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CRYSTAL GROWTH AT A $Bi_2Sr_2Ca_1Cu_2O_8$/Ag INTERFACE*

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CRYSTAL GROWTH AT A $Bi_2Sr_2Ca_1Cu_2O_8$/Ag INTERFACE

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Abstract

Crystal growth at the interface between grains of $Bi_2Sr_2Ca_1Cu_2O_8$ and an overlay of 300 nm of Ag has been studied to better understand how new grains grow and why there is such good grain alignment of the c-axis in this part of tape conductors. In 50 torr of oxygen, hillocks grow at temperatures as low as 600°C, and by 800°C have grown to large grains at the interface.

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Introduction

Oxide-powder-in-tube (OPIT) methods of preparing $Bi_2Sr_2Ca_2Cu_3O_{10}$, Bi(2223), from $Bi_2Sr_2Ca_1Cu_2O_8$, Bi(2212), and other oxide powders have become very sophisticated, and high-performance materials in lengths of many hundreds of meters are fabricated by several groups. Despite this great progress, there are many mysteries concerning the phase evolution that occurs as Pb-doped Bi(2212) plus Ca_2CuO_3 and CuO are transformed into the high-performance Bi(2223) conductor. As discussed in a recent series of papers,[1-3] Pb-doped Bi(2212) is metastable at room temperature and begins to transform as the temperature is reaised at 200°C/h toward the reaction temperature of ≈830°C. As shown in Figure 1, the intensity of the (008) and (0010) X-ray lines begins to decrease at about 500°C, and by about 750°C has fallen by about 30%. The vertical line at 748°C indicates recrystallization as the sample is held at 748°C. In other work,[1] it has been reported that all of the X-ray lines decrease by about the same amount and that the magnitude of the decrease varies with ramp rate.

In an attempt to understand in more detail the cause of the amplitude decrease in X-ray peaks, samples were studied in the environmental scanning electron microscope (E-SEM) at a pressure of 7 torr of oxygen. It was found that the grains of Bi(2212) develop hillocks, or nuclei for new grain growth, on the c-axis face of the grain at the Ag interface.[3] In the microscope image, the 1000-nm-size grains coated with about 300 nm of Ag display spots typically 100 nm in diameter

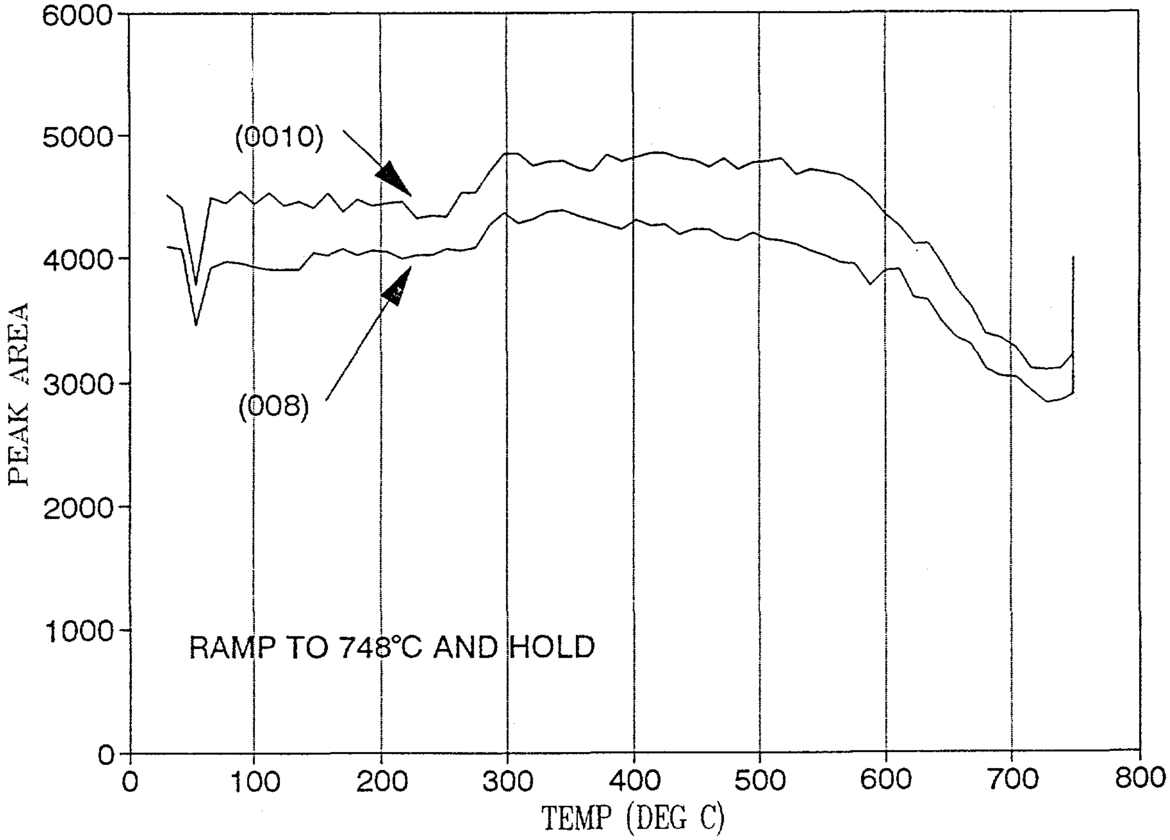

Figure 1. Plot of X-ray intensity of (008) and (0010) vs. temperature for Bi(2212).

and spaced about 300 nm apart. Hillocks develop only if there is an Ag overlay. The purpose of the work reported here is to describe further experiments done in a furnace where a full range of oxygen partial pressures could be used and the temperature is more accurately known.

Results and Discussion

Figure 2a shows an SEM picture of an Ag-coated pellet of the preliminary mixed powders that are used to make Bi(2223) tape after it has been annealed in 50 torr of oxygen at 510°C. There is a smooth Ag coating with the usual step structure in the Ag. A similar pellet annealed at 560°C shows some evidence of a few hillocks. At 601°C, there definitely are hillocks, while at 654°C, there definitely are several hillocks for each grain of Ag, as shown on Figure 2b. At 701°C, the hillocks spread in area and cover approximately 20% of the surface, and at 755°C, the hillocks appear to cover most of the surface. At 50 torr oxygen partial pressure, diminution of the Bi(2212) X-ray lines (illustrated in Figure 1) is accompanied by the growth of the hillock structure shown in Figures 2 and 3.

Chemical analysis of the hillock areas is complicated because there is an Ag overlay beneath which presumably the hillocks are growing at the Bi(2212)/Ag interface. Energy-dispersive X-ray studies to identify the chemical composition show that the "valleys" that look like Ag analyze as predominantly Ag. On the hillocks, the Ag lines are still strong but there clearly are also strong Bi and Sr lines, but no visible Ca or Cu lines.

Scanning around the sample in the SEM reveals holes in the Ag and that the regions near these holes are the first areas where the hillocks can be seen, presumably because the Ag is thinner there. An energy-dispersive X-ray study of these holes in the Ag indicate very high Cu, much higher than the Bi and Sr lines and much higher than would be expected for the Cu lines in a Bi(2212) sample. This could be CuO, but CuO lines do not appear in the crystal-structure powder pattern.

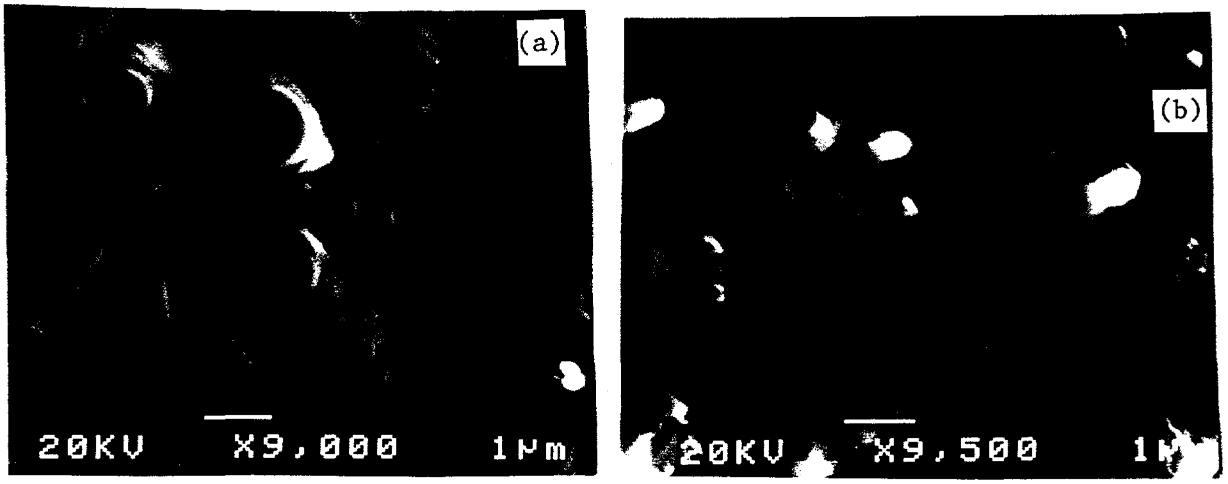

Figure 2. Growth of hillocks at 510 and 654°C for 1 h.

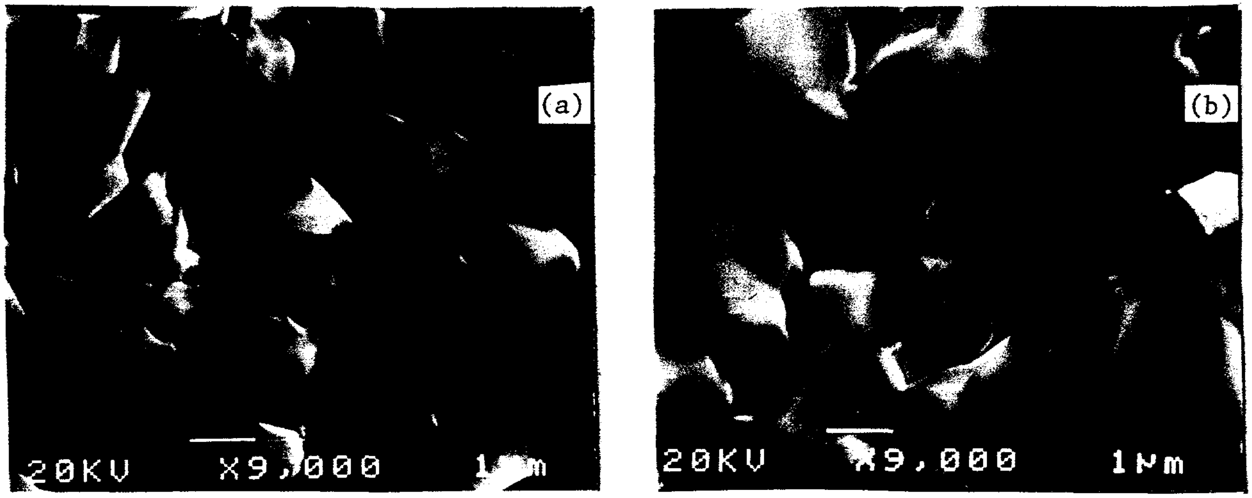

Figure 3. Growth of hillocks at 701 and 755°C for 1 h.

Conclusions

A series of measurements of hillock growth on the Bi(2212) grains has been carried out in a furnace with carefully controlled temperature and oxygen partial pressure, and the initial discovery of hillock growth in the E-SEM has been confirmed. The hillocks grow only when an Ag coating is on the pellet and they first appear at 500 to 600°C. The hillocks first grow as pillars about 100 nm in diameter to a height of several hundred nm in this low-temperature regime. About 700°C, the hillocks spread out to a width comparable to that of the Bi(2212) grain size. At 760°C, the hillocks cover most of the surface.

Acknowledgments

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