OPTIMIZING PERFORMANCE OF NEUTRON INTERFEROMETERS

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Abstract: The perfect crystal neutron interferometer is an extremely sensitive device for verifying new concepts in quantum theory, the precise measurement of nuclear constants and non-destructive material analysis. The performance of such interferometers is strongly deteriorated in case of geometric imperfection. We at the Atominstitut of the Austrian Universities prepare the worldwide largest interferometers with lengths up to 21 cm cut from 4 inch monolithic perfect single crystal silicon ingots. The precision requirements are so demanding that we cannot rely on the precision of the cutting machines which is several microns for large crystals. Therefore we have developed an iterative procedure of precise determination and correction of geometric imperfections in the sub-micron range accompanied by numeric simulations of the fringe visibility. We will present improvements in interferometer quality using precise geometric measurements, numerical simulations and selective etching.

1 INTRODUCTION

Interferometers are some of the most sensitive measuring devices in fundamental physics [1,2], condensed matter research [3] and in technology. The first interferometers cut from silicon were produced from Bonse [4] and used for x-rays. The first successfully operating neutron interferometer was manufactured and tested 1974 by Rauch, Treimer and Bonse [5, 6]. The interferometer shown in Fig. 1 is optimized for neutrons with a wavelength of 2.6 Å. The beam intensities in neutron interferometry are rather low therefore we increased the size of the monolithic interferometer crystals continuously in order to utilize larger beam areas, bigger samples and to increase the path length of the neutrons inside the now possible larger samples. Various designs have been realized during the past years, now we have developed a new type of a 45° Mach-Zehnder interferometer (Fig. 1) which enables longer flight path and a better beam separation compared to other geometry. It is actually the world’s largest neutron interferometer with a beam path of 21 cm and a beam separation of 5 cm. The two middle plates are designed only for testing purpose and will be removed later (Fig. 5). The hyperpure and distortion free monolithic perfect silicon crystal ingot was supplied by Wacker Silitronic in Burghausen and selected among their best pieces.

The intensive neutron beams used for these experiments are generated in nuclear reactors or spallation sources. Fig. 2 shows the flight path of the neutrons through the interferometer. The crystal planes are perpendicular to the plate’s and basis’ surface. In

![Image](Figure 1. Worlds largest neutron interferometer.)
each plate some neutrons are reflected at the crystal planes while the rest is transmitted which results in splitting the beam into two beams. The lost neutrons at the mirrors are not shown. The beams are widened because of the reflections inside the plate. After the usable parts of the beam were split in the beam splitter into beams I and II and reflected in the mirrors they recombine in the analyzer. For measurements a sample is put into one beam (I or II) and the phase shifting of the interfering output beams o and h is measured. To achieve maximum sensitivity the path lengths of beam I and II have to be the same within the plates and between the plates. This means that some symmetrical conditions must be met. The thickness of the mirrors and the distances between the plates have to be the same and their surfaces must be parallel. In order to achieve 80% contrast of the interference fringes the cutting tolerances have to be below ±2.5 µm over the full length of 21 cm (Fig. 3).

The exact lattice orientation is determined with a Laue camera or an x-ray diffractometer. For the necessary precise machining of the large silicon ingot for high quality neutron interferometers the cutting machines have to be specially prepared. According to the crystal structure we cut the ingot with a diamond wheel of a surface grinding machine with the highest precision available. The cutting procedure of the interferometer plates lasted 30 hours and had to be performed without a break. Several factors have to be kept in mind to obtain highest quality, i.e., the temperature of the coolant and air must be stable, no vibrations may occur to the cutting machine and the cutting sequence has to be optimized. Fig. 4 shows a flow chart of a modern production process. After cutting we measure the geometry of the newly born interferometer with a coordinate measuring machine (CMM).

We developed a software package [7] which visualizes the geometry in the sub-micron range, performs ray tracing, calculates contrast, animates etching and plots possible improvements if the neutron interferometer’s geometry is not perfect as shown in Fig. 3 where one plate is 15 µm too thick. Etching this plate 15 µm will at first decrease and after 4.25 µm increase the neutron interferometers contrast to 99% at 15 µm. It demonstrates the critical influence of the geometric imperfection of only one plate.

The cutting process destroys the crystal structure on the surface therefore the crystal must be etched about 15 - 25 µm. The etching solution removes more silicon at the borders of the plates which is an additional contribution to the imperfection. With newly developed etching methods we are now
able to correct local geometric imperfections. But the etching process can never be controlled exactly therefore after each etching process the interferometer’s geometry is measured with a CMM and the new data is fed into the software again. If there are correctable imperfections, selective etching, measuring and evaluation is repeated several times.

If the final test with neutron beams at one of our interferometer test facilities at the Atominstitut or at the ILL in Grenoble was successful the interferometer is ready for use in experiments.

The aim of measuring the geometry of neutron interferometers with high precision is to continuously improve the performance of our interferometers. We want to control the cutting process within 1 µm [7], but this is only possible if we find out all systematic errors of the cutting machine, i.e. its systematic positioning errors, systematic guiding errors of the cutting wheel etc. We also want to control the etching process accurately. Learning its behavior with our special interferometer designs is crucial to achieve good geometry. This is done by studying each etching analysis thoroughly. To correct imperfect geometry the software predicts the necessary modifications that have to be done.

2 GEOMETRIC MEASUREMENT AND VISUALIZATION

Each interferometer is measured several times on a CMM (Tab. 1) either at the Institute for Production Engineering (P.H. Osanna) or at the Physikalisch Technische Bundesanstalt (in cooperation with P. Becker). The surface of 80 x 45 mm on each plate’s side is scanned by a matrix of 6 x 6 points. These are 36 points for each side and for our large interferometer with 12 sides 432 measurement points. Each measurement is repeated twice so the number of data points is 1296 (threelfold). The three measurements are interpolated to get most accurate data for the interferometer. The CMM may only use as less force as 0.1 N when touching the elastic plates.

Table 1. Technical specifications of CMMs.

<table>
<thead>
<tr>
<th>Location</th>
<th>Machine Name</th>
<th>Measuring Volume</th>
<th>Resolution</th>
<th>Uncertainty</th>
</tr>
</thead>
<tbody>
<tr>
<td>TU Vienna</td>
<td>Zeiss UMM 500</td>
<td>500x200x300 mm³</td>
<td>0.1 µm</td>
<td></td>
</tr>
<tr>
<td>PTB Braunschweig</td>
<td>Leitz PMM 866</td>
<td>800x600x600 mm³</td>
<td>0.1 µm</td>
<td>U3 = 1 µm + 2×10^{-6} × L</td>
</tr>
</tbody>
</table>

Fig. 6 shows the influence of etching a plate. Fig. 8 demonstrates the same plate but in the section profile showing that the maximum difference of thickness is 7 µm. By studying many etched surfaces we learn about the behavior of etching. We clearly see that the borders of the plates are etched more.
than the central part. This is a thermodynamic transportation effect. The etching solution is warming up during etching and therefore rises. The fresh acid is floating from the borders to the central region. So the etching solution is fresher at the borders.

One of the most important quality features of the neutron interferometer is the utilizable area. Usually each etching session decreases this range. But with our advanced knowledge about the etching process we could turn this usually negative side effect to good account and improved the crystal geometry. Our software package calculates the utilizable area of each plate. The grey area in Fig. 9 shows the usable area after cutting for a thickness tolerance within 3 µm. This is 42% of the total surface. After etching this area increased to 82% (Fig. 10).

![Figure 8. Section profile of Fig. 7.](image)

![Figure 9. 42% of surface is within a thickness tolerance of 3 µm (grey area). White indicates area is too thick, black area is too thin.](image)

![Figure 10. Same plate after etching 12 µm. 82% of surface is within the 3 µm tolerance.](image)

### 3 CONTRAST CALCULATION AND INTERFEROMETER OPTIMIZATION

The contrast or visibility is an objective measure for the quality of an interferometer device. The contrast depends on several factors like crystal quality, geometric errors, vibrations and thermal drifts. Hitherto contrast values up to 90% could be achieved in neutron interferometry. Geometric imperfections are supposed to be the main source for contrast reductions.

Experimental setups consume much time to build up, test and tune them, especially in neutron interferometry where the beam intensities are quite low. Our aim is to help the experimenter to reduce beam time by shifting several testing and preliminary procedures to the personal computer with the actual neutron interferometer geometry. With our software it is easy to find crystal areas with highest contrast and to calculate integral contrast values. The comparison with real measurements gives valuable hints for further improvements of the whole interferometer setup.

The calculation of the contrast is based on the Takagi approach [8 - 11] to the dynamical theory of x-ray diffraction and uses to a great extent the formalism of quantum mechanics in order to reduce the algebraic complexity of the Ewald and von Laue theory [12]. Based on this Mana and Vittone published a handsome formalism [13] which Kuetgens, PTB Braunschweig, implemented in a PC software. We adapted the parameters to the neutron field and developed a complete software solution for the quality control of neutron interferometer productions. This includes high precision geometric measurements, visualization of surfaces, calculation of all relevant sizes like thickness of plates and distances between plates by ray tracing for contrast calculation, utilizable surface areas and animated simulations of geometric influences to the interferometer quality for its improvement.

The contrast calculation with the scanned geometry points out geometry imperfections in the sub-micron range. It’s not practicable to correct these small errors by iterative cutting. Instead we etch the concerned areas selectively because it’s cheaper and less time consuming. To do so we cover the
good surface areas with a thin layer of Picein tar which is an effective shield against the HNO$_3$ / HF acid mixture and which can be removed easily after etching. Fig. 1 shows the Picein vessel before covering the interferometer plates. With only a few mouse clicks we tell the software which surfaces we want to etch. The result is displayed as a three dimensional graph showing the contrast in the vertical axis. Fig. 11 shows the contrast after a basic etching of 15 µm and Fig. 12 after an additional selective etching of 3.25 µm of one surface. The calculated contrast increased from 90.8% to 97.2%. In another case we could raise the contrast of an imperfect neutron interferometer’s experimental contrast from 11% to 43% by only one selective etching process (Tab. 1). With a further iteration an improvement to more than 70% is expected.

Our software is able to simulate the etching process thanks to today’s fast PCs in only a few hours.

![Figure 11. Calculated mean contrast after basic etching: 90%.](image)

![Figure 12. Calculated mean contrast after 3.25 µm selective etching: 97%.](image)

The expected increase of contrast caused by a simulated etching process is displayed visually and can be saved as a standard Windows AVI file for reviewing. You find an animation example on our Atominstitut site [14].

To find the optimal etching depth one can easily change the etching depth in the animation by moving the mouse.

4 COMPARISON OF SOFTWARE BUNDLE AND EXPERIMENT

First we produced an interferometer with a new cutting machine at the PTB Braunschweig. Unfortunately with this CNC controlled machine one positioning error occurred. After a basic etching of 20,1 µm we measured the neutron interferometers geometry and the software bundle calculated a contrast of 11% which was in good correspondence with the experimental contrast of 10%. The contrast animation calculated an optimal mean contrast of 91% after a selective etching of 15 µm (Fig. 2) on one side of an interferometer plate considering the whole surface. As etching cannot be done exactly we were cautious performing it and the resulting etching depth was between 9 and 11 µm. Tab. 2 compares the experimental and the calculated contrast values. The experimental contrast was measured at the Atominstitut which can offer a maximum contrast of 80%. Therefore the experimental contrast is always biased to the lower values. Fig. 13 demonstrates the beam splitter with the positions we used as beam entrance for testing purposes which correlates to Tab. 2.

![Table 2. Experimental / calculated contrast after selective etching.](image)

With except one point at the top border of the plate our measurements meet the calculations within the expected variation [7]. After all we can say that the first proof was successful. More accurate tests are running at the time of printing.
5 CONCLUSION
We examined geometric imperfections of large single crystal neutron interferometers and developed an iterative procedure to reduce them. Systematic and non-systematic errors of cutting are defined better and the understanding of etching is improved. Our procedure includes precise CMM measurements in the sub-micron range, surface visualizations, contrast calculations, etching simulations and finally selective etching for the reduction of geometric deviations. Our simulations show that small corrections can lead to a dramatic gain in interference contrast. First measurements justify our new methods. We now routinely use the software bundle and the new correction methods for neutron interferometer productions. We are already known as a reliable deliverer of high performance silicon devices for neutron experiments. With the new procedure we are now able to produce them with a granted tolerance of 2 µm.

An important and now completely new possibility for the researcher is that he can simulate whole experiments with his individual neutron interferometer because its geometry is known exactly. Future deliveries of neutron interferometers may include the software bundle and all geometric data to run experiments on the PC right from the start.

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