NONDESTRUCTIVE METHOD AND APPARATUS FOR IMAGING GRAINS IN CURVED SURFACES OF POLYCRYSTALLINE ARTICLES

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NONDESTRUCTIVE METHOD AND APPARATUS FOR IMAGING GRAINS IN CURVED SURFACES OF POLYCRYSTALLINE ARTICLES

Field of the Invention

The present invention relates to a method and apparatus for determining the grain flow in a curved surface of a polycrystalline article.

Background of the Invention

Although there are a number of established techniques for providing high resolution images of the internal structure of large single crystals, as is discussed below, these techniques are not suitable for verification of the grain flow in a curved or a convex surface of a polycrystalline sample, such as the curved surface of an article made of aluminum. In such an article, the grains of the curved surface of the formed article are flattened during the forming process and are elongated in one direction, with the relatively large faces thereof oriented parallel to the curved surface. It is noted that rotation of these grains about the longitudinal axis into a position perpendicular to the curved surface would make imaging of the grains easier to accomplish but such an approach is undesirable because of the resulting changes in the formed article so produced. On the other hand, the flattened elongated grains of the formed article present special problems insofar as providing high resolution imaging of the grains is concerned.
As mentioned above, there are established methods for providing high resolution images of large single crystals and in this regard, x-ray topography techniques have been used for several years to provide high resolution images of the internal structures of such large single crystals. These methods are used to produce a point-by-point correspondence between the incident x-rays striking the surface and the diffracted x-rays striking a film. Such x-ray topography methods are described, for example, by B.K. Tanner, in "X-Ray Diffraction Topography," Pergamon Press, New York, N.Y. (1976). A further reference in this field which describes x-ray metallography techniques of interest, including the Berg-Barrett method discussed below, is A. Taylor, "X-Ray Metallography," John Wiley & Sons, Inc., New York, London (1961). The Berg-Barrett method just referred to provides for locating the sample a long distance from the x-ray source so that the x-ray beam will appear to be nearly parallel, and then placing the film very close to the sample surface to limit the divergence of the diffracted beam. A Berg-Barrett method has been used for producing images of grains in polycrystalline uranium. This method is described by L. Le Naour, in "X-Ray Topography of Uranium Alloys," ORNL-tr-5069, (translated from the French CEA Report, CEA-R-3494), Union Carbide Corporation, Nuclear Division, Oak Ridge National Laboratory (May 1968). Methods based on crossed-soller slits for limiting the divergence of the diffracted beam have produced images which show texture variations in rolled aluminum samples. These methods have been described by Y. Chikauro, Y-Yoneda and G. Hildebrant, in "Polycrystal Scattering Topography," J. Appl. Cryst., 15, 48 (1982).

In each of these methods, parallel and/or divergent x-ray beams are directed toward samples with flat surfaces for diffraction thereby, and these
methods can not be used efficiently for imaging grains in a curved polycrystalline surface.

Summary of the Invention

A principal object of the present invention is to provide an efficient method for determining the grain flow in a convex, textured, polycrystalline surface.

Generally speaking, a first aspect of the invention concerns a method for determining the grain flow of the grains in convex curved surface of a polycrystalline article, wherein the method comprises: directing a monochromatic converging X-ray beam onto the curved surface of the polycrystalline article so that the converging beam is diffracted by the grains in that surface of the article; limiting the height of the diffracted beam; measuring the linear intensity of the height-limited diffracted X-ray beam at a plurality of positions along the surface of the article in a direction orthogonal to the width of the beam; and producing an image of the grain flow in the curved surface based on the intensity measurements.

In accordance with a preferred embodiment of the invention, a method is provided for determining the grain flow in a convex surface of a polycrystalline article, wherein the method comprises the following steps or operations:

i) aligning the curved surface of the polycrystalline article in a horizontal X-ray diffractometer;

ii) directing a monochromatic, converging X-ray beam onto the curved surface of the polycrystalline article so that the converging X-ray beam is diffracted by the crystallographic planes of the grains in the polycrystalline article;
iii) passing the diffracted x-ray beam through a set of horizontal, parallel slits;

iv) measuring the linear intensity of the diffracted x-ray beam after the beam passes through the set of horizontal, parallel slits, using a linear position sensitive proportional counter, and as a function of position in a direction orthogonal to the counter; and

v) providing an image of the grains in the curved surface of the polycrystalline article based on data from the intensity measurement.

A specific application of the x-ray diffraction imaging method of the invention is in determining the grain flow in a convex, textured, polycrystalline surface of aluminum. In this application, the preferential orientation of the (110) crystallographic planes is parallel with the convex surface. However, it will be understood that the method of the invention is not limited to imaging grains in this type of polycrystalline surface.

In accordance with a further aspect of the invention, an apparatus is provided for determining grain flow in a convex curved surface of a polycrystalline article, said apparatus comprising means for producing monochromatic, convergent incident x-ray beam for diffraction by the crystallographic planes in the grains of the polycrystalline article; a set of horizontal, parallel slits for limiting the height of the diffracted beam; a linear position sensitive proportional counter for measuring the linear intensity of the diffracted beam and producing a digital output in accordance therewith; and means for step-scanning the article through the x-ray beam in a direction orthogonal to the counter, i.e., orthogonal to the width of the height-limited beam.
Other features and advantages of the invention will be set forth in, or apparent from, the following detailed description of preferred embodiments of the invention.

**Brief Description of the Drawings**

Figure 1(a) is a schematic drawing showing x-rays diffracting from grains in a polycrystalline sample with a flat surface, wherein a parallel x-ray beam is used for diffraction;

Figure 1(b) is a schematic drawing showing x-rays diffracting from grains in a polycrystalline sample with a curved surface, wherein a parallel x-ray beam is used for diffraction;

Figure 2 is a schematic drawing illustrating an aspect of the method of the present invention involving the diffraction of a convergent, monochromatic x-ray beam from a curved surface of a polycrystalline sample;

Figure 3 is a schematic side elevational view, partly in a block diagram format, of one preferred embodiment of the apparatus of the invention;

Figure 4a is a gray-scale image of the intensity data collected using the method and apparatus of the invention in determining the grain flow in a contoured region near the equator of an aluminum hemisphere;

Figure 4b is a gray-scale image of the intensity data collected using the method and apparatus of the invention in determining the grain flow in a contoured region near the pole of an aluminum hemisphere.

Figure 5 is a perspective view of an apparatus similar to that of Figure 3, wherein the sample is cylindrical.

**Description of the Preferred Embodiments**

Before considering method and apparatus of the present invention, certain other approaches or techniques will be considered. Referring to Figure
1(a), the grains in a flat polycrystalline sample 10 are imaged using the Berg-Barrett technique referred to above, with the incident parallel beams of light being diffracted by surface 10 on a film 12. In Figure 1(a) (and Figure 1(b)) the angle $\Theta_s$ represents the Bragg angle, required for compliance with Bragg's Law, which is given by the equation $\lambda = 2d \sin \Theta$, wherein $\lambda$ equals the radiation wavelength, $d$ equals the d-spacing, or distance between diffracting planes, and the angle $\Theta$ equals the angle between the incident x-ray beam and the crystal planes in the sample.

Referring to Figure 1(b), the same technique is used with a convex specimen surface 10'. Figure 1(b) indicates that only the preferentially-oriented crystal planes near the point where angle $\Theta$ equals $\Theta_s$ will diffract from a curved surface of a polycrystalline sample when the usual Berg-Barrett technique is used.

As stated above, an important aspect of the present invention concerns the use of a convergent incident beam as the incident beam in imaging of the grains. The use of such a convergent incident beam is shown schematically in Figure 2. The convergent beam can be produced by a commercially available monochromator. In Figure 2, the position of the curved surface of the sample has been adjusted in accordance with the location of the focal point for the radiation beam so as to limit the incident angle to $\Theta_s$. This distance is provided by the equation: $R = R' \sin \Theta_s$, where $R$ is the distance from the sample surface to the focal point of the radiation beam, $R'$ is the radius of the curved surface of the polycrystalline sample, and $\Theta_s$ is the angle of diffraction for the crystal planes in the polycrystalline sample.

Before considering Figure 2 further, reference is now made to Figure 3, wherein some of the basic components of an apparatus constructed in
accordance with a preferred embodiment of the invention are shown. It will be
noted that some of these components or units are shown in somewhat more
detail in Figure 5 which is described below. These components, which are part
of a horizontal x-ray diffractometer and are basically conventional, include the
crystal 20 of a monochromator (not shown), and a position sensitive detector 22
including a horizontal slit housing 24, i.e., a housing which provides a set of
horizontal slits (not shown in Figure 3) in front of the window (not shown) of the
detector 22. A multichannel analyzer 26 is connected to the output of detector
22 while a stepping mechanism or device for stepping the sample 10" in a
direction perpendicular to the horizontal plane is indicated at 28. The output
signal from the position sensitive detector 22 is a signal whose amplitude is
proportional to the position of the x-ray along the detecting element.

The height of the diffracted beam from the polycrystalline sample 10" is
limited to a line tangent to the curved surface in the plane of Figure 2 by using,
referring to Figure 3, the set of horizontal, parallel slits referred to above. As
stated, these slits are provided in housing 24 in front of the window (not shown)
of the position sensitive detector 22, which preferably comprises a linear-position
sensitive proportional counter. As illustrated, the linear position-sensitive
proportional counter (detector) 22 is disposed or stationed as close as
physically possible to the polycrystalline sample 10". It will be understood that
the loss of resolution caused by the divergence of the diffracted beam is limited
by closeness of the sample 10" and counter 22. The sample is step-scanned
using stepping device 28 in a longitudinal direction from the equator to the pole
of the curved surface, and linear-intensity data are produced at each step, these
data being representative of grain images.
Considering a specific example, a hemispherical-shaped article was back-extruded from a plate of aluminum alloy for testing the apparatus of the invention. In a back-extrusion operation, the grains in a polycrystalline plate of aluminum alloy are flattened, elongated and oriented preferentially in a position with their flat surface and longitudinal axes parallel to the surface of the hemisphere. The back-extruded hemisphere 10" was aligned in a x-ray diffractometer of the type illustrated in the schematic showing provided in Figure 3.

In the test, the quartz focusing monochromator (represented in Figure 3 by monochromator crystal 20) diffracted the incident beam to a focal point about 210 mm from the monochromator crystal 20. The convergent nature of the beam was due to the geometry of the crystal, which is known as Johansson geometry. In Johansson geometry, the diffracting planes of the monochromator crystal are inclined to the surface at approximately 10°. In addition, the diffracting surface is ground with a slight radius and then bent to the same radius. The reflected beam was intercepted by the sample 10", i.e., the hemisphere of the aluminum alloy, at a theta angle of 32.38° which corresponds to the theta angle required for diffraction of the (220) crystal plane. The sample surface was struck with each proton at the same theta angle by properly positioning the sample along the converging incident beam. This ensured diffraction of only those grains which were nearly parallel with the surface.

The vertical divergence of the diffracted beam was limited by passing the beam through a pair of the horizontal slits of the housing 24 prior to entrance of the beam into the linear-position sensitive detector 22. The intensity data from the linear position sensitive detector 22 were collected in the multichannel analyzer 26 in which the channels represented position along the
wire in the linear-position sensitive detector 22. These data represented a one-dimensional image in the horizontal plane of the grains in which the (110) planes were nearly parallel to the surface. Two-dimensional imaging information was obtained by using stepping mechanism 28 to provide stepping of the sample in a direction perpendicular to the horizontal plane, in steps equal to the vertical divergence of 0.1 millimeter. After converting the channels and steps to x-y positions, the intensity data were used to produce images of the grains.

In the example being considered, the resolution of the data was limited by the resolution of the linear-position sensitive detector 22, by the vertical divergence of the diffracted beam, and by the departure of the diffracted beam from the divergent path, shown in Figure 3, for the horizontal plane. The resolution for the linear-position sensitive detector 22 is believed to be approximately 0.06 mm. The vertical divergence of the diffracted beam was limited by the set of horizontal slits of housing 24. The mosaic spread, as well as the diffusivity of the (110) texture, determined the extent of departure of the diffracted beam from the horizontal divergent path. As noted above, the detector 22 was stationed as close as physically possible to the sample for minimization of this effect. Photomicrographs of the sample revealed that many grains were as large as 1 to 2 mm. Consequently, it is believed that horizontal slits 0.1 mm in width can be used without significantly compromising the resolution. The step size in the y-direction corresponded to 0.108 mm, while each channel in the multichannel analyzer (1024 channels) represented 0.117 mm in the x-direction. The counting time at each step was 1200 seconds.

The sample 10" described above in connection with Figure 3 was examined in the finished condition near the equator and near the pole. The image of intensity data taken near the equator of the sample is shown in
Figure 4a. The image of intensity data taken near the pole of the sample is shown in Figure 4b. In the images of Figures 4a and 4b the horizontal direction is parallel to the equator and the vertical direction is from the equator to the pole. In the high-intensity regions near the equator of the sample, the grains were relatively large and elongated in the direction of the pole region. However, the grains in the high-intensity regions near the pole of the sample were relatively smaller and nondirectional. In other words, a distinct difference was obtained between the data in grain-flow near the equator and near the pole, with the data showing large, flat, elongated grains running parallel with the surface in the equatorial region, while in the polar regions, these grains were rotated so that their elongation directions were perpendicular to the surface, so as to give the appearance of having been flattened in the polar direction. It will be appreciated that quantitative relationships can be developed in terms of grain size and orientation from the data used for the grain flow images.

Referring to Figure 5, an embodiment of the apparatus of the invention is shown which is similar to that of Figure 3 but shows some of the components of the diffractometer of Figure 3 in somewhat more detail. In this embodiment, the sample, denoted 30, is cylindrical in shape and is mounted for rotation on a rotary table 32 which includes a stepping mechanism or device, indicated schematically at 34, for providing an up and down stepping movement or motion of the sample 30, as indicated by the double headed arrow 36. A monochromator 38 includes a crystal 40 (corresponding to crystal 20 of Figure 3) and an associated X-ray tube 42 which provides an X-ray focal spot 44 (corresponding to that of Figure 3). Beams diffracted by sample 30 are received by the slits, e.g., 46, of a slit array 48 (corresponding to housing 24 of Figure 3) mounted on the front of a detector 50 (corresponding to detector 22 of
Figure 3). The operation of the apparatus of Figure 5 is basically the same as described above.

Although the present invention has been described relative to specific exemplary embodiments thereof, it will be understood by those skilled in the art that variations and modifications can be effected in these exemplary embodiments without departing from the scope and spirit of the invention.
Abstract of the Disclosure

A nondestructive method, and associated apparatus, are provided for determining the grain flow of the grains in a convex curved, textured polycrystalline surface. The convex, curved surface of a polycrystalline article is aligned in a horizontal x-ray diffractometer and a monochromatic, converging x-ray beam is directed onto the curved surface of the polycrystalline article so that the converging x-ray beam is diffracted by crystallographic planes of the grains in the polycrystalline article. The diffracted x-ray beam is caused to pass through a set of horizontal, parallel slits to limit the height of the beam and thereafter. The linear intensity of the diffracted x-ray is measured, using a linear position sensitive proportional counter, as a function of position in a direction orthogonal to the counter so as to generate two dimensional data. An image of the grains in the curved surface of the polycrystalline article is provided based on the two-dimensional data.
**Fig. 1a**

- Flat specimen surface
- Preferentially oriented planes
- Incident parallel beam
- Diffracted beam
- Film

**Fig. 1b**

- Incident parallel beam
- Convex specimen surface
- Diffracted beam
- Upper surface
- Lower surface