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J. A. Lewis, M. Wegmann, C. E. Platt, and M. Teepe

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Platinum-enhanced densification of grain-aligned $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ films

J. A. Lewis and M. Wegmann

Science and Technology Center for Superconductivity and the Materials Science and Engineering Department, University of Illinois, Urbana, Illinois 61801

C. E. Platt and M. Teepe

Science and Technology Center for Superconductivity, University of Illinois, Illinois 61801

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Grain-aligned $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ films were fabricated by vacuum filtration in an applied magnetic field (7 T). Platinum (Pt) was shown to lower the peritectic temperature of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ by almost 75 °C, leading to dramatic microstructural differences between films densified on MgO (single crystal) substrates and those densified on Pt foil to a maximum temperature of 1020 °C in oxygen. Superconducting quantum-interference device hysteresis loops measured at 0–5.5 T at 5 K showed that films fired on Pt had $\Delta M (H_{\text{app}} \parallel c \text{ axis})$ values 15–60 times larger than those fired on MgO.

Melt texturing is among the most promising techniques developed to produce grain-aligned $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) components with superior superconducting properties. Melt texturing can be achieved either by a melt-growth process^{1–3} or by directional solidification;^{4,5} however, both of these processes have inherent limitations. For example, in melt-growth processing, the YBCO domains that form upon resolidification of a peritectic melt are poorly aligned within the bulk of the sample. Alternatively, directional solidification of YBCO produces highly oriented samples, but the growth rates required for grain-aligned materials are extremely slow ($\approx 1 \mu\text{m}/\text{sec}$). Previously we have shown that a combination of magnetic alignment of YBCO particles under ambient conditions followed by controlled heat treatment can be used to produce partially melted, grain-aligned YBCO films.⁶ Recently, several researchers have shown that the size distribution of the insulating Y_2BaCuO_5 (Y211) phase is affected by the presence of platinum during melt texturing.^{7–10} In this letter, we report on how platinum affects the densification and melting behavior of magnetically aligned YBCO films.

YBCO films were formed by vacuum filtration of a particulate suspension in an applied magnetic field ($H=7 \text{ T}$). 10 vol % $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ particles (diameter $\approx 2\text{--}6 \mu\text{m}$) were dispersed by dissolving an appropriate amount of an organic dispersant in an isopropanol solution. The as-cast films (diameter = 2.5 cm, thickness $\approx 0.5 \text{ mm}$) were fired either on magnesium oxide (MgO) single-crystal substrates with (100) orientation or on platinum (Pt) foil. Samples were heated in nitrogen to 650 °C at 1 °C/min, held at 650 °C for 2 h in oxygen, heated to 950 °C at 5 °C/min, held at 950 °C for 2 h, heated to 1020 °C at 5 °C/min in oxygen, rapidly cooled to 1010 °C, and then slow cooled at 1 °C/h to 950 °C. Samples were then furnace cooled from 950 to 450 °C, oxygen annealed for 24 h, and cooled to room temperature.

Differential thermal analysis (DTA) of as-received $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ powder, $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ powder mixed with 10 wt % MgO powder, and $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ powder mixed with 10 wt % Pt powder was conducted in flowing oxygen (flow rate $\approx 10 \text{ cm}^3/\text{min}$) at a heating rate of 5 °C/min to determine whether YBCO films would interact with either substrate material during heat treatment. In all DTA measurements, a large sample size ($\approx 150 \text{ mg}$) was used to simulate

the actual film conditions during heat treatment. While this may produce broader peaks at slightly higher temperatures due to heat and/or mass transfer effects, the differences in measured peak temperatures between samples should remain independent of sample size. Figure 1 shows the DTA curves for $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ powder, $\text{YBa}_2\text{Cu}_3\text{O}_{7-x} + 10 \text{ wt \% MgO}$ powder, and $\text{YBa}_2\text{Cu}_3\text{O}_{7-x} + 10 \text{ wt \% Pt}$ powder heated in an oxygen atmosphere. For the pure $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ powder sample, an endothermic peak was observed at 1030 °C corresponding to the incongruent melting of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ phase (i.e., $\text{YBa}_2\text{Cu}_3\text{O}_{7-x} \rightarrow \text{liquid} + \text{Y}_2\text{BaCuO}_5$). With $\text{YBa}_2\text{Cu}_3\text{O}_{7-x} + 10 \text{ wt \% MgO}$ powder, this endothermic peak was observed at a slightly lower temperature (1027 °C). However, with $\text{YBa}_2\text{Cu}_3\text{O}_{7-x} + 10 \text{ wt \% Pt}$ powder, this endothermic peak was observed at a much lower temperature (954 °C). Clearly, platinum has a dramatic impact on the melting behavior of Y123, reducing the onset of incongruent melting by almost 75 °C.

Figures 2(a) and 2(b) show polished cross sections of aligned YBCO films fired on MgO and Pt substrates, respectively, as viewed by scanning electron microscopy (SEM).

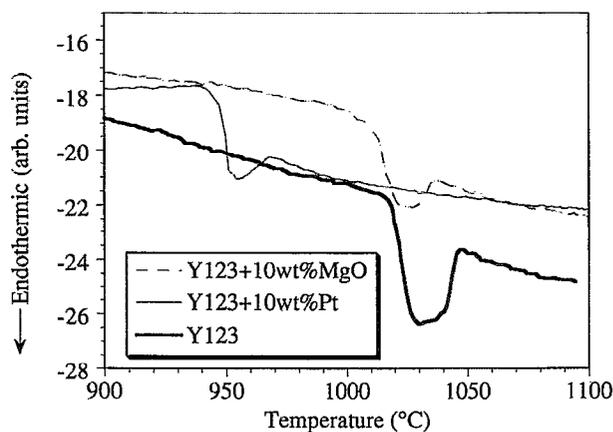


FIG. 1. Differential thermal analysis results for Y123 powder, Y123+10 wt % MgO powder, and Y123+10 wt % Pt powder samples heated at 5 °C/min in flowing oxygen.

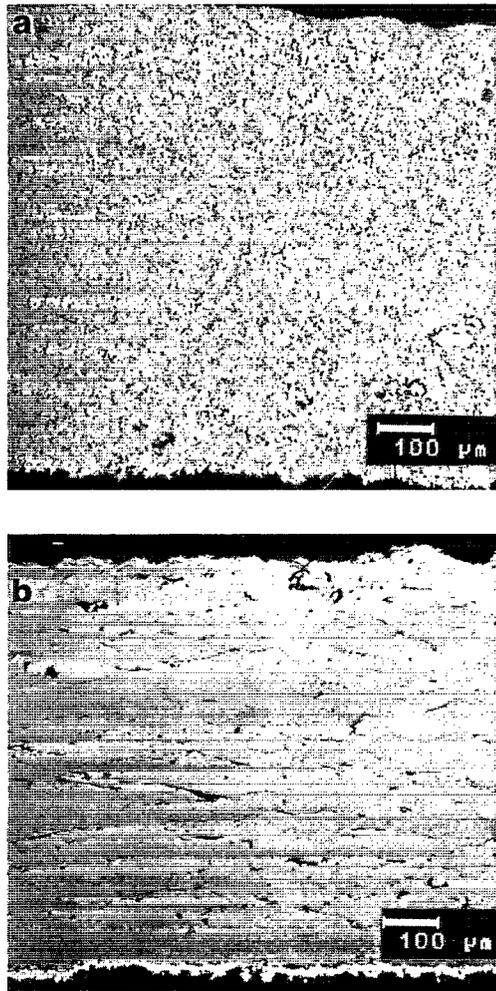


FIG. 2. SEM of polished cross sections of magnetically aligned YBCO films heated to 1020 °C on (a) MgO and (b) Pt. (Note: The YBCO film-substrate interface corresponds to the bottom surface of each sample.)

Each film was removed from their respective substrates prior to SEM analysis. The YBCO films fired on MgO have a small average grain size ($\approx 10 \mu\text{m}$) and a large amount of open porosity, features indicative of a conventionally sintered material. Based on the DTA results for pure $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$, it is expected that these films did not undergo incongruent melting during heat treatment, since the maximum firing temperature (1020 °C) remained below the peritectic temperature (1027 °C) of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ heated in oxygen in contact with MgO. In contrast, YBCO films fired on Pt have a much larger average grain size ($>50 \mu\text{m}$) and are nearly 100% dense, features indicative of a melt-processed material. Hence, a significant interaction between the Y123 film and the Pt substrate must have occurred to produce this type of microstructural development.

Figure 3 shows a schematic representation of the proposed mechanism for densification of the YBCO films fired on Pt substrates. Below 950 °C, the YBCO film is not affected by the Pt substrate and no liquid is present. At 954 °C, incongruent melting of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ phase occurs at the film-substrate interface due to the interaction with Pt as

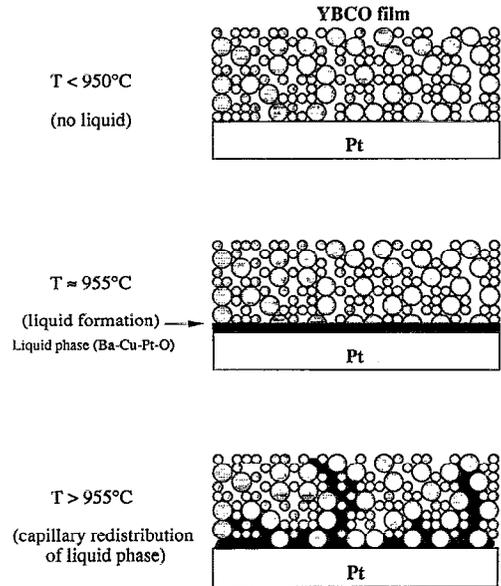


FIG. 3. Schematic representation of the proposed densification mechanism of YBCO films fired on Pt substrates.

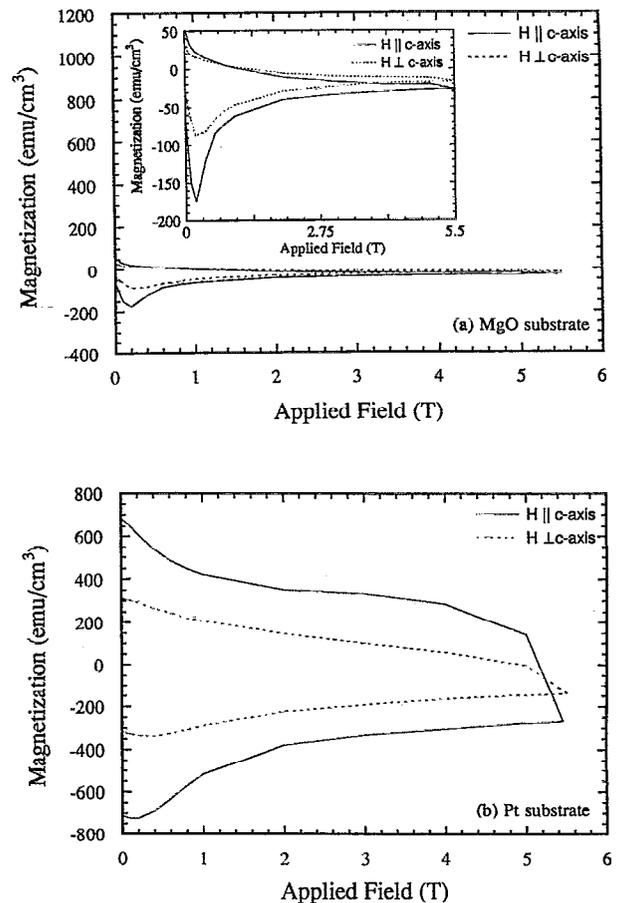


FIG. 4. Magnetization hysteresis loops at 5 K of YBCO films heated to 1020 °C on (a) MgO and (b) Pt.

shown by differential thermal analysis. This results in the formation of a liquid phase, rich in barium, copper, platinum, and oxygen, which is then distributed throughout the film via capillary forces. Yoshida *et al.* have identified three possible phases, $\text{Ba}_4\text{CuPt}_2\text{O}_9$, $\text{Y}_2\text{Ba}_2\text{CuPt}_2\text{O}_8$, and $\text{Y}_2\text{Ba}_3\text{Cu}_2\text{PtO}_{10}$, which form in the Y-Pt-Ba-Cu-O system.¹¹ Thus, the composition of this liquid phase is expected to be $\text{Ba}_4\text{CuPt}_2\text{O}_9$ based on their observations. Upon further heating, additional liquid may be formed at the film-substrate interface and redistributed within the porous network of the film. This proposed process explains not only how partial melting and densification occurred, but how the observed compositional and microstructural uniformity was developed throughout the film [refer to Fig. 2(b)]. Alternatively, if capillary redistribution of the liquid phase did not occur during densification, then one would expect a large compositional and microstructural gradient to be present.

Hysteresis measurements of aligned YBCO films were made using a high-field superconducting quantum-interference device magnetometer at 5 K and were normalized with respect to sample volume. This data are shown in Figs. 4(a) and 4(b) corresponding to films fired on MgO and on Pt, respectively. The bulk anisotropy (or degree of *c*-axis texture) within these films was evaluated from the ratio of $\Delta M (H_{\text{app}} \parallel c \text{ axis})$ over $\Delta M (H_{\text{app}} \perp c \text{ axis})$. The value of this ratio ($\Delta M_{\parallel} / \Delta M_{\perp}$) was averaged over the applied magnetic field (0–5.5 T) and found to be 1.65 and 2.20 for YBCO fired on MgO and on Pt, respectively. More importantly, the magnitude of $\Delta M (H \parallel c \text{ axis})$ was found to be approximately 15 times higher at 1.0 T and 60 times higher at 5.0 T for YBCO films fired on Pt as compared to those fired on MgO. Clearly, this results from the large difference in microstructural development (e.g., grain size, alignment, and connectivity) between these films during densification.

In summary, platinum has a strong influence on the den-

sification and melting behavior of YBCO films. We have presented the first observations of platinum induced local melting of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ below the peritectic temperature of the pure material. Production of magnetically aligned, partial melt-processed YBCO films is greatly enhanced through this approach by allowing aligned grains to remain unmelted and, hence, serve as templates for nucleation and growth upon recrystallization.

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