**Experiment title:**
Structure investigation of embedded nanoclusters in SiO\textsubscript{2} films by x-ray diffraction

**Experiment number:**
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**Report:**

1. **Nanoclusters in thin SiO\textsubscript{2} films**

Ion beam synthesized Au, Ge, and Si nanoclusters embedded in SiO\textsubscript{2} films of 30 or 100 nm thickness onto a (100) Si substrate have been investigated by grazing incidence X-ray diffraction. The best experimental conditions have been found to use an incident angle of 0.3° and a Soller collimator in front of the szintillation detector. The results can be summarized as follows:

(i) Au nanoclusters can be clearly detected even in thin SiO\textsubscript{2} films < 100 nm. Depending on the Au impurity content in the oxide (7-20 at.%) and the annealing temperature of ion beam synthesis the nanocluster size varies between 1.5 and 3 nm which is in reasonable agreement with TEM results (Fig. 1).

(ii) As derived from the existance of multi-reflexes [(111), (200), (220), (311)] the Au nanoclusters are nanocrystals with a random crystalline orientation within the matrix. The crystalline structure has been found even in the as-implanted state, which can be explained by the high mobility of gold under the beam during the synthesis process. No texture effects have been observed.

(iii) From the comparison of Au, Ge and Si nanocrystals it has been found that Ge nanoclusters could be identified only for the highest impurity content of 20 at%, whereas the detection of Si nanocrystals failed (Fig. 2).
The use of an asymmetrical cut Ge crystal in front of the sample has only a minor influence on the SN-ratio of the diffraction reflexes (Fig. 3). The idea behind was to enlarge the divergence of the incoming beam to fulfil the condition for X-ray diffraction for an increased number of nanocrystals with respect to their random orientation within the SiO₂ film.

![XRD pattern of Au nanocrystals embedded in a 30 nm SiO₂ film on (100) Si for different annealing conditions (left) and cross-sectional TEM micrographs of the film annealed at 1050°C for different magnifications (right) which enable information about the position of the nanoclusters in the oxide and their crystalline structure due to the identification of the lattice planes (inset).](image1)

![Comparison of the XRD pattern of Au nanocrystals in SiO₂ films for different measuring modes. The use of an asymmetrical cut Ge crystal results in only a minor improvement in the SN-ratio (10-15%), whereas small angle scattering significantly reduces the intensity.](image2)

**Fig. 1:** XRD pattern of Au nanocrystals embedded in a 30 nm SiO₂ film on (100) Si for different annealing conditions (left) and cross-sectional TEM micrographs of the film annealed at 1050°C for different magnifications (right) which enable information about the position of the nanoclusters in the oxide and their crystalline structure due to the identification of the lattice planes (inset).

![Comparison of the XRD pattern of Au nanocrystals in SiO₂ films for different measuring modes. The use of an asymmetrical cut Ge crystal results in only a minor improvement in the SN-ratio (10-15%), whereas small angle scattering significantly reduces the intensity.](image3)

**Fig. 2:** Comparison of the XRD pattern of Au, Ge, and Si nanocrystals in thin SiO₂ films (Au, Ge for dox=30 nm; Si for dox=100 nm). Despite the higher annealing temperature and the larger oxide thickness no significant reflexes have been observed for Si.

**Fig. 3:** Comparison of the XRD pattern of Au nanocrystals in SiO₂ films for different measuring modes. The use of an asymmetrical cut Ge crystal results in only a minor improvement in the SN-ratio (10-15%), whereas small angle scattering significantly reduces the intensity.

### 2. Investigation of Si/SiO₂ superlattices

In addition, X-ray reflectometry (XRR) has been successfully applied to determine the properties of superlattice structures made by ~ 45 periods of SiO₂ (5nm) and thin Si nanocrystal films (with variable nanocrystal size between 2-5 nm). The structures were produced by reactive physical vapour deposition. The evaluation of the XRR interference pattern gave information about the total thickness of the superlattice and allowed to determine independently the thickness of the SiO₂ layers and the mean nanocrystal size, which corresponds to the Si layer thickness. For these investigations the high angle resolution of the ROBL beamline enables considerably advantages in comparison to laboratory equipment.