Recent polarizing supermirror projects at the ILL

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Abstract. We present a status of recent projects involving the in-house production of neutron multilayer optics, mainly polarizing supermirrors, at the ILL. Our main “mass production” project is for the wide-solid angle analyzing benders for the future instrument WASP (Wide Angle Spin Echo). The current status of this project based on Co/Ti supermirrors, which spans several years, will be presented. Some parameters of polarizing supermirrors for cavity polarizers, mainly based on Fe/Si supermirrors and produced in the past few years for various ILL instruments, are also reported. Some supermirror samples produced in order to study depolarization effects are also mentioned.

1. Introduction
The multilayers were fabricated by magnetron sputtering, on the two large area coating machines run by the ILL multilayer group. Reactive sputtering is used in some cases in order to adjust scattering length densities and interface structures [1]. The first chapter is a progress status of the supermirror production for the WASP analyzers. The second one presents supermirrors produced for several cavity devices, with more focus on the D33 polarizers. The last one is a short report on the type of multilayers produced for the depolarization study.

2. Status of the WASP project
The production of supermirror analyzers for the future ILL spin-echo instrument WASP is in progress. The analyzer design, based on Schärpf-type C-shaped benders and described in [2], is inspired by the design of the D7 analyzers upgrade [3]. We remind a few parameters from the design: the supermirrors are Co/Ti with m=2.8, double-side coated on borated float glass. The coating process and typical supermirror performance are the same as in [3]. The substrate dimensions, 254x141x0.2 mm³, were chosen to fit the instrument geometry and to maximize the number of substrates coated per process run. The mirrors are then mounted into cassettes which hold them with a bending radius of about 7 m. Each cassette, consisting of 37 mirrors, corresponds to a horizontal angle of analysis of 1° from the sample. The full angular analyzer coverage of the designed instrument would be 240°, which corresponds to 1.25 m² of detector coverage at 3 m from the sample, or 636 m² of supermirror to be coated. The production started in 2008, with an annual rate steadily increasing from then on; today enough mirrors have been produced to cover more than 50° (Figure 1). The instrument commissioning is foreseen at the end of 2015, with a target of 90° of analyzer coverage. There will be a deviation from the extrapolation in 2013 because the control system of the coating machine was renewed. This upgrade is expected to improve the process reliability in the following years.
Figure 1: Supermirror analyzers’ production status for WASP represented in terms of detector angular coverage. The number of cassettes produced (or degrees covered) is indicated in parentheses. The extrapolated production for 2013, 2014 and 2015 is the average over the last 3 years.

Figure 2: (a) Photograph of the “old” cassette design. (b) Coordinate measurement on the external surface of the side mirror of a cassette of the type shown in (a) (red circles), and best circle fit (blue solid curve) corresponding to a radius of 8.1m. The z-axis is along the neutron beam path. (c) Photograph of the cassette design under development.

Some measurements were performed with the first mirrors produced, on the SPAN instrument at the BERII reactor in Berlin. The mirrors were mounted into three cassettes, which were used as an analyzer in front of a 2D position sensitive detector. A velocity selector was used to characterize the cassettes over the wavelength range to be used by the WASP instrument. Two π-flippers were used in order to perform polarization corrections. Across the wavelength range covered by the design (0.4 – 1.2nm), the performance of the analyzer block could be estimated from the measurements by correcting from instrumental effects. This gives an analyzing efficiency with a maximum of about 95% at 0.5nm with possibly a slight decrease for shorter and longer wavelengths, and a roughly constant transmission of about 51% of polarized beam. When taking into account the detection area effectively covered by the analyzers, this is within the design specifications and close to the analyzer performance predicted by Monte Carlo simulations. More details concerning the measurement procedure, data analysis and comparison with simulations can be found in [2].

Nevertheless, dimensional metrology revealed that the mechanical design of the cassettes, also inspired from the D7 project, presents some bending non-homogeneity along the mirrors (Figure 2-b).
The best-fit circle has a radius of 8.1m, instead of 7m expected from the design. Still, this radius is low enough to prevent direct line of sight through the 1mm channels between the mirrors, which would allow transmitting some neutrons without analyzing them. From the difference between the measured coordinates and the best-fit circle, an RMS slope error of 4mrad was calculated. This imperfection should affect the transmission performance of the cassettes. This could for instance contribute to the small difference between simulated and measured cassette performance observed in [2].

The design is being revised and a new prototype is being tested (Figure 2-c), in order to reach more uniform bending. The essential change is in the type of mechanical spacers that are inserted between the mirrors before pressing them, in order to define the neutron channels. Vertical aluminum wires placed in the neutron path and perpendicular to it were replaced by horizontal shims placed out of the neutron path and parallel to it. The stack of mirrors and spacers is pressed through continuous contacts along the mirrors edges, instead of using discrete contact points in the old design. A small part of the mirrors height is lost for placing the shims, but this is compensated by the suppression of the absorption by aluminum wires. The characterization of the shape errors resulting from this new mechanical design is in progress.

3. Cavity devices
From 2011, several supermirror coatings were produced in order to fabricate transmission polarizers in cavities (see e.g. [4] for the operating principle) or wavelength filters in cavities.

3.1. Design parameters
The cavities were designed by taking into account the operating wavelength range of each instrument, the m-value of the supermirrors that could be achieved with satisfying quality, the characteristics of the incident beam, and the available space on the instrument. Resulting parameters for these various devices are presented in Table 1. In the case of D33, the polariser design was given by the calculations performed in [5], together with wavelength filters which we also produced. There are two interchangeable Fe/Si polarisers in order to cover the whole wavelength range of the instrument. Similarly, two interchangeable wavelength filters based on non-magnetic Ni/Ti are used to cut the longer wavelengths, with two different cut-off values. Other parameters like the characteristics of the coating on the guide faces, not detailed here, were deduced from [5].

3.2. Fabrication
The devices consist in guide sections with grooves in opposite faces in order to maintain polarising mirrors at a given angle from the incident beam. The guide sections, including the internal guide non-depolarizing coatings when needed, were produced by guide manufacturing companies. We present here the supermirror coatings on silicon substrates that were produced and inserted into the assembled guide sections. In every case, the substrates were single crystal double-side polished silicon substrates, 0.55 mm in thickness, and identical supermirrors were coated on both sides. The supermirrors required for the devices in Table 1 were coated in several coating runs. As an area of 800x190mm² can be coated at once, several substrates were coated simultaneously in each run together with a test glass.
Table 1. Parameters of cavity polarizers or wavelength filters, fabricated for various ILL instruments.

| Instrument and device type | Geometry    | Incidence angle (°) | Supermirror m-value | Operating wavelength range (nm) | Supermirror materials | Device overall Length (mm) |
|---------------------------|-------------|---------------------|---------------------|---------------------------------|----------------------|---------------------------|
| IN11 polariser (2)        | V-shape     | 0.908               | 3.2                 | 0.29 – 1.43                     | Fe/Si                 | 2000                      |
| D22 polariser (3)         | V-shape     | 1.04                | 3.2                 | 0.33 – 1.64                     | Fe/Si                 | 1242                      |
| D33 polariser             | V-shape     | 0.72                | 3.6                 | 0.20 – 1.13                     | Fe/Si                 | 1200                      |
| D33 polariser             | single blade| 1.4                 | 3.6                 | 0.39 – 2.20                     | Fe/Si                 | 1200                      |
| D33 wavelength filter     | V-shape     | 1.32                | 1.2                 | 0 – 1.11                        | Ni/Ti (4)             | 842                       |
| D33 wavelength filter     | single blade| 2.2                 | 1.2                 | 0 – 1.85                        | Ni/Ti (4)             | 842                       |

(1) Assuming a parallel beam incident on mirror, without divergence. For the wavelength filters, wavelengths greater than the indicated positive cutoff are rejected.
(2) Replacement.
(3) Upgrade.
(4) Non-depolarizing supermirror.

3.3. Characterization: the example of mirrors for the D33 polarisers

At least one test glass from each run was measured by polarized neutron reflectometry with our test instrument T3. As an example, Figure 3(a) shows an average over several of these measurements, weighted by the proportion of mirrors from each coating run actually mounted in the D33 V-shaped polarizer. Figure 3(b) shows polarized neutron transmission measured on a double-side coated mirror that was mounted afterwards in the D33 V-shaped polarizer. In both cases the only correction to the data is background subtraction, as the polarizer and flipper efficiency are not known. Thus the measured polarization (“P” in Figure 3) has to be taken as a lower limit.

In order to estimate the cavity polarizer performance with realistic mirror characteristics, the transmission data presented in Figure 3(b) were converted into transmission for both spin states as a function of wavelength. The incident angle is set to the design values of the D33 V-shaped polarizer and single-blade polarizer, and the attenuation in the substrate is included. The results are presented on Figure 4 for both devices, in terms of polarization efficiency, total transmission of a non-polarized beam, and usual figure-of-merit $P^2T$. Again, the polarization has to be taken as a lower limit, due to the previous remark on T3 measurement and to the fact that the applied magnetic field is about 100mT on D33, instead of 60mT on T3. The curves on Figure 4 only characterize the properties of the double-side coated mirrors in transmission at given incidence angles for a perfectly collimated beam. Other effects, like incoming beam divergence and reflections on the guide faces, can be taken into account by performing a ray-tracing simulation with the full geometry, using these data as an input. This work is in progress, by using the approach described in [5], and the results will then be compared with the performances of the complete devices on D33. The measurement of these performances on the instrument is also in progress.
Figure 3: (a) Polarised neutron reflectivity measurements on single-side coatings, averaged over the coating runs made for the D33 V-shaped polariser. (b) Polarised neutron transmission measurement of a double-side coated D33 mirror. The wavelength is 0.75nm, and a field of 60mT was applied along the mirrors hard magnetization axis. See text for details.

Figure 4: Polarisation efficiency $P$ (a), total transmission of a non-polarised beam $T$ and figure-of-merit $P^2T$ (b) deduced from measured transmission of Figure 3(b), in the case of the D33 V-polariser and single polariser, with double-side coated mirrors at respective incidence angles of 1.4° and 0.72°. This would be in the case of a single transmission through the mirror of a perfectly collimated beam.

4. Multilayers for the depolarization study
The new generation of neutron beta decay experiments necessitates a very high polarization of large, cold neutron beams [6]. This puts high requirements on the performance of polarizing supermirrors, which becomes limited by depolarization. Due to this effect, the polarization of a perfectly polarized neutron beam can be decreased when it is reflected by or transmitted through a supermirror coating. A systematic experimental study of parameters influencing such depolarizing effects is in progress. The measurement setup is a test bench developed on PF1B at the ILL, based on $^3$He opaque spin filter cells, which can measure depolarization of the neutron beam down to $10^{-4}$ accuracy. For this study, a series of samples was produced to investigate the effect of several coating parameters. It was based on Co/Ti and Fe/Si material pairs, using the same deposition conditions as for usual supermirror production. The number of individual layers was bounded by 1 and 541 in the case of Co/Ti, or by 1 and 903 in the case of Fe/Si. Single layer coatings were either Co or Fe with thickness ranging from 50 to 200 nm. In most cases the layer thickness sequence was given by a standard supermirror design.
Three samples were produced in order to isolate the effect of the magnetic layer thickness: one was coated according to the thickness sequence of an \( m=2.1 \) Fe/Si supermirror, and two others were coated with a constant Fe thickness of 2 and 10 nm, respectively.

Details about the method and the results of the first experiment can be found in [7], where mostly Co/Ti supermirrors were studied. The main conclusions of this work are that the depolarization increases with the \( m \)-value, and that it can be reduced by increasing the magnetizing field, up to about 1 T. At 0.1 T, the depolarization can be of the order of \( 5 \times 10^{-3} \) or more. The remaining samples, including Fe/Si coatings, “non-supermirror” coatings and supermirrors from other sources, were measured in a second beamtime [8]. Among other results, the measurements performed on the multilayers with a constant Fe thickness indicate a more important depolarization when the Fe layers are thinner and much less magnetic field dependence than for supermirrors. This suggests that the magnetic layer thickness is a critical parameter for depolarization by multilayers.

This method gives interesting insights into the mechanisms of magnetization in supermirrors, which are complex multilayer structures, in an applied magnetic field. This is of interest both for fundamental understanding and for applications in neutron optics. For instance, this study reveals some depolarization mechanisms occurring up to applied magnetic field of 1 T in some cases, much higher than field intensities commonly used to “saturate” supermirror polarizers. The present method then allows quantifying small depolarization levels that probably reveals incomplete saturation, which would be challenging to measure by other techniques like SQUID magnetometry or polarized neutron off-specular scattering, as explained in [9]. An extensive report on the results from the neutron depolarization study can be found in [9], where depolarization dependence on many parameters is reported: applied magnetic field, \( m \)-value, incidence angle, wavelength, reflection/transmission geometry, layer material, etc.

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