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Magnetic anisotropy of thin sputtered MgB2 films on MgO substrates in high magnetic fields

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We investigated the magnetic anisotropy ratio of thin sputtered polycrystalline MgB2 films on MgO substrates. Using high magnetic field measurements, we estimated an anisotropy ratio of 1.35 for T = 0 K with an upper critical field of 31.74 T in the parallel case and 23.5 T in the perpendicular case. Direct measurements of a magnetic-field sweep at 4.2 K show a linear behavior, confirmed by a linear fit for magnetic fields perpendicular to the film plane. Furthermore, we observed a change of up to 12% of the anisotropy ratio in dependence of the film thickness. © 2014 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution 3.0 Unported License. [http://dx.doi.org/10.1063/1.4869291]

One possible application for superconductors is the use as a spin detector in ferromagnet/insulator/superconductor tunneling junctions. The so-called Meservey-Tedrow method (M-T method) offers the possibility to measure the spin polarization of the tunneling electrons due to the different Zeeman energies of the spin up and spin down electrons.1,2 In particular, the superconductor requires a moderate transition temperature, a large penetration depth, and a composition of light elements. The moderate transition temperature leads to an energy gap larger than the Zeeman energy in magnetic fields of approximately 3 T. The large penetration depth allows the magnetic field to enter the superconductor homogeneously, while the magnetic field is aligned in parallel to magnetization of the ferromagnet, i.e. in the film plane. The spin orbit scattering of the quasi particles scales with the fourth power of the atomic number. Until now, aluminum has been used as the superconducting spin detector, because it fulfills all the described requirements. In particular, there are few compounds consisting of only lighter elements (compared to Al).

Consequently, Magnesium diboride (MgB2) is an attractive candidate for M-T tunneling experiments, because both constituents are lighter than aluminum. Furthermore, its larger penetration depth (λAl = 15.7 nm, λMgB2 > 30 nm)3,4) would allow a measurement of perpendicularly magnetized materials,5,6 if the junctions structures are defined well below 50 nm. On top of that, MgB2 has been a topic of interest for technical applications ever since the discovery of its superconductivity more than 10 years ago.7,8

In pursuit of a suitable measurement method for these materials, we investigated the magnetic anisotropy and tunneling properties of thin MgB2 films sputtered onto cubic (001) MgO substrates. Beside the high quality films made by HPCVD,9 we chose the sputtering technique due to a better compatibility to our established M-T devices.10 In addition, MgO is the most commonly used substrate for, e.g., half metallic Heusler compounds.11,12 Unfortunately, sputtered MgB2 films are

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MgB$_2$ crystals are perpendicularly oriented on the surface plane independent of the choice of substrate. Using the Scherrer formula, a perpendicular grain size of 7 nm could be estimated. However, despite the clear c-axis MgB$_2$ (0002) peak, it was not clear whether most of the grains were regularly or randomly oriented. Therefore, it was necessary to conduct field-cooling experiments that were dependent on the orientation of the applied magnetic field.

Figure 1 shows the field-cooling transport measurements for a 60 nm thick MgB$_2$ sample with a magnetic field applied parallel to the film plane, and Figure 2 shows the curves corresponding to a magnetic field applied perpendicular to the film plane. The critical temperature was defined by the 90% criteria at the normalized resistance at 40 K. For magnetic fields up to 16 T for the perpendicular case and up to 18 T for the parallel case, a residual resistivity was observed at 4.2 K because of an increase of the transition width dependent on the applied magnetic field. However, because of the strong decrease in the resistance within a small temperature interval, we assume that the samples are approaching the phase transition.

Figure 3 shows the approximate dependence of the upper critical field $H_{c2}$ on the normalized transition temperature. These data points could be well described by a linear fit. Usually, the upper critical field can be estimated using the Ginzburg-Landau relation for a superconductor by assuming the dirty-limit extrapolation.

$$H_{c2} = -0.69T_c \frac{dH_{c2}(T)}{dT} |_{T_c}$$
FIG. 1. Transition curves of 60 nm MgB$_2$ films with an applied magnetic field parallel to the film plane.

FIG. 2. Transition curves of 60 nm MgB$_2$ films with an applied magnetic field perpendicular to the film plane.

FIG. 3. The estimated upper critical field at 0 K. Using the 90% criteria. The line is a linear fit to the data. Due to the constant slope the anisotropy ratios is still constant.

The upper critical field $H_{c2}(0)$ for the $a$-$b$-axis direction is estimated to be 22.18 T, and the $H_{c2}(0)$ in the $c$-axis direction is 16.37 T. These values imply an anisotropy factor $\gamma$ of 1.35 at 0 K.

Using Ginzburg-Landau theory and these critical fields, the coherence length at 0 K can be estimated from a magnetic field applied parallel to the $c$ axis with

$$H_{c2}^c = \frac{\phi_0}{2\pi \xi_{ab}^2}$$
and a magnetic field applied parallel to the a-b plane with

$$H_{C2}^{cb} = \frac{\phi_0}{2\pi \xi_{ab} \xi_c}$$

where $\phi_0 = 2.07 \times 10^{-15}$ Vs is the magnetic flux quantum. By using these formulas, the coherence lengths can be estimated to be $\xi_{ab} = 4.48$ nm and, for the perpendicular c axis, $\xi_c = 3.31$ nm.

However, these values are underestimated, as has previously been shown by Gurevich for 400 nm films. Furthermore, the behavior of the curvature of the upper critical fields differs for single crystals in the clean limit and polycrystalline samples in the dirty limit. Lyard et al. have observed a positive curvature close to $T_c$, which becomes negative with a saturation at higher fields for an upper critical field applied perpendicular to the a-b plane of a single crystal with dimensions of $50 \times 50 \times 10$ $\mu$m$^3$. Theoretical calculations by Dahm et al. describe this behavior for single crystals.

Due to the polycrystalline structure and therefore the many scattering centers of our film, this saturation could not be observed by our measurements. Therefore, we fit our measured points linearly and compared the results to a magnetic sweep measurement.

A linear extrapolation for both fields resulted in 31.74 T for the parallel case and 23.5 T for the perpendicular case at 0 K. With these values, the anisotropy ratio was again found to be 1.35. Although there are different approaches to estimating the upper critical fields, the calculated anisotropy ratios are the same because of the linear fit. Despite the agreement of the anisotropy factor, the estimated upper critical fields are strongly different. However, as a consequence of the fact that both slopes of the fit curves are constant, the anisotropy factor of 1.35 is also constant over the entire critical temperature range. Only in the region of small applied fields should the anisotropy factor increase.

To confirm this value of the anisotropy ratio estimated above, measurements were performed for a sweep of the magnetic field at a constant temperature of 4.2 K, as shown in Figure 4. For the linear case, the critical fields of the fitted slope should agree very well with the measured sweep. This type of measurement has the advantage that the phase transition, and therefore the anisotropy ratio $\gamma$, can be directly observed as a function of the applied magnetic field. The disadvantage is that the values are limited by the constant temperature of 4.2 K.

In the non-linear case, a strong deviation from the linear extrapolation should be observed. By using the 90% criteria, the critical field in the perpendicular case was found to be 19.48 T, which represents a deviation of less than 1% from the estimated field of 19.6 T at 4.2 K. However, the deviation in the parallel case was found to be 26.28 T, which represents a deviation of 3 T compared to the linear approximation. Here, the linear extrapolation is clearly overestimated at lower temperatures, so saturation could be expected in the parallel case for temperatures near 0 K.
as suggested by Gurevich. Therefore, a decrease in $\gamma$ is common for temperatures below 4.2 K and samples in the dirty limit.

The curves of the sweeps exhibit a transition width of approximately 5 T, which indicates an apparent retardation of the superconductivity on several grains as a function of the magnetic field. For clean single crystals, a small upper critical field of $\approx 4$ T for an applied field perpendicular to the film plane and $\approx 15$ T for an applied field parallel to the film plane is expected, which would result in an anisotropy ratio of up to 3.75 at 0 K. Any impurity results in an increase of the upper critical fields in both directions, which has previously been observed by Gurevich et al. on polycrystalline MgB$_2$ samples.$^{16}$

However, the constant distance of the sweep curves between the parallel and perpendicular magnetic fields with respect to the resistivity demonstrates that the grains are distributed homogeneously, i.e., the anisotropy ratio is nearly constant for the grains, independent of the transition temperature.

Only at approximately 10% of the resistance, the slope of the curve corresponds to the field perpendicular to the film plane becomes stronger than in the parallel case. This behavior could be caused by different crystal sizes, which was also observed by Lyard et al.$^{19}$

Table I shows how the anisotropy factor at 4.2 K depends on the film thickness. A decrease in the anisotropy factor of up to 12% was observed. The decrease of the anisotropy factor can be explained by an increase of the crystal size. Handstein et al. observed an anisotropy ratio of 1.1 for imperfect hot deformed bulk samples, and an anisotropy factor of 1.8 to 2 was observed by Patnaik et al. for c-axis-oriented thin films.$^{17, 21}$ However, an anisotropy factor of up to 9 was observed by Simon et al. for randomly oriented powder samples.$^{22}$ Our values are comparable to the values of Jung, who predicted an anisotropy factor of 1.25 for thin films.$^{15}$

In summary, we fabricated sputtered polycrystalline MgB$_2$ films in which most of the grains are c-axis orientated. The anisotropy ratio was nearly constant in the investigated temperature and field range, which was determined using a linear extrapolation as well as the Ginzburg Landau theory (at 0 K). Overall, the thin films in the thickness regime between 30 nm and 120 nm are an ideal starting point for further experiments utilizing Meservey-Tedrow tunneling. Textured films enable us to tunnel preferably in the $\pi$ as well as the $\sigma$-band of the two-band superconductor MgB$_2$.

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| film thickness (nm) | 30  | 60  | 120 |
|---------------------|-----|-----|-----|
| anisotropy ratio $\gamma$ | 1.32 | 1.21 | 1.15 |

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