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ABSTRACT

$TlBa_2Ca_2Cu_3Dy_xO_{8+\delta}$ superconductor samples ($x = 0$–0.075) were prepared by a two-step solid-state reaction technique to investigate the impact of Dy_2O_3 (dysprosium sesquioxide) addition on the formation and the transport properties of the Tl-1223 phase. The samples were analyzed using X-ray powder diffraction, AC susceptibility, and high-harmonics temperature dependence to study the effects of Dy_2O_3 on phase formation, superconducting transition temperature T_c, and the intergrain critical current density J_c. XRD results revealed that the prepared samples are nearly single-phases Tl-1223 with a tetragonal structure. T_c is not suppressed as Dy content increased, while J_c increased up to $x = 0.025$, then decreased with a further increase in x. Our findings show that the presence of dysprosium sesquioxide promotes the high-T_c phase and enhances the transport critical current densities.

1. INTRODUCTION

Tl-based high-temperature superconductors (HTS) are among the materials with the highest critical temperatures. However, the synthesis of these materials is considered complex due to thallium's high volatility and toxicity. During the synthesis process, these materials can form many phases with different numbers of copper oxide layers: Tl-2201 (90 K) → Tl-2212 (110 K) → Tl-2223 (125 K) → → Tl-1223 (118 K).[1–5] Temperature treatments in vacuum, oxygen, or other reactive and neutral gases have become a standard practice in the synthesis of high-temperature superconductors. This method can modify oxygen stoichiometry and carrier concentration, which is essential for achieving an optimal critical temperature.[5–8] Various studies have shown that synthesizing high-purity Tl-based superconductive materials critically depends on the properties of the precursor. There are generally two approaches to address this challenge. The first approach involves synthesizing precursor with preferred characteristics through different chemical methods, such as sol-gel,[9–11] in-situ polymerization,[5] urea combustion,[12] and metal-organic methods.[13] The second approach involves adding or substituting various precursor elements to enhance chemical stabilization and reactivity.[14–17]

The effects of rare-earth elements on the superconducting properties of various materials have been reported as effective pinning centers, enhancing the current-carrying capability, improving the purity of high-temperature phases, and reducing impure phases.[18–21] Currently, research into the Tl-1223 high-temperature superconductors focuses on numerous topics, including the partial substitution or addition of rare-earth elements to enhance the critical current density and stabilize the Tl-1223 phase[22–30]

Awad *et al.* reported in 2013 that partial replacement of Ca^{2+} by Sc^{3+} ions enhanced the formation of Tl-1223 and reduced the secondary phases. Also, the authors observed that Sc-substitution did not affect the tetragonal structure of Tl-1223 but changed the lattice parameters. In addition, the grain size decreased as Sc content increased.[22] However, Muntaz *et al.* studied the partial substitution of Tl by Y and proved that adding Y did not improve the superconducting properties. The magnitude of diamagnetism sharply decreased with increasing Y content in the charge reservoir layer of the final compound. This localization at Y^{3+} reduced the number of carriers in the CuO_2 planes and promoted antiferromagnetism.[23] In another study of the (Tl, Pb)-1223 system, A. Cigaň and colleagues observed the low-level La^{3+} doping ($x = 0.04$) $(Tl_{0.6}Pb_{0.5})(Sr_{0.8}Ba_{0.2})_2Ca_2Cu_3O_{8+\delta}$–$x$$LaO_{1.5}$ increased critical temperature T_c by about 2 K and enhanced the intergrain critical current density J_c, compared to undoped samples. However, increased La concentration also raised the amount of the (Tl, Pb)-1212 impurity phase.[24] Samples with nominal composition $(Tl_{0.5}\ Pb_{0.5})Sr_{2-x}Ce_xCa_2Cu_3O_y$ with varying cerium (Ce) content were discussed by Hamdan. The onset transition temperature increased gradually by about 6 K as Ce content increased from 0 to 0.2, while the zero resistance transition temperature rapidly decreased. These results were attributed to controlling the hole concentration in the CuO_2 planes through the Ce^{4+} replacement of Sr^{2+}.[25]

In another study by Abou-Aly *et al.*,[26] the authors studied the effect of partial replacement of Ca^{2+} by Pr^{3+} (magnetic ions) and La^{3+} (non-magnetic ion) on the superconducting properties of $Cu_{0.5}Tl_{0.5}Ba_2Ca_{2-y}R_yCu_3O_{10-\delta}$ (where R = Pr and La) with $0 \le y \le 0.2$ phase. The authors found that minor substitutions of Ca by Pr and La (0.025) improved and enhanced the volume fraction of (Cu, Tl)-1223 phase and increased T_c. The enhancement in T_c was explained by the transition of the sample from an overdoped to an optimally doped state and the increase in the volume fraction of (Cu, Tl)-1223. The investigation showed that the sample with $y = 0.025$ had the best superconducting parameters, particularly for Pr^{3+} ions. The effect of partially replacing Ca^{2+} ions with Sm^{3+} in bulk $Tl_{0.8}Hg_{0.2}Ba_2Ca_{2-x}Sm_xCu_3O_{9-\delta}$ superconducting samples with $x = 0.00$–0.20 was investigated by Abou-Aly and colleagues. The results emphasized the formation of a nearly single phase of (Tl, Hg)-1223 and provided strong evidence of the successful substitution of Sm^{3+} ions in the microstructure of (Tl, Hg)-1223. The superconducting transition temperature decreased as Sm content increased, while the intergrain critical current density increased with Sm content up to $x = 0.05$, after which it declined.[27] Awad[28] investigated the effect of Cd substitution on the formation and transport properties of the $Cu_{0.5}Tl_{0.5}Ba_2Ca_2Cu_3O_{10-\delta}$ phase. It was found that low levels of Ca substitution by Cd ($x = 0.2$) enhanced the volume fraction of the (Cu, Tl)-1223 phase and consistently increased T_c and J_c. However, an increase in the concentration of Gd led to the degradation of superconductivity.

The influence of samarium (SmF_3) and lanthanum (LaF_3) fluorides on the physical and mechanical properties of $Tl_{0.8}Hg_{0.2}Ba_2Ca_{2-x}R_xCu_3O_{9-\delta-y}F_y$ superconducting phases, where R = Sm and La, with $0.00 \le x \le 0.10$, was investigated in references by Khattar *et al.*[29,30] Both substitutions did not alter the crystal structure of the (Tl, Hg)-1223 superconductor. Up to $x = 0.025$, improved superconducting transition temperature and transport critical current density were observed, but these properties decreased substantially beyond that point. La substitution resulted in significantly higher values for T_c, J_c, and (Tl, Hg)-1223 volume fractions compared to Sm substitution. These findings establish that incorporating LaF_3 is notably more effective in enhancing superconducting characteristics than SmF_3.

The motivation for the present work stems from the significant effects rare-earth oxides have on the physical and chemical properties of superconducting materials. The present study focuses on the impact of doping dysprosium (III) oxides (Dy_2O_3) on Tl-1223 material. A small amount of Dy addition is expected to enhance stability and improve superconductivity properties. The characterization of Dy_2O_3 doping on the superconducting parameters of $TlBa_2Ca_2Cu_3Dy_xO_{8+\delta}$ is investigated through X-ray diffraction, ac linear susceptibility, and third harmonics nonlinear susceptibility analyses.

2. EXPERIMENTAL

The two-step method was used to synthesize dysprosium-free and dysprosium-doped $TlBa_2Ca_2Cu_3Dy_xO_{8+\delta}$ superconductors. First, dysprosium-free and dysprosium-doped $Ba_2Ca_2Cu_3Dy_xO_y$ precursors were synthesized. Tl_2O_3 was then added to the precursor before the final sintering. The following powder materials were used: $BaCO_3$ (99.0%, Oxford Chem. Serve), $CaCO_3$ (99.98%, Oxford Chem. Serve), CuO (99.999%, Sigma-Aldrich), Dy_2O_3 (99.99%, Sigma-Aldrich), and Tl_2O_3 (99.99%, Sigma-Aldrich). Dysprosium-free and dysprosium-doped precursors were synthesized in the stoichiometric ratio Ba:Ca:Cu:Dy = 2:2:3:x, where x represents the concentrations of Dy_2O_3. Four different compositions were synthesized; namely, samples were synthesized when $x = 0.000$, 0.025, 0.050, and 0.075, respectively. Powders were mixed in these stoichiometric ratios and then ground carefully in an agate mortar. The resulting powder mixtures were calcined in an alumina crucible in air, with four intermediate grindings at 900 °C for 60 h. The calcined powders were then ground and pressed into pellets using a hydraulic press at about 500 MPa. To eliminate CO_2 from the precursors, the pellets were annealed in a tube-type furnace at 900 °C in flowing oxygen at a partial pressure of 0.2 bar for 24 h.

In the final step, the precursors were mixed with Tl_2O_3 in a ratio of 1.2:1 and ground in an agate mortar. After final grinding, the powders were pressed into discs 10 mm in diameter and 3 mm thick using a hydraulic press under a pressure of 500 MPa. The resulting samples were placed in gold crucibles, sealed in evacuated quartz tubes (up to 10^{-3} Torr), and then oxygen was pumped in at a pressure of 2 atm before sealing.[5] Subsequently, the quartz tubes were then inserted into a programmed muffle furnace, where the temperature was raised at a rate of 7 °C/min up to 700 °C and held at this temperature for 3 h, then increased at a rate of 9 °C/min to 880 °C and held at this temperature for 10 min. Finally, the samples were rapidly cooled to room temperature.

The prepared samples were characterized by X-ray diffraction (XRD, Dron-3 + PC) with CuK_α radiation. The phase method was used to study the real parts ($-4\pi\chi'$) of the linear susceptibility.

Errors in determining χ' at frequencies higher than 1 kHz did not exceed 1% when $-4\pi\chi > 0.1$, but increased proportionally with decreasing susceptibility and frequency for $4\pi\chi < 0.1$. High harmonics were used to measure the intergranular critical current densities, with a measurement error of approximately 2% when the measured signal was less than 0.2 μV, and no more than 0.5% when the signal was higher. Measurements were mainly performed at $h = 1$ Oe, $f = 20$ kHz, and $H = 0$. The Earth's magnetic field was shielded to less than 10^{-3} Oe using permalloy screens.[31–33]

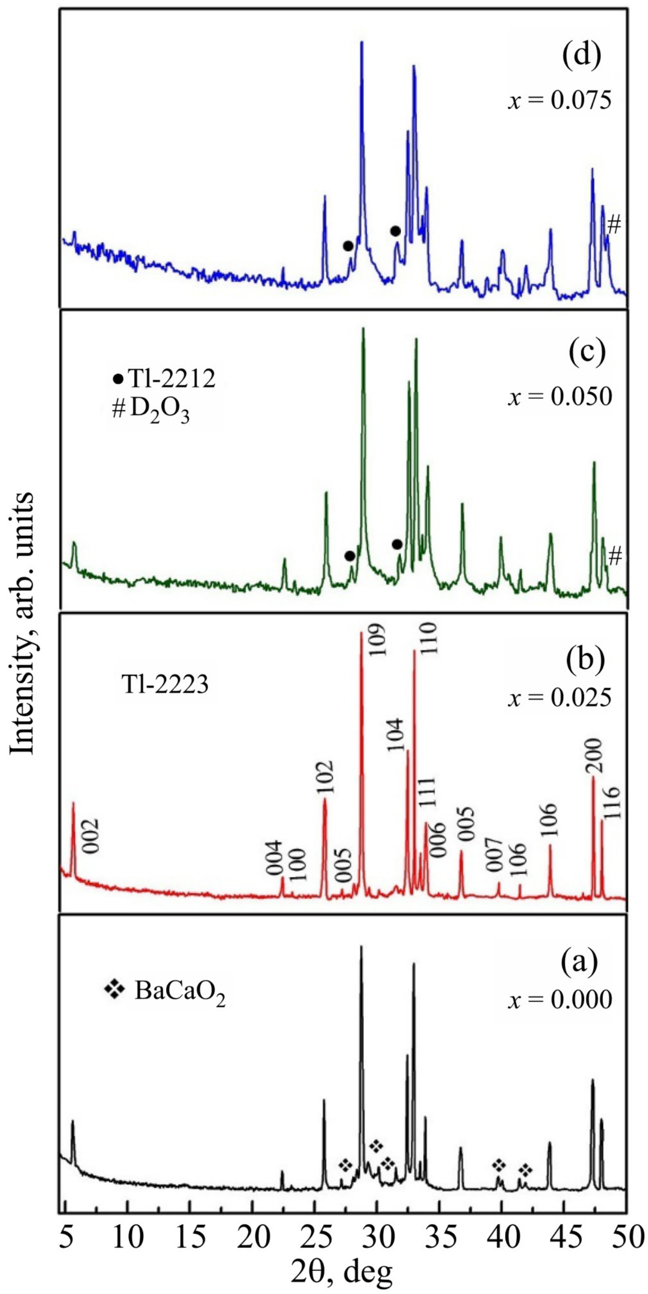

FIG. 1. XRD patterns of the undoped and doped samples $TlBa_2Ca_2Cu_3Dy_xO_{8+\delta}$, with $x = 0.000$, 0.025, 0.050, and 0.075.

3. RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction patterns of the as-sintered samples with the nominal composition of $TlBa_2Ca_2Cu_3Dy_xO_{8+\delta}$ undoped and doped samples with varying concentrations of dysprosium (III) oxides, specifically $x = 0.000$, 0.025, 0.050, and 0.075. The XRD patterns indicate that the samples are well indexed to the tetragonal unit cell of the Tl-1223 phase, with space group *P*4/*mmm*.[22,34] The undoped sample exhibits small amounts of the precursor $BaCuO_2$ phase, commonly present during the preparation of the Tl-1223 phase by solid-state reaction.[35] In the sample with a low Dy_2O_3 concentration ($x = 0.025$), a nearly single phase was formed. However, at higher doping concentrations ($x = 0.050$ and 0.075), the impurity phase increases, and the superconducting phase decreases. Additionally, at these higher Dy_2O_3 concentrations, the Tl-2212 phase appears, with its intensity increasing as the dysprosium concentration rises. The high-intensity XRD peaks of Dy_2O_3 occur at the angles 28.974°, 33.586°, and 48.242°,[36] which overlap with the peaks of the Tl-1223 phase. Consequently, in the XRD analysis of the Tl-1223 phase, only a peak at the angle 48.248° is visible.

Figure 2 presents the temperature dependences of the real part ($-4\pi\chi$) of ac susceptibility for undoped and doped samples. Measurements were conducted in zero magnetic field ($H = 0$, with $h = 1$ Oe and $f = 20$ kHz). The diamagnetic onset temperature of the superconducting transition for all samples is the same, and approximately $T_c = 118$ K. However, full diamagnetism occurs at different temperatures for each sample. The complete screening of the applied ac magnetic fields is observed at ≈ 97 K for $x = 0.000$, 101 K for $x = 0.025$, 90 K for $x = 0.050$, and 87 K for $x = 0.075$.

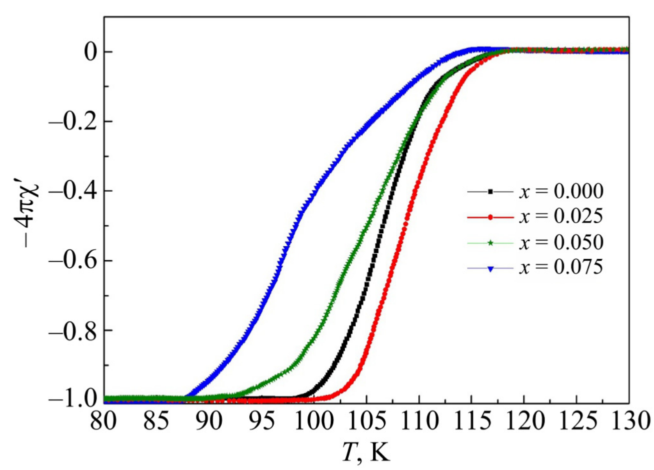

FIG. 2. Temperature dependences of the real $-4\pi\chi'$ parts of ac susceptibility for undoped and doped samples, at $h = 1$ Oe, $f = 20$ kHz, and $H = 0$.

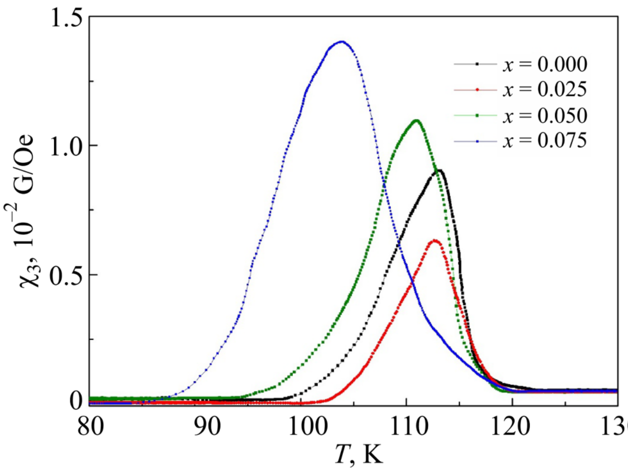

FIG. 3. Temperature dependences of the amplitude of the third harmonics $\chi_3(T)$, at $h = 1$ Oe, $f = 20$ kHz, and $H = 0$.

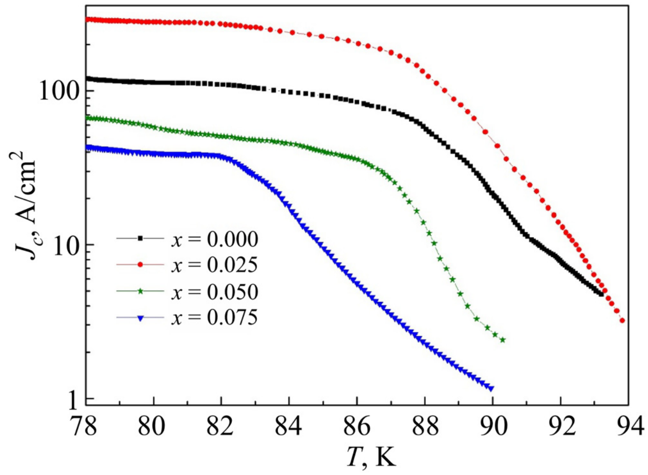

FIG. 4. Dependence of the critical current densities $J_c(T)$ on temperature for undoped and doped samples.

The high harmonics nonlinear susceptibility was first interpreted using Bean's critical-state model,[37,38] which suggests that the amplitude of each harmonic is inversely proportional to the critical current density ($V_n \sim 1/J_c$). It is well known that applying a sinusoidal excitation to a nonlinear system, it results in a non-sinusoidal response. The amplitudes of the high harmonics decrease only slightly as the harmonics number increases. Therefore, it is more practical to use harmonics with low numbers; in this study, we use the third harmonic. It should be noted that measurements of higher harmonics are performed at frequencies that are multiples of the fundamental frequency, effectively creating a zero-background scenario. High harmonics are widely used to characterize superconducting materials, providing important information such as the critical temperature and transport critical current density.[24,32,39]

Figure 3 presents the temperature dependence of the magnitude of the third harmonics χ_3 at $h = 1$ Oe, $f = 20$ kHz, and $H = 0$. $\chi_3(T)$ curve exhibits a one-peak plot, which is attributed to the penetration into the Josephson medium formed by weak links between the crystallites, associated with intergranular critical current density. Similar to the $-4\pi\chi'$ dependence for undoped and high-doped samples, the critical temperature T_c remains the same, but there are differences in peak intensity and width. The location and characteristics (intensity, width) of the peak on the $\chi_3(T)$ curves indicate that a dysprosium concentration $x = 0.025$ positively influences on the electrical properties of Tl-1223 materials.

Figure 4 compares the values of the transport critical current density J_c versus temperature for Dy-free and Dy-doped Tl-1223 samples derived from the $\chi_3(T)$ data.[31,32] The figure shows that the J_c for the Dy-doped sample (0.025) increases significantly more than for the Dy-free sample. The J_c value for the undoped sample is approximately 120 A/cm^2, while the highest value of critical currents is observed in the $x = 0.025$ sample at 290 A/cm^2. Thus, a small addition of dysprosium to Tl-1223 significantly enhances the transport critical current density.

4. CONCLUSION

We have successfully prepared $TlBa_2Ca_2Cu_3Dy_xO_{8+\delta}$ superconductor samples, with x ranging from 0 to 0.075, using a two-step solid-state reaction method. X-ray results revealed that the undoped sample contained small amounts of the precursor $BaCuO_2$ phase. In contrast, the sample with a dysprosium concentration of 0.025 formed nearly a single phase with a tetragonal structure. The temperature dependence of the ac susceptibility and high-harmonics data indicated that T_c does not suppress as Dy content increased, while J_c increased up to $x = 0.025$ and then decreased with further increases in x. Consequently, a Dy concentration of 0.025 enhances phase purity, diamagnetism, and the value of J_c.

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