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Application of confocal X-ray fluorescence micro-spectroscopy to the investigation of paint layers

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HIGHLIGHTS

- The performance of the confocal micro X-ray fluorescence was studied.
- Confocal micro X-ray fluorescence was used for identifying paint layers.
- The multilayered paint fragments of a car were analyzed nondestructively.

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ABSTRACT

A confocal micro X-ray fluorescence (MXRF) spectrometer based on polycapillary X-ray optics was used for the identification of paint layers. The performance of the confocal MXRF was studied. Multilayered paint fragments of a car were analyzed nondestructively to demonstrate that this confocal MXRF instrument could be used in the discrimination of the various layers in multilayer paint systems.

1. Introduction

All vehicles are painted, and most automobiles are painted with multiple layers of different paints. Such multilayer paints can be used in the forensic discrimination of evidence for traffic accidents. In some traffic accidents, the drivers at fault run away to avoid punishment. After these drivers escape, they can repair and repaint their vehicles. Because such repainted surfaces may appear as they did before an accident, the ability to analyze such vehicles nondestructively is significantly challenging. In fact, although the surfaces of repainted parts may appear as they did initially, it is difficult for repair technicians to ensure that the elemental composition of every layer of paint in repaired parts is the same as that in the original parts. However, layers with various elemental compositions would produce different X-ray fluorescence (XRF) spectra (Tiwari et al., 2014; Melquiades et al., 2013; Sun et al., 2011). Thus, these XRF spectra corresponding to the different paint layers could be used in the examination of repaired vehicles. Moreover, the XRF spectra of different paint layers can be obtained nondestructively by using a confocal micro XRF (MXRF) spectrometer based on polycapillary X-ray optics.

XRF analysis is nondestructive and therefore widely used in the analysis of criminal evidence. In recent years, confocal MXRF devices based on polycapillary X-ray optics have become increasingly popular (Peng et al., 2014a; Schoonjans et al., 2012). These devices feature a polycapillary focusing X-ray lens (PFXRL) in the excitation channel and a polycapillary parallel X-ray half-lens (PPXRHL) in the detection channel. The PFXRL in the excitation channel is used to focus the divergent or parallel X-ray beam into an output focal spot. When the output focal spot of the PFXRL and the input focal spot of the PPXRHL are adjusted in a confocal configuration, only the X-rays from the confocal volume defined by the overlap of these foci can be detected. By the relative movement between the confocal volume and sample located at the confocal position, the micro-volume to be analyzed can be

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displaced laterally or in a direction perpendicular to the surface of the sample. Therefore, point-to-point, three-dimensional (3D) XRF information about the sample can be obtained nondestructively (Lühl et al., 2013; Peng et al., 2014b). This so-called confocal method based on polycapillary X-ray optics was first proposed in the early 1990s by Gibson and Kumakhov (1992). Today, the method is widely used in many fields, such as 3D micro X-ray absorption fine structure analysis of chemical speciation in stratified systems (Lühl et al., 2012), full-field transmission confocal X-ray imaging for small samples (Sun and MacDonald, 2013), confocal energy-dispersive small-angle X-ray scattering for the identification of milk powders (Sun et al., 2013), confocal X-ray diffraction for the identification of plastics (Liu et al., 2013; Sun et al., 2014), and confocal MXRF analysis of microSD cards (Nakazawa and Tsuji, 2013). This confocal method can also be adapted to synchrotron radiation (Sun et al., 2009; Lühl et al., 2012; Menzel et al., 2013). However, synchrotron radiation requires large facilities and accordingly is not convenient for users (Desouza et al., 2013).

In this work, a confocal MXRF spectrometer based on polycapillary X-ray optics and a conventional laboratory X-ray source was used to nondestructively discriminate between multiple paint layers.

2. Experimental setup

Fig. 1 shows a schematic diagram of the confocal MXRF spectrometer, which features a PFXRL and a PPXRHL. The divergent X-ray beam from the X-ray source, which was positioned at an input focal distance F_1 away from the input of the PFXRL, was focused by the PFXRL into an output focal spot at an output focal distance F_2 away from the output of the PFXRL. The PPXRHL was placed confocally at an input focal distance F_3 away from the output focal spot of the PFXRL. The X-ray source used was a Mo rotating-anode X-ray generator (RIGAKU RU-200, 60 kV–200 mA) whose spot size is $300 \times 300\ \mu m^2$. The detector system is composed of an XFlash Detector 2001 RÖNTEC and a RÖNTEC MAX Spectrometer. The crystal thickness of the detector is 0.3 mm. The peak-to-background-ratio is better than 400:1 for MnKα radiation at 5.89 keV. The maximum count rate of the detector system is 4×10^5 counts/s. The energy resolution of this detector system is 142 eV at 5.9 keV, and the entrance size of the detector is 5 mm^2.

3. Results and discussions

3.1. Characterization of PFXRL and PPXRHL for confocal MXRF

The input focal distance and output focal distance of the PFXRL were 77.2 and 12.1 mm, respectively. The energy dependence of the gain in power density and output focal spot size of the PFXRL is shown in Fig. 2. The output focal spot size of the PFXRL was measured using a combination of a sharp edge and organic glass as a scatterer (Sun and Ding, 2005). The input focal distance of the PPXRHL is 12.3 mm. The energy dependence of the gain in power density and input focal spot size of the PPXRHL is shown in Fig. 3. The gain in power density of the PPXRL was measured using a pinhole with a diameter of 500.0 μm (Sun et al., 2007). The input focal spot size of the PPXRHL was measured using an X-ray source scan (Sun et al., 2007). The focal spot size of the PFXRL and the PPXRHL decreased with the increase in the X-ray energy because the focal spot size is almost directly proportional to the critical angle of total reflection of X-rays, and the critical angle of total reflection is inversely proportional to the X-ray energy. The spatial resolution of the confocal MXRF spectrometer depended on the focus sizes of the PFXRL and PPXRHL and the relative position between the PFXRL and the PPXRHL. This spatial resolution can be characterized by the full width at half-maximum (FWHM) values of the overlapping focal profiles. The profile size of the confocal volume along the Z direction (Fig. 1), d_Z, was the minimum between the output focal spot size of the PFXRL, ϕ_1, and the input focal spot size of the PPXRHL, ϕ_2, i.e., $d_Z = \min\{\phi_1, \phi_2\}$. When the angle between the central axes of the PFXRL and PPXRHL was 90°, the profile size of the confocal volume along the X and Y directions (Fig. 1), d_X and d_Y, respectively, could be approximately determined by ϕ_1 and ϕ_2 as follows (Peng et al., 2013):

$$d_X = d_Y = (\phi_1 + \phi_2)/\sqrt{2} \quad (1)$$

where ϕ_1 and ϕ_2 are known (Figs. 2 and 3) and the confocal volume profile size could be determined. For example, At 17.5 keV, the confocal volume profile sizes along the X, Y and Z directions

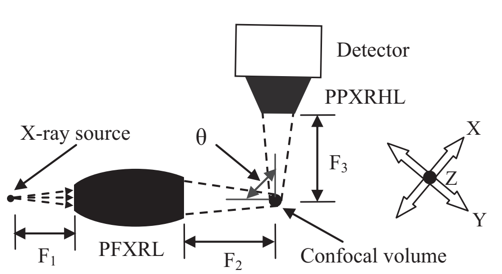

Fig. 1. Schematic diagram of the confocal MXRF spectrometer.

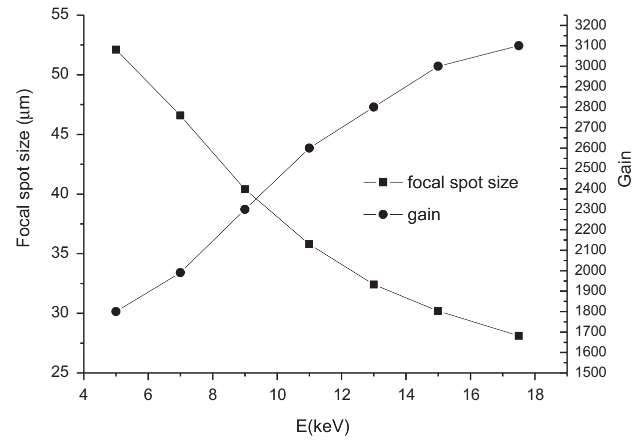

Fig. 2. Energy dependence of the gain in power density and input focal spot size of the PFXRL.

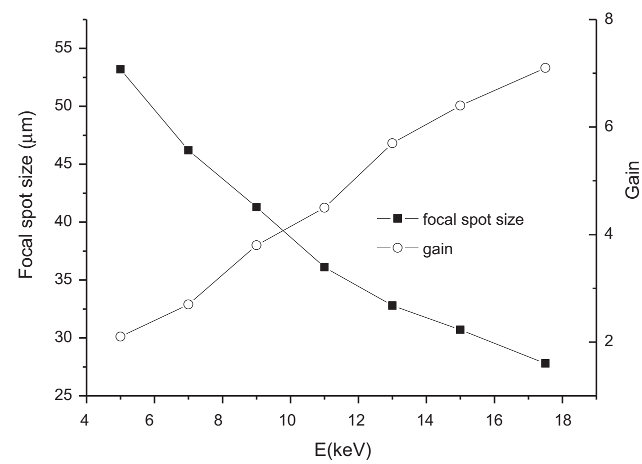

Fig. 3. Energy dependence of the gain in power density and input focal spot size of the PPXRHL.