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# Nanoparticulate precipitates in Y:123 thin films

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Y:123 high temperature superconductors thin films were obtained by DC magnetron sputtering method. The structure and crystalline orientation is determined by $\theta$–$2\theta$ scan obtained by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The $Y_2O_3$ nanoprecipitates were evidenced through the complete thickness. The critical current density function of temperature is estimated from a.c. magnetic susceptibility.

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*Keywords*: Y:123 thin film, Nanoparticulate, XRD, TEM, a.c. susceptibility

## 1. Introduction

For the c-axis oriented $YBa_2Cu_3O_{7-\delta}$ (Y:123) thin films, a frequently observed feature is the presence of outgrowths as: needle –like a-axis grains, epitaxial grains such as $Y_2O_3$, $CuYO_2$ and $Y_2BaCuO_5$,nonepitaxial grains of CuO or$Y_2O_3$ [1-2]. It has been shown that a decrease of the deposition temperature from 740 to 720 $^oC$, can lead to a drastic increase of a-axis oriented grains on the surface of the films [3].The presence of $Y_2O_3$ precipitates as a defect is also observed in Y:123 thin films prepared by different techniques [4-5].These precipitates grow semi coherently in a cubic form having a volume between 100 and 1000 $nm^3$, with a density estimated at $10^{16}$ particles/$cm^3$ [4]. These defects can be considered as good candidates for the effective pinning centers. Close to the surface of the film also some rectangular shaped $Y_2O_3$ precipitates have been identified [6].

## 2. Experimental

Y:123 thin films were deposited onto MgO single crystal substrate by using DC magnetron sputtering system. In order to increase the deposition rates and the uniformity of the resulting films the target is a cylinder which is fixed inside of cathode, itself placed inside a ring magnet. The sputtering system works in the DC mode with a power of 50-70 W. The sputtering gas was a mixture of oxygen and argon, with partial pressures 0.4 mbar Ar and 0.3 mbar oxygen.

Before each deposition the target was presputtered for 40 minutes. The substrate temperature was kept at 780 $^oC$ (an optimized temperature regarding the epitaxial and compositional properties of the films), for all depositions in this study. After deposition, the films were annealed at 450 $^oC$ for 30 minutes in an oxygen atmosphere. The deposition time was 1 h, leading to thin films with thickness of approximately 200 nm.

EDX (energy dispersive X-Ray) shows that the target composition is Y:Ba:Cu = 1.02:2:3 .The film are Y rich, and composition is Y:Ba:Cu = 1.3:2:3.For reliable composition measurements of the film it is necessary to prepare 1μm thick films.

X-Ray studies were performed with a Brucker diffractometer using a copper anode. The acceleration voltage is typically 30kV.

The transmission electron microscopy (TEM) was used for study the local structure and for the observation of very small defects, down to nanometer size.

The real ($\chi'$) and imaginary ($\chi''$ ) parts of the a.c. susceptibility were simultaneously collected with a Lake Shore Model 7000 a.c. susceptometer in the temperature range from 77K to 110 K, by using frequencies f and a.c. field amplitudes $H_{ac}$ situated in the ranges from 20 Hz to 1000 Hz and from 20 A/m to 800 A/m respectively. When the system is cooled down to 4.2 K an additional DC magnetic field of maximum 1 Tesla is applied.

## 3. Results and discussion

The peak indexation of the X-ray diffraction pattern obtained for the best quality Y:123 thin film deposited in optimal conditions indicates that only (00l) reflexions and the reflexions (h00) for MgO substrate. This behavior characterizes the highly oriented c-axis thin film. The c = 1.1681 nm value for lattice parameter was calculate from the X-Ray diffraction pattern.

The epitaxy was investigated by the measurement of rocking curve. The $\theta$–$2\theta$ angle is fixed on (003) peak and the rocking curve is obtained by oscillating the sample holder with an angle $\omega$ around the axis of diffractometer , what allow probing the contribution of the misoriented crystallites. Fig. 1 shows the rocking curve for the Y:123 film deposited on optimal conditions. The FWHM (full width for high maximum) of rocking curve obtained by Lorentz model for the lineshape is equal to $0.247^0$, and confirmed the excellent epitaxial quality of thin film. The substitution of Cu by Zn increases the FWHM value indicating the decrease of the epitaxy of the film [7].

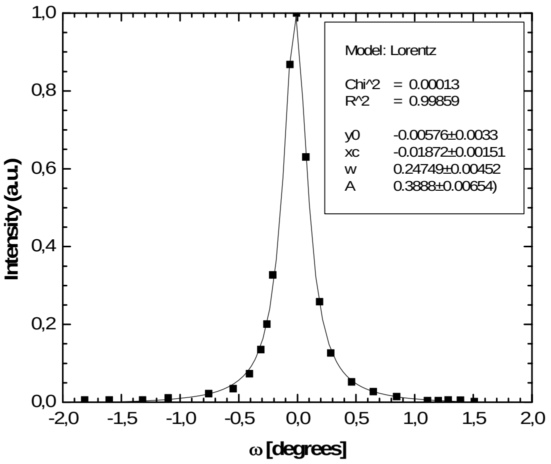

*Fig. 1. The rocking curve around the (003) line of Y:123 thin films obtained in optimal conditions on MgO substrate.*

Fig. 2 shows the cross section of thin films obtained by TEM micrograph. The lines observed in Fig. 2 correspond to the $CuO_2$ planes, while the bright part at the bottom of the figure is the MgO substrate. The parallel arrangement of these lines reflects the c-axis orientation of film. The ellipses reveal the presence of nanoprecipitates throughout the film thickness. Similar results were obtained for similar compositions of Y:123 thin films [5,6].The global film composition determined previously is Y rich, and suggest that more probably the nanoprecipitates are$Y_2O_3$.Recentely artificial pinning centers were introduced perpendicular to the Y:123 film surface using nanosized $Y_2O_3$ islands prepared on substrate [8].These artificial defects enhanced $J_c$ at 77K from 1.8 to 2.7 $MAcm^{-2}$. Trapped flux and critical current were enhanced by using nanoscale $Y_2BaCuO5$ and $Y_2Ba_4CuMO_y$ (M=Nb,Ta,Mo,W,Ag,Sb,Sn,Bi) phases embedded in Y:123 films [9,10].

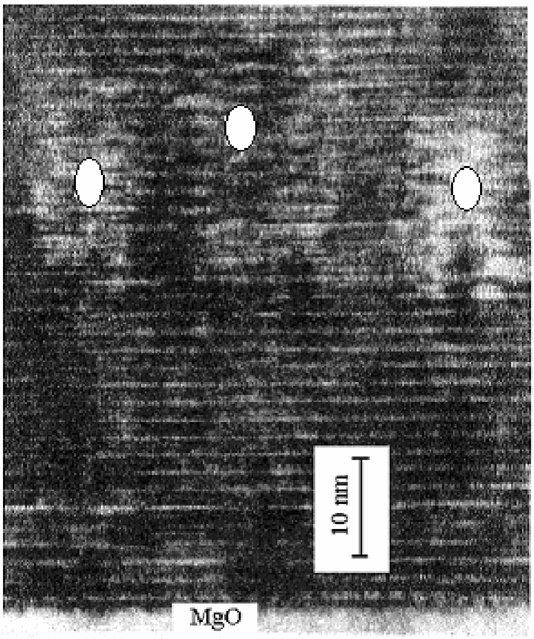

*Fig. 2.Cross section TEM micrograph of Y:123 thin film. The ellipses reveal the nanoprecipitates.*

Fig. 3 show the temperature of imaginary part$\chi''$ of a.c.susceptibility for a.c. field increasing from $H_{ac}$= 0.4 A/m to 800 A/m.$\chi''$(T) exhibit a single peak at temperature $T_p$ indicating a maximum hysteressis losses due to the motion of the vortices. With increasing the amplitude $H_{ac}$ of alternative field, the shape of $\chi''$(T) broadening and shifts to lower temperatures.

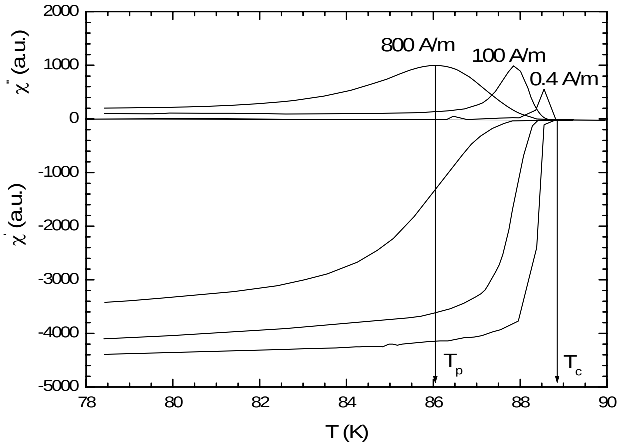

*Fig. 3.The real and imaginary part of the A.C. susceptibility of the Y:123 thin film.*

By performing AC susceptibility measurements at different external fields we can deduce the temperature dependence of the critical current density from equation [11]:

$J_c$ ($T_p$) = 3.157 $H_{ac}$/d, where $T_p$ is the temperature of the $\chi''$(T) peak and d is the thin film thickness.

Fig. 4 show the $J_c$ (T) deduced from the experimental curves of $\chi''$ (T).

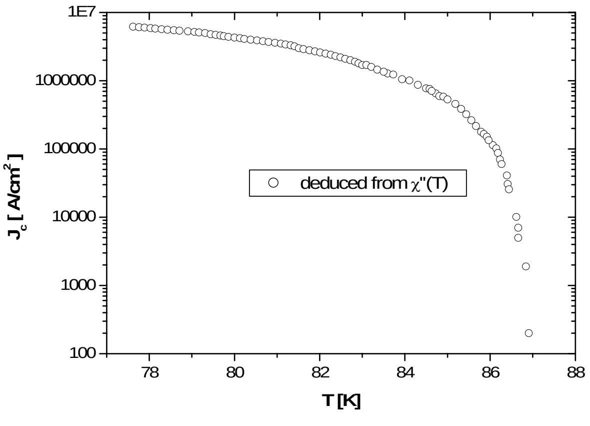

*Fig. 4. $J_c$ (T) deduced from the experimental curves of $\chi''$(T).*

The high value of critical current density (up to 7× $10^6$ $A/cm^2$ at 77K) suggest that the nanoparticulate precipitates are responsible for the strong pinning of vortices in Y:123 film.

## 4. Conclusions

The Y:123 films synthesized by d.c. sputtering method are Y-rich and presents nanoparticulate precipitates (more probable $Y_2O_3$).

The films have their c-axis perpendicular to the substrate surface and good epitaxial quality.

A.C. magnetic susceptibility versus temperature shows a single peak in the $\chi''$(T) dependence which broadened and shift to lower temperature by increasing the applied A.C. amplitude.

The critical current density $J_c$(T) deduced from $\chi''$ (T) curves shows high values at 77K, and suggest that the nanoparticulate precipitates are strong pinning centers.

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