Microwave properties of MgB$_2$ thin films prepared *in situ* by thermal evaporation combined with sputtering

A.G. Zaitsev, R. Schneider, R. Hott, F. Ratzel, G. Linker and J. Geerk

Forschungszentrum Karlsruhe, Institut für Festkörperphysik, P.O. Box 3640, D-76021 Karlsruhe, Germany

E-mail: alexander.zaitsev@ifp.fzk.de

Abstract. Superconducting MgB$_2$ thin films were prepared *in situ* using a combination of rf magnetron sputtering of B and thermal evaporation of Mg. The films exhibited $T_c$ of up to 36 K. The microwave measurements were performed on 14×14 mm$^2$ films using both Cu-shielded and Nb-shielded sapphire puck resonators at the frequency of 18.8 GHz. The hf surface resistance ($R_s$) and the change of the hf surface reactance ($\Delta X_s$) were determined. The films exhibited low $R_s$ matching the literature results for high-quality MgB$_2$ films. Below 3K $R_s$ reached 3-5 $\mu\Omega$ which was the resolution limit of our measurement. The temperature dependences of both $R_s$ and $\Delta X_s$ were in good agreement with BCS theory. From the $R_s(T)$ dependence we obtained an energy gap $\Delta(0) = 3$ meV. The measured variation of the London penetration depth with temperature, $\Delta\lambda_L(T)$, was also in good agreement with the BCS model. Using the BCS relation between the energy gap and the penetration depth we fitted our experimental $\Delta\lambda_L(T)$ data and obtained $\lambda_L(0)$ values, which ranged for different films from 85 to 100 nm.

1. Introduction

Microwave measurements on MgB$_2$ films continue to be of interest from both fundamental and technical viewpoints. On one hand, they allow an accurate estimation of several physical quantities [1,2]. Especially, due to the BCS-like behaviour of MgB$_2$ the superconducting gap $\Delta(0)$ and the absolute value of the London penetration depth $\lambda_L(0)$ can be evaluated. On the other hand, it has been shown recently [3], that at temperatures below 4.2 K high-quality *in situ* MgB$_2$ films may exhibit lower microwave surface resistance ($R_s$) than the high-temperature superconducting films and even Nb films. Using the parallel-plate resonator measurements and subtracting the external losses, $R_s$ of the MgB$_2$ films was estimated to be smaller than 1 $\mu\Omega$ at 10 GHz below 3 K.

So far high-quality *in situ* MgB$_2$ films were obtained by hybrid physical-chemical vapour deposition process [4], by co-sputtering of Mg and B targets [1,5] and by co-evaporation of Mg and B [3,6]. Recently we reported the preparation of *in situ* MgB$_2$ films by thermal evaporation combined with sputtering [7]. In the present contribution we report on the microwave properties of these films.

2. Experimental

Superconducting MgB$_2$ thin films were prepared *in situ* in a ‘one-step’ process using a combination of rf magnetron sputtering of a planar B target and thermal evaporation of Mg [7]. The films exhibited
critical temperatures of up to 36 K and critical current densities of more than 10 MA/cm$^2$ at temperatures below 20 K. The normal-state resistivity above $T_c$, $\rho(40K)$, ranged from 50 to 100 $\mu\Omega$cm. For the hf measurements 250-300 nm thick films on 14$\times$14 mm$^2$ large $r$-cut sapphire substrates were used. The measurements were performed using a sapphire puck resonator at a frequency $f_0 = 18.9$ GHz in the TE$_{011}$ mode. Two resonator shieldings of identical geometry were made of copper and of niobium. The surface of the Nb shielding was cleaned by etching in a HF:HNO$_3$:H$_3$PO$_4$ mixture and annealing by an electron beam for 4 hours at temperature of $\sim 1200^\circ$C in vacuum of $\sim 10^{-7}$ mbar. The microwave measurements were performed in a He bath cryostat evacuated by a roots pump in order to reach temperatures down to 2 K. The microwave surface resistance ($R_s$) and the change of the microwave surface reactance ($\Delta X_s$) of the MgB$_2$ films were determined as a function of temperature using the temperature dependences of the unloaded quality factor and the resonant frequency, respectively.

3. Results and Discussion

The examination of the hf losses in the MgB$_2$ films was started by measuring their temperature dependences by the Cu-shielded resonator, which is suitable for temperatures downwards from $T_c$. Of advantage are also the reproducible hf losses in Cu, which can be straightforwardly calibrated and subtracted from the measured losses. The examined films exhibited low $R_s$ which match the literature results for high-quality MgB$_2$ films [1-3]. In particular, $R_s(20K) = 0.9 - 1$ m$\Omega$ and $R_s(10K) = 0.1 - 0.2$ m$\Omega$ were typically reached. The measurements below 10 K turned to be insufficiently accurate, since the small losses in MgB$_2$ could not be resolved from the large losses in Cu.

Both $R_s$ and $\Delta X_s$ of the MgB$_2$ films revealed the temperature dependences compatible with the predictions of the BCS theory [8]. In particular, $R_s$ decreased exponentially with temperature, as shown in fig. 1. According to the theory an exponential fit of the low-temperature part of the $R_s(T)$ curve provides the estimation of the energy gap as $R_s(T) = \exp(-\Delta(0)/k_B T)$. For the data of fig. 1 the best fit was obtained for $\Delta(0) = 3$ meV with the accuracy of $\pm 10\%$. This $\Delta(0)$ value corresponds to the upper limit of the theoretical predictions for the small $\pi$-gap of the two-gap energy band structure of MgB$_2$ [1,2,9]. No direct indication of the large $\sigma$-gap with $\Delta(0) = 7$ meV was observed. These results are in good agreement with our tunnel junction measurements performed on the same MgB$_2$ films [9]. Most of the tunnel characteristics measured for over 80 different films showed one gap with $\Delta(0) = 3$ meV. Only in a few cases the characteristics exhibited tiny shoulders at 7 meV.

Figure 1: Evaluation of $\Delta(0)$ for a MgB$_2$ film from $R_s(T)$ measurement data (symbols). The line shows a BCS exponential fit for $\Delta(0) = 3$ meV.
Measured $\Delta X_s(T)$ dependences allowed us to estimate the variation of the London penetration depth with temperature, $\Delta \lambda_s(T)$. A direct output of this estimation is an effective value: $\Delta \lambda_{\text{eff}} = \Delta X_s / 2 \pi f_0 \mu$, where $\mu$ is the magnetic permeability of vacuum. Obtaining the actual penetration depth requires a correction for the finite film thickness, $d$, as $\lambda_{\text{eff}} = \lambda_s \coth(d/\lambda_s)$, followed by the integration of $\Delta \lambda_s(T)$ data. Generally this procedure requires the knowledge of $\lambda_s(0)$ value. However, if a superconductor behaves in agreement with the standard BCS model the penetration depth is given by:

$$\lambda_{\text{eff}}^2(T) = \lambda_s^2(0) \left[ 1 - 2 \int_{\Delta(T)}^{\infty} \left(-\frac{df(E)}{dE}\right) \frac{E}{(E^2 - \Delta^2(T))^{1/2}} dE \right],$$  

(1)

where $f(E)$ is the Fermi function and the temperature dependence of the energy gap is given by [9,10]:

$$\Delta(T) = \Delta(0) \sqrt{1 - (T/T_C)^2},$$  

(2)

Using $\lambda_s(0)$ as a fit parameter we calculated first the $\lambda_s(T)$ dependence and then the $\Delta \lambda_{\text{eff}}(T)$ one. The comparison of the calculated $\Delta \lambda_{\text{eff}}(T)$ data with experiment yields the required $\lambda_s(0)$ value. As it is shown in fig.2 the measured and the calculated $\Delta \lambda_{\text{eff}}(T)$ data reveal a good agreement, which confirms the applicability of the BCS theory for describing the $\lambda_s(T)$ dependence of the MgB$_2$ films. The resulted $\lambda_s(0)$ values were estimated between 80 nm and 100 nm for different films. Similar values were reported in literature for the MgB$_2$ films prepared by other methods [1,2].

Fig.2(b) shows the temperature dependence of $\lambda_s^2(T)/\lambda_s^2(0)$, i.e. of the normalized superfluid density [8]. Obviously, the curves calculated by eq.(1) for different $\lambda_s(0)$ values and the same $\Delta(0)$ value coincide in fig.2(b). The measurement data of different MgB$_2$ films match this line very well for $T/T_C > 0.5$. At lower temperatures the match between the experimental and the calculated data becomes less well, which can be partly explained by degrading measurement accuracy due to the prevailing effect of the hf loss in Cu. Fig. 2(b) also shows the curve $\lambda_s^2(0)/\lambda_s^2(T) = 1 - (T/T_C)^4$ according to the ‘two-fluid’ model, which is usually a good approximation for the conventional low-temperature superconductors, like NbN [2]. The data for our MgB$_2$ films clearly disagree with the ‘two-fluid’ model.

**Figure 2:** (a) Evaluation of $\lambda_s(0)$ for three different MgB$_2$ films using $\Delta \lambda_{\text{eff}}(T)$ measurement data, marked with crosses, circles and triangles. The lines show the fits using eqs (1) and (2) and different $\lambda_s(0)$ values: $\lambda_s(0) = 80$ nm (solid line, coincides with circles), $\lambda_s(0) = 100$ nm (dashed line, coincides with crosses and triangles) and $\lambda_s(0) = 120$ nm (dotted line).

(b) Comparison of the experimentally obtained $\lambda_s(T)$ data with the dependences predicted by BSC (solid line) and two-fluid (dashed line) models.
The low-temperature part of the $R_s(T)$ dependence was examined using the Nb shielded resonator. For the sake of calibration, a bulk Nb sample was measured with the same device. The results for this Nb sample, which are shown in fig.3, agree well with the literature data, see e.g. [3]. Remarkably, the same resonator with a MgB$_2$ film exhibited higher quality factors than in the case of Nb. Thus, we confirm the conclusion of ref.[3], that the high-quality MgB$_2$ films can provide lower hf loss than Nb at temperatures down to 2 – 3 K. Furthermore, due to the exponential $R_s(T)$ behaviour, reaching the highest possible $T_c$ of MgB$_2$ films does not seem to be necessary for reaching low $R_s$ at low temperatures. In our case very low hf losses were measured for a MgB$_2$ film with relatively moderate $T_c$ of 32.5 K, see fig. 3. In particular, $R_s \leq 3 – 5 \mu \Omega$ was reached at $T \leq 3$ K.

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Figure 3: $R_s(T)$ measurement data obtained for a MgB$_2$ film at 18.8 GHz using a Cu shielding (crosses) and a Nb shielding (open circles). The error bars show the measurement uncertainty due to losses in Cu. The solid squares display $R_s$ of a bulk Nb sample measured in the Nb shielding.