Nuclear Instruments and Methods in Physics Research A 764 (2014) 1–6

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research A

journal homepage: www.elsevier.com/locate/nima

Technical Notes

Measurement of grain size of polycrystalline materials with confocal energy dispersive micro-X-ray diffraction technology based on polycapillary X-ray optics

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ARTICLE INFO

Article history:
Received 24 April 2014
Received in revised form
10 June 2014
Accepted 4 July 2014
Available online 15 July 2014

Keywords:
Confocal micro-energy dispersive X-ray diffraction
Polycapillary X-ray optics
Measurement of grain size for the polycrystalline materials

ABSTRACT

The confocal energy dispersive micro-X-ray diffraction (EDMXRD) based on polycapillary X-ray optics was used to determine the grain size of polycrystalline materials. The grain size of a metallographic specimen of nickel base alloy was measured by using the confocal EDMXRD. The experimental results demonstrated that the confocal EDMXRD had potential applications in measuring large grain size.

1. Introduction

The grain size is an important parameter for the polycrystalline materials, which has effects on many of their properties, such as thermal conductivity [1], microwave properties [2], electrical conductivity [3], mobility [4], magnetic properties [5], corrosion behavior [6], stress [7] and ferroelectric behavior [8]. This makes the measurement of grain size to be important for the designer and user of the polycrystalline materials. Many methods of measuring grain size have already been used, such as, microscopic examination [9], X-ray diffraction [10], ultrasonic velocity [11] and electron backscattering diffraction [12]. Among such methods, the microscopic examination is generally the most accurate. However, it is difficult to measure large grain with size more than a few hundred microns for some engineering materials [13] by using the microscopic technology. The X-ray diffraction pattern can yield semi-quantitative information about grain size together with information about crystal quality and orientation, and therefore, the X-ray diffraction is often used to obtain grain size [14,15]. Most of existing X-ray diffraction methods of measuring the grain size are used to analyze small grains [10,14,15]. In this paper, the confocal technology based on polycapillary X-ray optics was used to measure the large grain size.

In recent years, the confocal technology based on polycapillary X-ray optics has been popular. The principle of this technology was first proposed in the early 1990s by Gibson and Kumakhov [16]. This confocal technology is now widely used in three-dimensional (3D) micro-X-ray fluorescence [17,18], 3D X-ray absorption fine structure [19–21], energy dispersive micro-X-ray diffraction technology (EDMXRD) [22], small angle X-ray scattering [23], and X-ray imaging [24]. In the confocal EDMXRD technology, a polycapillary focusing X-ray lens (PFXRL) is used to focus the X-rays from the X-ray source to obtain a micro-output focal spot for exciting the sample, and a polycapillary parallel X-ray lens (PPXRL) with a micro-input focal spot is used in the detection channel for collecting the XRD signals from the sample. The output focal spot of the PFXRL and the input focal spot of the PPXRL are overlapped. This confocal configuration ensures that only the XRD signals from

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http://dx.doi.org/10.1016/j.nima.2014.07.012
0168-9002/

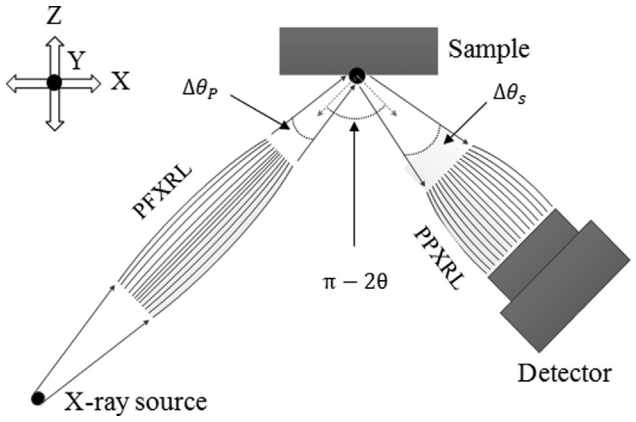

Fig. 1. Realization of confocal EDMXRD.

the confocal volume overlapped by the foci of the polycapillary X-ray optics can be detected. By moving the sample located at the confocal position, the micro-volume to be analyzed can be displaced laterally or in a direction perpendicular to its surface. Therefore, the three dimensional (3D) XRD information about the sample can be obtained point to point [25].

In this paper, the confocal EDMXRD based on polycapillary X-ray optics was proposed to be used to measure the grain size. The performances of the application of the confocal EDMXRD in measuring the large grain size were presented.

2. Experiments and results

2.1. Experimental setup

Fig. 1 schematically shows the realization of the confocal EDMXRD based on a PFXRL in the excitation channel and a PPXRL in the detection channel. The PPXRL was confocally placed with the PFXRL. The confocal configuration was helpful in improving the signal-to-noise ratio of the EDMXRD [22]. The X-ray source was an Oxford Ultra-Bright micro-focus Mo source. The energy resolution, ΔE, of the detector system was 150 eV at 5.9 keV. The convergence, $\Delta\theta_p$, of the output beam from the PFXRL and the divergence, $\Delta\theta_s$, of the beam which could be collected by the PPXRL were 85.6 and 84.3 mrad, respectively, at 5.9 keV. The resolution of lattice spacing $\Delta d/d$ can be calculated by the following equation [25]:

$$\frac{\Delta d}{d} = [(\Delta\theta \cot\theta)^2 + (\Delta E/E)^2]^{1/2} \quad (1)$$

where θ is the Bragg angle, d is the lattice spacing, E is radiation energy, and $\Delta\theta$ can be calculated by $\Delta\theta = (\Delta\theta_p{}^2 + \Delta\theta_s{}^2)^{1/2}$. The resolution of lattice spacing of the confocal EDMXRD at $\theta = 43^\circ$ was 13.1%.

2.2. Measurement of grain size

A piece of metallographic specimen of nickel base alloy was analyzed. The position of the diffraction peak changes with different diffraction angles, which can be used to distinguish the diffraction peak from the fluorescence peak with a fixed position. By this and the Bragg equation for diffraction, we confirmed that the peak at 4.73 keV in Fig. 2 was the (200) for the nickel base alloy. And the size of the confocal micro-volume depends on the size of the output focal spot of the PFXRL and input focal spot of the PPXRL. D_X, D_Y and D_Z were the size of the confocal micro-volume along the X, Y and Z axis (Fig. 1), which could be measured by using a knife-edge made of polymethyl methacrylate as a scatterer [26], and they were 76.3, 50.2 and 76.5 μm, respectively, at 4.73 keV which was the energy of the XRD signals of (200) (Fig. 2).

When the sample was scanned, a series of XRD spectra were obtained, and some of such spectra were shown in Fig. 2 (a)–(c) where the intensity of the (200) varied at different scanning points. The intensity of the diffraction peak corresponding to the scanning distance could be obtained by the sample scan (Fig. 3). The distribution of the primary X-rays incident on the sample lactated at the confocal micro-volume overlapped by the foci of the polycapillary X-ray optics is Gaussian distribution. Therefore, when a grain with a size smaller than that of confocal micro-volume scanned through this confocal volume, the distribution of intensity of the XRD signals from the sample at various scanning distances was also Gaussian distribution (Fig. 3(a)).

The full width at half-maximum (FMHM) of the scanning curve in Fig. 3(a) was related to the size of both grain and confocal micro-volume, and the theoretical relationship among them was simulated as following. Fig. 4 shows the scheme of the sample scan through the confocal micro-volume. In Fig. 4, the size of the section of the confocal micro-volume along the Y and X direction were $D_Y = 50.2$ μm and $D_X = 76.3$ μm. In the simulation, the section of the confocal micro-volume was corresponding to a two-dimensional matrix with a Gaussian-distribution. The values of the matrix elements were corresponding to the intensity of the incident X-rays. At one step in the sample scan, the corresponding intensity of the X-ray diffraction peak was proportional to the sum of the values of the matrix elements coverd by the simulative grain sample. Therefore, for given sizes of both confocal micro-volume and grain, the theoretical relationship between the size of FMHM of the scanning curve in Fig. 3(a) and the grain size S could be obtained. For a given confocal micro-volume with a size of $D_Y = 50.2$ μm and $D_X = 76.3$ μm, respectively, the theoretical relationship between the size of FMHM of the scanning curve in Fig. 3 (a) and the grain size S was shown in Fig. 5.

With the known D_X and D_Y, we obtained different fitting results for the theoretical relationship between the FMHM and the grain size S based on two different kinds of fitting formulas as following:

$$\mathrm{FWHM} = bS^2 + c \quad (2)$$

$$\mathrm{FWHM} = \sqrt{kS^2 + D^2} \quad (3)$$

where S is grain size, D is profile size of the confocal micro-volume, and the b, c and k are different constant coefficients for various S and D. As shown graphically in the fitted lines (Fig. 5), the fitting results for the theoretical computational data using two kinds of fitting formulas both have acceptable error ranges, however, the fitting result based on Eq. (2) was better than that based on the fitting Eq. (3).

According to the fitted line in Fig. 5(a) for the scanning direction Y, the diameter S of the grain with a size smaller than that of the confocal micro-volume could be obtained by following equation:

$$\mathrm{FWHM} = 0.0046S^2 + 50.1498 \quad (4)$$

According to the fit line in Fig. 5(b), the diameter S of the grain along the scanning direction X could be written as:

$$\mathrm{FWHM} = 0.0031S^2 + 75.9626 \quad (5)$$

The FWHM in Eqs. (4) and (5) was known for a given scanning curve. Therefore, the grain size, which was smaller than that of the confocal micro-volume, along the scanning direction Y and X could be obtained by using Eqs. (4) and (5), respectively. The center of the Gaussian curve in Fig. 3(a) was corresponding to the center of