



Source apportionment of aerosol particles using polycapillary slightly focusing X-ray lens

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ABSTRACT

A micro-X-ray fluorescence (Micro-XRF) spectrometer based on a polycapillary slightly focusing X-ray lens (PSFXRL) and laboratory X-ray source was designed to carry out the source apportionment of aerosol particles. In the distribution curve of the X-ray intensity in the focal spot of PSFXRL, there was a plateau with a diameter of about 65 μm . The uniformity of this plateau was about 3%. This was helpful in measuring the XRF spectrum of a single aerosol particle in which the element distributions are not uniform. The minimum detection limit (MDL) of this Micro-XRF spectrometer was 15 ppm for the Fe- K_{α} . The origins of the aerosol particles at the exit of a subway station and a construction site were apportioned. This Micro-XRF spectrometer has potential applications in analysis of single aerosol particles.

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1. Introduction

Single aerosol particle analysis is a useful method of identifying origins of aerosol particles [1,2]. It can provide more detailed and reliable information about aerosol particles, which would be impossible to obtain from bulk analysis. The main single aerosol particle analysis methods are electron probe X-ray microanalysis (EPXMA), laser mass microanalysis (LAMMA), microsynchrotron X-ray fluorescence (Micro-SXRF) and microproton-induced X-ray emission (Micro-PIXE) [1–5]. The detection limit of EPXMA and LAMMA is higher than that of Micro-SXRF and Micro-PIXE. However, although the Micro-SXRF and Micro-PIXE can provide elemental analysis with $\mu\text{g/g}$ sensitivity for almost all the elements of the periodic table even for a very small, micrometer size samples, they require large and expensive facilities. In fact, the XRF analysis of a single aerosol particle can be carried out by using a laboratory Micro-XRF spectrometer [6–9]. Moreover, in Refs. [6–8], a polycapillary focusing X-ray lens (PFXRL) is used. It is well known that the distribution of the X-ray intensity in the focal spot of the PFXRL is Gaussian distribution, which is not helpful in measuring

accurately the elemental XRF spectrum of a single aerosol particle in which the element distribution is not uniform. The reason for this is that the size of the focal spot of the PFXRL is larger than that of a single aerosol particle. Therefore, the elemental XRF spectrum of a single aerosol particle varies when it locates at different positions in the focal spot of the PFXRL. In order to tackle this problem, a uniform X-ray microbeam as an exciting source is necessary.

In this paper, a uniform X-ray microbeam obtained by using a PSFXRL (polycapillary slightly focusing X-ray lens) combined with a pinhole from a laboratory X-ray source was proposed to be used in the XRF analysis of single aerosol particles. The performances of the Micro-XRF spectrometer based on a PSFXRL were studied. As an example of the application of the Micro-XRF spectrometer based on the PSFXRL in the origin identification of single aerosol particles, the particles at the exit of a subway station and a construction site in Beijing, Chinese capital, were studied.

2. Experiments and results

2.1. Experimental setup

Fig. 1 schematically shows the realization of the Micro-XRF spectrometer based on the PSFXRL combined with a pinhole. The X-ray source is a Mo rotating anode X-ray generator (RIGAKU RU-200, 60 kV–200 mA) whose spot size is $300 \times 300 \mu\text{m}^2$. The pinhole with a diameter of about $50 \mu\text{m}$ was placed close to the focal spot of the

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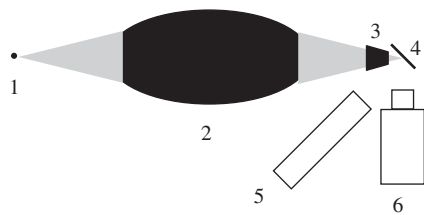


Fig. 1. Scheme of Micro-XRF spectrometer based on PSFXRL: (1) X-ray source, (2) PSFXRL, (3) pinhole, (4) sample, (5) microscope, and (6) detector.

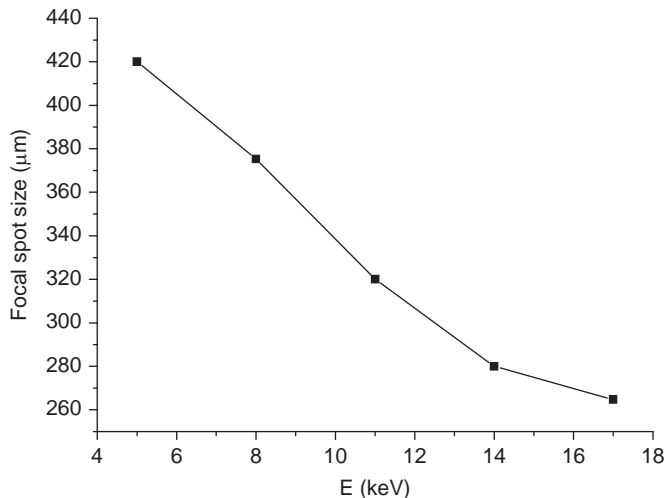


Fig. 2. Energy dependence of the focal spot size for PSFXRL.

PSFXRL in order to confine the X-ray beam hitting the sample. The detector system is an XFlash[®] Detector 2001 RÖNTEC and RÖNTEC MAX Spectrometer. The energy resolution of this detector system is 140 eV at 5.9 keV.

2.2. Performances of PSFXRL for Micro-XRF experiments

The energy dependence of the focal spot size of the PSFXRL is shown in Fig. 2. It is measured with a knife-edge scan where, in order to reduce dead time in the detector system, an organic glass is used as a scatterer. The properties of the PSFXRL are measured by using the backscattered spectra from the organic glass [10]. As shown in Fig. 2, the focal spot size of the PSFXRL decreases with the increasing energies. This is explained by the well-known $1/E$ energy dependence of the critical angle of total reflection of X-ray [10]. Fig. 3 shows the differential curve corresponding to knife-edge scan for measuring the diameter of the focal spot of PSFXRL at 17.0 keV. As shown in Fig. 3, there is a plateau in the distribution curve of the X-ray intensity in the focal spot of this PSFXRL. The energy dependence of the diameter of this plateau is shown in Fig. 4. The uniformity of this plateau is 2.8% and 3.1% at 8.0 and 17.0 keV, respectively. The gain in power density in this plateau is 347.2 and 432.7 at 8.0 and 17.0 keV, respectively. Other parameters of the PSFXRL are reported in Table 1.

2.3. Minimum detection limit of Micro-XRF spectrometer based on PSFXRL

The MDL (minimum detection limit) can be calculated by the following formula [11]:

$$\text{MDL} = 3 \cdot C_i N_B^{1/2} / N_A \quad (1)$$

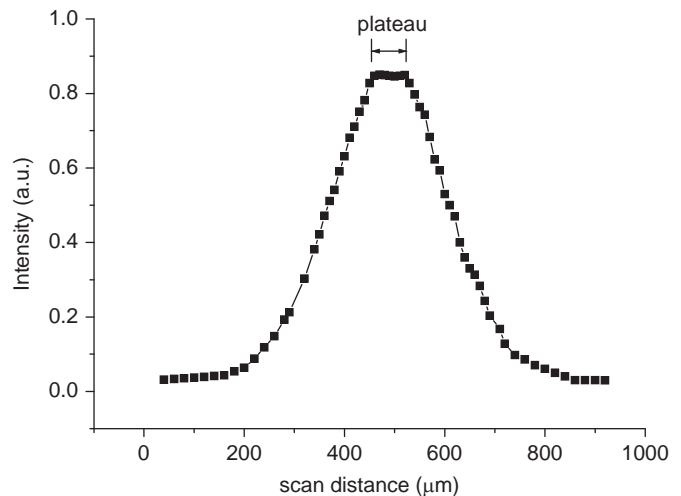


Fig. 3. Differential curve corresponding to knife-edge scan for measuring the diameter of the focal spot of PSFXRL at 17.0 keV.

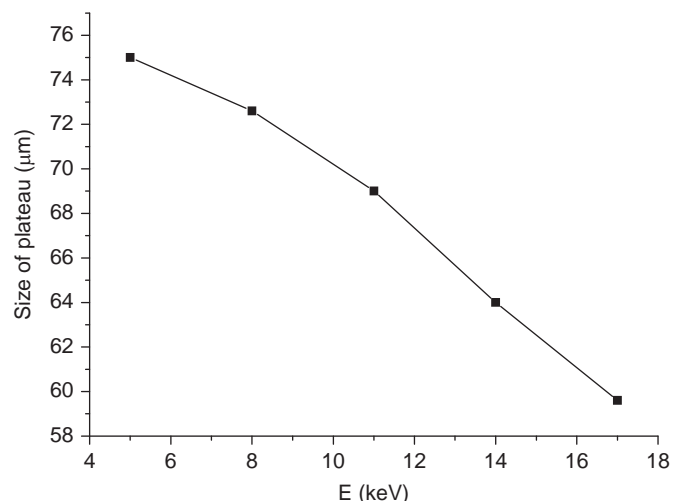


Fig. 4. Energy dependence of the size of the plateau in the focal spot of PSFXRL.

Table 1

The parameters of the PSFXRL.

Length (mm)	102.6
Input diameter (mm)	4.9
Output diameter (mm)	5.8
Input focal distance at 17.0 keV (mm)	60.2
Output focal distance at 17.0 keV (mm)	118.4
Number of the capillaries composing the PSFXRL	278,000

where C_i is the concentration of element i in the standard sample, N_B and N_A are the areas of background and net peak of the analyzed element, respectively. The MDLs of the Micro-XRF spectrometer based on the PSFXRL were 15 and 19 ppm for Fe- K_{α} and Cu- K_{α} , respectively, which were measured using standard samples prepared by pipetting standard solutions on a Mylar film with a thickness of 3 μm. The measurement time was 360 s when the operating voltage and current of the X-ray tube was 22.0 kV and 50.0 mA, respectively. The MDLs of the Micro-XRF spectrometer based on PSFXRL were higher than that of LAMMA, Micro-SXRF and Micro-PIXE, and lower than that of EPXMA.

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