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Technical Notes

Performances for confocal X-ray diffraction technology based on polycapillary slightly focusing X-ray optics

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ABSTRACT

The confocal X-ray diffraction (XRD) technology based on a polycapillary slightly focusing X-ray lens (PSFXRL) in excitation channel and a polycapillary parallel X-ray lens (PPXRL) with a long input focal distance in detection channel was developed. The output focal spot of the PSFXRL and the input focal spot of the PPXRL were adjusted in confocal configuration, and only the X-rays from the volume overlapped by these foci could be accordingly detected. This confocal configuration was helpful in decreasing background. The convergence of the beam focused by the PSFXRL and divergence of the beam which could be collected by the PPXRL with a long input focal distance were both about 9 mrad at 8 keV. This was helpful in improving the resolution of lattice spacing of this confocal XRD technology. The gain in power density of such PSFXRL and PPXRL was about 120 and 7 at 11 keV, respectively, which was helpful in using the low power source to perform XRD analysis efficiently. The performances of this confocal XRD technology were provided, and some common plastics were analyzed. The experimental results demonstrated that the confocal diffraction technology base on polycapillary slightly focusing X-ray optics had wide potential applications.

1. Introduction

The X-ray diffraction (XRD) has wide applications in such as material, medicine, life science and so on [1–3]. How to efficiently and accurately carry out XRD analysis of the sample is very important for investigators. The high power X-ray source such as synchrotron radiation is often used to obtain efficiently the XRD spectra of the sample. Such slit as Soller slit and pinhole are often used to improve the resolution of lattice spacing of the XRD technology and to decrease the effect from the background on the spectra of the sample in order to improve the accuracy of the XRD analysis. However, the slit and the pinhole are not focusing X-ray optics. In fact, there are many focusing X-ray optics which can be sued to focus efficiently X-rays from the source for XRD analysis [4–7]. Among them, polycapillary X-ray optics are especially suitable to be used to focus the X-rays from the conventional X-ray source for the XRD technology [7]. Such polycapillary X-ray optics, which was also named after Kumakhov lens [8], works on the total external reflection of X-rays, and can be used to collect the X-rays in a wide energy band from a relatively large source angle. They could be divided into three types: polycapillary focusing X-ray lens (PFXRL), polycapillary slightly focusing X-ray lens (PSFXRL) and polycapillary parallel X-ray lens (PPXRL). The confocal technology based on polycapillary X-ray optics was first proposed in the early 1990s by Gibson and Kumakhov [9]. In recent years, this confocal technology has been widely used in the 3-dimensional micro-X-ray fluorescence (3D-MXRF) technology [10–12]. In such 3D-MXRF spectrometer, a PFXRL is used in excitation channel in order to obtain a micro-output focal spot with diameter of about 30 μm, and a PPXRL with a short input focal distance and accordingly with a smaller input focal spot is used in the detection channel. Such confocal 3D-MXRF based on the PFXRL and PPXRL with a short input focal distance are used to obtain the point-to-point 3D XRF information of the sample. In order to study the potential application of such confocal micro-technology based on the PFXRL and PPXRL with a short input focal distance in obtaining the point-to-point 3D XRD information of the sample, the performance of such confocal microtechnology for XRD analysis is studied [13]. In this confocal micro-XRD based on

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the PFXRL and PPXRL with a short input focal distance, the convergence of the X-ray beam incident on the sample and the divergence of X-ray beam excited from the sample which can be collected by the PPXRL are both about 112 mrad at 8 keV. This is not helpful in improving the resolution of lattice spacing of the confocal XRD technology [13].

In this paper, in order to improve the resolution of lattice spacing of the confocal XRD technology we proposed to use the PSFXRL with small convergence and the PPXRL with small divergence to perform the confocal energy dispersive XRD (EDXRD) analysis of the sample. The performances of this confocal EDXRD were presented, and the application of this confocal diffraction technology in analysis of the plastics was studied.

2. Experiments and results

2.1. Experimental setup

Fig. 1 is a schematic diagram of the confocal EDXRD diffractometer based on the PSFXRL, PPXRL and the conventional X-ray source. As shown in Fig. 1, the divergent X-ray beam from the X-ray source positioned at an input focal distance f_1 away from the input of the PSFXRL was focused by the PSFXRL into an output focal spot at output focal distance f_2 away from the output of the PSFXRL. The PPXRL was placed confocally at an input focal distance f_3 away from the output focal spot of the PSFXRL. This PPXRL could restrict the detector field of view to ensure that only the X-rays from the confocal volume defined by the overlap of output focal spot of the PSFXRL and input focal spot of the PPXRL could be detected. This confocal configuration was helpful in decreasing background as shown in Fig. 2 with measurement time of 10 s at a working voltage of 20 kV and current of 10 mA. The X-ray source was a Mo rotating anode X-ray generator (RIGAKU RU-200, 60 kV-200 mA) whose spot size was 300 × 300 μm². The detector system was an XFlash Detector 2001 RÖNTEC and RÖNTECMAX Spectrometer. The maximum count rate of the detector system was 4 × 105 counts/s. The entrance size of the detector was 5 mm². The energy resolution of this detector system was 142 eV at 5.9 keV.

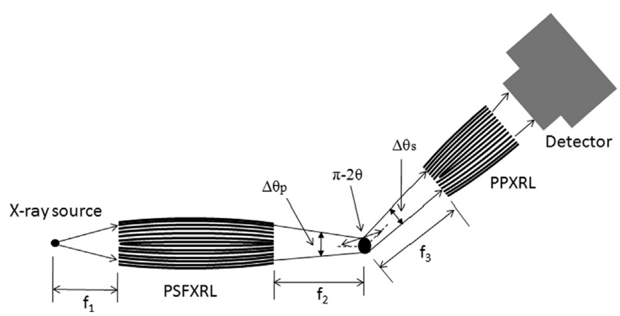

Fig. 1. Schematic diagram of confocal EDXRD diffractometer.

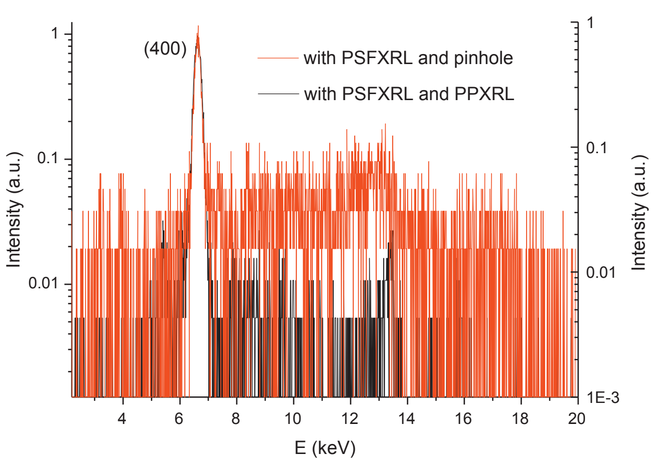

Fig. 2. EDXRD spectra of Si obtained with PSFXRL between the source and the sample and a pinhole with a diameter of 0.8 mm and PPXRL before the window of detector, respectively.

2.2. Performance of PSFXRL and PPXRL for confocal EDXRD

The input focal distance and output focal distance of the PSFXRL were 76.8 and 170.0 mm, respectively. The energy dependence of the gain in power density and output focal spot size of the PSFXRL is shown in Fig. 3. The input focal distance of the PPXRL was 160 mm. The energy dependence of the gain in power density and input focal spot size of the PPXRL is shown in Fig. 4. The PSFXRL and PPXRL used here had a longer output focal distance and input focal distance, respectively. This resulted in the smaller convergence of the X-ray beam from the PSFXRL and the smaller divergence of the X-ray beam which could be collected by the PPXRL. The energy dependence of the convergence $\Delta\theta_p$ determined by the PSFXRL (Fig. 1) and divergence $\Delta\theta_s$ determined by the PPXRL (Fig. 1) is shown in Fig. 5, which was measured using the knife-edge scan method [14]. The decrease of the convergence and the divergence with higher energies was explained by the well known $1/E$ energy dependence of the critical angle of total reflection.

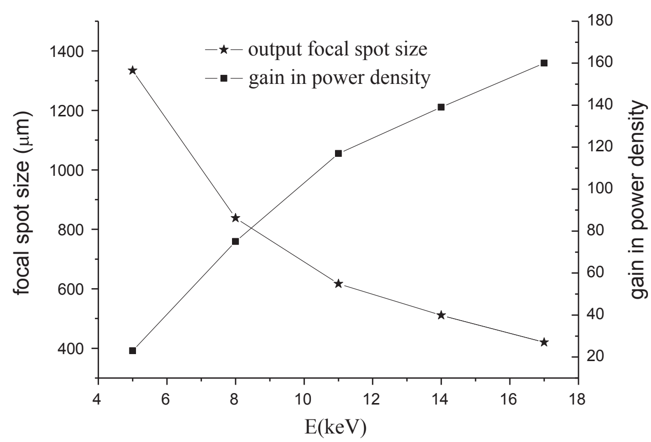

Fig. 3. Energy dependence of output focal spot size and gain in power density for PSFXRL.

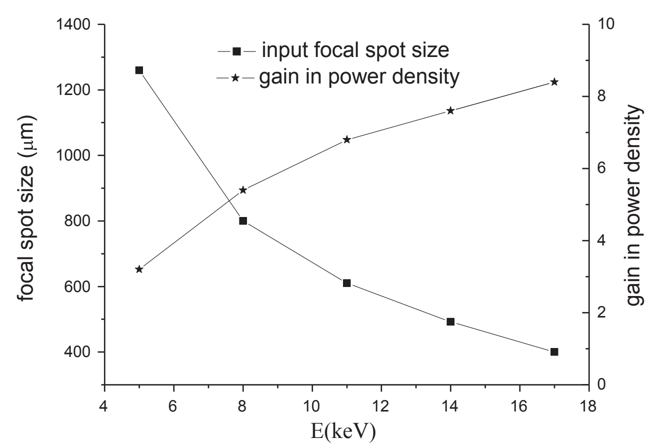

Fig. 4. Energy dependence of input focal spot size and gain in power density for PPXRL.