

**EFFECT OF NANOSIZE MgO PARTICLES ADDITION
ON FORMATION AND SUPERCONDUCTIVITY OF
Tl_{0.9}Bi_{0.1}Sr_{1.95}Ta_{0.05}Ca_{0.9}Y_{0.1}Cu₂O_{7- δ} CERAMICS**

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ABSTRACT

In this paper, nanosize MgO particles were added to Tl_{0.9}Bi_{0.1}Sr_{1.95}Ta_{0.05}Ca_{0.9}Y_{0.1}Cu₂O_{7- δ} superconductor in various weight fraction between 0 wt.% to 0.8 wt.% before sintering using the conventional solid state synthesis method. The phase formation and the microstructure of the samples were studied by XRD and SEM, respectively. The highest critical temperature $T_{c\ zero}$ of 80 K was observed at 0.2 wt.% MgO and T_c was gradually suppressed for higher MgO additions. The effect of the MgO addition on J_c revealed maximum J_c at 0.2 wt.% MgO addition. SEM investigations revealed no difference in microstructure for the pure sample and the MgO added samples. The results showed that a small amount of MgO addition (0.2 wt.%) in Tl-1212 enhanced J_c and this is suggested as due to increased flux pinning in the sample due to the MgO addition.

INTRODUCTION

The most challenging development of high- T_c superconductors are the practical usage and application of the materials. Intensive research worldwide has been done on the desired transport properties of superconductors for their wider practical application. One of the most important transport properties of high- T_c superconductors is the behavior of high critical current density, J_c in magnetic field. For example, previous reports revealed that J_c in polycrystalline Bi-2212 is dominated by weak-link behavior where increasing magnetic field causes a rapid drop in J_c . [1]. In addition, a large anisotropy and weak coupling across grain boundaries can also caused current density to decrease [2]. However, the introduction of artificial flux pinning centers can improve the flux pinning capability [3]. Several reports have shown that the introduction of nanosize particles of MgO [3], Al₂O₃ [2], SiC [4], ZrO₂ [5] and SrZrO₃ [6] into high- T_c superconductors improve effectively J_c value of several high-temperature superconductors. However, there are very few reports about the effect of nanosize MgO particles addition on Tl-based superconductors. In this paper, the effects of addition of nanosize MgO particles in various weight fractions on critical current density, J_c and flux pinning of Tl1212 superconductor synthesized from Tl_{0.9}Bi_{0.1}Sr_{1.95}Ta_{0.05}Ca_{0.9}Y_{0.1}Cu₂O_{7- δ} starting composition was studied. The behaviour of critical current density, J_c in external magnetic field and XRD and SEM investigations are presented and discussed.

EXPERIMENT DETAILS

Superconducting powder with nominal composition $Tl_{0.9}Bi_{0.1}Sr_{1.95}Ta_{0.05}Ca_{0.9}Y_{0.1}Cu_2O_{7-\delta}$ were prepared by the two-step precursor and solid state synthesis method. Appropriate amounts of high purity ($\gg 99.9\%$) powder of $SrCO_3$, Ta_2O_5 , CaO , Y_2O_3 and CuO were mixed and ground in an agate mortar followed by calcination in air at $900^\circ C$ for 48 hours with several intermittent grindings. Appropriate amounts of Tl_2O_3 and Bi_2O_3 were then mixed to the precursor. The mixture was then pressed into pellets under the load of 3.5 - 4 tons using a hydraulic press. The pellets were then heated at $1000^\circ C$ for around 3 minutes in a box furnace with oxygen flow and slow cooled at $10^\circ C/hours$ to room temperature. The samples were reground and added with appropriate amounts (0.1 to 0.8 wt.%) of MgO powders with average size of 36 nm purchased from Nanostructured & Amorphous Materials, Inc., USA. The mixture was reground and repressed into pellets before final sintering at $1000^\circ C$ for 3 minutes under oxygen flow.

Temperature dependent room temperature resistivity was measured using the standard DC four-point-probe technique in conjunction with a Janis closed cycle He cryostat. To measure the critical current density J_c , all the samples were carefully cut into bar shapes of approximate dimensions around $13.00mm \times 1.15mm \times 2.85mm$. The critical current densities were measured by the standard DC four-point-probe technique using a criterion of $1\mu V/mm$. The measurements were carried out in zero and applied magnetic field directed in two directions: parallel and perpendicular to the sample's wide surface (SWS). The powder X-ray diffraction (XRD) analyses were performed using Bruker Model D8 Advanced Diffractometer with $Cu-K\alpha$ source. The microstructures of the samples and EDX were analyzed using JEOL model JSM 6400 scanning electron microscope.

RESULTS AND DISCUSSION

The powder XRD patterns of all samples revealed $Tl-1212$ as the major phase and was indexed based on a tetragonal unit cell (space group, $P4/mmm$). The 1201 phase constitutes the minor phase in addition to several unidentified peaks. Figure 1 shows XRD patterns of $Tl_{0.9}Bi_{0.1}Sr_{1.95}Ta_{0.05}Ca_{0.9}Y_{0.1}Cu_2O_{7-\delta}$ with 0 and 0.2 wt.% MgO . Reflection peaks due to the 1201 phase are indicated by asterisk (*). $T_{c\ onset}$, $T_{c\ zero}$, room-temperature resistivity at 300K, 1212:1201 phase volume ratio and lattice parameters of all samples are listed in Table 1. An initial increase in the 1212 phase was observed for the 0.1 wt. % MgO added sample but decreases for samples with 0.2 to 0.8 wt. % MgO . It was also observed, that there is an increase in both a - and c - lattice with addition of small amount of MgO (0.1 and 0.2 wt. %). The increase in both the lattice parameters indicates that some form of low level substitution may have taken place. However, there is no systematic change in the lattice parameters for higher MgO addition.

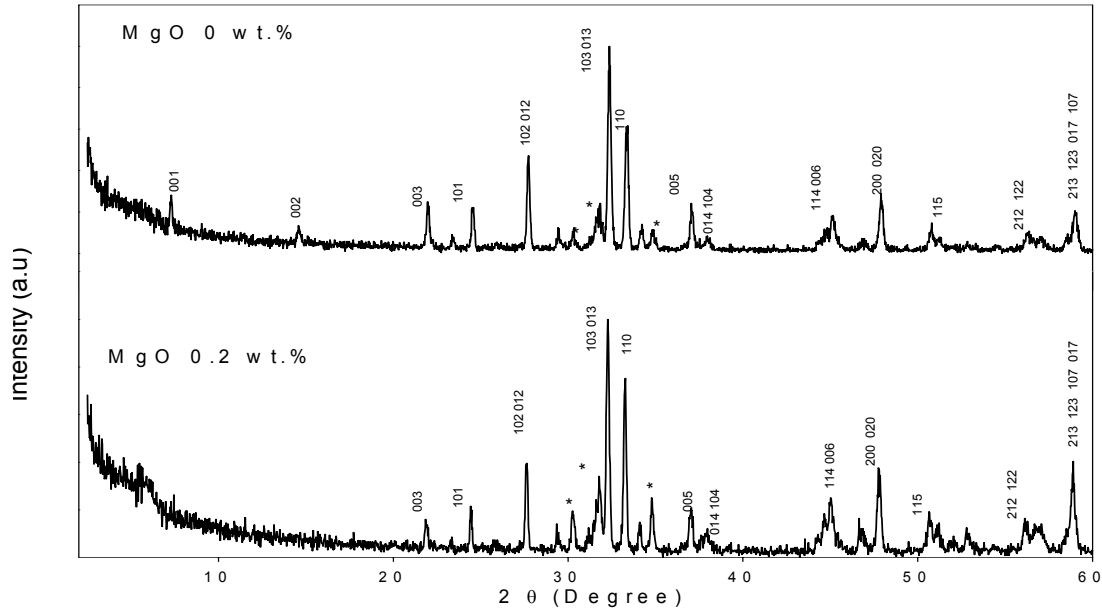


Figure 1: Powder XRD patterns for 0 wt.% MgO and 0.2 wt.% MgO showing dominant 1212 phase. Peaks due to 1201 phase are indicated by asterisk (*).

Table 1: $T_{c \text{ onset}}$, $T_{c \text{ zero}}$, room-temperature resistivity at 300K, 1212:1201 phase volume ratio and lattice parameters of $Tl_{0.9}Bi_{0.1}Sr_{1.95}Ta_{0.05}Ca_{0.9}Y_{0.1}Cu_2O_{7-\square}$ with various amounts of added MgO.

Sample MgO content (wt.%)	$T_{c \text{ onset}}$ (K)	$T_{c \text{ zero}}$ (K)	Resistivity at 300K ($\times 10^{-3} \Omega \cdot \text{cm}$)	1212:1201 volume ratio (vol %)	1212 unit cell a (Å)	c (Å)
0 wt.%	95	65	0.7	81 : 19	3.788	12.108
0.10 wt.%	76	60	0.8	83 : 17	3.794	12.116
0.20 wt.%	96	80	1.5	76 : 24	3.821	12.138
0.40 wt.%	70	45	0.4	75 : 25	3.801	12.124
0.60 wt.%	87	68	0.4	75 : 25	3.809	12.145
0.80 wt.%	80	58	0.3	73 : 27	3.815	12.141

The results of the temperature dependent resistance measurements on all samples are shown in Figure 2. All samples showed metallic normal state behavior with the highest $T_{c \text{ zero}}$ of 80K recorded for the 0.2 wt.% MgO added sample. Further addition of MgO caused $T_{c \text{ zero}}$ to decrease. The increase in $T_{c \text{ zero}}$ for the 0.2 wt.% MgO added sample may be due to the low level substitution mentioned above which caused changes in internal pressure of the system.

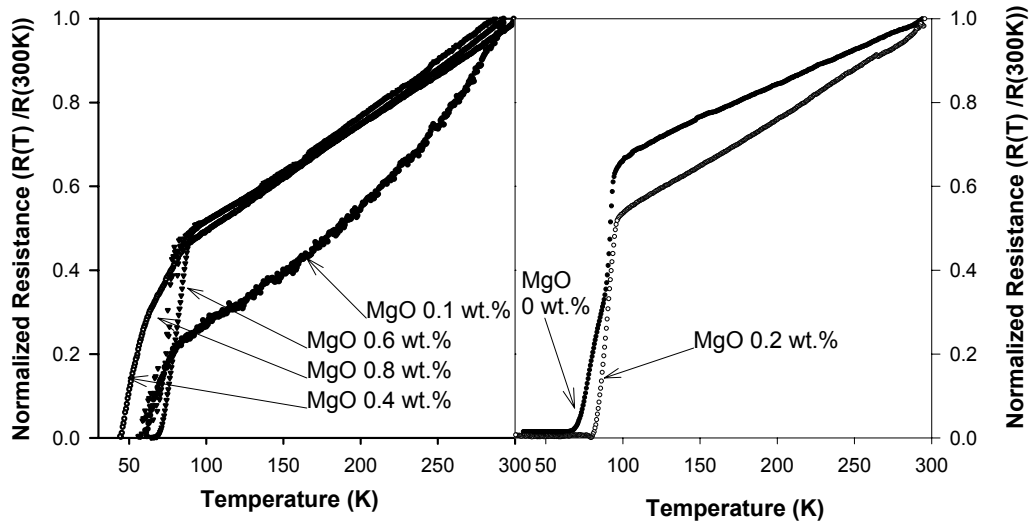


Figure 2: Normalized resistance versus temperature curves for $Tl_{0.9}Bi_{0.1}Sr_{1.95}Ta_{0.05}Ca_{0.9}Y_{0.1}Cu_2O_{7-x}$ with various amounts of MgO.

SEM micrographs for all samples showed porous microstructure consisting irregular shaped grains with average grains size between 2-5 μm . Figure 3 shows SEM micrographs for samples with 0 wt.% and 0.2 wt.% MgO. It can be observed that the 0.2 wt. % sample showed more regular shaped grains and higher porosity compared to the pure sample. Similar shaped grains are observed for all the MgO added samples. The presence of Mg was confirmed by EDX analysis.

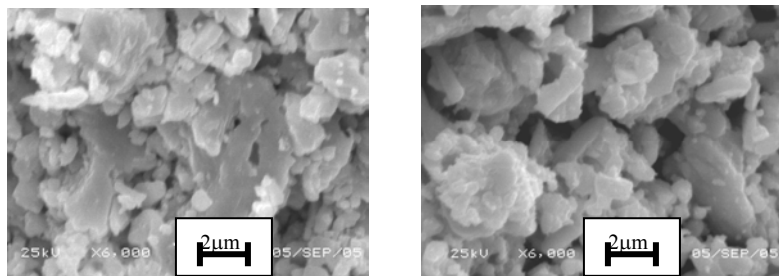


Figure 3: SEM micrographs for Tl1212 samples with 0 wt.% MgO (left) and 0.2 wt.% MgO (right)

Figure 4 shows critical current density measured between 20 K and 50 K for all samples. The highest J_c recorded for all temperatures (Figure 4(a)) is for the sample with 0.2 wt.% MgO. It is clear that addition of a small amount of MgO enhanced the current density capacity of the Tl-1212 ceramic. At 20 K, J_c value for 20 wt. % MgO added sample was more than double that of the 0 wt. % MgO. However, when larger amount of MgO (> 0.2 wt.%) was added, J_c drops gradually. Figure 4(b) shows the reduction of J_c with increasing temperature for all samples. This behaviour is probably due to thermally activated flux creep as previously suggested for other high-temperature superconductors [7].

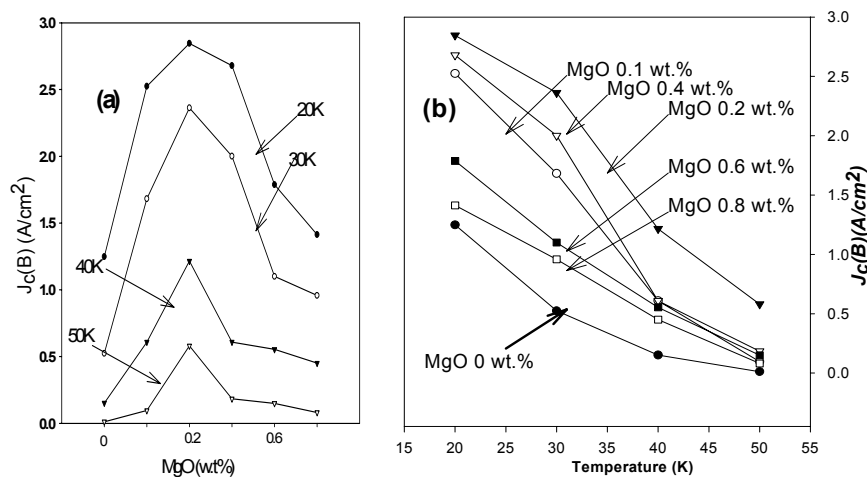


Figure 4: Critical current density, J_c of Tl-1212 versus a) added MgO (wt.%) and b) temperature (K)

Figure 5 shows the normalized critical current density versus applied magnetic field applied parallel and perpendicular to samples wide surface (SWS) for samples with 0, 0.2 and 0.4 wt.% MgO. The samples showed rapid drop of J_c at low fields (<0.1 Tesla) before a slower deterioration at higher fields (>0.1 Tesla). This indicates that all the samples display effects of presence of weak links at lower fields and at higher fields (above 0.1 T) the weak links are decoupled and a network of strong links takes over for transportation of supercurrent. Addition of MgO effectively reduced the J_c drop at low fields with the 0.2 wt.% MgO sample showing the smallest drop of J_c . The enhanced J_c with 0.2 wt.% MgO added sample is probably due to increased magnetic flux pinning. The enhanced J_c cannot be due to the volume of 1212 phase as there is only a small difference in the 1212 phase volume between the 0.2 wt.% MgO added sample and that of the 0 wt.% MgO sample. Microstructural differences cannot be considered as a factor as the samples did not show significant difference in microstructure. On the other hand, the MgO particles may not directly act as flux pinning centers as the MgO particles size of 36nm is larger than the coherent length of Tl-1212 which is around 3nm. As such, it is suggested that introduction of nanosize MgO into Tl-1212 may cause secondary defects at the interfaces between MgO particles and the superconducting matrix which act as flux pinning centers in Tl-1212 phase [2].

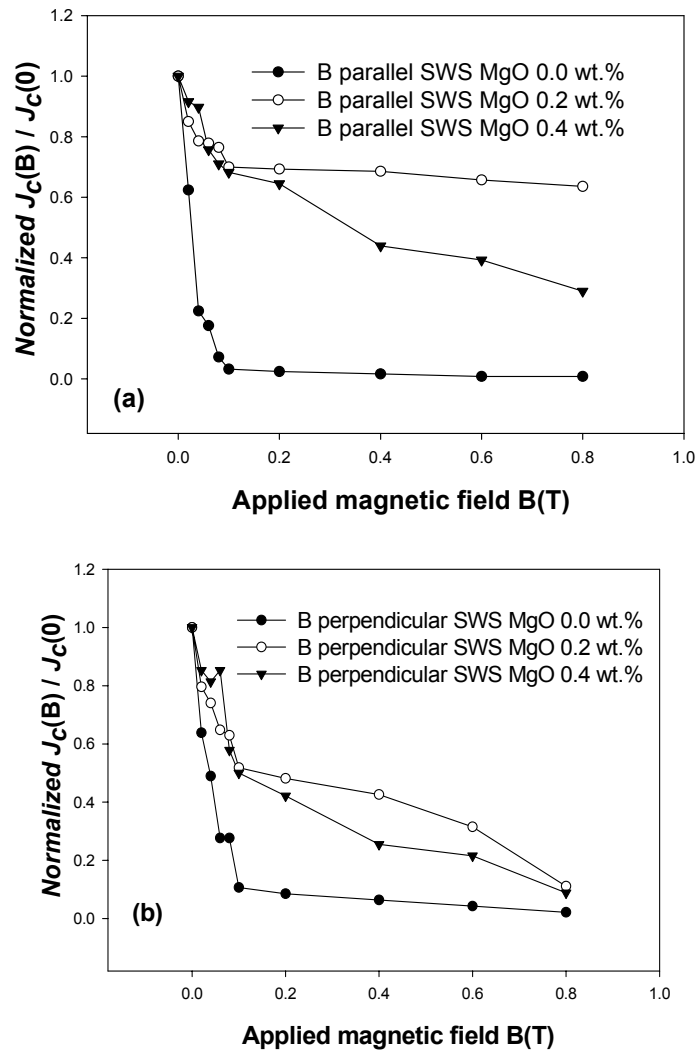


Figure 5: Normalized critical current densities, J_c versus applied magnetic field in a) parallel SWS direction b) perpendicular SWS direction for 0 - 0.4 wt.% MgO at 20K.

CONCLUSION

In conclusion, effects of nano MgO particles on formation and superconductivity of $Tl_{0.9}Bi_{0.1}Sr_{1.95}Ta_{0.05}Ca_{0.9}Y_{0.1}Cu_2O_{7-8}$ have been investigated. The best superconducting behavior and highest J_c was observed for the sample with 0.2 wt. % MgO. The behavior of J_c in external magnetic field indicates presence of weak links at lower fields (< 0.1 Tesla) and strong links at higher fields (> 0.1 T). Addition of 0.2 wt. % MgO produced faster decoupling of weak links at low fields. The enhanced J_c behavior as a result of MgO addition is suggested as due to increased magnetic flux pinning of the sample.

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