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Improvement Study on Heat Resistance of Multilayer-Coated Replica Gratings

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Abstract. We have been developing aperiodic W/B_4C multilayer-coated replica gratings for use in the tender X-ray range 2 to 4 keV. It has become apparent that surface degradation was caused by heating multilayer gratings at around 60°C; the multilayer structure has been well maintained, whereas the grating grooves formed in epoxy resin of the replica grating were significantly deformed. Such degradation has not been observed in conventional gold-coated replica gratings as far as we know. In this study, replica gratings made with a high glass transition temperature resin were prototyped to improve the heat resistance of multilayer-coated replica gratings. Diffraction efficiencies of multilayer gratings with and without heat treatment at approximately 95°C for 60 hours were measured and compared. The fact that the change rate between the peak values is slight, being less than 3%, indicates that the heat resistance has been improved.

INTRODUCTION

X-ray emission spectroscopy (XES) is a powerful tool for studying the electronic structure of materials [1]. Recently, with the advent of high-brilliance synchrotron radiation sources, operando XES studies in the photon energy range between roughly 1 and 5 keV, the so-called tender X-ray range, have increased attention for nondestructively providing bulk-sensitive and element-selective information [2]. High resolution wavelength-dispersive X-ray spectrometry (WDS) is frequently used in XES measurements. For the sake of sophisticated analysis of a variety of materials, the acceptance energy in WDS instruments has been expanded by employing simultaneous multiple crystals [3]. On the contrary, diffraction gratings have been often used on account of the lack of crystals with appropriate lattice constants satisfying the Bragg condition in the energy range below 2 keV. A flat-field spectrometer equipped with a spherical varied-line-spacing (VLS) grating has a large acceptance of a few hundreds of eV and more at a fixed angle of incidence [4]. The diffraction efficiency in the range above 2 keV is considerably decreased, however, it can be enhanced by multilayer coating. Since multilayer gratings have high wavelength-selectivity, they are suitable for wavelength-scanning monochromators rather than flat-field spectrometers [5,6].

To develop a flat-field spectrometer for tender X-ray emission spectroscopy, we conceived an aperiodic W/B_4C multilayer-coated grating having uniform diffraction efficiency over the range 2 to 4 keV [7,8]. This layer structure is analogous to supermirrors for hard X-rays, but significantly simplified [9]. Not only a replica grating but also the master grating were coated with the aperiodic multilayers, and their performance was comparable each other. In fact, this is desirable for end users of flat-field spectrometers as well as the diffraction grating manufacturer, because replica gratings with high quality and uniformity can be provided inexpensively.

A grating spectrometer chamber is often baked out prior to experiments to get rid of residual gas. In spite of low-temperature baking even at 60°C, we experienced a critical problem that the surface of the multilayer grating was seriously damaged. We consider that this phenomenon has resulted from the heat stress between the multilayer and the base grating, because it has not been seen in other conventional gold-coated replica gratings as far as we know.

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The result of the cross-sectional observation of the layer structure by transmission electron microscopy (TEM) shows that the multilayer structure has been well maintained, but the grooves formed in resin have been significantly deflected. It indicates that we need to overcome the problem between the multilayer coating and the epoxy resin by improvement of the heat resistance of multilayer-coated replica gratings. Therefore, we have performed a feasibility study of a new replication process using a high glass transition temperature (T_g) epoxy resin compared to the conventional one that we have used so far.

In this paper we report the results of atomic force microscopy (AFM), laboratory X-ray reflectivity (XRR) and X-ray diffraction efficiency of the periodic W/B_4C multilayer-coated replica gratings before and after heat treatment, and then the heat resistance improvement of the replica gratings fabricated based on the new replication process is shown.

HEAT TREATMENT OF MULTILAYER-COATED REPLICA GRATINGS

Laminar-type replica gratings were fabricated from an identical negative master grating with a new replication process using a high T_g epoxy resin. The fabrication technique will be detailed elsewhere. The nominal parameters of the master grating are as follows: radius curvature of 11,200 mm, grating constant of 1/2400 mm, groove depth of 2.8 nm, and duty ratio of 0.5 [7]. A periodic W/B_4C multilayer was deposited on the 50 nm thick gold-coated replica gratings by ion beam sputtering: the periodic length is 5.6 nm; the ratio of the W thickness to the period is 0.5; the number of layers is 41; the topmost layer is W. For the sake of simplicity to analyze the results of XRR and diffraction efficiency measurements, the periodic multilayer coating was chosen. One of the two multilayer gratings, named A, was kept as-deposited. The other grating, named B, was heat treated with a vacuum furnace. Figure 1 shows plots of the surface temperature of grating B and the pressure in the vacuum furnace against heating time. The temperature was monitored by a thermocouple probe during heating test. The surface temperature reached a target temperature of 90°C after taking approximately four hours, was kept constant for 60 hours, and then was cooled down to room temperature by natural cooling. In consequence, the average temperature for 60 hours was 94.7°C slightly higher than the target temperature, and the pressure was below 10 Pa. As a result of visual inspection, the surface of grating B after heat treatment was still specular.

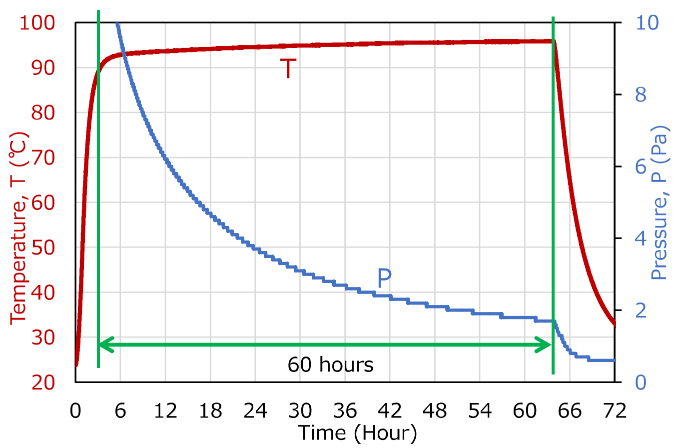

FIGURE 1. Plots of the surface temperature of the multilayer grating, B, and the pressure in the vacuum furnace as a function of heating time.

Figure 2(a) shows AFM images of the A (as-deposited) and B (heat-treated) gratings. It is found that the clear grooves of grating B are observed, and the surface roughness of 0.4 nm root-mean-square (rms) is comparable to that of grating A. Figure 2(b) shows a comparison of the peak reflectivities of grating B before and after heat treatment plotted as a function of the Bragg diffraction order. The Cu-*K*α radiation was incident parallel to the grating grooves in the XRR measurements. Taking into account the results of AFM and XRR measurements, it is considered that the interface roughness of the multilayer has been increased owing to heat treatment. Nevertheless, the multilayer period has almost remained unchanged before and after heating, being from 5.660 nm to 5.652 nm. Therefore, the above suggests that the layer structure is well maintained, regardless of the diffusion at the interfaces of the multilayer.

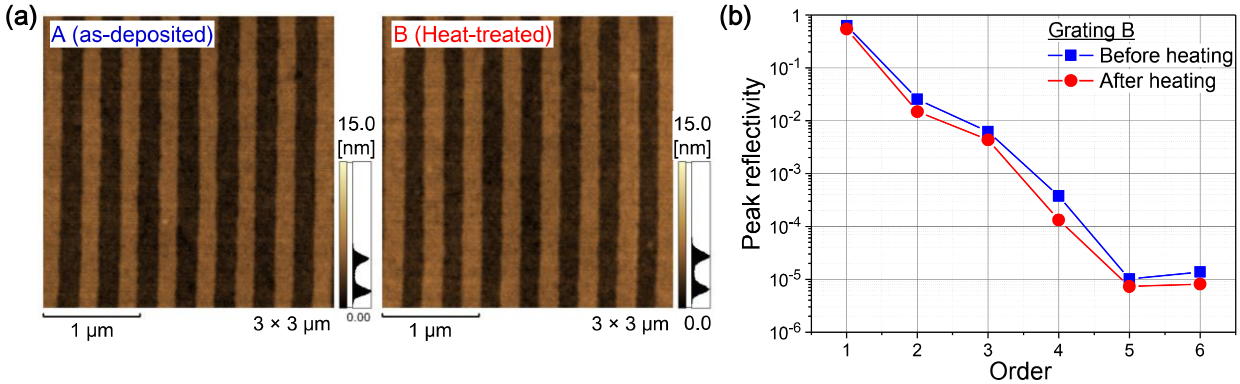

FIGURE 2. (a) AFM images of the A (as-deposited) and B (heat-treated) gratings. (b) Peak reflectivities of grating B before and after heat treatment plotted as a function of the Bragg diffraction order.

DIFFRACTION EFFICIENCY MEASUREMENTS

Diffraction efficiency measurements of the multilayer gratings were performed using our in-house reflectometer at the BL-11B beamline of the Photon Factory, KEK, Japan [10]. A double-crystal monochromator with Si(111) crystals was chosen to use the X-ray energy range 2.8 to 4.2 keV. The beam size was estimated to be approximately 0.6 mm (V) × 4 mm (H) at the sample stage in the reflectometer. An X-ray photodiode detector (AXUV100 Si/Zr, IRD Inc.) was used, and the detector acceptance area of 10 mm-square was restricted to 2 mm (V) × 8 mm (H) to improve the angular resolution. The multilayer grating was set at a grazing angle of incidence of 1.35° that is complementary angle of 88.65°, and the diffracted light from the sample grating was measured as a function of detection angle ranging from 2.4 to 5.0°. Figure 3 shows comparisons of the zeroth and first order diffraction efficiencies obtained from the A (as-deposited) and B (heat-treated) gratings. Note that two gratings are not identical. The periodic W/B_4C multilayer coating on grating A has enhanced the first order diffraction efficiency, but the spectral width, being approximately 0.4 keV, is less wide to be used for flat-field spectrometers without mechanical scanning. This means that it is necessary to expand the spectral width with the aperiodic multilayer technique, as described above [7]. The diffraction efficiencies for the zeroth and first orders of grating B are almost comparable to those of grating A. Focusing on the peak positions at 3.5 keV of the 1st order diffraction efficiencies, there seems to be a slight difference in the two gratings. Also, the change rage of the peak values is small, it is less than 3%. Actually, it is difficult to deny the possibility that the efficiency change originates from an individual difference in the gratings. However, taking into account that grating grooves formed in the conventional resin were deformed by heating even at 60°C, resulting in significant efficiency degradation, the above result indicates that the heat resistance of the multilayer-coated replica grating has been successfully improved up to 90°C.

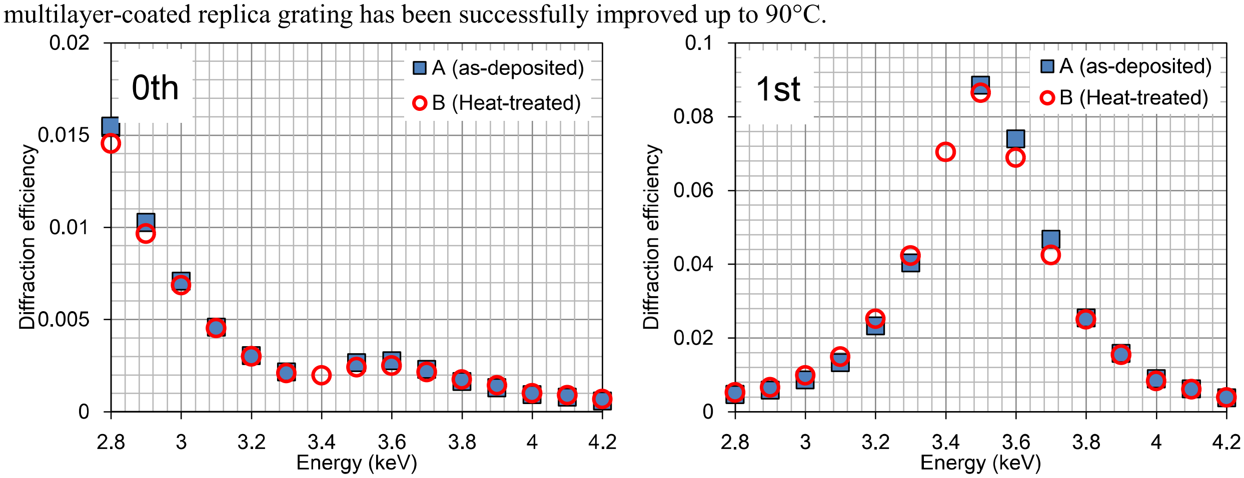

FIGURE 3. Comparisons of the zeroth and first order diffraction efficiencies obtained from the A (as-deposited) and B (heat-treated) gratings. There is a lack of data at 3.4 keV regarding the A grating, however, the unmeasured data would be almost in agreement with those of the B grating.

CONCLUSION

Replica gratings were fabricated based on a new replication process using a high T_g epoxy resin and were coated with periodic W/B_4C multilayers. One of the multilayer gratings was heat-treated at 94.7°C for 60 hours in vacuum. As a result, it has been confirmed that the multilayer structure and the grooves of the heated multilayer grating have been well maintained, and the diffraction efficiencies are in agreement with those of another as-deposited multilayer grating. Considering that the former multilayer-coated replica gratings fabricated by our conventional replication process were crucially damaged at 60°C, it is shown that the new process is a promising technique for developing multilayer-coated replica gratings with high heat-resistance (up to 90°C).

The detailed description associated with the new replication technique has been omitted in this paper. For more discussion concerning the heat resistance improvement, it would be necessary to exclude the individual difference between samples and clarify the correlations among the surface roughness, diffraction efficiency and heating temperature in addition to the repetition performance and the imaging performance.

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