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Report:

During the 4 days of our experimental time at ESRF, in total 823 data files, each being one 2D-XRD image (about 2 MBytes/file), were collected. This was mainly possible because part of the data acquisition could be done automatically using a macro file written by Florian Heidelbach. The experiments were performed in December 1996, and the evaluation of the results is not finished yet. This report is therefore a preliminary report, and lists the experiments done, some practical problems and the first results. No publications were made yet using these data.

Sample preparation and mounting.

The sample used for the first experiments was a small piece from a Si wafer (about 1x1x0.5 mm^3), containing 140 nm thick $CoSi_2$ lines (fabricated by annealing 30 nm Co during a two step rapid thermal anneal (450 °C, 100s; 700 °C, 30s) with different dimensions, separated by a 100 nm thin oxide.
This sample was glued on a thin glass bar and the bar was mounted in a plastic holder which fitted in the goneometer head. The mounting was such that the $CoSi_2$ lines were perpendicular to the phi rotation axis. This is important, because, when the sample is rotated along this axis, the XRD beam will elongate along the length of the lines, and not along the width. As we do not expect a strain variation along the length of the lines, elongation of the beam does not give a problem. However, if the beam would elongate along the width direction, we would measure different regions along the width of the line (-> different strain) when changing phi, which would make the results unreliable.
The XBD beam was focused through a glass capillary on the sample. The beam radius changes with distance from the capillary. In order to have a small beam (we aimed at a beam diameter < 5 µm), it is important to bring the sample as close as possible to the capillary. Further, this should be done in such a way that the sample does not hit the when rotating. For this reason, the sample was moved, in horizontal position, as close as safely possible to the capillary (see Fig. 1, distance d_1), and then turned to the correct phi angle for XRD measurements. We did overlook one important detail here, which is important to remember for later measurements. It is explained in Fig. 1. The rotation axis for phi is determined by the place on the sample to be measured. In our case, it was close to the left side of the sample, as shown in Fig. 1 by the black dot. When rotating the sample around this phi axis, the distance increases (d_2). A shorter distance (d_3), and thus a smaller beam spot, would have been obtained when the rotation axis is close to the right side of the sample as shown in Fig. 1, bottom. Because the result of the experiment is better for a smaller beam dimension, this mounting of the sample is very important and should be done as good as possible.

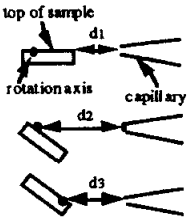

Fig.1: ***Distance of sample to capillary versus position of the phi axis***

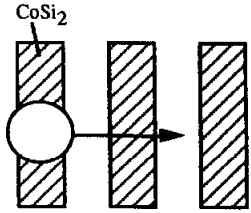

Fig.2: ***Scanning experiment across 5 μm wide $CoSi_2$ lines.***

Experiments and first results

Besides many tests on the Si (HKL = 440) and on the $CoSi_2$ (HKL = 440) lines to obtain a good XRD signal, three main experiments were performed:

1. Scanning across 5 μm wide $CoSi_2$ lines

The intention of this experiment is to measure the variation of mechanical stress along the width of 5 pm lines. A scan was performed across 5 μm wide $CoSi_2$ lines, starting on top of the first line, and scanning across the second line. This is shown in Fig. 2. Each 1 μm, an XRD signal was measured at five different angles phi: 12.5.27.5.35.50 and 62. This is necessary to extract strain data from the XRD signal. Further, a small relative phi scan was done with an relative angle +0.5 and -0.5 around each of these five angles, for example at 12. 12.5, 13 etc.. The XRD images obtained at these three angles will be added to improve the statistics of the experiment. So, at each position of the beam on the sample, 15 spectra were collected, with an integration time of 300 sec for each measurement. In total, data were obtained at 16 adjacent positions on the sample. The further processing of these data is still going on. However, to have a first estimation of the magnitude of the XRD beam diameter, the intensity of the $CoSi_2$ signal as function of the position was extracted from the data. This is shown in Fig. 3. It is clear from this figure that the $CoSi_2$ signal does not go to zero in between the lines. This indicates that the XRD beam diameter is larger than 5 μm. This is probably due to a too large distance between sample and capillary. However, there is a clear variation of the intensity with position on the lines, indicating that the beam size << 10 μm. The position of the lines can be estimated from the sign of the 2th derivative of a polynomial lit to the data (dotted line).

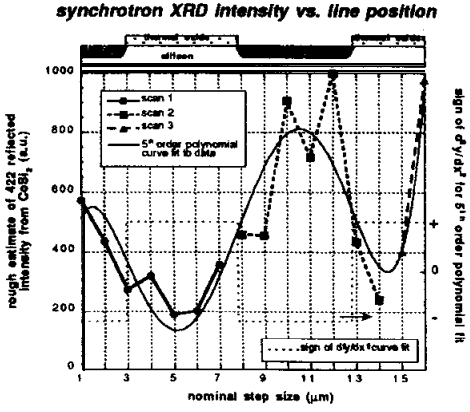

Fig. 3: ***Intensity of $CoSi_2$ signal as function of the position on the sample.***

2. Zoom-out experiment.

In a second experiment the beam was positioned on the sample just outside the first 5 pm $CoSi_2$ line, so that no $CoSi_2$ signal was obtained. Next, the distance from sample to capillary was increased in steps of 0.43 mm. This resulted in an enlargement of the beam diameter, so that the beam did probe a small part of the $CoSi_2$ line. The larger the distance, the larger the beam diameter, the larger the part of the $CoSi_2$ line which was probed. In this way, an alternative "zoom-out scan" across the $CoSi_2$ line was performed. Also here, the data still have to be further processed.

3. $CoSi_2$ line width

In order to control whether the strain in the $CoSi_2$ lines changes with line width, XRD measurements were performed on lines with wtdth 3 μm and 2 μm. Also here one of the main problems was probing the correct lines on the sample. No optical tool was available to control this. Again, data were collected at different phi in order to extract strain information

Expected progress

The data have to be further processed using the FIT2D program of A.P. Hammersley, of which is a version available at IMEC. There is still a lot of work to do: summing the data for small Phi in order to obtain better statistics, correcting.for the Si, signal which sometimes overlaps with the $CoSi_2$ signal, and extracting the relevant data.for strain calculations. The experiments were very interesting and promising, good signals from the thin $CoSi_2$ could be obtained. We are convinced that we will get some nice results. A lot of know-how was collected during these first experiments with the micro beam, which will be very important for further similar experiments we hope to do with this micro-beam at ESRF.