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Fabrication of X-Ray Optics for a Portable Total Reflection X-Ray Fluorescence Spectrometer Using Electrolytic in Process Dressing Grinding and Magnetorheological Finishing

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Abstract: Problem statement: A portable X-ray elemental analyzer (total reflection X-ray fluorescence spectrometer) has been developed since 2006 and this spectrometer made it possible to perform ultra trace elemental determination. The intensity of scattered X-rays that become background noise in a spectrum is reduced with the improvement in surface accuracy of an X-ray reflector used in the portable spectrometer and therefore using an X-ray reflector with an ultra-precision specular surface can lead to further improvement in detection limits obtained by the portable spectrometer. **Approach:** In the present paper, a combination of electrolytic in process dressing (ELID) grinding and Magnetorheological Finishing (MRF) is applied to fabricating an X-ray reflector for the portable spectrometer. Magnetorheological finishing is used as final finishing after ELID grinding. **Results:** A peak to valley value of 107 nm and a root mean square value of 17 nm in a surface cross section with a length of 27 mm are obtained with the use of ELID grinding and MRF. **Conclusion:** X-ray reflectors having a large specular surface can be fabricated using a combination of ELID grinding and MRF. Using a grinding wheel containing diamond abrasive grains finer than those in the present paper in ELID grinding can lead to further improvement in surface accuracy of an X-ray reflector.

Key words: Electrolytic in process dressing grinding, magnetorheological finishing, total reflection X-ray fluorescence, Portable spectrometer, X-ray reflector

INTRODUCTION

Total reflection X-Ray Fluorescence (TXRF) spectrometry (Yoneda and Horiuchi, 1971) is now an established method for ultra trace elemental determination and various types of samples such as river water, aerosol particles, semiconductor and thin-layer films are analyzed by using this spectrometric method. Figure 1 shows a schematic view of TXRF spectrometry. When X-rays are incident on a specular surface at a glancing angle below a critical angle for total reflection (e.g. 0.1°), the incident X-rays are totally reflected. In TXRF spectrometry, incident X-rays irradiate a sample on a sample holder (X-ray reflector) at a glancing angle below a critical angle and excite fluorescent X-rays from the sample. A high power X-ray source such as synchrotron radiation was usually used for improving detection sensitivity in TXRF analysis and fg detection limits (1 fg = 10^{-15} g) (Wobrauschek *et al*., 1997; Sakurai *et al*., 2002) were obtained by synchrotron radiation. On the other hand, a portable TXRF spectrometer (Kunimura and Kawai, 2007; 2010) with a low power X-ray tube (1-5 W X-ray tube) has been developed since 2006. Figure 2 shows the present portable spectrometer. The use of such low wattage X-ray tube made it possible to downsize TXRF spectrometers. Detection limits down to 10 pg (10^{-11}g) were achieved by the present portable spectrometer and this result shows that ultra trace elemental determination is performed even when a low power X-ray tube is used. Improvement in surface accuracy of an X-ray reflector can lead to further improvement in the detection limits obtained by the present portable spectrometer. Figure 3 shows a relationship between X-ray reflectivity on quartz glass and surface roughness (Root mean square (rms) value). X-ray reflectivity increases with the improvement in surface roughness as shown in Fig. 3. X-ray reflectivity on a reflector with a surface roughness of sub-nanometers can be as high as that on an ideal specular surface (i.e., flat surface with a surface roughness of zero).

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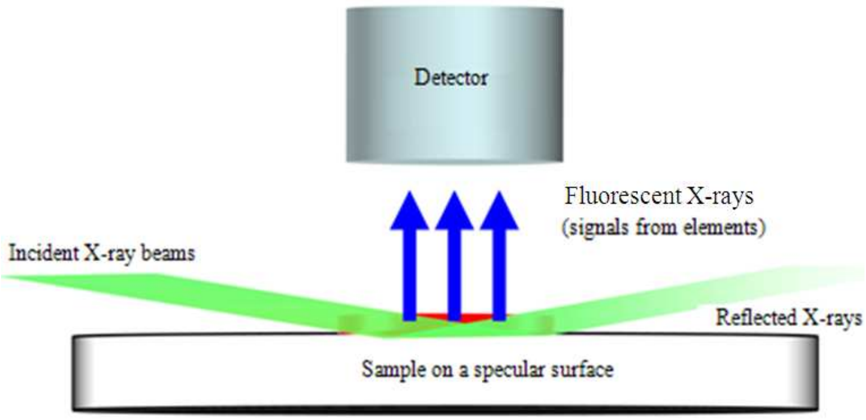


Fig. 1: Schematic view of total reflection X-Ray Fluorescence (TXRF) spectrometry

Fig. 2: Portable TXRF spectrometer

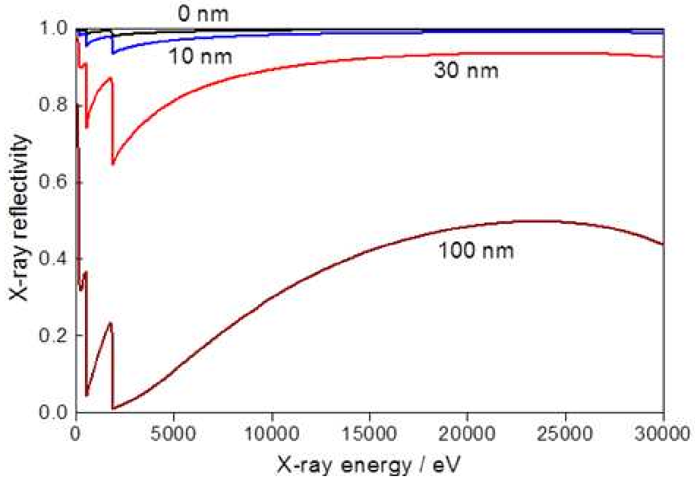


Fig. 3: X-ray reflectivity on a quartz glass as a function of X-ray energy at a glancing angle of 0.05°. The X-ray Reflectivities in Fig. 3 were calculated using the on-line resource at the web page of Center for X-Ray Optics in Lawrence Berkeley National Laboratory (http://henke.lbl.gov/optical_constants/)

Increasing X-ray reflectivity results in a decrease in the intensity of scattered X-rays that become background noise in a TXRF spectrum, leading to an increase in detection sensitivity. Using electrolytic in process dressing (ELID) grinding (Ohmori and Nakagawa, 1990), nano-surfaces are obtained in a short time (a few to several tens of minutes). Ohmori *et al*. (2006) reported that optical materials with an ultra-precision specular surface are fabricated in a short time using a combination of ELID grinding and Magnetorheological Finishing (MRF) (Golini *et al*., 1999). In this combination, MRF was used as final finishing after ELID grinding and a flatness (peak to valley (PV) value) of several nanometers and an rms value of sub-nanometers were achieved in a measured area of 144 μm×108 μm. In TXRF analysis, an area where incident X-rays illuminate is usually a few centimeters square and therefore a flat reflector having a large ultra-smooth surface is needed for reducing scattered X-ray intensity. There is a possibility that surface roughness of sub-nanometers in a few centimeters square is achieved using this combination. In the present paper, this combination is applied to fabricating a quartz X-ray reflector for the portable TXRF spectrometer.

MATERIALS AND METHODS

An ELID grinding machine "HSG-10A2" (Nachi-Fujikoshi, Co., Toyama, Japan) was used. Figure 4 shows a schematic view of ELID grinding. As shown in Fig. 4, a metal-resin bonded abrasive wheel played a role in a positive electrode and a negative electrode was placed in front of the abrasive wheel. A D.C pulse voltage was applied between the positive and negative electrodes. The abrasive wheel was efficiently dressed by electrolyzation, and this dressing was continuously performed during an ELID grinding in order to avoid decreased wheel sharpness from wear. In the present paper, two kinds of metal-resin bonded diamond abrasive wheels (#4000, #8000) were used. After ELID grindings with each abrasive wheel, surface roughnesses (Ra value) were measured with a contact-type surface tester "Surftest-701" (Mitsutoyo Kiko Co., Ltd., Kasugai, Japan).

An MRF machine "Q22-Y" (QED technologies, Inc., Rochester, NY) was used. Figure 5 shows an inside of the MRF machine and a schematic view of MRF.

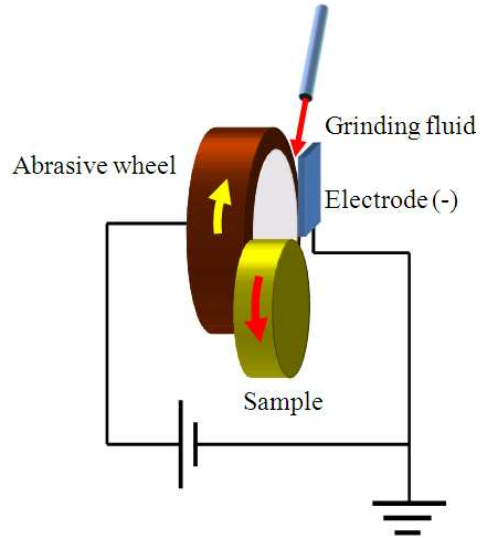
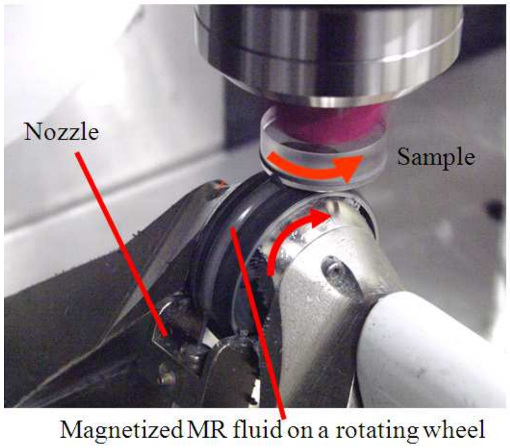
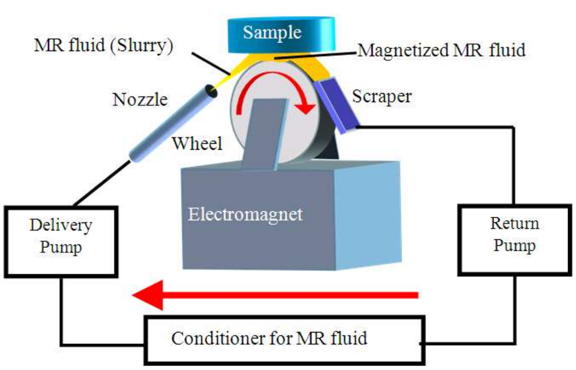


Fig. 4: Schematic view of electrolytic in process dressing (ELID) grinding



(a)



(b)

Fig. 5: (a) Inside of a magnetorheological finishing (MRF) machine, (b) schematic view of MRF

One liter of a magnetorheological (MR) fluid containing diamond or CeO_2 abrasive grains was injected into the fluid circulation system in the MRF machine. As shown in Figure 5, this fluid was ejected onto a rotating wheel, and magnetic fields were applied to the fluid. The application of the magnetic fields led to stiffening of the MR fluid. This magnetized fluid was used for polishing a sample. This stiffened MR fluid returned to a slurry after it passed through the magnetic fields, and then it was collected via suction. Before and after MRF, surface shapes were measured with a Zygo GPI laser interferometer (Zygo, Co., Middlefield, CT).

RESULTS

Figure 6 shows improvement in figure accuracy and surface roughness of a quartz glass (diameter: 30 mm) by using MRF. An MR fluid containing CeO_2 abrasive grains was used and a total time for polishing was 17 min. As shown in Fig. 6, using MRF, an rms value in the whole surface area was improved from 17-8 nm and a PV value was improved from 152-103 nm. Figure 7 shows Ra values of a quartz glass with a diameter of 30 mm after ELID grindings. ELID grindings with each abrasive wheel were performed for 30 min. As shown in Fig. 7, surface roughness was improved with the decrease in a size of abrasive grains. A surface roughness of several nm was achieved when using the #8000 metal-resin bonded abrasive wheel. After ELID grinding with the #8000 abrasive wheel, MRF was performed. An MR fluid containing diamond abrasive grains was used and a total time for polishing was four hrs. Figure 8 shows shapes of surface cross sections with a length of 27 mm before and after MRF. Brittle fractures were caused by ELID grinding because quartz glass is a brittle material. As shown in Fig. 8a, grooves with a depth of a few hundred nanometers were due to the brittle fracture. As shown in Fig. 8, MRF was useful to remove grooves and to improve surface accuracy. Using MRF, a PV value was improved from 279-107 nm and an rms value was improved from 69-17 nm.

DISCUSSION

Using ELID grinding, an X-ray reflector with a surface roughness of several nanometers was produced in a short time. Magnetorheological finishing (MRF) was able to be used as final finishing for further improvement in flatness and surface roughness of an X-ray reflector fabricated by ELID grinding. Using a combination of ELID grinding and MRF, an X-ray reflector with a flat and smooth surface for the portable TXRF spectrometer can be efficiently produced.

(a) (b)

Fig. 6: Surface shapes of a quartz glass (a) before and (b) after MRF

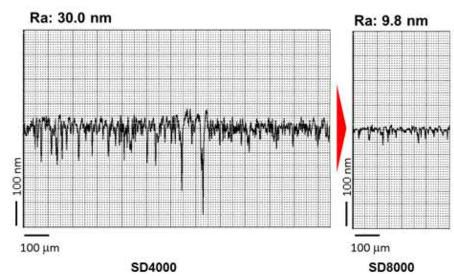
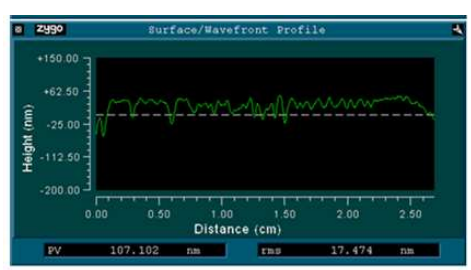


Fig. 7: Surface roughnesses (Ra) of a quartz glass after ELID grindings

(a)



(b)

Fig. 8: Shapes of surface cross sections (a) before and (b) after MRF

CONCLUSION

The first result of the application of a combination of ELID grinding and MRF to fabrication of a quartz X-ray reflector for a portable TXRF spectrometer was reported. In the present paper, MRF was used for further improvement in flatness and surface roughness of a quartz reflector fabricated by ELID grinding and a PV value of 107 nm and an rms value of 17 nm were achieved in surface cross section with a length of about 3 cm. This result indicates that using this combination makes it possible to produce a large specular surface. For further improvement in flatness and surface roughness of an X-ray reflector, the use of a grinding wheel containing diamond abrasive grains finer than those in the present paper is needed in ELID grinding as preceding process.

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