Insitu annealing experiment in Indus-1 reflectometer station

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The multilayer mirrors are used in high brilliance third generation synchrotron beamlines where high heat load is generated. High thermal environment induces structural and chemical modifications in mirrors concerned [1] that ultimately deteriorates the optical performance. Performance of these optical elements under control thermal load needs to be evaluated in the laboratory. Designing an in situ annealing arrangement for reflectivity experiments is complicated due to high precision angular motion of the sample & detector. The reference surface for sample is to be maintained perfectly otherwise the reflected signal will be lost from the acceptance of the detector. In addition the heater operation should not interfere the detector arrangement used in reflectivity setup for detection of reflected signal in terms of radiative background. Furthermore the heater should be able to generate temperature ~900°C needed for most of thermal annealing experiments.

Different kinds of techniques are in use to heat the sample [2]. In conventional annealing setup the evacuated quartz tube is heated from outside using heating element, radiative heaters using focused infrared lamp are also used. We have designed a heater based on resistive heating. Tungsten wire has been employed as it can be used for e-beam heating for rapid thermal annealing also. The temperature on sample surface can goes up to 1000°C with Molybdenum base plate.

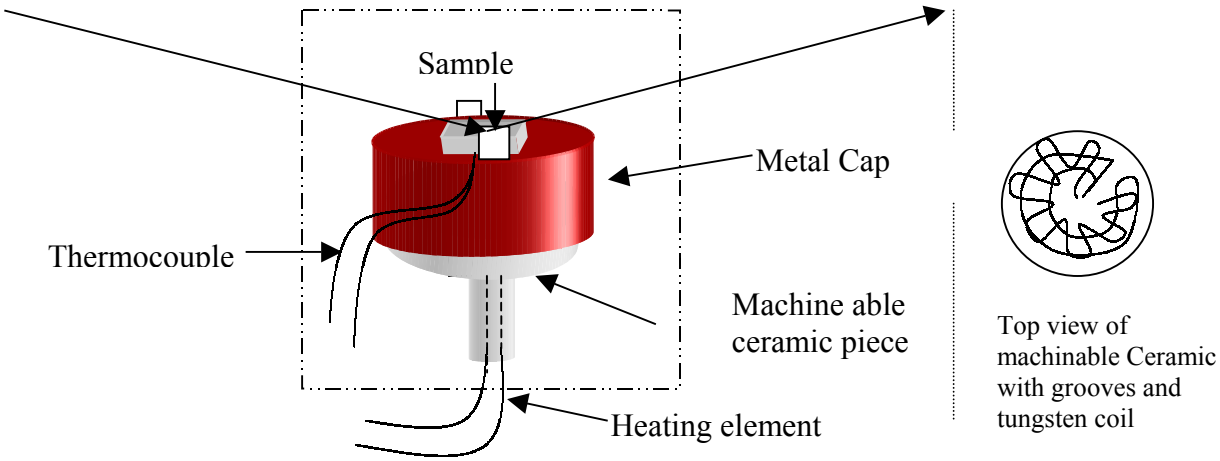

Figure 1: Schamatic of heater designed for insitu annealingexperiment in Indus-1 reflectometer.

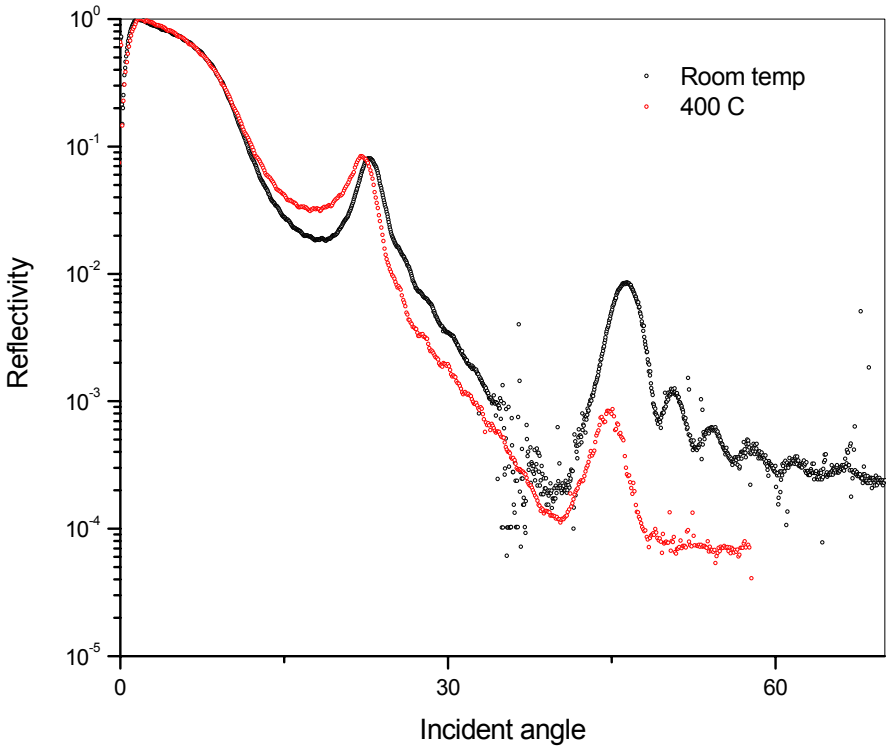

Figure 2: Change in reflectivity spectrum of W/C multilayer having period of 110Å (C 77Å/W 33Å)$_{10}$ measured atλ=80Å upon annealing at 400°C for 2 hours.

Heater design

We have used machinable ceramic as an insulator. A groove for coil placement was cut as shown in figure1. Tungsten coil made of 0.3mm diameter and 1.6ohm resistance is employed. A metal cap made of molybdenum/copper is placed on top of ceramic base. The sample sits on metal cap. Cr-Al thermocouple is used for temperature measurement.

Electrical power of heater is controlled with relay supply in feedback of temperature monitor. With 70 Watt of electrical power temperature upto 700°C is recorded. This whole heater is mounted on a special holder that ultimately mounts on goniometer rotary stage. Provision for alignment with SR beam is provided. Schematic of designed heater is shown in figure1. First annealing experiment is carried out on W/C multilayer of period d=110Å. In figure 2 the change in soft x-ray reflectivity of multilayer upon annealing is shown, with the reflectivity curve corresponding to annealed sample shifts toward lower angle side as the multilayer period expands. Detail study to understand structural modification in W/C sample is taken up using soft x-ray and hard x-ray reflectivity techniques.

Refrences:

1) P. Ruterana, J.P. Chevalier, P.Houdy, J.Appl. Phys. 65 (1989) p3907.
2) P. Paturi, H. Huhtinen, R. Laiho, Rev. Sci. Instr. 69 (1998) p3945.