A non-depolarizing CuTi neutron supermirror guide for PERC

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Abstract

Neutron guides are used to transport slow neutrons from sources to experiments. Conventional neutron supermirror guides use alternating thin layers based on nickel and titanium. Due to the magnetic properties of nickel, their neutron reflectivity properties are spin-dependent, in particular when exposed to high magnetic fields. Motivated by the requirements of precision experiments on neutron beta decay, we present novel supermirrors based on copper and titanium, which preserve the neutron beam polarization. These show excellent reflectivity and prove to be very stable even when exposed to high temperatures.

\textit{Keywords:} neutron supermirror, neutron guide, cold neutrons, magnetron sputtering

1. Introduction

Neutron guides are long glass channels whose inner surfaces are coated with a reflecting layer for angles of incidence up to a critical angle. They allow to transport cold and thermal neutrons away from a reactor or spallation source to experimental sites as far as 200 m away from the source [1, 2]. Supermirrors, first devised by Mezei and Daugleish [3], consist of a large number of thin double layers of typically Ni/Ti and varying in thickness such that the Bragg condition for neutrons with grazing incidence is fulfilled for a broad band of neutron de Broglie wavelengths. They hence increase the maximum angle of reflection at a given wavelength beyond the critical angle of total reflection of the best element Nickel by some factor \( m \).

Within the neutron decay experiment PERC currently under construction at the MEPHISTO [4] beam line of the Heinz Maier-Leibnitz Zentrum (MLZ), Garching, Germany, an 8 m long guide inside an approximately 12 m long magnet system serves as decay volume [5, 6], with the guide surface largely in direction of the magnetic field. PERC will measure correlation coefficients in neutron 

\[ \lambda_g = \lambda_0 \approx V_{\text{ud}} \]

and to search for physics beyond the Standard Model of particle physics like novel scalar and tensor couplings. For a recent review of the field see [7].

In a guide, neutrons are reflected on the walls due to certain Bragg conditions that are defined by the layer thicknesses and material composition of a multilayer supermirror system. In the PERC experiment, the neutron spin of a highly polarized neutron beam has to be preserved on the level of \( 10^{-4} \) per bounce in order to match the precision requirements, see [5]. Supermirror coatings made of layers of ferromagnetic Ni and layers of paramagnetic Ti can depolarize the polarized neutron beam due to magnetic scattering if the magnetization has a component perpendicular to the scattering vector inside the coating [8]. Nickel with added molybdenum is commonly used for non-depolarising neutral guides [8, 9, 10]. Single layers of NiMo and NiP alloys are used for non-depolarizing guides for ultra-cold neutrons (UCNs), as well as a number of other materials, see [11, 12] and references therein.

In this paper, we present a novel neutron supermirror system made of Cu/Ti layers, the optimization procedure and its properties. A coating made of diamagnetic Cu and weakly paramagnetic Ti should be nearly non-magnetic even in strong magnetic fields of \( \approx 2 \text{T} \) and can hence maintain the neutron polarization on the highest level.

In order to optimize the new supermirrors, we make use of different characterization techniques. We present the experimental results of various Cu/Ti single layers, multilayers of alternating bilayers of constant thickness, and neutron supermirrors produced by pulsed DC and RF magnetron sputtering. We studied the effects of pure nitrogen inside the Ti with X-ray reflection (XRR) and X-ray diffraction (XRD). The thermal stability was evaluated by neutron reflectivity measurements before and after heat...
treatment. The model developed for the roughness growth inside a supermirror structure agrees well with the experimental data. The material composition of the layers was investigated using elastic recoil detection (ERD) [13] at the Q3D instrument [14] of the Maier-Leibnitz Laboratory (MLL), Garching. Depolarization measurements of early supermirrors were performed at the PF1b beam line [15] of the Institut Laue-Langevin (ILL), Grenoble, France, using an opaque test bench with polarized $^3$He spin-filter cells [16, 17].

2. Experimental details

The substrate material has max. dimensions of $200 \times 300 \text{mm}^2$ and the max. capacity of the sputtering machine is $300 \times 1200 \text{mm}^2$. The Cu films were deposited by RF magnetron sputtering with a frequency of 13.56 MHz. The Ti films were deposited using pulsed DC magnetron sputtering with a frequency of 50 kHz and an additional substrate bias voltage. The base pressure of the system was $8 \times 10^{-7} \text{mbar}$ and the pressures during sputtering were $5.4 \times 10^{-3} \text{mbar}$ for Ti and $1.9 \times 10^{-3} \text{mbar}$ for Cu. The sputtering power for both materials was set to 500 W. The distance between substrate and sputtering head was 150 mm. During the Ti sputter process, N$_2$ gas was admixed with the Ar sputter gas.

The samples consisting of TiN$_x$ single layers (with $x$ up to $\approx 30\%$) and Cu/TiN$_x$ multilayers and neutron supermirrors were deposited on float glass. Before coating, the glass mirrors were first cleaned in an ultra-sonic bath using alkaline mucasol cleaner and then processed further using isopropanol. In the sputtering chamber, they were cleaned in-situ using an Ar ion beam. The TiN$_x$ single layers have thicknesses of 32 nm, 165 nm, and 300 nm. The multilayers consist of 40 bilayers of equal thickness of 6.6 nm for TiN$_x$ and 6.9 nm for Cu. The neutron supermirrors consist of 95 bilayers Cu$_x$/TiN$_x$ with varying thickness.

The density, thickness, and surface roughness of each film were determined by XRR measurements in $\Theta/2\Theta$ mode using CuK$_{\alpha}$ radiation (1.54 Å) on a Bruker D8 diffractometer. The lattice parameters were determined by XRD measurements on the same device in $\Theta/2\Theta$ mode and in gracing incidence mode (GIXRD).

The neutron reflectivity (NR) curves of the multilayers and supermirrors were measured at the instrument TREFF at FRM II [18]. The NR investigations were performed at a fixed neutron wavelength of 4.8 Å in $\Theta/2\Theta$ mode. The resistance to heat treatment of the neutron supermirrors was also verified by NR at TREFF.

3. Results and discussion

3.1. Effects of impurities on single- and multilayers

A previous attempt at a Cu/Ti based supermirror with 20 bilayers is documented in Ref. [19]. The quality of the data did not allow conclusions on the effect of the high mobility of Cu, which we find to be significant as shown below. The TiN$_x$ layers can form an effective interdiffusion barrier [20, 21, 22, 23]. Using air as reactive sputtering gas as is commonly done for Ni layers [24] could lead to ferromagnetic clusters caused by copper oxides [25]. We consequently investigated the effects of a pure nitrogen admixture on the microstructure of the Ti layers by using XRD on samples with a nominal thickness of 300 nm deposited on float glass.

The addition of nitrogen to the Ar sputtering gas leads to a dilatation of the Ti lattice. The pure Ti target material shows two preferential planes, namely [002] and [101]. The Ti layer grown under pure Ar atmosphere shows the [002] plane, which gradually changes to the [111] plane of the TiN compound [26] with increasing nitrogen content. The [101] plane of pure Ti disappears. Generally, the reactively deposited Ti layers tend to grow more in amorphous structure. Fig. 1 shows the results of the XRD measurements on samples with different admixtures of N$_2$.

We expect that the observed change in crystal structure and the nitrogen inside the Ti limit the interdiffusion for the multilayer structure and reduce roughness growth from layer to layer. As a downside, an increasing N$_2$ concentration also lowers the neutron optical contrast between the layers.

Fig. 2 shows three NR measurements of multilayers deposited with different nitrogen admixtures. As the mirrors each consist of 40 bilayers with equal layer thicknesses, we
obtain constructive interference at a particular Bragg angle of reflection, as well as total reflection from the top layer below a critical angle.

The interpretation of such NR measurements is non-trivial. The roughness of the layers and interdiffusion of the different layer materials have very similar impact on the neutron reflectivity. It is hence not possible to distinguish between them from NR data. The high neutron reflectivity and the smallest peak width of the mirror sputtered with 2 sccm N$_2$ indicate that both, interlayer roughness and interdiffusion, are the lowest for this sample. With lower N$_2$ concentration, the peak width increases and the peak position shifts to larger angle of reflection. If the interface roughness increases, the proportion of diffuse scattered neutrons increases, which broadens the reflected peak. Assuming that two layers diffuse into one another, the effective thickness of the layers ‘seen’ by the incoming neutrons gets smaller. The thinner the layers are, the larger is the angle of Bragg reflection.

3.2. Single surface roughness

Based on the above results, we conclude that sputtering Ti reactively with 2 sccm N$_2$ would be the most promising option to obtain a Cu/TiN$_x$ neutron supermirror with highest possible reflectivity. We investigate the influence of a substrate bias voltage on the surface roughness of single TiN$_x$ layers. Fig. 3 shows the roughness of six TiN$_x$ single layers deposited on float glass with a thickness of 32 nm each, as calculated with our model, see section 3.3.

The surface roughness shows a clear dependency of the substrate bias voltage, which can be explained as follows: By applying a negative bias voltage to the substrate, some fraction of the ionized Ar atoms are accelerated onto the substrate surface. The surface roughness of the layer is the smallest between $-115$ V and $-150$ V, when the ions have enough energy to remove loosely bound atoms to smooth the surface. This can be interpreted as a nanopolishing procedure. For a bias voltage between $-100$ V and 0 V, the surface polishing effect decreases with the smaller energy of the Ar ions. For a voltage below $-150$ V, the ions have enough energy to kick out Ti atoms from the deposited layers. This effect is called re-sputtering and causes the layer roughness to increase [27].

The effect of applying a substrate bias voltage of $-150$ V is confirmed by the XRR measurement results shown in Fig. 4. A 165 nm thick TiN$_x$ layer, deposited without bias voltage is compared to a 167 nm layer deposited with additional bias voltage of $-150$ V. For the sample deposited without bias voltage, the green curve in the figure, which is calculated according to our model, indicates a surface roughness of $\approx 6.5$ nm. The difference between the green theoretical curve and the measurement is presumably coming from an $\approx 7$ nm thick TiN$_x$ interlayer formed by oxidation in the near surface region. For the sample sputtered with bias voltage, a surface roughness of 2.15(3) nm was determined (red curve). Taking the roughness of the float glass substrate of 0.6 nm into account, and assuming a linear growth of roughness with thickness, this corresponds to a roughness growth rate of only 0.09 Å nm$^{-1}$.

3.3. Neutron supermirror

Taking into account our investigations on single- and multilayers, as well as improving the sputtering conditions, we produced Cu/TiN$_x$ neutron supermirrors with a high reflectivity of $> 90 \%$ at the critical angle of reflection in the best case. For the sample shown in Fig. 5, this reflectivity is 92%.

The layer sequence for the mirrors was calculated by the following method: We approximate the layer sequence we obtain according to Ref. [3] for each material separately by a 5 point interpolation function. Then we calculate the reflectivity including our roughness growth model and optimize the reflectivity by varying the supporting points of
Figure 4: XRR measurement of 165 nm thick TiN$_x$ layers deposited with a N$_2$ gas flow of 2 sccm and with or without substrate bias voltage. The red curve shows the fit result, with a surface roughness of only 2.15(3) nm. The green curve is not a fit, but a calculation assuming a roughness of $\approx 6.5$ nm.

Figure 5: Neutron reflectivity measurement of a Cu/TiN$_x$ $m = 2$ supermirror with reflectivity of above 90%. The red curve is a fit using our roughness growth model in Eq. (1). The reduced $\chi^2$-value of the fit is 6.8.

Figure 6: NR measurement of a Cu/TiN$_x$ $m = 2$ supermirror consisting of 95 bilayers with a reflectivity of $R = 85\%$ before and after baking for 48 h at 100$^\circ$C. The green curve is not a fit, but a calculation assuming a roughness of $\approx 6.5$ nm.

Figure 7: NR measurement of an early Cu/TiN$_x$ $m = 1.95$ supermirror after subsequent baking for 12 h at 80 to 300$^\circ$C. Only after baking at 300$^\circ$C the reflectivity decreases significantly. The dip in reflectivity is due to a missing top-layer. Measurement points are connected by lines to help distinguish the data sets.

3.4. Temperature stability

Stability of the mirrors at elevated temperatures is important for two reasons. First, it indicates that the interdiffusion is indeed reduced by the addition of N to the Ti layers. Additionally, the guide will be located in the $\approx 12$ m long warm bore of the PERC magnet. To improve the vacuum conditions there, the bore can be backed at 100$^\circ$C, see Ref. [6]. But this procedure must not degrade the neutron guide.

In Fig. 6 we present the NR measurement of one mirror with slightly lower reflectivity, which was used for thermal stability evaluation. The mirrors were deposited on standard float glass. The supermirror coating consists of 95 Cu/TiN$_x$ bilayers with varying thickness. The mirror shows an excellent temperature stability. Still after baking for 48 h at a temperature of 100 $^\circ$C, the neutron reflectivity remains practically unchanged.

Tests at higher temperatures were performed with an earlier sample. This was produced using low-purity targets and unfortunately suffered from a production error, a missing top-layer. The overall performance of this early sample is still worse than the recent result shown in Fig. 5. The sample was subsequently heated for 12 h at 80, 100, 150, 200 and 300$^\circ$C, and the neutron reflectivity was measured after each treatment. The results in Fig. 7 show that only baking at 300$^\circ$C has a significant
effect.

3.5. Roughness growth model

To describe the roughness growth inside the Cu/TiN$_x$ supermirror stack, we need to account for the effect of interdiffusion. The new model was expanded and integrated into an existing fit routine used for the description of Ni/Ti supermirrors. The roughness of the $i$-th layer is given by the roughness of the substrate $r_{\text{sub}}$, a constant (effective) roughness due to interdiffusion $r_{\text{diff}}$ and a term which describes the roughness accretion $r$ with thickness:

$$r_i = r_{\text{sub}} + r_{\text{diff}} + r \cdot \left(\frac{d_i}{d_{\text{tot},i}}\right)^p,$$

where $d_i$ and $d_{\text{tot},i}$ are the thicknesses of the $i$-th layer and all layers below, respectively, and $p$ is the power of the roughness growth. We include roughness in our reflectivity calculation by folding the step function of the neutron optical potential at the layer borders with a Gaussian function where the standard deviation is given by the roughness except for $r_{\text{diff}}$. Here, the Gaussian function is replaced by a double-sided decaying exponential which leads to a better approximation of a diffusion profile.

$$r_{\text{sub}} \text{[nm]} = 0.80(2)$$
$$r \text{[nm]} = 2.41(9)$$
$$r_{\text{diff}} \text{[nm]} = 0.79(2)$$
$$p = 1.41(6)$$

| red. $\chi^2$ of the fit | 6.8 |

Table 2: Roughness parameters from a fit to the reflectivity data of the Cu/TiN$_x$ ($m = 2$, 190 layers) neutron supermirror shown in Fig. 5 as parametrized by Eq. (1).

For sputtering without ion plating, the deposited layer follows purely statistical roughness growth with $p = 0.5$, similar to snow flakes accumulating on smooth surfaces. For our Cu/TiN$_x$ supermirrors produced with a bias voltage of $-150$ V we obtain $p > 1$, as the surface is polished in-situ by the incoming accelerated particles. The resulting roughness growth parameters for the red curve shown in Fig. 5 are listed in Tab. 2.

For a total thickness of the supermirror coating of $\approx 2$ µm, the surface roughness ($r_{\text{sub}} + r_{\text{diff}} + r$) of the supermirror is only 4 nm. We observe the parameter $p > 1$, which indicates that the roughness growth is small in the beginning and increasing with the overall coating thickness. This effect can be explained by the small surface roughness of the substrate, which helps to keep the roughness low from the beginning and as well as by the fact, that the first layers have only a thickness of a few nanometers, where amorphous growth is favored and crystal growth suppressed. This roughness model might also prove helpful for the understanding of conventional supermirrors with higher $m$-values.

3.6. Magnetic properties

The presence of magnetic roughness in layers, as is typical for ferromagnetic materials, causes depolarization of neutrons. We investigate early mirrors produced using a less pure Ti target (99.5%), which is known to contain traces of iron due to the production process. At the PF1b beamline of the ILL, we characterise these samples with $m$-values up to 1.2 with an in-plane magnetic field of up to 0.82 T using an opaque test bench with two $^3$He neutron spin filter cells. The setup is described in Fig. 4 of Ref. [17], where it was used to characterize neutron polarizers. The measurement technique is sensitive to polarization and depolarization effects below the $10^{-4}$ level. With these early mirror samples, we find depolarization of $6(4) \times 10^{-5}$. More details on this experiment can be found in [28].

We note that Ref. [11] finds depolarisation at the level of a few times $10^{-5}$ per bounce for UCNs on single NiMo and Cu layers, but the application to our case with cold neutrons and high magnetic fields is not straightforward.

Given the demanding (de)polarization criteria of the neutron decay measurement with PERC and its high magnetic field of 1.5 T, and the in total thicker layer sequence of the envisaged $m = 2$ supermirror, we switch to highly pure Ti targets for further development. The material composition of the layer sequence was investigated using elastic recoil detection (ERD) using an 170 MeV $^{127}$I ion beam at the Q3D instrument at the MLL. The impurities Na, Mg and Fe, which were found in the Ti Target with 99.6% purity, can be explained by the Kroll production process [29] and are consistent with the material’s technical data sheet. In the Ti targets with 99.999% purity, no Fe impurity was detectable, see Fig. 8. Such targets are typically produced by the Van-Arkel-de-Boer process [30]. Validation measurements with the opaque test bench using the new $m = 2$ mirrors are planned.

4. Conclusion

We demonstrate that the addition of N to the Ti layers results in an effective interdiffusion barrier for multilayer
structures of Cu/TiN$_x$. X-ray diffraction measurements indicate a dilatation of the Ti lattice and a reorientation of the Ti grains from preferential [002] to [111] with increasing nitrogen content. The addition of nitrogen also increases the long-term and temperature stability of the multilayers. X-ray reflectivity measurements show very smooth layer interfaces for layers produced with ion-plating sputtering technology.

Neutron reflectivity investigations confirm that $m = 2$ Cu/TiN$_x$ non-depolarizing neutron supermirrors with a reflectivity above 90% can be produced. Compared to Ni/Ti mirrors, the reflectivity at the maximum angle is lower by a few percent only. Background due to neutron absorption in PERC will hence be as low as envisaged in Ref. [5]. The excellent thermal stability will enable baking-out the neutron guide inside the warm bore of the PERC magnet [6] to obtain very good high vacuum conditions. Our new model for roughness growth of the supermirror layers enables an excellent description of the neutron reflectivity measurements.

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