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Germanium Single-Crystal Wafers for Composite Neutron and X-Ray Monochromators

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Plastically deformed germanium single crystal wafers have been produced routinely at the Risø facility for plastic deformation of wafers and used to make composite germanium monochromators since early 1996. The majority of the processed wafers are cut as (511) slabs, but also several (311) and (111) and a few (711) and (155) wafers have been processed and tested. Results of these tests are presented.

KEYWORDS: neutron and X-ray monochromators, germanium composite wafers, large mosaicity, deformation of germanium

§1. Introduction

The Risø facility for plastic deformation of germanium wafers have been in routine operation since early 1996. Until now about 700 wafers have been processed and used to make composite monochromators for neutron scattering instruments at Risø National Laboratory, Denmark, Institute for Energy Technology, Norway, Hahn Meitner Institute, Germany, Oak Ridge National Laboratory, Tennessee and Demokritos Research Centre, Greece. The majority of the processed wafers are cut as (511) slabs, but also several (311) and (111) and a few (711) and (155) wafers have been processed and tested. In a white beam, the Bragg reflected neutrons from the composite monochromators have Gaussian like symmetric line shapes with a full width at half maximum of about 15 to 20 minutes of arc.

§2. Equipment and Process

The development of the Risø facility was inspired by the deformed germanium composite wafer monochromator produced by Axe *et al.*[1] at Brookhaven some years ago. The facility is semi-automatic and requires minimal attention. Figure 1 shows a CAD drawing of the present facility. It consists of a commercial tube furnace with a maximum operating temperature of 1200 °C (marked A). The furnace can radiate heat to the tools used for deformation or flattening of the wafers. These tools are exchangeable and mounted inside evacuated quartz glass enclosures (marked B and C) which fit into the furnace. All movements are done by a robot controlled by a PLC-control system (marked E) which also controls and monitors the temperature. In Fig. 1, one of the quartz tubes with deformation tool (B) is being inserted in the the furnace. The other (C) is stored in the cooled storage box (marked D) while cooling the processed wafer from the operating temperature (870 °C for germanium) to room temperature. The deformation process and the making of the composite monochromators are described in de-

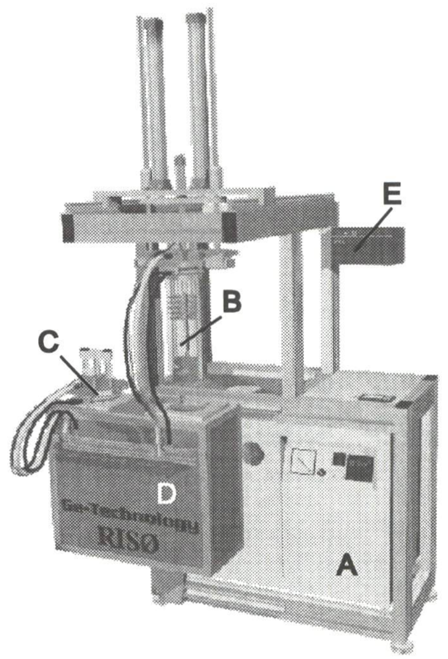

Fig. 1. CAD drawing of the Risø facility for plastic deformation of germanium wafers. See text for details and explanation of the lettering.

tail by by Lebech *et al.*[2,3] and illustrated schematically in the upper right hand part of Fig. 2. The left hand part of Fig. 2 shows a close up photo of the bending tool mounted with a single wafer and ready for insertion into the quartz tube. The hole behind the tool is the entrance to the furnace. A photo of two finished slabs with twenty plastically deformed germaniun wafers in each slab is shown in the lower right hand part of Fig. 2. The slabs are approximately 1 cm thick and may be used as flat neutron monochromators or cut into several strips that may be mounted in a focusing device. A single bend, but not flattened wafer may be used as focusing monochromator for X-rays provided that the bending curvature has been properly chosen.

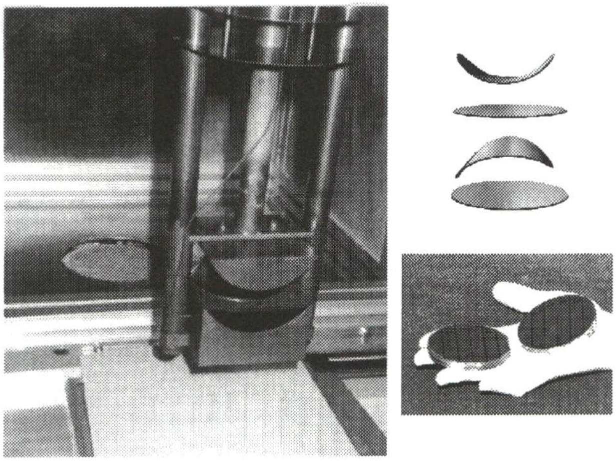

Fig. 2. Left hand: Photo showing the bending tool with one wafer ready for loading into the quartz tube. Right hand, top: Illustration of the deformation process. Right hand, bottom: Two finished slabs with twenty deformed germaniun wafers in each slab.

§3. Experimental Results

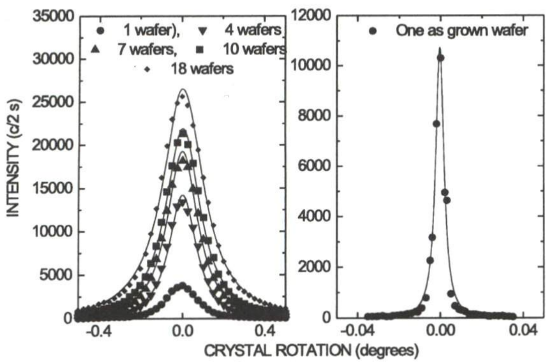

Fig. 3. Left hand: Rocking curves for several stacked plastically deformed (511) germanium wafers. Right hand: Rocking curve for an as grown (511) germanium wafer. The solid curves represent least squares fits to Lorentzian line shapes.

About 400 of the processed wafers have been carefully tested by neutron diffraction before being made into composite slabs suitable as a neutron monochromator. Within the experimental accuracy the test results for the individual wafers are consistant and reproducible. The wafers are 75 mm in diameter and 0.4 mm thick. They were delivered cut with the (511) reflection perpendicular to the wafer plane within an accuracy of 0.05°. Two mutually perpendicular 25 and 15 mm cut flats allowed preparation of composite crystals simply by careful alignment of these faces. As an illustration, Fig. 3 (left hand part) shows the rocking curves obtained when stacking several wafers to form a composite crystal slab. The right hand part of Fig. 3 shows the much narrower rocking curve obtained for an as grown wafer. Figure 4 shows the experimental set-up where we used a perfect Ge(333) monochromator with open incident and exit collimation. This geometry gives perfect focusing[4] for the Ge(511) reflection with extreme resolution as illustrated by the full width at half maximum of 0.23 minutes of arc (see Fig. 3, right hand part) obtained for a single as grown wafer.

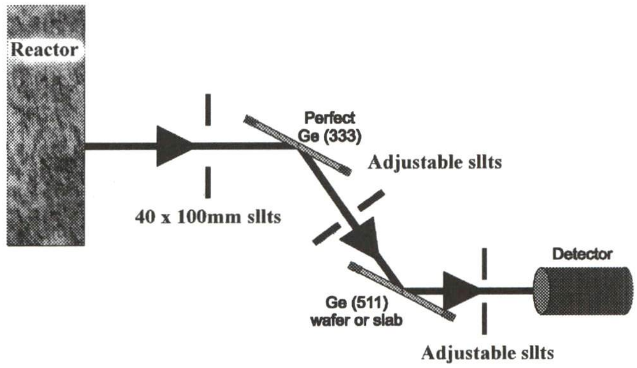

Fig. 4. Experimental set-up used to check the (511) germanium wafers and composite monochromators.

Figure 5 shows the performace of the finished composite monochromator strips. The strips have been cut from five composite slabs of plastically deformed wafers. The composite slabs were made by stacking twenty wafers separated by tin foil and soldering the stack together by heating the stack to about 400 °C while applying pressure. The rocking curves of the twenty strips (four from each composite slab) have been superimposed. The peak profiles fit better to Lorentzian line shapes than Gaussians similarly to the results obtained for the individual wafers. Close inspection of Fig. 5 shows also that the rocking curves for the strips cut from the top and bottom of the composite slabs have a tendency to be narrower and more intense than the rocking curves for the strips cut from the central part of the composit slab.

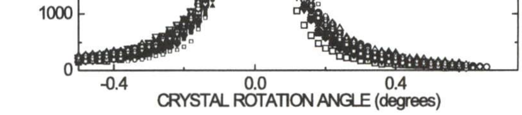

Fig. 5. The superimposed rocking curves of twenty strips cut from five (511) composite germanium monochromator slabs each composed of twenty plastically deformed wafers.

The experimental fact that, when using the set-up shown in Fig. 4, the rocking curves for the individual wafers and the composite monochromator slabs and strips are best described by Lorentzian line shapes is somewhat surprising. Although we can not offer any satisfactorily explanation for this observation, experience shows that the peak shape seems to depend on the surface treatment of the the as grown wafers. Anyway, the peak shapes, measured by placing a detector in the monochromator exit beam and rocking the composite germanium monochomators through the (511) reflection in the white reactor beam, are rather perfect Gaussians. This is exemplified in Fig. 6 which shows the performance of the composite monochromator made at Risø for the Demokritos Research Centre in Greece. The left hand part shows the Gaussian peak profile (solid curve) of the rocking curve obtained when using the un-collimated incident beam produced by the Risø (511) germanium composite monochromator. The right hand part shows the rocking curve obtained for the set-up shown in Fig. 4 using the perfect as grown (333) germanium monochromator. Here the line shape is clearly Lorentzian (solid curve).

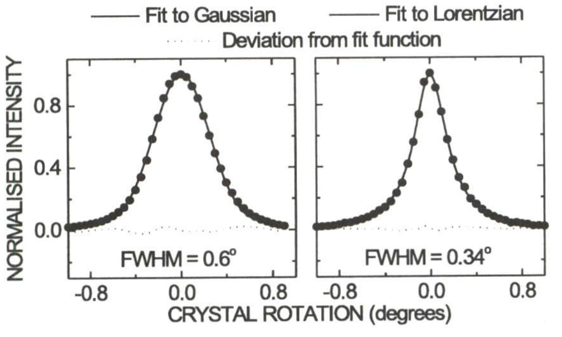

Fig. 6. Result obtained for the composite monochromator delivered to Demokritos Research Centre in Greece. Left hand: Gaussian peak profile obtained when using the un-collimated incident beam produced by the Risø deformed (511) germanium composite monochromator. Right hand: Peak profile obtained when using an extremely well collimated incident neutron beam produced by an naturally grown Ge (333) monochromator with perfect mosaicity.

Figure 7 is a further illustration of the performance of the composite germanium monochromators. Here we compare the performance of two germanium monochromators both having increased mosaicity. The data is determined by placing a detector in the monochromator exit beam and rotating the monochromator crystals through a rather large angle in the white reactor beam. Hereby, Bragg peaks from several reflections are picked up by different wavelengths as illustrated by the section shown in Fig. 7. The lower diffraction pattern is obtained from the a bulk pressed germanium monochromator produced at Risø many years ago.[5] The upper diffraction pattern is obtained from the presently used Risø composite wafer germanium monochromator. The solid curves are the results of least squares fits to the sum of three Gaussians. The composite germanium monochromator gives a considerably higher peak and integrated intensity than the bulk pressed monochromator. In addition the peak shapes are perfect Gaussians for the composite monochromator whereas the peaks shapes for the bulk pressed monochromator are asymmetric and rather poorly fitted by Gaussians.

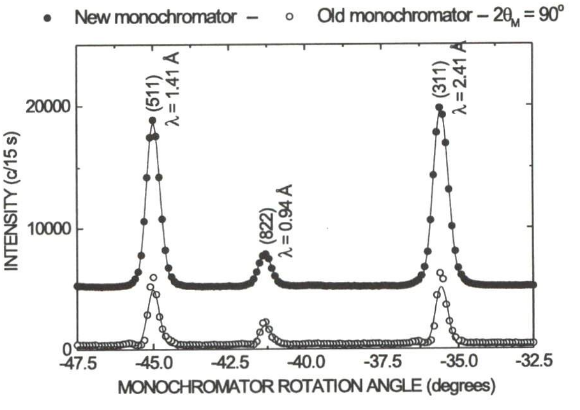

Fig. 7. Comparison between a composite germanium monochromator made from twenty plastically deformed wafers and a bulk pressed germanium monochromator. The solid curves represent least squares fits to the sum of three Gaussians. The scattering angles of the monochromators were 90 °.

§4. Summary

For several years, the Risø facility for plastic deformation of composite germanium wafers have produced high quality monochromator crystals for neutron scattering instrumentation with a large degree of reproducibility. In the past we have mainly concentrated on processing wafers cut with the (511) perpendicular to the wafer. The full width at half maximum of the rocking curves are about 15 to 20 minutes of arc. Tests have shown that the presently used bending radius 47.6 mm for 75 mm diameter wafers is close to the upper limit for breaking the wafer. A few (311), (711) and (155) have also been processed and four (111) wafers have been processed and tested. With the limited number of wafers cut differently than (511) it is difficult to draw definite comclusions, but the results indicate that the full with at half maximum of the rocking curves are about 20 % larger for the (111) wafers than for the (511) while the full with at half maximum of the rocking curves for (311), (711) and (155) are of the same order of magnitude as those obtained for the (511) wafers.

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