DUBBLE	**Experiment title:** **Surface structure of Si(111) during wet chemical etching**	**Experiment number**: 26-02-261
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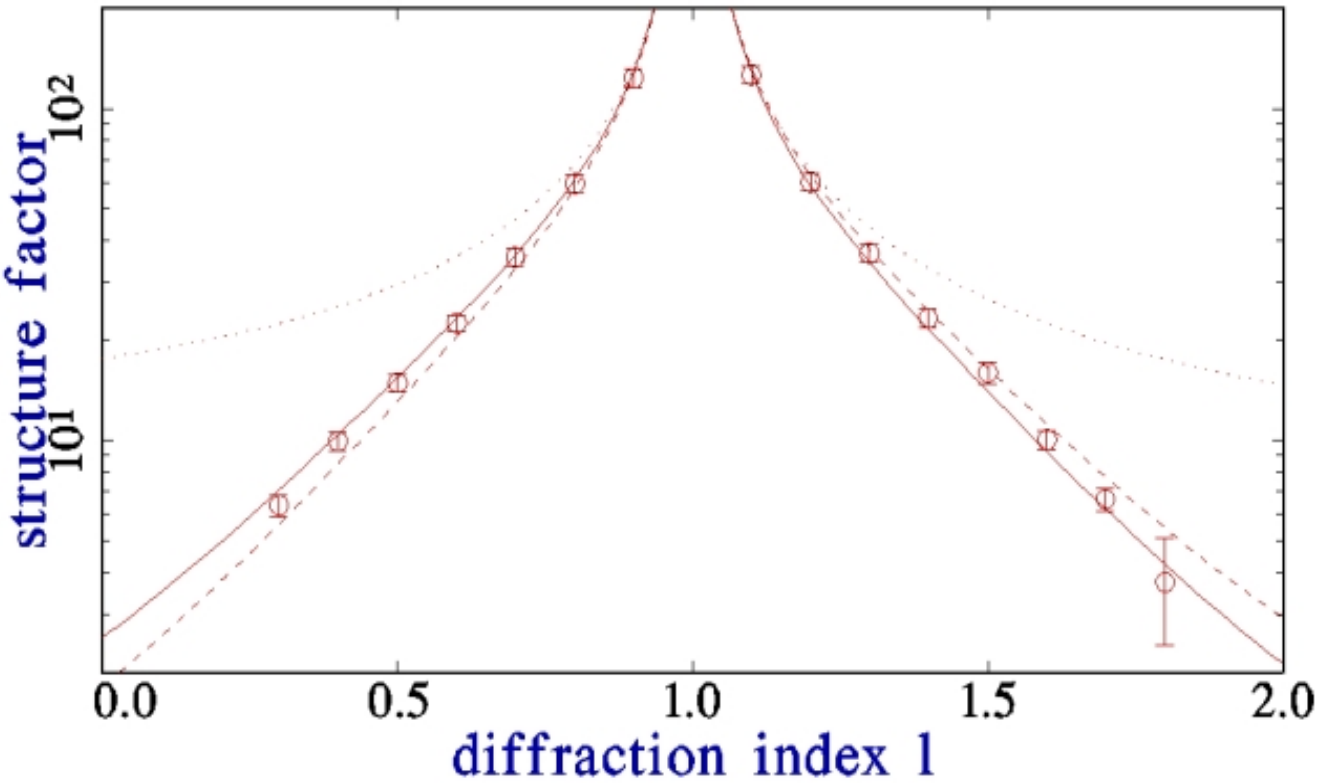
Names and affiliations of applicants (* indicates experimentalists): **D.Nguyen*, H.Philipsen*, I.A.Shah*, B.M.A. van der Wolf* * and E. Vlieg***		

Report: (max. 2 pages)

A well-known anisotropic etching solution for creating Micro Electro-Mechanical Structures (MEMS) in silicon is potassium hydroxide (KOH). Because of its importance, the etching process has been studied by several methods and models have been developed for the various chemical steps involved. The silicon surface is expected to be predominantly hydrogen-terminated in KOH solutions. For the etching reaction to start and continue, the surface has to be partly OH terminated. The aim of the experiment was to check the validity of these models by determining the interface structure and surface termination of Si(111) under etching conditions.

We used a new experimental cell, designed to do *in situ* X-ray investigation of the silicon(111). The cell uses a transmission geometry in order to have optimum etch conditions and was found to work very well. Using an X-ray energy of 20 keV (higher than used before on this branch of DUBBLE), the X-ray absorption is around 50% (the same value as calculated) and thus enough intensity is available to do the experiment. Sample preparation was done in a fume-cupboard of the ESRF Chemical Lab, and always the entire assembly was mounted on the diffractometer. Thus no chemical preparation was done inside the hutch.

In order to obtain good data, the surface roughness has to be kept to a minimum and we tested several etching conditions and samples to find the optimum circumstances. The KOH concentration needs to be kept low, because during etching the surface will eventually roughen kinetically. A quite low concentration of 0.01 M KOH was found to be already too high, and only extremely low concentrations near 10^{-4} M allowed long measuring times. Samples with a special low miscut turned out to give worse results than standard polished wafers, thus miscut is not an important parameter.



The (10) rod of Si(111) during KOH etching.. Circles represent the data. The solid curve is calculated for an OH terminated surface. The dashed curve represents an H-terminated surface. The dotted curve represents the non-roughened (flat) surface.

Even for the most optimum conditions we could find, the surface is still far rougher than surfaces prepared in vacuum. Together with the high background signal from the liquid, this means that it was not possible to measure full rods, only the range near a bulk Bragg peaks was accessible. The figure shows a (10) rod that we measured. Despite the limited range, it is clear that the experimental data agree much better with the calculation for an OH-terminated surface than for a H-terminated one. This is an unexpected result, because it was generally assumed that this surface would be predominantly H-terminated. The full analysis of the data is in progress.

Because the optimization in experimental conditions required many days of beamtime, we did not have time to investigate the influence of the additive isopropanol. In subsequent experiments we will control the electrochemical potential in the system as well. This is not only of interest in order to find the influence of this potential on the surface termination, but it may also help in the preparation of surfaces with minimum roughness.