X-ray capillaries with a lacquer-metal internal bilayer for focusing hard X-rays

Robert Mroczka[1], Agnieszka Chrzanowska[1], Emilia Fornal[1], Grzegorz Żukociński [2], Andrzej Kuczumow[1]

[1]Department of Chemistry, The John Paul II Catholic University of Lublin, Al. Kraśnicka 102, 20-718 Lublin, Poland, rmroczka@kul.pl

[2]Department of Chemistry, Maria Curie-Sklodowska University, Lublin, Poland

The quality of a copper mandrel used for manufacturing of x-ray metallic capillaries was improved by the deposition of an acrylic lacquer. Since complete removal of acrylic lacquer from capillaries appeared to be impossible, we redesigned the sahape of capillries in order to allow the X-ray penetration through a lacquer layer and thus to receive effective X-ray reflection from metallic layer.

EXPERIMENTAL PROCEDURES - ELECTROFORMING

In the first step **a stainless steel wire** was passivated **(Fig.1a)**.The parabolic shape of copper layer was formed by electroplating, **a mandrel (Fig.1b)** was withdrawed from solution bath at different speed. The **mandrel** was **covered with acrylic lacquer** and **then gold was deposited by vacuum deposition (Fig.1c).** In the next step a capillary wall was formed from nickel sulphmate bath and then the mandrel was strained and placed between two pieces of the laminate. Finally, the core steel wire was removed and the copper layer was etched with an acid . Lacquer due to its very high resistivity to chemicals remains inside of capillaries.

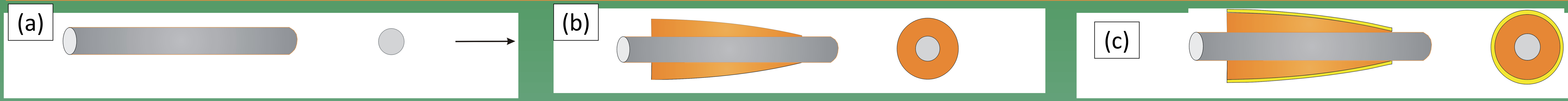

Fig. 1 Scheme of metallic capillary manufacturing (the first three steps).

MEASUREMENTS

Surface roughness of the mandrel covered by acrylic lacquer was tested using an atomic force microscope (AFM). Measurements were corried out in 30 places with the steps of 2 mm on the area 20x20 µm (Fig. 2 and Fig. 3).

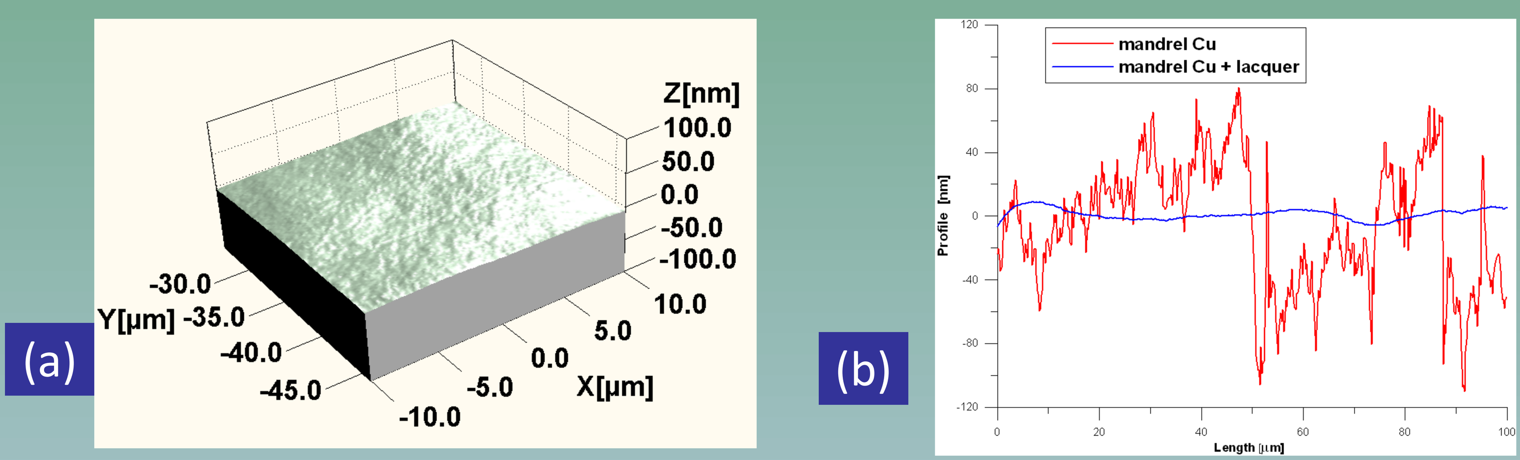

Fig. 2 Effectivity of levelling by an acrylic lacquer a) copper after lacquering, Sq = 1.91 nm, AFM b) profile comparison before and after lacquering

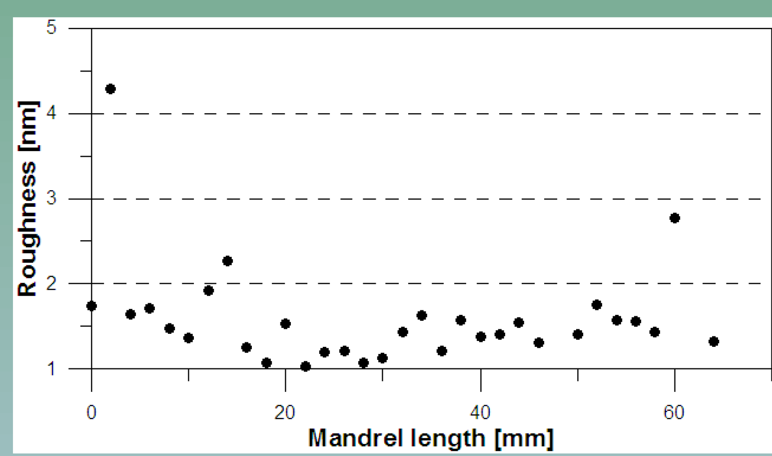

Fig. 3 Surface roughnhess evaluation along a mandrel with acrylic lacquer Mean surface roughness ca. 1.5 nm is achived,

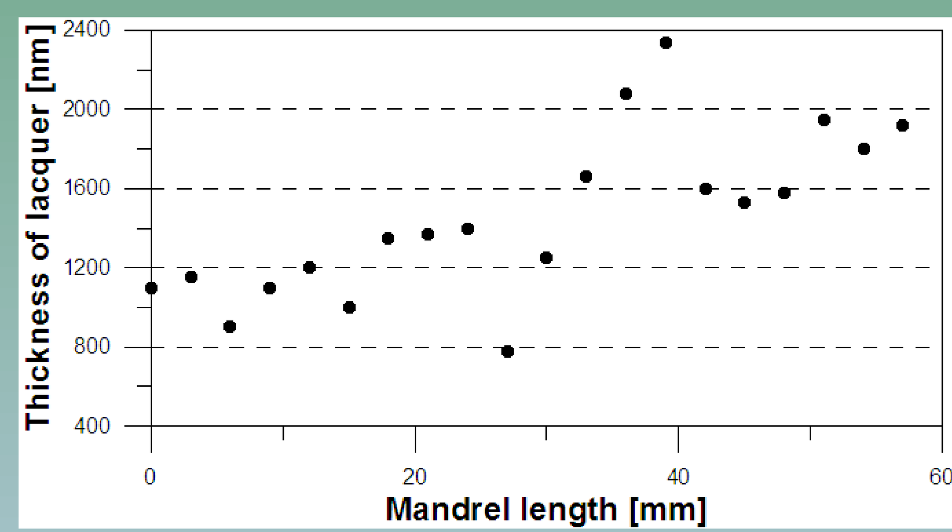

Fig. 4. Thickness of a lacquer layer along mandrel measured by optical profilometr.

Thickness of a lacquer layer was determined using an optical profilometer (OP) and a laser scan micrometer (LSM). To ensure a good quality of data measurments were carried out after the deposition of gold on lacquer. Lacquer layer thickness was calculated by subtracting fitted profiles, a fitted profile of the mandrel without lacquer was substracted from a fitted profile of the mandrel covered with lacquer.. Then the results were divided by the group refraction index of the material to obtain real thickness vaues. In the case of an optical profilometer thickness was determined by dividing the optical distance by the group refraction index of the film material. When the thickness of a layer was below 0.8-1 µm masurment was not possible. For LSM measurments the thickness limitation is about 0.1 µm if proper metallic layer is deposited. Results obtained by OP i LSM are comparable, however, LSM method is faster, more accurate and can determine profile of mandrel with bilayer lacquer/Au. Analysing Fig. 3 we can notice that roughness along the mandrel is highly uniform and it is not affected by non uniform lacquer thickness distribution (Fig.4, thickness ranges from 0.5 to 2 um). It means the lacguer thickness can be reduced to ~ 1 um and any redunction in smoothness of surface should not be observed.

CALCULATIONS

X-ray reflectivity calculations (http://www.cxro.lbl.gov/) were performed to determine the transmission of X-rays through metallic capillaries with lacquer/Au bilayer. Geometrical parameters of capillaries (350 um inlet diamater, 200 um outlet diamater, 65 mm length, parabolic shape described by the equation y=sqrt(0.22X)) were used for 2D simulations. For such capillaries incident angles ranges from 0.05 to 0.07 degress. Fig. 5 presents the results of the calculations of reflectivity coefficient for 5, 10, 15, 20 i 25 keV for acrylic lacquer (2um)/Au(50nm) bilayer deposited on Ni substrate. Roughness of interface lacquer/Au layer was set at .5 nm (rms) which correspond to the mean roughness value of lacquered mandrel (Fig.3). Blue and green fill of graph on Fig. 5 stand for optimal region where enetring X-rays were able to penetrate through lacquer layer (2 um thick) and then can be reflected from gold at reflectivity coefficient ~ 0.7 – 0.8.

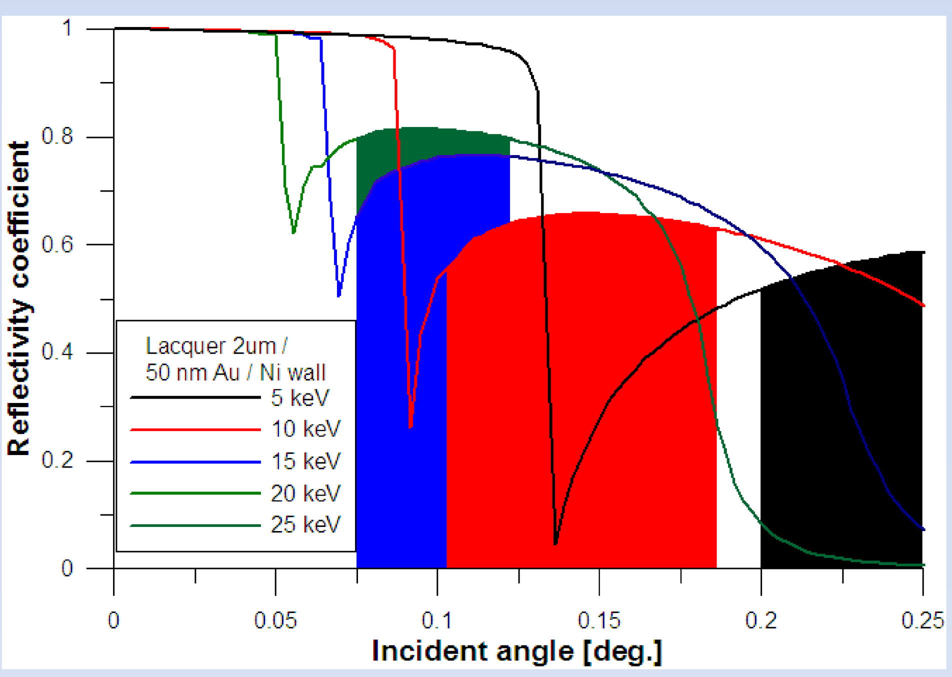

Fig. 5 X-ray reflectivity form acrylic (2um)/Au (50 nm) bilayer deposited on Ni substrate for different X-ray energy vs. Incident angle. Roughness (rms) of acrylic/gold interface - 1.5 nm.

CONCLUSIONS

1. **Surface roughness can be reduce about 15 times by coating a mandrel by acrylic polymer layer, from 30-40 nm up to 1.5 nm (Fig. 2b).**
2. **Thickness of acrylic layer ranges from 0.5-2.5 um on the mandrel (Fig. 4), but deviations of mandrel from desired parabolic profile are mantain at the same lavel as before the deposition of lacquer (results not presented here).**
3. **Vacuum deposition of thin gold layer does not degrade acrylic layer (AFM micrograpgs not presented here).**
4. **Due to high resistivity lacquer layer cannot be removed from capillaries. If its thickness is not higher than 3um for 15 – 20 keV energy of X-ray we can still predict good reflectivity (Fig.5). Geometrical shape of capillaries was redesigned and new capillaries were produced to fullfill these requiments.**
5. **Previously our method of manufacturing capillaries was limited only to noble metals due to the use of nitric acid for etching an mandrel. Now it is expanded to many metals (e.g.) Mo,Co,Ni) because the lacquer layer is excellent protection barrier against nitric acid**
6. **X-ray metallic capillaries with acrylic lacquer/Au bilayer will be tested very soon at synchrotron line (HASYLAB).**