ESRF	**Experiment title:** **X-ray investigations on CoSi$_2$ nanowires manufactured by focused ion beam synthesis**	**Experiment number:** **SI-1548**
Beam line: **ID 01**	**Date of experiment:** **from:** 05 December 2007 **to:** 11 December 2007	**Date of report:** 18.03.2008
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Report:

Nanowires and chains of nanoparticles are of emerging interest in nanoelectronics, nano-optics and plasmonics as well as for their monolithic integration into microelectronic devices; CoSi$_2$ is a promising material due to its CMOS-compatibility micro-electronics technology that shows metallic behaviour with low resistivity and high thermal stability. It is well known that cobalt disilicide films can be formed in silicon by implanting Co in stoichiometric concentration and a subsequent annealing procedure. It has been shown that ion beam synthesis allows the fabrication of epitaxial buried or surface CoSi$_2$ layers on silicon. Submicron patterns can be directly produced by writing focused ion beam (FIB) cobalt implantation. Si(111) wafers were implanted with 60 keV Co^{++} ions in the dose range of 2×10^{16} to 2×10^{17} cm^{-2}. A subsequent annealing was done in a two step process, namely 600°C for 60 minutes and 1000°C for 30 minutes in a N$_2$ ambient.
To improve the visibility of the fabricated nanowires a short reactive ion etching in CF4 for ~30 s was applied to remove the ~20 nm thin silicon top layer covering the buried CoSi$_2$ nanostructures. SEM investigations showed the formation of continuous nano wire structures following the <110> direction. The formation of CoSi$_2$ nanostructures is explained by precipitation, Ostwald ripening and coarsening that causes a shrinking of the initial implanted profile. Sizes down to 10 nm are demonstrated. For the formation of single-crystalline of cobalt disilicide it is necessary to avoid amorphization of the silicon on top of the implanted region. To reach the necessary dose in the range of 10^{16} – 10^{17}cm^2 a lot of repetitions for each pixel irradiation is required. Additionally the temperature of the sample was kept constant at about 420°C during implantation.

We have studied the strain of the Si host lattice in the surrounding area of a single nanostructure using high resolution x-ray diffraction in combination with a highly focused (0.5µmx3µm) x-ray beam at the beam line ID01 at the ESRF. We could show that it is possible to investigate a single nano wire of a size of below 50 nm.

The pattern measured directly on the wire shows a small peak indicating tensile strain (approx. -1.4%). This feature can be only found if the X-ray beam focused on a single nano wire whereas its intensity changes with the layer width. We found that the CoSi$_2$ peak change slightly if we move from one wire to an other. Moreover the diffuse scattered intensity around the Si bulk reflection is strongly enhanced.

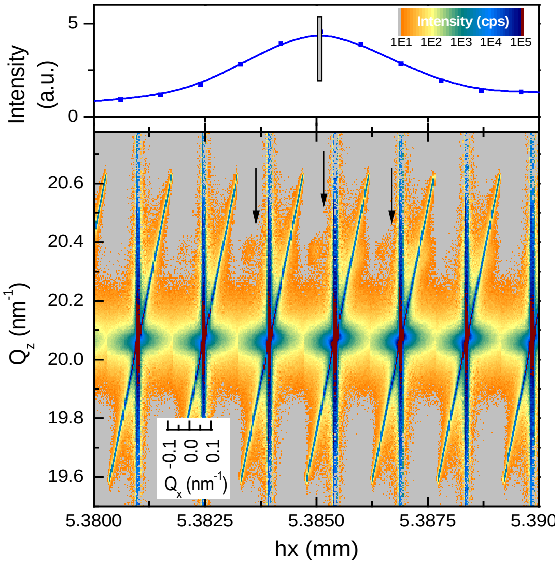

Figure 1: Reciprocal space maps measured across one singe CoSi$_2$ nano wire; the beam width was about 3µm. The CoSi$_2$ peak is indicated (arrow).

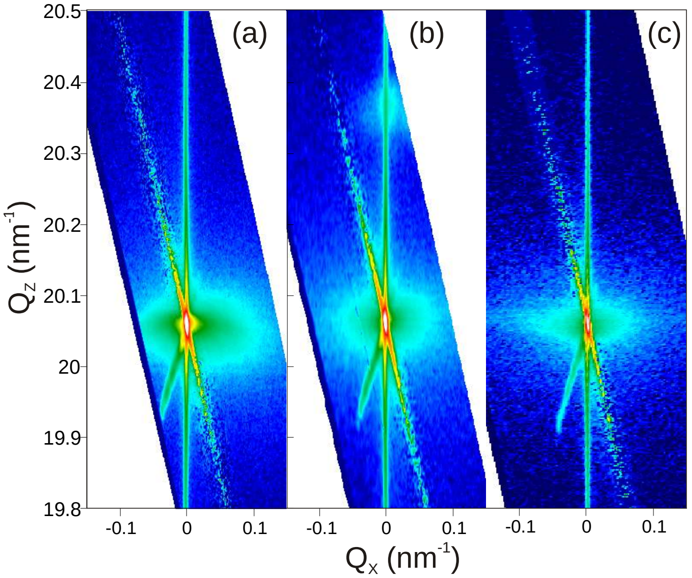

Figure 2: Reciprocal space maps measured with a smaller focus width of 0.5µm (a) between by 10µm separated wires, (b) on a wire and (c) several 100µm away from any wire.

Figure 1 shows a series of reciprocal space maps recorded at different positions while moving the X-ray beam over a singe nano wire. The CoSi$_2$ was clearly identified. However the beam size was to large to see more details, therefore the beam size was further reduced down to 500nm vertically resulting in a foot print of about 2µm. Figure 2 shows the corresponding reciprocal space maps around the Si (111) reflection recorded at different distances from the nano wire. These are the first measurements where we were able not only to detect a single wire but as well as to find a difference in the diffuse scattering measuring between wires or far away from them. The diffuse scattering around the Si(111) reflection in the vicinity of a wire seems to be increased (Fig.1a) whereas it is again reduced measuring directly on a wire (Fig.1b). For comparison a reciprocal space map measured about 350µm away from any wire shows the lowest diffuse scattering, comparable to a non treated substrate (Fig.1c). These findings may indicate the possible mechanism lying behind the formation of CoSi$_2$ rods, their spontaneous and self-aligned growth along the <110> directions: The implantation of Co into the Si creates not only a supersaturated Co solution but as well as a large number of excess Si interstitials. Upon annealing they agglomerate, form clusters and at the end transform into stable dislocation loops enclosed between {311}-planes and elongated in <110> direction (rod-like defects). During annealing the solved Co atoms diffuse and getter in these rod like defects and form the silicide.

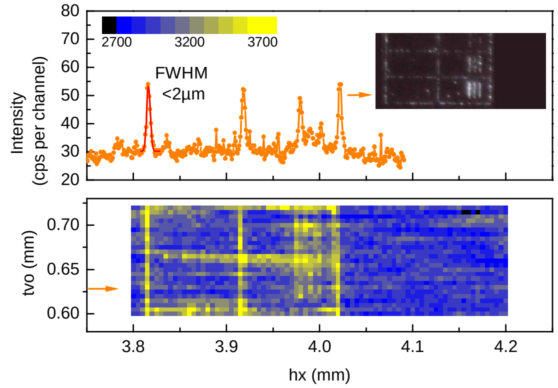

Figure 3: Real space pattern of the investigated CoSi$_2$ nano wires, single wires within a FWHM of 2µm can be detected; the inset shows a microscopy pattern.

Figure 3 demonstrate the spatial resolution of the experiment. The change of intensity exactly resembles the structure of the CoSi$_2$ nano wire pattern (see microscopy picture in the inset). The resolution of this real space pattern is below 2µm and is mainly limited by the accuracy of the mechanical sample movement and the beam size. A further reduction the foot print of the X-ray beam on the sample and an increase the mechanical stability as well as of the accuracy of the sample movement would allow one strongly to increase the sensitivity of the experiment. Therefore we would be able to study the change of the diffraction pattern in the close vicinity of a nano wire to learn more about the influence of the defects on the formation of CoSi$_2$ nanowires itself.