ESRF	**Experiment title: Study of kinetics of interdiffusion in NbSi multilayers measured by x-ray reflection during thermal annealing**	**Experiment number**: HS-1104
Beamline:	**Date of experiment**: from: 8 nov 2000 to: 12 nov 2000	**Date of report**: 20 jul 2001
Shifts:	**Local contact(s)**: Dr. Till Metzger Dr. Angel Mazuelas	*Received at ESRF:*

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Report:

The proposal was originally concentrated to thermal stability of NbSi multilayers. The beam time was finally allocated to later scheduling period than it was expected. In that time the study of thermal stability of both amorphous and partially crystalline NbSi multilayers was finished and published (or accepted for publication). That is why, after some consultation with local contact, the aim of the experiment was changed a little bit and concentrated to NbTi systems.
The NbTi multilayers prepared by magnetron sputtering at Institute of Electrical Engineering of Slovak Academy of Science, Bratislava. We measured two type of samples: Sample denoted 81 – Titanium thick Nb(30Å)Ti(50Å) and sample 52 – Niobium thick Nb(80Å)Ti(50Å). All multilayers have ten periods and was deposited on silicon substrate. Small chips used for measurement was cut from a single larger plate to get the same starting conditions for each annealing cycle.
Three different types of experimental scans were realized. For all of them PSD detector was used in vertical position (parallel to diffraction plane).
1) Small angle in plane reflection – th + del scan in angular scale up to 4° in th. The measurement was provided both ex-situ (before and after annealing) and in-situ (during annealing). For ex-situ scans with longer acquisition time (5s – 20s) we measured a map with PSD detector fully opened. This scans had an off-set 0.45°. We preferred to reach with PSD further from the specular region. For better time resolution in in-situ measurement the acquisition time was reduced to 1s. There was no chance to get a map with good statistics and only specular intensity was measured by closing the vertical slit before PSD to 1mm.
2) Large angle in plane specular diffraction – th + del scan in angular scale up to 48° in th.
3) Off-plane gamma scan covering the area from 2nd to 4th Bragg peaks (approx from 1 ° to 2.2° in th).
Both large angle and gamma scans were measured only ex-situ before and after annealing. The wavelength 1.66 Å was used.

The high temperature vacuum furnace with berylium hemisphere was used for annealing. The turbomolecular pump provided a vacuum about 1.10^{-5} mbar. Some manual corrections of automatic temperature stabilization procedure was useful to get the fastest temperature grow at the begining of annealing. During annealing at the highest temperatures the berylium hemisphere was cooled by fan (temperature of Be hemisphere at 800°C was 140°C).

The experiments are listed in the following table.

sample No	81_4[*+]	81_4[*]	81_4[*]	81_3	81_2	81_5	81_7	52_1
temp. (°C)	500	600	700	700	800	600	750	700
time (min)	35	38	35	120	60	90	75	85

[*] The same chip annealed at three different temperatures

[+] ex-situ measurement only.

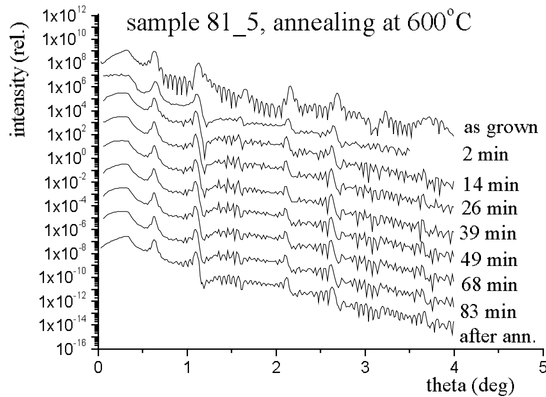

Fig. 1: *In situ specular reflectivity in annealing at 600°C*

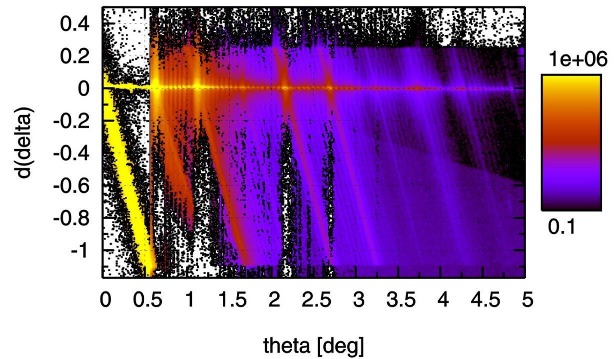

Fig. 2: *Map along specular scan, as grown sample*

In Fig. 1 is the example of the results of in-situ measurement. This figure shows a typical features obtained for all temperatures: Practically the whole structure changes happened before the first in-situ scan was measured (only a few minutes of isothermal annealing) even at the lowest temperature we used. During other prolongation of annealing time the structure remained stabilized. For this reason probably no kinetics of this process could be possible to identify from these measurements. On the other hand the temperature dependence can be observed easily. The higher the temperature, the large structure change occurs, as expected.

By combination of data from specular and diffuse scattering the pure interdiffusion mixing and growth of interface roughness should be possible to separate. The diffuse scattering data shows enormous growth of interface roughness and it is clear, that process cannot be treated as pure interdiffusion through the interfaces. The situation is rather complex and its interpretation is complicated by possible oxidation of Titanium sublayers due to worse quality of vacuum during annealing. The idea about creation of some kind of TiO_x phase is probable due to the significant increase of multilayer period during thermal treatment at all temperatures.

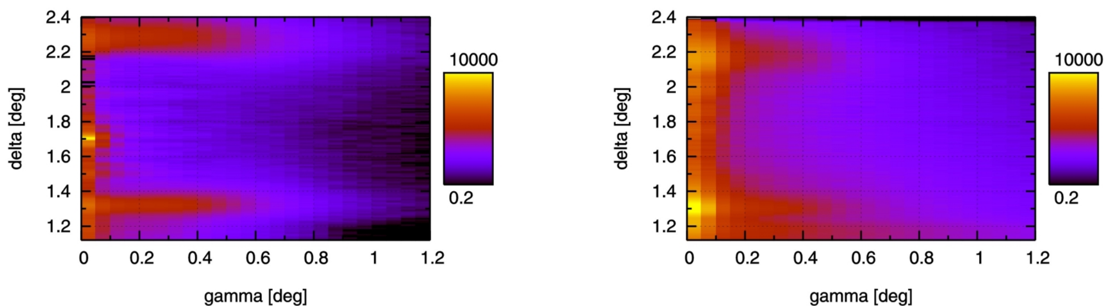

Fig 3: *Gamma scan around 3rd Bragg peak before (left) and after annealing at 750°C (right).*