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Arun Singh Dev, Dileep Kumar, Satish Potdar, et al.

Portable Mini-Chamber for Temperature Dependent Studies using Small Angle and Wide Angle X-ray Scattering

Arun Singh Dev[1], Dileep Kumar[1, a)], Satish Potdar[1], Pallavi Pandit[2], Stephan V. Roth[2,3] and Ajay Gupta[4]

[1]UGC-DAE CSR, University Campus, Khandwa road, Indore 452017, India
[2]Photon Science, DESY, Notkestrasse 85, 22607 Hamburg, Germany
[3]KTH Royal Institute of Technology, Department of Fibre and Polymer Technology, Teknikringen 56-58, SE-100 44 Stockholm, Sweden
[4]Amity Centre for Spintronic Materials, Amity University, Sector 125, Noida-201 303

[a)]Corresponding author: dkumar@csr.res.in

Abstract. The present work describes the design and performance of a vacuum compatible portable mini chamber for temperature dependent GISAXS and GIWAXS studies of thin films and multilayer structures. The water cooled body of the chamber allows sample annealing up to 900 K using ultra high vacuum compatible (UHV) pyrolytic boron nitride heater, thus making it possible to study the temperature dependent evolution of structure and morphology of two-dimensional nanostructured materials. Due to its light weight and small size, the chamber is portable and can be accommodated at synchrotron facilities worldwide. A systematic illustration of the versatility of the chamber has been demonstrated at *beamline P03, PETRA-III, DESY, Hamburg, Germany*. Temperature dependent grazing incidence small angle x-ray scattering (GISAXS) and grazing incidence wide angle x-ray scattering (GIWAXS) measurements were performed on oblique angle deposited Co/Ag multilayer structure, which jointly revealed that the surface diffusion in Co columns in Co/Ag multilayer enhances by increasing temperature from RT to ~ 573 K. This results in a morphology change from columnar tilted structure to densely packed morphological isotropic multilayer.

INTRODUCTION

Thin films, in particular nanometer scale structures, have been the subject of numerous studies because of their potential applications in the field of thin film technology [1]. The surface/interface roughness, stresses, strain, morphology and structure of thin films control many important physical and chemical properties of such type of structures [2,3,4,5]. Particularly, in case of ferromagnetic thin films, surface and interface morphologies significantly influence the magnetic properties, such as magnetic hysteresis, magnetic anisotropy, coercive force, magnetoresistance as well as structure of the magnetic domains. Besides the preparation conditions [2], temperature is one of the important parameter which strongly affects structure and morphology of the thin films [6]. For example, depending on substrate temperature during deposition, good quality films with smooth surface can be deposited [7]. In case of FePt thin film, annealing leads to transformation of fcc disorder phase to highly L10 ordered fct phase which is responsible for the appearance of hard magnetic properties in FePt layer [8]. Annealing in the presence of magnetic field is used for inducing strong magnetic anisotropy in thin films, where strength of the magnetic anisotropy sensitively depends on structure and film morphology. A number of experiments in the literature aiming to correlate the magnetic properties with interface structure and morphology have been performed [9]. In such studies, evolution of surface morphology and structure has been studied by depositing series of samples and post annealing at selected temperatures. However, the possible unwanted variation in deposition conditions or post-deposition treatments in preparing series of samples may induce changes in the morphological features like grain size and grain texture. Therefore, in such experiments, it is difficult to separate the effect of temperature on morphological and structural properties unambiguously.

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In order to avoid such problems and to study temperature dependent properties on a single sample, a dedicated mini chamber has been designed and developed, where temperature dependent morphological and structural changes can be studied simultaneously during annealing under high vacuum (HV) condition. A systematic illustration of the potential of the set-up has been demonstrated on multilayer sample: $Co_{oblique}$(44Å)/Ag_{normal}(15Å) multilayer (10 bilayers) deposited on Si(100) substrate. To follow the evolution of the structural and morphological features, temperature dependent grazing incidence wide angle x-ray scattering (GIWAXS)and grazing incidence small angle x-ray scattering (GISAXS) measurements were carried out respectively at MiNaXS P03 beamline, Petra III, DESY, Germany [10], which were later correlated with the magnetic properties of the multilayer.

DESIGN AND DESCRIPTION

The overall design of the vacuum compatible mini chamber is decided according to the demand for simultaneous structural characterization using grazing incidence GISAXS and GIWAXS of thin films with an option of in-situ heating. A photograph of the chamber along with port description is shown in Fig. 1(a). SS304L material is used for fabrication of the chamber. It has a diameter of 80 mm and a height of 170 mm. The chamber has number of ports to accommodate various components namely; heater, vacuum pump, an UHV gauge, electrical feedthrough and Kapton windows for x-rays. The top dome is prepared by fixing it on a 63mm CF flange. It houses two Kapton windows for x-ray entrance and exit. The designed chamber has a volume of about 2 liters and surface area exposed to vacuum is about 66100 mm^2. Considering the gas load due to the surface area, a rough estimate of time required evacuating complete chamber to a pressure ~10^{-8} mbar with a 100 l/s turbo molecular pump is less than an hour, whereas the time required to pump out the gas load due to the volume of the chamber is almost negligible. A compact cold cathode gauge (Model: FRG-700) has been used for vacuum measurement in a pressure range up to 1×10^{-8} mbar. The ultimate vacuum ~ $5\text{x}10^{-8}$ mbar is achieved in the chamber within 30 minutes using 100 l/s TMP. Figure 1(b) shows the photo of the mini chamber at P03 beamline. For sample heating, an UHV compatible pyrolytic boron nitride heater has been used, which offers a quick heating of the sample [11] with a rate of about 100 K/s. It has very low thermal mass with chemically inert and dielectric surface. Two stainless steel (S.S) rods are used for providing the base to the heater and to keep the heater surface at the level of Kapton windows for x-ray scattering experiments. The heater surface dimensions are about of $35\times25\times2$ mm^3. K-type thermocouple is attached to the surface of heater to record the sample temperature. Two Kapton windows of size $50\times25mm^2$ and $80\times35mm^2$ have been made on the top dome of the chamber in opposite direction for incident and scattered x-rays. Exit window size is kept bigger so as to cover suitably large scattering angle along exit direction of the x-ray, which is essentially required for GIWAXS measurements. A copper pipe is welded with the chamber body for the water flow so as to maintain its temperature near room temperature (RT) during annealing of the sample. Water flow also prevents the damage to the Kapton window due to possible increase in the surrounding temperature.

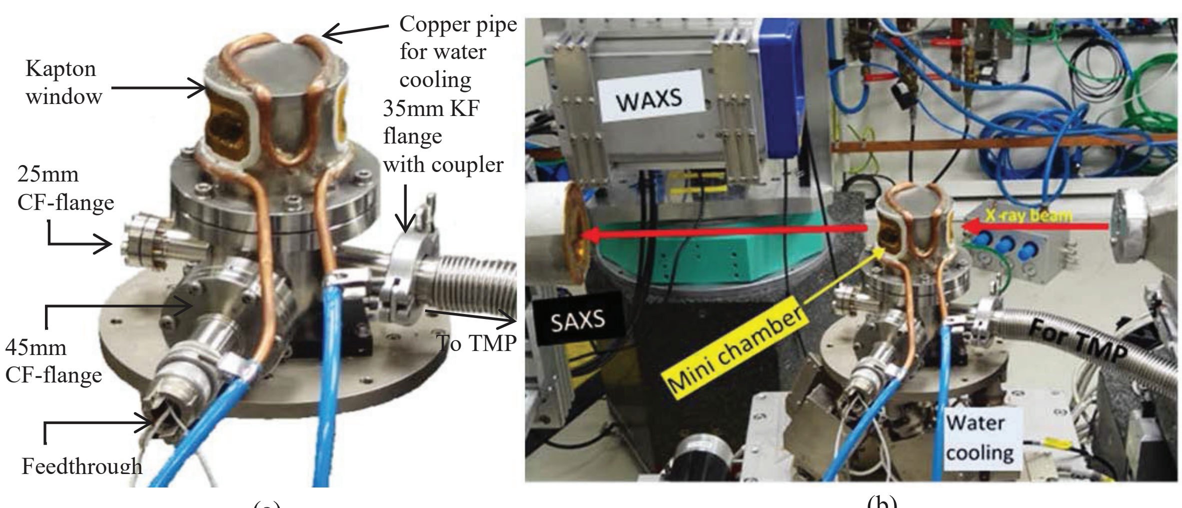

FIGURE 1(a) Actual photograph of the chamber and its various components (b) Mini chamber attached at beam line P03 at Petra III, Hamburg.

RESULTS AND DISCUSSION

In order to demonstrate the strength of the chamber, a multilayer structure [$Co_{oblique}$(44Å)/Ag_{normal}(15Å)]$_{10}$ was deposited on Si(100) substrate in a separate UHV chamber using e-beam evaporation technique. To prepare this multilayer, angle of incidence (θ) was kept alternatively at θ=75° (oblique angle) and θ=0° during evaporation of Co and Ag materials respectively. Here θ is the angle of deposition flux direction with respect to substrate normal. Since obliquely deposited Co layers are expected to be in columnar structure, therefore final multilayer structure is expected to be consists of alternative layers of columns (Co) and isotopic Ag layers. It may be noted that, this multilayer is named as Co_{obl}/Ag_{nor} in the rest of the manuscript.

For temperature dependent GISAXS and GIWAXS measurements, multilayer was attached to the heater plate inside the chamber. As shown in the Fig. 1(a), the mini chamber was placed on an sample stage at P03 beamline, PETRA-III, DESY, Hamburg [10] with the capability of aligning the sample using various motorized motion (6-axis motions in HEXA pod). Geometry for the GISAXS measurements is shown in Fig. 2(a), where α_i and α_f are the incident and reflected angles of x-ray with respect to sample surface and $\overrightarrow{k_i}$ and $\overrightarrow{k_f}$ are the incident and outgoing wave-vectors. Centre of rotation of the chamber was kept at the sample surface height in such a way that two–dimensional GISAXS and GIWAXS patterns can be measured through the Kapton windows in a vertical and horizontal scattering plane without variation in the beam position on the sample surface (in case of change in α_i). For the present experiments, incident photon energy of 13 keV was used at the sample position. PILATUS 1-M (Dectris Ltd., Switzerland) and PILATUS 300k detectors with a pixel size of (172 × 172) μm^2 were used for GISAXS and GIWAXS data recording respectively. The sample-to-detector distance (SDD) was set as 2245 mm for GISAXS and 128 mm for WAXS measurements. Incident angle α_i was fixed to 0.45°, which is well above the critical angle of the

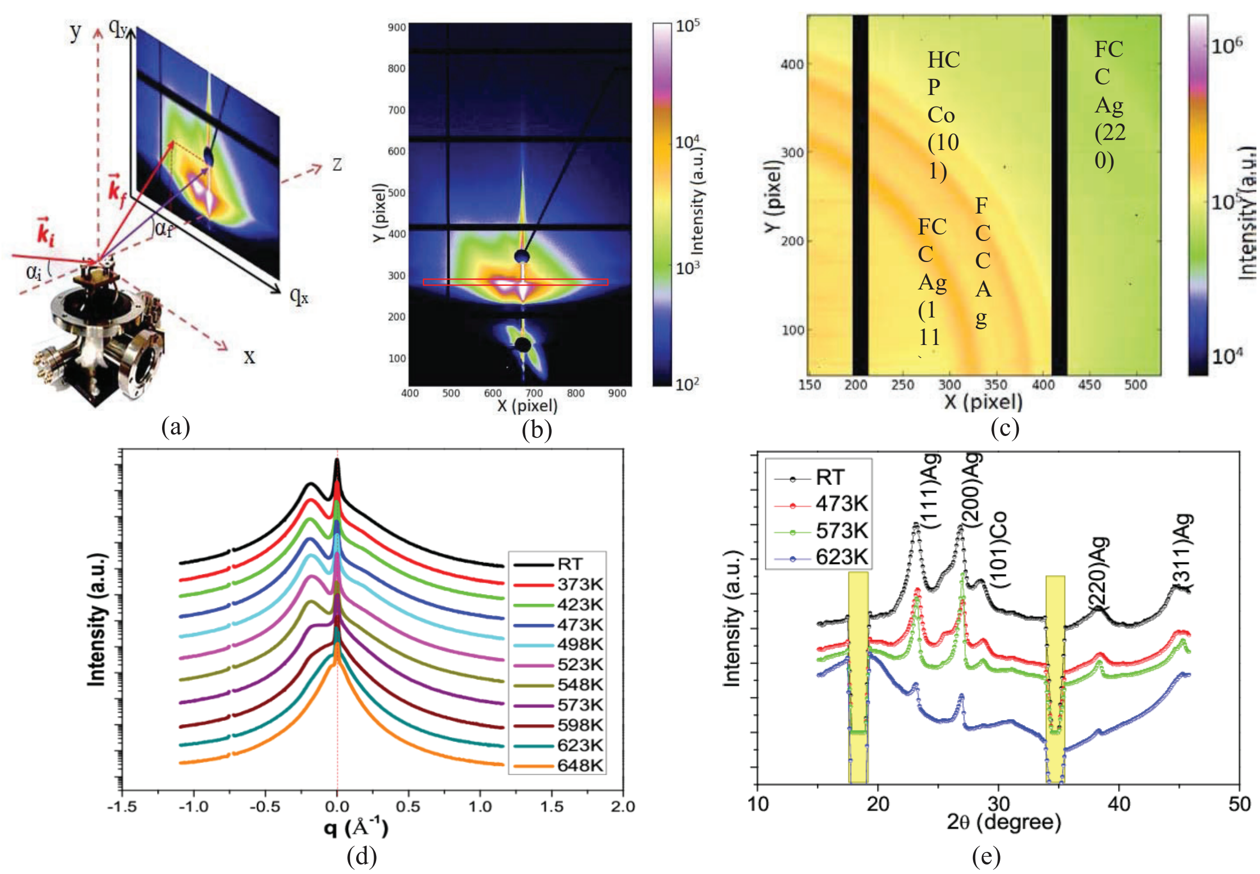

FIGURE 2(a) the geometry for GISAXS measurements, (b) and (c) show GISAXS and GIWAXS patterns of Co_{obl}/Ag_{nor} multilayer at room temperature (RT). **FIGURE 2**(d) and (e) gives GISAXS and GIWAXS 1-D profiles with increasing temperatures. GISAXS 1-D profiles are taken at red marked area in Fig. 2(b) corresponding to yoneda wings region. The yellow areas in GIWAXS 1-D profiles correspond to the intermodular gap (IDG) of the detector, where no photons can be detected.

Co film at 13keV (corresponding λ=0.95373Å) in present study. Measurements were performed on Co_{obl}/Ag_{nor} multilayer with increasing temperature under base vacuum of 2×10^{-5} mbar.

Representative GISAXS and GIWAXS patterns of Co_{obl}/Ag_{nor} multilayer at room temperature are shown in Fig. 2(b) and 2(c) respectively. In the GISAXS pattern, an asymmetric distribution of the scattered intensity on both sides of the specular rod has been observed. Continuous arcs in GIWAXS image confirm the polycrystalline nature of the Co_{obl}/Ag_{nor} multilayer. It may be noted that full diffraction ring in GIWAXS measurements are not visible due to the fixed opening of Kapton window. Arcs with corresponding planes are marked in the image. In order to understand the temperature dependence more clearly, 1D profiles extracted from the temperature dependent 2D GISAXS and GIWAXS images are shown in Fig. 2(d) and 2(e) respectively. In Fig. 2(d), broken 1D profiles (zero photon counts) near q= -0.74Å^{-1} correspond to the intensity drop underneath module of modular detector and does not influence the data analysis. 1D GISAXS profile present two intensity maxima visible along the q direction, separated by the specular rod. It may be noted that the maxima (satellite peaks) at left side of the specular peak is relatively intense. It may be noted that this maxima shifts toward lower q values (towards specular), and become weaker as temperature increases. Interestingly, at temperature above 648 K, both side of specular rod becomes symmetric. Extracted temperature dependent 1D data from simultaneous GIWAXS measurements using DPDAK software, confirms growth of Co layers in hcp phase and Ag layers in fcc phase in Co_{obl}/Ag_{nor} multilayer. Furthermore, sharpening of the diffraction peaks with temperature confirm increase in particle size. In GISAXS measurements, decreasing asymmetric distribution of the scattered intensity with increasing temperature confirms the termination of tilted columns (formed due to oblique angle Co deposition) [12] due to diffusion at temperature above 573K.

CONCLUSION

The present work describes the design and the performance of a mini chamber for temperature dependent studies of thin films and multilayer structures using simultaneous grazing incidence small and wide angle x-ray scattering measurements. The water cooled body of the chamber allows sample annealing up to 900 K using UHV compatible pyrolytic boron nitride heater, thus making it possible to study the temperature dependent evolution of structural and morphological properties of 2D nanostructured materials. A systematic illustration of the versatility of the chamber has been demonstrated on oblique angle deposited Co_{obl}/Ag_{nor} multilayer structure on Si(100) substrate at P03 beamline, PETRA-III, DESY, Germany. In-situ temperature dependent GISAXS and GIWAXS measurements were done to study the morphology and structure respectively. Combined analysis revealed that the increase in temperature enhances the diffusion in Co columns. Temperature about 648K resulting in a morphology change from columnar tilted structure to densely packed morphological isotropic multilayer.

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