraction is an integer, "n". For diffraction, "n" is never less than 1. It is well known in the art that for X-ray diffraction to occur, lambda, theta, and d must have the relationship described by the Bragg equation.

In the typical XRD apparatus, the x-ray source and its collimator remain fixed. The X-ray wavelength, λ, is limited to a single value. The point detector and/or the specimen are moved. The angle 2θ, is thus measured allowing the detection of the various "d" spacings present in the specimen. Alternatively, 2θ may be held fixed and the wavelength, λ, varied. The prior art XRD apparatus may be utilized in one of the following XRD methods.

Powder Method

The XRD apparatus may be used with a powdered or polycrystalline specimen. It is assumed in the analysis that the crystallites of the powdered specimen are randomly oriented and that there are a great many crystallites illuminated by the incident beam. This allows the detector to be scanned in one dimension rather than two-dimensions. If the crystallites in the specimen are not randomly oriented, the emerging diffraction pattern will not be a series of uniform rings, but a constellation of spots or perhaps mottled rings. A linear scan would not correctly gather all the information contained in these mottled rings or spots. Specimen preparation for this type of analysis is time-consuming and requires skill and care.

When a specimen has few crystallites, or non-random orientation of its crystallites, the specimen can be rotated about one or more axes during the test. This simulates a specimen with random crystalline orientation distribution. However, the required movement often increases the time needed to perform the test. The data collected during a test where the specimen is rotated may be collected in a manner that preserves the rotation orientation information. The preferred crystalline orientation (also known as "texture") may be sensed using this technique.

Laue Equations and Method

The Laue method exploits the full, three-dimensional nature of X-ray diffraction. The Bragg equation is a simplification of the three-dimensional Laue Equations:

$$a1\cdot(S-So)=h\lambda \quad (2)$$

$$a2\cdot(S-So)=k\lambda \quad (3)$$

$$a3\cdot(S-So)=l\lambda \quad (4)$$

X rays diffracted from a single incident beam, So, are diffracted in three-dimensions, not just in a single plane. The directions of the incident and diffracted beams are represented by So and S, respectively. The crystal lattice vectors are a**1**, a**2**, and a**3**. The Miller indices are h, k, and l. The variable λ is wavelength, as it is for the Bragg equation, (1).

In the Laue method, the broad-spectrum ("white") X-ray source is collimated to a thin, pencil-like beam. A specimen, usually a single crystal, is placed in the path of the pencil x-ray beam. X rays are diffracted by the specimen and emerge in a variety of directions, as described by the Laue equations. A sheet of photographic film, or some other area detector, is placed near the specimen. The sheet of film may be placed behind the specimen, between the specimen and the X-ray source, or nearly anywhere as required by an operator and as dictated by the application. X rays that are diffracted by the specimen travel to the film and produce a "Laue pattern." If the specimen is a single crystal, the Laue pattern is a set of small spots. The film (or a typical position-sensitive area detector) does not record wavelength information, only intensity and location information. By knowing the relative location and orientation of the X-ray beam, the specimen, and the plane of the film, it is possible to accurately calculate 2θ for each of the spots.

Since there are usually a great many spots on the film, it is possible, by trial and error, to ascertain the crystalline orientation of the specimen, the crystal structure of the specimen and the dimensions of the crystal unit cell. However, it can be a tedious process.

Since film, and other position-sensitive area detectors cannot achieve high resolution for both position measurement and wavelength measurement, the Laue method is used principally on single crystals. This is because, without wavelength information, it is impossible to use the trial-and-error method if more than one crystal is illuminated by the X-ray beam. The lack of a practical means of gathering wavelength information as part of a Laue measurement severely limits the application of this method.

Area Detectors Vs Point Detectors

An "area" detector is sensitive to X rays in a plane, generally oriented normally to the diffracted X-ray beam. A line detector can also be scanned to function as an area detector. Typically, area detectors are not able to measure the wavelength of the X rays, only the intensity and position within the detector plane.

A "point" detector, as it is known in the art, is insensitive to position within its sensing region. The detector does not detect X-rays at an actual point, but over its volume. It does not distinguish where, within its volume, the X-ray photon was detected. Point detectors are often "energy-discriminating." That is, they produce a signal that can be processed to determine the wavelength of the detected X-ray photon.

Mono-chromatic vs. "Color" Detectors

An "area" X-ray detector may be substituted for the "point" X-ray detector described in the XRD methods above. An area detector is sensitive to X rays in a plane, generally oriented normally to the incident X-ray beam,