Investigation of performance of x-ray capillaries internally covered with a lacquer-metal reflectivity layer

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The metallic parabolic capillary with an internal lacquer/gold layer for hard X-ray optics was manufactured by (i) deposition of a layer of acrylic lacquer (2μm) on a copper mandrel, (ii) deposition of a layer of gold with a thickness of 100 nm by vacuum evaporation, (iii) electrodeposition of a nickel layer (100 μm), and (iv) removing the copper mandrel. The surface roughness of the copper mandrel was reduced approximately 10 fold to approximately 1.5 nm and did not increase after the deposition of the gold layer (ii). In this manner, the capillary obtained in step (iv) has an inner lacquer/gold layer with interface roughness of about 1.5 nm. The geometric parameters of the surface were measured using an atomic force microscope (roughness), optical profilometer (waviness), and a laser scan micrometer (figure error). Because of the difficulty in removing lacquer layer from the inside of the capillary after step (iii), the curvature of the parabola was optimized in a way allowing complete transmission of the X-ray beam with energy of 21 keV through the lacquer layer. In order to design a proper parabolic curve of X-ray, reflectometric curves were calculated for the lacquer/gold bilayer with thickness and roughness obtained for the manufactured capillaries. The capillaries were tested on the L line (DORIS, Hamburg) using energy 21 keV. For one of the capillaries, the flux gain of 1.6 was recorded. The poor efficiency of the capillaries is mainly caused by waviness that leads to an increase in the incident angles. In consequence, reduction of beam intensity and beam diffusion occurs.

Keywords: x-ray metallic capillary optics, roughness, waviness

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1. Introduction

X-ray capillary optics require a very smooth surface for efficient x-ray reflection. Glass capillaries produced in a traditional way by drawing from a piece of tube are characterized by a smooth usually ca. 1-nm surface, depending on the surface size [1,2]. Manufacturing of metallic X-ray optics requires using different techniques, such as electroforming or replication [7]. In this method, the metallic mandrel is replicated into a final X-ray focusing element. In comparison with the very sophisticated, time-consuming, and expensive method of polishing metallic surface, the lacquering technique seems to be cheap and relatively easy to apply. In this method, a thin acrylic layer is uniformly applied on the surface and then dried. The lacquer-coating technique was used for diamond-turned polishing of surfaces [3,4]. Kopecky et al. [5] showed that acrylic lacquer is an effective way of polishing figured optics to obtain X-ray reflectivity. However, they noticed that prior to electroforming application complete removal of remaining lacquer without damaging the mandrel surface may be required due to the very high cost of manufacturing of the mandrel. In our technology, the mandrel is formed with the electroplating method, which is very cheap and non- time-consuming [6]. To address the problem with complete solving the remaining lacquer inside capillaries, we produce capillaries with a lacquer-metal internal reflectivity bilayer. We concentrated our effort on deposition a lacquer layer with a uniformly distributed thickness below 2 μm.

2. Experimental

Acrylic lacquer was deposited on the electrolytic copper surface using the dip-coating method. The speed of withdrawal of the mandrel from the lacquer solution was 20 mm/min. After deposition, lacquer was dried at 60 ^{o}C for 30 min. Copper had been previously electrodeposited on a stainless steel wire (304 grade, supplied by Knight Wire, England) with a diameter of 0.2 mm.

Then, a gold layer with a thickness of about 100 nm was deposited by vacuum evaporation. Roughness on the interface lacquer/gold was maintained during this process. Finally, a nickel layer without internal stress (~ 100 μm) was electrodeposited. The mandrel (a stainless steel wire together with the copper layer) was mechanically removed due to very low adhesion between copper and lacquer. In consequence, the capillary had a lacquer (2 μm)/Au(100 nm)/Ni(100 μm) structure.

Roughness of the deposited copper and lacquer layers was examined using an AFM (5600LS AFM, Agilent Technologies) in non-contact mode (tip radius < 7 nm, resonance frequency 280 kHz) with a resolution of 512×512 and scan area of 20 μm×20 μm. The measurements were carried out at 30 places, located

2 mm apart along the mandrel wire. The separation corresponded to the 1.5 – 1.6 µm increase in copper layer thickness due to the parabolic curvature of the mandrel. The surface roughness, S_q, – the root mean square of arithmetic average of the absolute values of the surface roughness – was calculated using the Scanning Probe Image Processor (SPIP) v. 5.1.4 software (Image Metrology A/S, Denmark) according to the formula

$$S_q = \sqrt{\frac{1}{MN}\sum_{k=0}^{M-1}\sum_{l=0}^{N-1}\left[\, z(x_k, y_l)\right]^2}$$

where x and y are the coordinates, z is the perpendicular deviation from the ideally smooth surface, M is the number of points in the x direction, and N is the number of points in the y direction.

A laser scan micrometer (LSM 500H, Mitutoyo) and an optical profilometer (WYKO NT9800, Veeco) were used to determine surface waviness, the shape of the mandrel, and the thickness of acrylic lacquer. Figure 1 shows a representative mandrel roughness after lacquering. Figure 2 shows a roughness of the mandrel before (red line) and after (blue line) deposition of acrylic lacquer. The roughness of the mandrel was reduced about 10 times after deposition of the lacquer layer.

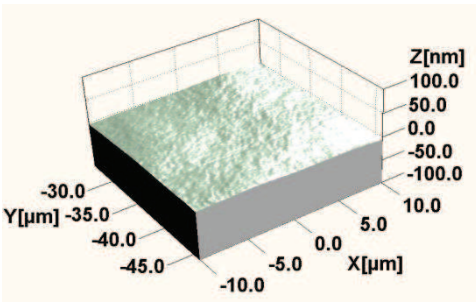


Figure 1. AFM micrograph of mandrel surface after lacquering. Sq = 1.9 nm.

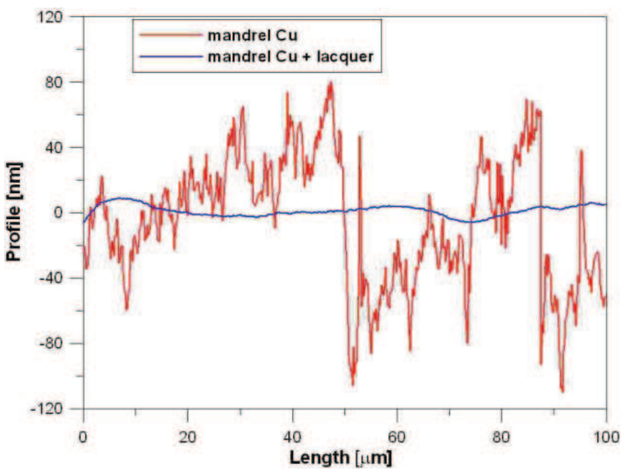


Figure 2. Profile of the surface before (red line) and after (blue) lacquering.

It is clearly seen that the levelling abilities of lacquering reduce surface roughness significantly (about ten times). Another relevant factor is uniformity of lacquer thickness distribution over the whole surface of the mandrel. This is shown in Fig. 3. It can be seen that the mean thickness is about 1.5 µm. The thickness of the lacquer layer was calculated by subtraction of the mandrel profile after and before lacquering. Data from the laser scan micrometer and optical profilometer were used for this operation. Waviness and thickness of the lacquer layer was measured after deposition of a 100-nm gold layer by vacuum deposition owing to the transparency of the lacquer layer.

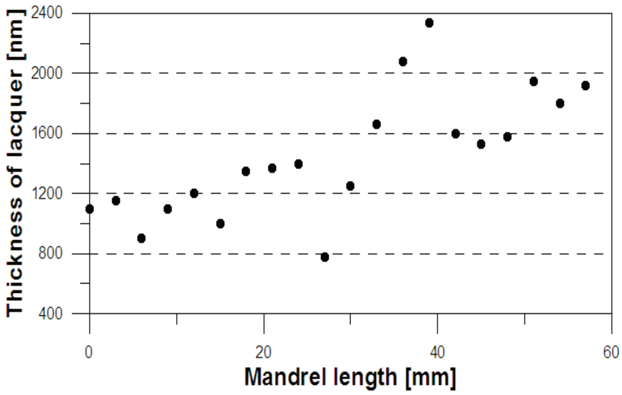
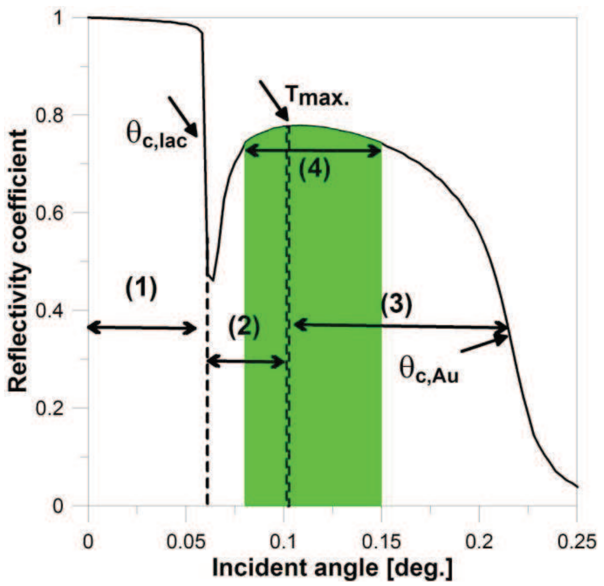


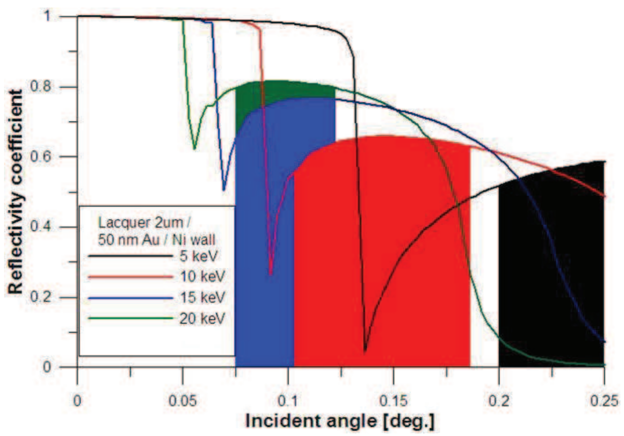
Figure 3. Distribution of acrylic lacquer thickness over the mandrel

3. Calculations of X-ray reflectivity of capillaries

X-ray reflectivity calculations (http://www.cxro.lbl.gov) were performed to determine transmission of X-rays through the lacquer/Au interface present on the internal wall of the metallic capillaries. Figure 4a shows X-ray reflectivity of 21 keV on the acrylic lacquer/gold bilayer. We can distinguish four characteristic regions for the X-ray reflectivity curve. The first region (1) represents X-ray reflectivity from acrylic lacquer with 2-µm thickness. X-rays are totally reflected below the critical angle ($\theta_{c,lac.}$) characteristic for acrylic lacquer. At $\theta_{c,lac,}$ the intensity of x-rays falls to 1/e of its value at the surface of lacquer. Above the critical angle $\theta_{c,lac}$ (region (2)), X-rays are not reflected from the lacquer surface but the transmission coefficient gradually increases up to point T_{max} and simultaneously X-rays are reflected from the gold layer. Starting from point $T_{max,}$ the shape of the X-ray reflectivity curve becomes very similar to the curve characteristic for X-ray reflection from a single gold layer. If we assume that our requirement is reflectivity e.g. above 70% for the entire internal surface of the capillary, the incident angle has to range from ~ 0.075 to 0.15 ^{0} (region (4) green fill), which corresponds to the specific curvature of the parabolic shape. Fig. 4b shows the reflectivity coefficient as a function of the incident angle for X-ray energy of 5, 10, 15 and 20 keV. The solid backgrounds correspond to optimal X-ray reflectivity regions for different X-ray energy. Different incident angles correspond to different curvature of capillaries, e.g. the angles ranging from 0.05 to 0.07 correspond to maximum output beam divergence about 4 mrad. This region of X-ray reflectivity is not useful, as it covers the absorption edge (~ 0.06 ^{0}) for 20 keV. For 20 keV, it is recommended that the incident angles should be located between 0.08 and 0.12, which corresponds to a capillary with maximum output beam divergence about 8 mrad. Taking this fact into account, the curvature of parabolic capillaries was adjusted in order to receive an incident angle in capillaries in the range between 0.09 and 0.12 degrees.



(a)



(b)

Figure 4.

(a) X-ray reflectivity from the acrylic (2 μm) /Au (50 nm) bilayer deposited on Ni substrate for 20 keV X-ray energy as a function of the incident angle. Roughness (rms) of the acrylic/gold interphase - 1.5 nm.

(b) X-ray reflectivity from the acrylic (2 μm) /Au (50 nm) bilayer deposited on Ni substrate for different X-ray energy as a function of the incident angle. Roughness (rms) of the acrylic/gold interphase - 1.5 nm. function of the incident angle. Roughness (rms) of the acrylic/gold interface - 1.5 nm.

According to X-ray reflectivity calculations for these values of angles (see Fig. 4), X-ray beams with energy of 20 keV can be transmitted through 2 μm-thick acrylic lacquer with transmission at ca. 80% from the top gold layer (rms roughness of gold equal 1-1.5 nm). In our experiment, the acrylic lacquer had 1.1 g/cm3 density and 1 – 2.7 μm thickness

4. Focusing properties of metallic capillaries – results and discussion

The main aim of the experiment on the capillary focusing abilities was to investigate the performance of a single-bounce lacquer-metallic capillary at beamline L (DESY, DORIS III) as a focusing element for hard X-ray synchrotron radiation providing an alternative to single glass capillaries. Four capillaries were prepared for measurements. The shape of capillaries was parabolic and the maximum output beam divergence was about 8 mrad. Other parameters of capillaries are listed in Table 1. All the capillaries were tested using a monochromatic synchrotron beam with photon energy 21 keV. Since no ring structure was observed in the far-field image, scans were performed using a 4-μm thick tungsten wire in the location of a potential focusing point to determine the beam parameters.

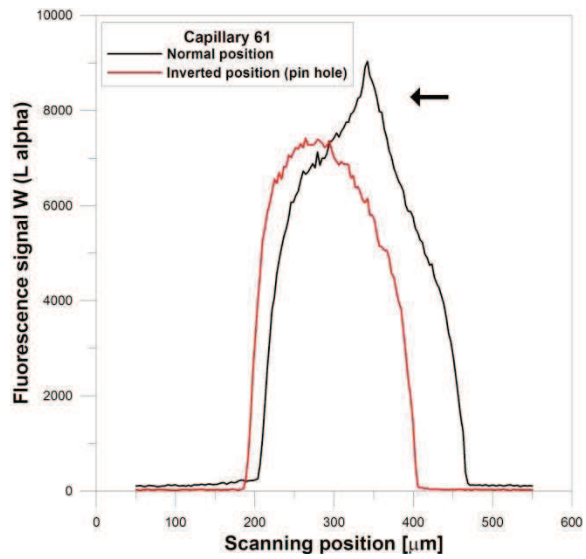


Figure 5. Profile of the beam in the focus point for capillary 61. Black line – beam from capillary No 61 in the normal position, red line – in the inverted position. Fluorescence signals (L alpha) from W wire were recorded.

Two measurements were made for each of the capillaries. One measurement was performed when the synchrotron beam entered the capillaries from a bigger aperture (normal position) and the other - in an inverted mode, when the entrance had a smaller aperture. After normalization of the beam profile in the potential focus point, both cases were compared. For three capillaries, the profiles recorded in the normal and inverted positions were nearly the same, which implies that the capillaries work as a usual collimator. A peak formed by the focusing beam was observed for capillary 61 (Fig. 5, black arrow). In this case, the maximum intensity is a little higher in the normal than in the inverted position of the capillary. Based on this result, the gain for the capillary was estimated as equal to 1.6. Such poor performance of the capillary is mainly determined by the waviness. In comparison to glass capillaries, the waviness is about one order of magnitude higher. The two basic sources of waviness include (i) waviness of the substrate (stainless steel wire) that was used for electroplating and (ii) waviness of the electrodeposited copper layer (for the mandrel). The best result obtained up to now is close to 1.2 mrad for the tested capillaries but recently we have reduced these values to 0.5 mrad (the capillaries have not been tested yet). Importantly, deposition of acrylic lacquer in the best case does not improve the waviness but only reduces roughness by ca. ten times. Therefore, there is a need for further studies focused on reduction of waviness by at least one order of magnitude to obtain better performance of metallic capillaries than that of the glass ones.

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Table 1. Parameters of gold/lacquer metallic capillaries.

No	Material of reflecting surface	Length [mm]	Aperture of entrance diameter [μm]	Aperture of exit diameter [μm]	Waviness [mrad]	Mean roughness rms [nm]	Lacquer thickness [μm]
60	gold	40	350	220	1.75	2.1	2.7
61	gold	69	330	220	1.8	1.9	2.0
63	gold	65	332	220	1.35	2.0	2.7
66	gold	70	335	220	1.28	-	1.0