	Experiment title: Solid-liquid eutectic transition in Au-Ge catalysts for the growth of semiconductor nanowires.	**Experiment number**: SI-1947
Beamline: ID03	**Date of experiment**: from: 28/10/2009 to: 03/11/2009	**Date of report**: 10/03/2011
Shifts: 18	**Local contact(s)**: Roberto Felici, Didier Wermeille	*Received at ESRF:*

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Report:

Semiconductor nanowires are produced via the vapour-liquid-solid mechanism [1] in which a low temperature semiconductor/metal eutectic is used to enable their nucleation and their growth. The base of this mechanism is the liquid state of these alloys which present in general a deep eutectic point that confers catalytic properties even at low temperature. For Au deposited on Si(111) substrate, which is the most used catalytic system, we have already reported several results concerning the structural evolution of the thin film upon annealing/cooling cycles. The most interesting result is the observation of an enhanced supercooling behaviour of the system depending on the surface/interface structure [2]. It is often assumed in literature that Si and Ge present very similar characteristics. It is therefore important to compare these two materials by proceeding to the same kind of experiments with a Ge(111) substrates. To do so a deposit of 7 ML of gold was performed at room temperature on a clean Ge(111) substrate. Then the sample was heated to the AuGe eutectic temperature (T_e=640 K) and the melting was observed.

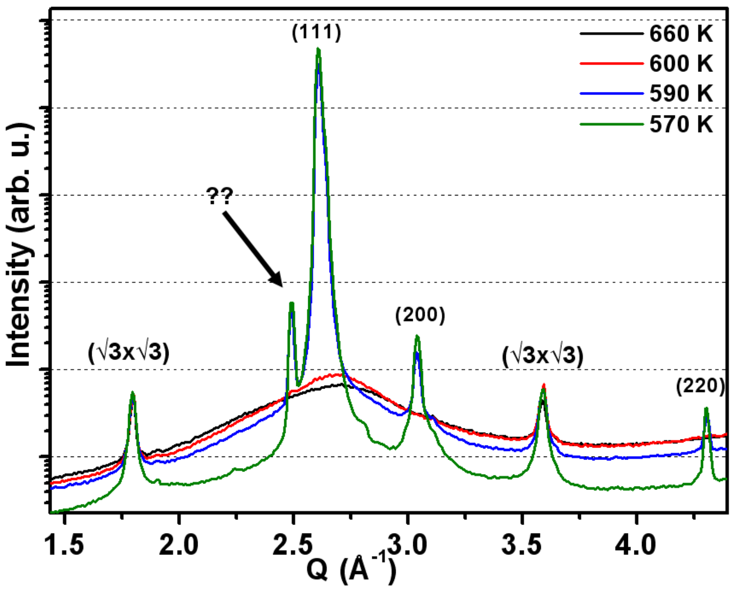

Figure 1: Radialscan performed along the Ge[1$\bar{1}$0] azimuth upon cooling. The solidification process takes place around 580 K.

In order to stick to our experimental procedure on the AuSi system, the sample was annealed to 685 K (50 K above T_e) and then cooled down to follow the liquid-solid transition. Figure 1 shows radialscans performed along the Ge[$1\bar{1}0$] azimuth for several temperatures. At 660 K (20 K above T_e) the AuGe liquid droplets give rise to a clear liquid signal with the presence of Au-(√3x√3) reconstruction peaks. At 600 K, the system is still liquid (40 K below T_e) as no gold peak has re-appeared, but at 590 K most of the liquid signal disappear and polycrystalline Au Bragg peaks arise. Cooling down further leads to a complete disappearance of the liquid phase, the supercooling value for this experiment was thus about 50~60 K.

A reciprocal space map was also recorded at 600 K upon cooling (Figure 2(a)). This map displays the first and second order of the liquid structure factor together with Au-(√3x√3) reconstruction peaks and other thin spots that could have not been assigned until now (as well as the one on Figure 1).

During the experiment, the Au/Si(111) system was also studied to investigate a potential structure of the liquid close to the substrate/droplet interface. Figure 2(b) is a reciprocal space map of the Au/Si(111) system, also in the supercool regime. The difference with the Au/Ge(111) system is the presence of Au-(6x6) reconstruction peaks which is known to form on Si(111) substrates [3]. The diffuse ring (Figure 2(b)) binding the (6x6) peaks around the Au-(√3x√3) spot has already been observed [2,4] but its origin is still unclear. As we do not observe this diffuse ring for the Au-(√3x√3) on Ge, it is a reasonable to assume that it may arise because of the existence of a link between the Au-(√3x√3) and Au-(6x6) reconstructions which has not been discovered yet as their structure at the atomic scale are still under debate. This calls for additional experiments and structural models to get a complete understanding of these two systems.

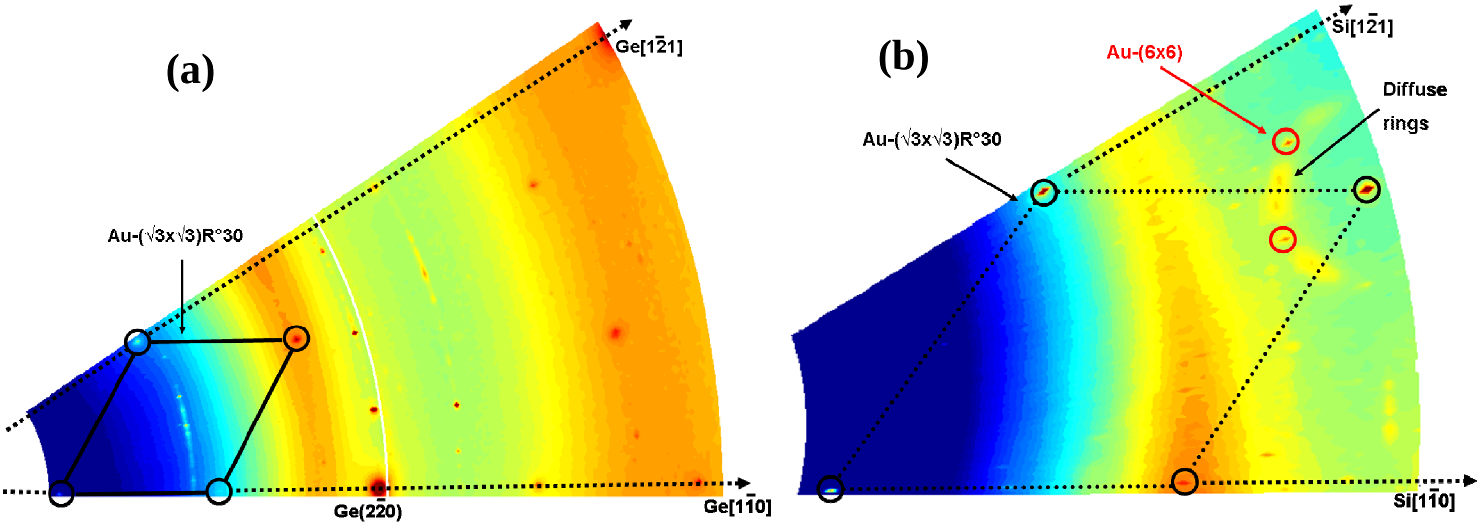

Figure 2: Reciprocal space map in the supercool regime for the (a) AuGe and (b) AuSi system. Red colour are for high intensity, blue for low and yellow for intermediate.

References:

[1] R. S. Wagner, W. C. Ellis, Appl. Phys. Lett. **4**, 89-90 (1964).
[2] T. U. Schülli, R. Daudin, G. Renaud, A. Vaysset, O. Geaymond, A. Pasturel, Nature **464,** 1174-1177 (2010).
[3] D. Grozea, E. Landree, L.D. Marks, R. Feidenhans'l, M. Nielsen, R.L. Johnson, Surf. Sci. **418**, 1 (1998).
[4] S. Takahashi, Y. Tanishiro, et K. Takayanagi, Surface Science **242**, 73-80 (1991).