Comparative study of Fe(Se,Te) thin films on flexible coated conductor templates and single-crystal substrates

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Abstract
In this paper, we compare epitaxial Fe(Se,Te) thin films grown by pulsed laser deposition on CaF2, SrTiO3, MgO single crystals as well as on different metallic templates having a CeO2 based top surface. In particular, we performed a detailed structural and superconducting analysis. X-ray diffraction studies showed highly textured films on all templates. The superconducting transition temperatures are between 21 K and 14 K for Fe(Se,Te) films on CaF2 and on MgO single-crystal substrates, respectively, whereas films on the metal templates show \( T_c \) values up to 18 K. The critical current density \( (J_c) \) was determined from magnetization loops in fields up to 7 T. Calculations in the framework of the extended critical state model showed \( J_c \) values over 2 MA cm\(^{-2}\) and 0.9 MA cm\(^{-2}\) at 5 K in self-field on single-crystal substrates and metal templates substrates, respectively. Detailed transmission electron microscopy studies reveal smooth layers on all templates. Whereas small defects were found in films on single crystals, pronounced grain boundaries with higher misorientation angle were visible in the layers on metallic templates.

Keywords: thin films, superconductor, coated conductor, Fe(Se,Te), epitaxy

1. Introduction
Since the discovery of superconductivity in iron-based superconductors (IBS) with a transition temperature above 20 K in 2008 [1], this family of materials attracts great attention both due to their unconventional superconducting properties as well as due to the prospect of possible applications of these materials in various fields of science and technology [2–5]. So far, cuprate superconductors seem to be more promising for application aspects due to the higher transition temperature and the high critical magnetic field \( (H_{c2}) \). However, even in spite of these remarkable properties, it is challenging to realize high critical current density \( (J_c) \) values in magnetic fields for superconducting wires or tapes based on cuprates partially due to a significant magnetic-field anisotropy \( (\gamma) \) [6]. Moreover, the critical angle \( (\alpha_c) \) of about 4° for cuprates at which the critical current density across a grain boundary starts to decrease exponentially, requires highly textured layers for the development of superconducting tapes [7, 8]. Therefore, the IBS might be particularly promising for high magnetic field applications requiring a high \( J_c \) at low temperatures due to their high \( H_{c2} \), low \( \gamma \) and larger \( \alpha_c \) [2, 9].
Among the different families of IBS, the so-called 11-type materials (Fe$_{1-x}$A$_x$A = Se, Te, S), such as FeSe or FeSe$_{1-x}$Te$_x$ (Fe(Se,Te)) are interesting from the point of fundamental physics as well as the applied science. One of the main advantages of these materials is the simplest structure among the IBS materials. Although, the typical 11-type superconductors have significantly lower $T_c$ compared to cuprates with about 8 K in bulk β-FeSe, up to 21 K in FeSe$_{0.5}$Te$_{0.5}$ films, and above 30 K in thin FeSe layers, they exhibit a rich electronic phase diagram and a high tunability by external parameters [5]. As a result, the $T_c$ of Fe(Se,Te) films is highly dependent on the substrate and growth conditions [10–14]. In addition, the thermal expansion coefficient might also play a major role by introducing compressive or tensile strain states in the film during cool-down and thereby enhancing or reducing the transition temperature of Fe(Se,Te) thin films [3, 15].

To avoid the detrimental effects of large angle grain boundaries on current transport, so-called second generation conductors were developed for the applications of high temperature superconductors in science and technology, which are mainly based on thin film processes. There are two major methods, which are most commonly used for the production of such textured metal templates: the rolling assisted biaxially textured substrates (RABiTS) approach or templates with a textured buffer using ion-beam assisted deposition (IBAD).

Within the last years, significant research efforts have been devoted to investigate also IBS materials on such metal templates: the rolling assisted biaxially textured substrates (RABiTS) approach or templates with a textured buffer using ion-beam assisted deposition (IBAD). Within the last years, significant research efforts have been done for state-of-the-art YBCO coated conductors. The films were grown by pulsed laser deposition (PLD) using a KrF excimer laser ($\lambda = 248$ nm) and a stoichiometric FeSe$_{0.5}$Te$_{0.5}$ target in a ultra high vacuum (UHV) chamber with a base pressure below $10^{-8}$ mbar. The distance between the target and the substrate is set to about 38 mm. The film thickness is controlled by the pulse number while monitoring the energy density on the target. The growth rate was estimated on the basis of regular focused ion beam (FIB) cuts and TEM images. Prior to deposition, the substrates were preheated to 800 °C under UHV conditions in order to clean the surface and then cooled down to the deposition temperature. The growth of the films was carried out in a two stage process described previously [28] using a seed layer deposited at 400 °C with a frequency of 4 Hz before the deposition of the main layer at 300 °C at a frequency of 7 Hz. The thin film growth process was in-situ monitored by the reflection high energy electron diffraction. After deposition, the as-grown thin film was cooled down to room temperature in ultra-high vacuum.

### 2. Experimental details

#### 2.1. Synthesis

Fe(Se,Te) films were prepared on (001) oriented CaF$_2$, MgO, SrTiO$_3$ (STO) single crystalline substrates as well as on Ni-$\%$ W tapes with a La$_2$Zr$_2$O$_7$/CeO$_2$ buffered layer architecture (provided by Deutsche Nanoschicht GmbH) and Hastelloy C276 tapes with an IBAD-MgO based buffer stack having a top Gd-doped CeO$_2$ layer (provided by S-Innovations), which were both developed for state-of-the-art YBCO coated conductors. The films were grown by pulsed laser deposition (PLD) using a KrF excimer laser ($\lambda = 248$ nm) and a stoichiometric FeSe$_{0.5}$Te$_{0.5}$ target in a ultra high vacuum (UHV) chamber with a base pressure below $10^{-8}$ mbar. The distance between the target and the substrate is set to about 38 mm. The film thickness is controlled by the pulse number while monitoring the energy density on the target. The growth rate was estimated on the basis of regular focused ion beam (FIB) cuts and TEM images. Prior to deposition, the substrates were preheated to 800 °C under UHV conditions in order to clean the surface and then cooled down to the deposition temperature. The growth of the films was carried out in a two stage process described previously [28] using a seed layer deposited at 400 °C with a frequency of 4 Hz before the deposition of the main layer at 300 °C at a frequency of 7 Hz. The thin film growth process was in-situ monitored by the reflection high energy electron diffraction. After deposition, the as-grown thin film was cooled down to room temperature in ultra-high vacuum.

#### 2.2. Characterization

The structural characterization of the Fe(Se,Te) films was performed by x-ray diffraction (XRD) with Co-K$_\alpha$ radiation using a Bruker D8 Advance in a $\theta$–2$\theta$ geometry. The texture of the films was analyzed with a Philips X’pert diffractometer using Cu–K$_\alpha$ radiation, whereas the in-plane lattice parameters were determined by reciprocal space maps (RSM) in a similar device. The local microstructure of selected samples was studied in detail together with the interface structure between films and substrates by TEM using a FEI Tecnai T20 operated at 200 kV acceleration voltage. The orientation and crystalline quality of the different layers was investigated with selected area diffraction (SAD). For the preparation of the TEM lamellas, the FIB method was applied using a dual beam system Helios 600i from FEI.

At first, the superconducting properties of the films were measured in a Quantum Design Physical Property Measurement System by a standard four-probe method using press contacts. Simultaneously, a Quantum Design Magnetic Property Measurement System was used for magnetic measurement to study the bulk superconducting properties. A film thickness of 280 nm ± 50 nm was used for these investigations, which also allowed to estimate the critical current density $J_c$ at different temperatures.
3. Results and discussions

As already mentioned in the introduction, we aim to compare epitaxial Fe(Se,Te) films grown on different templates. In order to do this, we use the optimized deposition conditions for layers on single crystalline substrates to prepare similar superconducting films on the textured metallic templates. Whereas initially a film thickness of about 70 nm was used for basic studies, thicker films were prepared for magnetic measurements.

3.1. Structural characterization

Figure 1(a) compares the XRD pattern of thin Fe(Se,Te) films grown on the single-crystal substrates and metal templates. Only (00l) Fe(Se,Te) peaks are present in the standard θ–2θ scans (marked by light blue bars) indicating that the obtained films were grown with a preferred c-axis orientation. All additional peaks in the different diffractograms arise from the substrate or the buffer layers, i.e. no additional peaks associated with different orientation of Fe(Se,Te) or additional phases were found. It is obvious that the (00l) peaks on the metallic templates are broader and have less intensity if compared with the layers on single crystals. This might result from a larger orientation spread and a higher defect density as will be discussed below. The c-axis lattice parameter of the films summarized in table 1 was determined by a Nelson Riley approximation from this data [29]. Additionally, the a-axis lattice parameter was determined by a reciprocal space mapping for the films on single crystals. Unfortunately, such measurements were not possible for Fe(Se,Te) layers on the metallic templates due to the low peak intensity of the (204) peaks used for the RSM analysis.

The c-axis lattice parameters of the films vary typically between 5.8 Å and 5.95 Å for the different substrates, which is slightly smaller than the value for the single crystalline FeSe0.5Te0.5 (c = 5.954 Å). At first, it was shown that PLD-grown films typically have a Se-rich stoichiometry, which leads also to a reduction of the c-axis parameter in comparison to the target value [13, 30]. TEM-EDX studies on the films studied in this paper revealed a composition close to FeSe0.5Te0.5; however, with a large uncertainty due to the small volume measured. Secondly, the higher c-axis lattice parameter for the Fe(Se,Te) films on the CaF2 substrate compared to SrTiO3 and MgO might be also the result of compressive strain due to thermal mismatch, i.e. different thermal expansion coefficients of substrate and film. This results also in the smallest a-axis value for CaF2 despite the fact that the in-plane lattice constant of all used single crystals is larger than the a-axis lattice parameter of single crystalline FeSe0.5Te0.5 (a = 3.800 Å) [15]. For the films on metallic tapes, the surface morphology of the top CeO2 layer might also influence the defect densities and the lattice parameters of the Fe(Se,Te) film (see TEM study below). Finally, also differences in the layer thickness might play a role [11].

The epitaxial growth of the Fe(Se,Te) films has been proven by pole figure measurements as shown in figure 1(b), where a clear epitaxial relationship was detected for all samples. All measured (111) or (101) pole figures and the corresponding ϕ scans of Fe(Se,Te) reveal no satellite nor additional reflections other than sharp and strong reflections at every 90° indicating a strong biaxial texture. The 45° in-plane rotation of the layer on CaF2 is due to the smallest lattice mismatch towards the single crystal. Again, the peaks in the pole figures of the Fe(Se,Te) films are broader on metal templates compared to single-crystal substrates. The ω-scan of the 001 reflection characterizing the out-of-plane orientation shows a sharp full width at half maximum of about ∆ω = 1.0° a for Fe(Se,Te) films on single-crystalline substrates but is significantly broader for RABiTS and IBAD-MgO metal templates with values of about ∆ω = 2.9° and ∆ω = 2.6°, respectively. Nevertheless, these values are similar to the one for the CeO2 top buffer layer of the template indicating a good texture transfer. The average in-plane orientation Δφ of Fe(Se,Te) is up to 2° on single-crystal and up to 5° on metal templates, respectively. In summary, the XRD results show an epitaxial growth and strong biaxial texture of the Fe(Se,Te) films on all substrates.

3.2. Superconducting properties

The superconducting properties of the films were evaluated by measuring the resistivity of unpatterned samples as well as the magnetization using a SQUID magnetometer. The onset of the superconducting transition, Tc,90, in the transport measurements is defined at 90% of the resistance in the normal state. Figure 2(a) shows the dependence of the resistance on temperature R(T) for the different substrates. The superconducting transition temperature is highest with about 20.6 K on CaF2 and lowest with about 13 K and 14 K for the thin films on RABiTS and IBAD-MgO templates, respectively. For the RABiTS substrates, the transition temperature was improved significantly with thickness. Thick Fe(Se,Te) films on the RABiTS template exhibit a resistance anomaly with a peak near to the superconducting transition. A similar behavior was found by other groups in Fe(Se,Te) [25] or Co-doped BaFe2As2 films [31] on metal tapes as well as in metallic superconductors [32]. This feature is typically related to parallel conducting paths for the bias current due to partially conducting buffer layers, sample inhomogeneities or technology-related issues in the patterning process. As we observed this peak on unpatterned samples, we assume that our thin buffer layer provides some conducting paths towards the metal substrate similar to the case of Co-doped BaFe2As2 on thin IBAD-MgO based buffer architectures [31].

Figure 2(b) shows the magnetization measured after zero-field cooling at an external magnetic field of 2 Oe applied along the c-axis for samples with a thickness of (280 ± 50) nm. The magnetization changes of the different films are in good correlation with the R(T) data in figure 2(a) for most substrates. The presence of a strong diamagnetic signal indicates that all Fe(Se,Te) layers exhibit a superconducting state at low temperatures. Additionally, some positive net magnetization is observed above the superconducting transition for the film on the RABiTS tape originating from the ferromagnetic properties of the used Ni-W substrate at low temperatures.
Figure 1. (a) Comparison of θ–2θ scans for selected Fe(Se,Te) films grown on different substrates showing a pure c-axis alignment (marked with light blue bars); single crystal peaks are marked with an asterisk (*); (b) pole figure of the (111) Fe(Se,Te) peak for films on CaF$_2$, IBAD-MgO, RABiTS and (101) peak on MgO, respectively.

Table 1. Structural parameters for the Fe(Se,Te) thin films grown on single-crystal substrates and metal templates determined from XRD measurements.

| Substrate | c-axis lattice (Å) | a-axis lattice (Å) | Δφ (111) (°) | Δω (001) (°) |
|-----------|-------------------|-------------------|--------------|--------------|
| CaF$_2$   | 5.91              | 3.74              | 1.4          | 1.1          |
| SrTiO$_3$ | 5.86              | 3.75              | 2.0          | 1.4          |
| MgO       | 5.83              | 3.76              | 2.0          | 1.5          |
| RABiTS    | 5.93              | 5.0/4.6           | 2.9/3.9      |
| IBAD-MgO  | 5.91              | 4.6               | 2.6          |

Figure 2. (a) Temperature dependence of the resistance for Fe(Se,Te) thin films grown on different substrates normalized to the resistance at 30 K. All films have a thickness of about 70 nm. Additionally, a film on RABiTS with 290 nm is shown. (b) Temperature dependence of the magnetization using a SQUID magnetometer with the applied field of 2 Oe (film thickness: 280 nm ± 50 nm).

The magnetization curves for the different samples indicate that the $T_c$ of the film on the RABiTS substrate (around 15 K) is just slightly lower than that of the sample on CaF$_2$; however, with a significantly broader transition width. Such a broad transition on RABiTS might be associated with the weak link behavior of large angle grain boundaries or with sample inhomogeneities. This needs to be clarified with a combination of local EBSD studies combined with scanning Hall probe microscopy planned for the future [27]. In case of Fe(Se,Te) on STO, which shows a higher transition temperature for thin
films in resistivity measurement compared to values obtained in magnetic measurements for thick films, additional influences need to be taken into account. At one hand, we observed a higher probability of crack formation in thick layers on STO which is a sign for internal strain states. On the other hand, oxygen diffusion might play a role for thicker films due to longer processing times compared to thin films. More detailed studies are required to clarify this behavior.

The critical current density \( J_c \) of the Fe(Se,Te) films was determined from the magnetization loops and calculated using the Bean model (figure 3). The thickness of each layer was determined by FIB cuts or from TEM lamella prepared in parallel. The magnetization loops were measured at 5 K in an external magnetic field up to 7 T applied along the c-axis. The value of \( J_c \) at self-field was evaluated using a fitting function of \( J_c(H) \) in the framework of the extended critical state model \([33]\). This model is a significantly better description for \( J_c \) in an external magnetic field than the standard Bean model. Fe(Se,Te) films grown on single-crystalline substrates showed a significantly larger \( J_c \) compared to the samples on metal templates. The Fe(Se,Te) film on the CaF\(_2\) substrate has the highest \( J_c \) at self-field of about 2.4 MA cm\(^{-2}\) and almost 0.4 MA cm\(^{-2}\) at \( \mu_0 H = 7 \) T. In comparison, the film grown on MgO has a similar field dependence, but lower values. A similar behavior with higher \( J_c \) values was observed for both films at 2 K, whereas a stronger field dependence is found for higher temperatures (figure 3(b)). In contrast, the film grown on STO has still a high \( J_c \) in self-field, but a significantly stronger magnetic field dependence, which is consistent with the broad superconducting transition observed in SQUID measurements. Even though the Fe(Se,Te) film on RABiTS has only a moderately lower \( T_c \) than the film on CaF\(_2\) and a strong biaxial texture, its \( J_c \) in self-field is significantly lower (about 0.92 MA cm\(^{-2}\)). Moreover, \( J_c \) on RABiTS decreases more drastically with increasing magnetic field than \( J_c \) on the CaF\(_2\) or MgO single crystalline substrates probably due to the presence of the grain boundary network. The Fe(Se,Te) film on the IBAD-MgO template exhibits a \( J_c \) value of 1.25 MA cm\(^{-2}\) at 5 K in self-field showing a similar \( J_c \) tendency with increasing magnetic field as the film on the RABiTS template. This is somehow in contrast to results reported by other groups on IBAD-MgO templates, where \( J_c \) decreases very slowly with increasing field \([24]\). Again, such behavior might be associated with the influence of the grain boundary network on this substrate, which was different in our films compared to the films in literature. It was shown above that the in-plane orientation of the film strongly depends on the substrates used for the deposition of the Fe(Se,Te) films. Whereas XRD gives an information on the global orientation distribution, more localized studies are required to analyze this behavior on a local scale. Therefore, a detailed microstructural study was performed to identify possible defect structures, which govern the superconducting behavior of the different films.

3.3. Detailed microstructure analysis using TEM

Lamellas of selected samples were prepared for a detailed TEM study in order to analyze the general microstructure, emerging defects as well as the interface between the superconducting film and upper part of the template. The final goal is to correlate the structural properties with the superconducting characteristics.

Figures 4–6 show TEM cross sections for Fe(Se,Te) films with a thickness of about 75 nm grown on the different single crystalline substrates. In all cases, we observe a homogeneous film thickness and flat surfaces. Furthermore, the results of SAD measurements confirmed an epitaxial relationship for all substrates. The high magnification TEM images reveal more localized defect structures. The high-resolution image for the film grown on STO shows only minor defects and a thin bright interface layer with some dislocations. Nevertheless, the interface is sharp as shown in figure 4(b). SAD patterns of Fe(Se,Te) films indicate that the film on STO is
well aligned. The corresponding fast Fourier transformations from the film and the substrate area confirm the high epitaxial quality of Fe(Se,Te) films on STO. In case of the Fe(Se,Te) films grown on MgO, the interface between the film and the substrate was smooth as seen in figure 5(b). The higher resolution image indicates a number of dislocations as well asren regions of the high-resolution image appear to be disturbed at a local scale. At the same time, SAD indicates some misorientation spread as shown in figure 4(c). An extra layer seems to be formed at the interface between the superconductor and the CaF$_2$ substrate. A similar behavior has been reported in a number of papers [34, 35]. The layer is attributed either to an interdiffusion of light elements as fluorine or strained areas. However, the Fe(Se,Te) film seems to have a high structural quality as indicated in the SAD image in figure 6(a). Accordingly, only minor defects are visible in the high-resolution image in figure 6(d), where the lattice planes seem to be uniform above the blurred interface layer.

In general, these results for films on single crystalline substrates seem to correlate well with the superconducting and transport properties described above. Whereas the Fe(Se,Te) films on MgO substrates have the lowest $T_c$ and self-field $J_c$ as well as the highest number of structural defects among all samples on single-crystal substrates, it is the opposite for the layers on CaF$_2$, which have the best superconducting, transport and structural properties. In contrast, the high structural quality of the Fe(Se,Te) films on STO fits with the observed high transition temperature, but does not explain the low $J_c$ data obtained in the magnetic measurements. Here, additional TEM studies on thick films are required to identify the origin of this behavior.

In the next step, different films grown on the textured metallic templates were studied with TEM. The cross-section of the layered architecture on a RABiTS substrate is shown in figure 7(a). Here, the 5 at.% W doped nickel tape was covered with an about 300 nm thick epitaxial La$_2$Zr$_2$O$_7$ layer and an additional thin CeO$_2$ buffer. Again, a homogeneous Fe(Se,Te) layer is grown on top of this template showing a slightly wavy surface. The nucleation on the CeO$_2$ surface seems to work fine as shown in figure 7(b). Furthermore, the film looks homogeneous over a larger scale on the lamella without any cracks or pores (not shown here). However, some high angle grain boundaries are visible in the cross section of the film on the RABiTS template, one example is shown in figure 7(c). They result in a clear tilt between the lattice planes on both sides of the grain boundary, which might be the reason for the lower $J_c$ values compared to the films on single crystalline substrates. Whereas such large grain boundaries were observed in TEM, no statement is possible on the density of these boundaries over the complete sample area, as only a small region was studied in detail. Whereas integral texture measurements indicate...
a clear biaxial texture as shown in figure 1(b), detailed EBSD measurements are required to get statistic values on the grain boundary distribution. It also needs to be clarified, if such high angle grain boundaries might be avoided by an improvement of the buffer architecture or an optimization of the film processing.

A similar analysis was performed for samples on IBAD-MgO templates having different thicknesses. Figures 8(a) and (c) shows the longitudinal cross-section of the Fe(Se,Te) on such a metal based template demonstrating the complex layer architecture. In this case, the top layer is an about 200 nm thick Gd doped CeO$_2$ film intended to transfer the biaxial texture to the superconducting layer. The Gd:CeO$_2$ shows columnar grains with a larger grain size compared to the final CeO$_2$ in the RABiTS buffer architecture. The interface between the top buffer layer and Fe(Se,Te) shown in figures 8(b) and (e) has a wavy appearance. Whereas for large areas an epitaxial growth is observed, again some tilted grains are visible even in the thinnest film. The resulting grain boundaries with a significant misorientation angle might be the reason behind the lower transition temperature and the lower $J_c$ values for films on IBAD-MgO. Again, more detailed studies are required to understand the nucleation behavior of Fe(Se,Te) on the CeO$_2$ layers. For thicker films, a columnar structure is apparent for the Fe(Se,Te) layer as indicated by the orientation contrast in figure 8(c). More detailed views on the surface layer of this film indicate visible grain boundaries, some of them with a larger misorientation angle (left side of figure 8(d)). However, as already discussed for the RABiTS based samples, no statistical information on the number of large angle grain boundaries can be achieved based on these TEM images. This might be only done by high-resolution EBSD maps, which allows to study the grain boundary distribution on a larger area.
4. Conclusion

In summary, we performed a detailed study on structural and superconducting properties of Fe(Se,Te) thin films grown on various single-crystalline as well as metal templates. XRD data showed that all the films on the different substrates are highly textured. Transport measurements showed a clear superconducting transition, the magnetic susceptibility of thicker layers proved a sharp diamagnetic signal for all films indicating bulk superconductivity. Among the samples studied, films on CaF$_2$ showed the highest transition temperature and critical current density with values above 2 MA cm$^{-2}$ at 5 K in self-field. The layers on MgO showed slightly smaller $J_c$ with a similar magnetic field dependence, which might be explained by a higher number of defects found in the TEM studies. In contrast, films on STO showed reduced $J_c$ values, which deserve further studies. Fe(Se,Te) layers on coated conductor templates showed smaller transition temperatures and critical current densities in magnetic fields for both RABiTS and IBAD-MgO templates. Detailed TEM studies indicated the presence of high angle grain boundaries in these films arising from the interface with the buffer layer. Therefore, further improvements are required to optimize the superconducting properties.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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