Influence of sintering temperature on microstructure, critical current density and pinning potential of superconducting Bi$_{1.6}$Pb$_{0.5}$Sr$_{1.8}$Dy$_{0.2}$Ca$_{1.1}$Cu$_{2.1}$O$_{8+\delta}$ ceramics

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ABSTRACT

The influence of sintering temperature on the microstructure, critical current density ($J_C$), pinning potential values ($U_0$) and flux pinning properties of Bi$_{1.6}$Pb$_{0.5}$Sr$_{1.8}$Dy$_{0.2}$Ca$_{1.1}$Cu$_{2.1}$O$_{8+\delta}$ superconductor has been investigated. The samples are prepared by the solid-state route and sintered at temperatures ranging from 846 to 860 °C. A systematic correlation between the sintering temperature, Lotgering index, $J_C$, $U_0$ and flux pinning properties has been found. The samples sintered at lower sintering temperature (846°C) have more grain boundaries with smaller grains while those sintered at a higher temperature (856°C) contain larger grains with good texturing. The flux pinning force ($F_P$) calculated from the field dependent $J_C$ values shows that the irreversibility lines (IL) of the Dy-doped samples shift towards higher fields to different extents depending on the sintering temperature. The maximum value of $F_P = 1697$ kN m$^{-2}$ is obtained for the sample sintered at 846°C and the peak position of $F_P$ is obtained at 0.96 T as against 616 kN m$^{-2}$ and 0.52 T for the sample sintered at 856°C. The $U_0$ values calculated by Anderson’s function is maximum for the sample sintered at 846°C. But the self-field $J_C$ value of this sample is lower than that of the samples sintered at 856°C. The samples sintered at 856°C show best self-field $J_C$ due to the improved microstructure. The changes in microstructure followed by very high enhancement of self-field $J_C$, $J_C(B)$ characteristics, $F_P$ and $U_0$ values within a narrow temperature range, are of great scientific and technological significance and the results are explained on the basis of microstructural variation with respect to sintering temperature, hole optimization and formation of point defects due to the doping of Dy atoms in Bi$_{1.6}$Pb$_{0.5}$Sr$_{1.8}$Dy$_{0.2}$Ca$_{1.1}$Cu$_{2.1}$O$_{8+\delta}$ system.

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1. Introduction

Since the discovery of Bi-based high temperature superconductors (HTSs), there have been many studies to improve the superconducting properties, in particular the critical temperature ($T_C$) and critical current density ($J_C$) of Bi-2212 and Bi-2223 systems. This is because $T_C$ and $J_C$ are the parameters of primary importance for potential applications of HTS. However the $J_C$ value is primarily limited due to the insufficient flux pinning properties [1]. Also the crystal structure of Bi-2212 is highly two dimensional (2D) with alternate stacking of superconducting CuO$_2$ layers and poorly conducting thick blocking layers, which reduce the Josephson coupling between the superconducting CuO$_2$ layers. The transport and magnetic properties are highly anisotropic and the in-plane conductivity along the CuO$_2$ layers is much higher than that of the out-of-plane conductivity along the blocking layers [2]. Due to the weak coupling between the superconducting layers, the 3D vortices melt into 2D pancake like vortices at higher temperatures and fields. The 2D pancake vortices are easily de-pinned causing flux flow and energy dissipation during transport current flow. In order to sustain high non-dissipative transport currents at higher temperatures and magnetic fields the vortices must be pinned. Therefore numerous efforts have been made to enhance the flux pinning properties of this system, and hence the $J_C$ value, by introducing effective pinning sites [3,4] into the system by different methods [5,6]. The irradiation technique is an effective way to introduce pinning centers [5], but it is not suitable for long wires. The other important technique is doping of the material which changes the charge carrier concentration. This change is one of the most important effects for the study of superconducting cuprates because it primarily determines the various physical properties of the system, such as transition temperature ($T_C$), electrical and

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magnetic properties [7]. Also, doping of impurity atoms creates
point defects in the system which can act as flux pinners. Therefore
doping is an effective method for improving the structural, trans-
port and superconducting properties of Bi-based superconductors
and to make the material suitable for the application at higher
temperatures and magnetic fields. Hence the effect of substitution
of cations on the superconducting properties of Bi-2212 system has
been a subject of great interest for both technologists and
theoricians.

It has been experimentally found that the Pb doping at Bi-site in
Bi-2212, increases the c-axis conductivity by more than one order
of magnitude and reduces the electromagnetic anisotropy significantly [8]. The flux pinning property is significantly improved by Pb
doping and the Pb doped Bi-2212 has shown enhanced $J_c$ in applied
fields, compared to the Pb free Bi-2212 system [9]. On the tech-
nological side the substitution of Pb for Bi improves the super-
conducting properties of both bulks and tapes [10] and the
nological side the substitution of Pb for Bi improves the super-
conductor sintered between 846 and 860 °C.

2. Experimental

The pure, i.e., Dy free (Bi$_{1.6}$Pb$_{0.5}$Sr$_{1.8}$Ca$_{1.1}$Cu$_{2.1}$O$_{8+\delta}$) and Dy-
doped (Bi$_{1.6}$Pb$_{0.5}$Sr$_{1.8}$Dy$_{0.2}$Ca$_{1.1}$Cu$_{2.1}$O$_{8+\delta}$) samples were prepared by
solid-state method using high purity oxides and carbonates (Aldrich
>99.5%). The ingredients were mixed and ground using a planetary
ball-mill (FRISCH Pulversette 6) and subjected to a three-stage
calcination process in air, at 800 °C/15 h + 820 °C/40 h + 840 °C/
60 h. Intermediate grinding was done at each stage of calcinations.
Samples were then pelletized at a pressure of 500 MPa. The pellets
of pure sample [undoped (Bi, Pb)-2212] were heat treated at 848 °C
(here after denoted as P48), which is the optimized sintering
temperature for this sample, for 120 h (60 h + intermediate
pressing at a pressure of 500 MPa + 60 h). The pellets of Dy-doped
samples were grouped into eight sets. The first set was heat treated
at 846 °C (D46) and the remaining sets at 848 °C (D48), 850 °C
(D50), 852 °C (D52), 854 °C (D54), 856 °C (D56), 858 °C (D58), and
860 °C (D60), respectively, for 120 h (60 h + 60 h), with one inter-
mediate pressing under the same stress. The heat treatment was
done in a large volume programmable muffle furnace with a Euro-
therm (Model: 2404) temperature controller. The stability and
accuracy of the temperature were ±0.5 K.

Phase analysis of the samples was done using XRD (Philips X’pert
Pro) equipped with an X’celerator and a monochromator at the
diffraction beam side. For all samples, scans were made in 2θ and at
0.01°/step. All the scans were done using Cu Kα radiation with a
tube voltage of 40 kV and current 30 mA by exposing a constant
area of the sample. The phase identification of the samples was
performed using X’pert High score software in support with the
ICDD-PDF-2 database. A measure of texture known as Lotgering
index ($F$) is calculated from the peak intensity of the XRD patterns
of sintered pellets and the corresponding randomized powder.
Lotgering index is given by,

$$ F = \frac{P_a - P_0}{1 - P_0} \quad (1) $$

where, $P = \Sigma I_{00l}/\Sigma I_{hkl}$ of the pellets surface and $P_a$ refers to P
measured for the pellets and $P_0$ for the corresponding randomized
powder. The microstructural and elemental analyses of the samples
were done using SEM (JEOL JSM 5600 LV) and EDAX, respectively.
For electrical measurements the samples were cut into rectangular
bar of dimension $12 \times 3 \times 1$ mm$^3$. The transition temperature ($T_c$)
of the samples was determined by the four-probe DC resistance
method, by cooling the sample in a liquid N$_2$ cryostat. In order
to minimize the contact resistance, four silver strips were fixed on
to one surface of the pellet during pressing. Leads of high quality
copper were soldered to silver strips. The outer two leads were
connected to a programmable DC current source (Keithley model:
220) and a constant current of 10 mA was used for the resistance
measurement. The voltage drop between the inner two leads was
measured using a programmable nano-voltmeter (Keithley model:
181). The temperature of the sample was monitored by a tempera-
ture controller (Lakeshore model: L340) using a temperature
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GPB–PC interfaced with a PC. The transport critical currents of the
samples in self and applied fields, large

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Fracture surface and cross-sectional view of pellets is shown in
Fig. 1 (a) The normalized XRD patterns of the pure and Dy-doped
(Bi,Pb)-2212 pellets after the last stage heat treatment and (b) the normalized XRD peaks of the pure and
Dy-doped (Bi, Pb)-2212 for the (008) plane at around 2θ~23.1°.

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The XRD patterns of the pellets obtained after the last stage heat treatment is normalized with standard XRD patterns of pure (Bi, Pb)-2212 sample and the resultant pattern is shown in [Fig. 1(a)]. It show that all the samples contain only the peaks corresponding to the (Bi, Pb)-2212 phase and no peaks of any secondary phase containing Dy or any other cation were detected at this stage within the detection limit of the instrument. The absence of secondary phases suggests that all the reactant phases are converted into the (Bi, Pb)-2212 and the doped Dy is incorporated into the crystal lattice of (Bi, Pb)-2212 superconductor. Also, Fig. 1(a) shows that when the sintering temperature of the Dy-doped samples is increased, the peak height corresponding to the (00l) planes also increases up to 856 °C. In order to bring out the increase in peak height of the (001) planes, the (008) peaks of all samples are separately shown with better resolution in Fig. 1(b). This shows that the texturing increases with increase of sintering temperature (up to 856 °C) and thereafter the peak height gradually decreases which show that the texturing decreases beyond 856 °C. This figure also shows that the (008) peaks of the Dy-doped sample shift towards higher angle [peak shift δ(2θ) = 0.23°] with respect to the position of undoped (Bi, Pb)-2212 sample. Even though all the XRD patterns are taken under identical condition, the Dy-doped samples show a peak shift of around 0.23°. This also supports that the doped Dy atoms entered in to the crystal structure of (Bi, Pb)-2212 system. The lattice parameter of the pure and Dy-doped samples were calculated by considering an orthorhombic symmetry for (Bi, Pb)-2212 system, which shows that the lattice parameter values are independent of sintering temperature. The c-axis lattice parameter of all the Dy-doped samples is 30.765 Å with in the accuracy of 0.002 Å. Similar behaviour has been observed in the case of a and b axes where 5.403 ± 0.002 Å and 5.396 ± 0.002 Å are the a-axis and b-axis parameters, respectively. A contraction in c-axis length is observed in all the Dy-doped samples as compared with the undoped samples (30.883 ± 0.002 Å), indicating the incorporation of Dy atoms in to the system. Because when the Dy3⁺ entered into the system, the interlayer distances Δz(Cu–Ca–Cu) and Δz(Ca–Sr) expand but Δz(Sr–Bi), Δz(Bi–Bi) and Δz(Sr–Bi–Bi–Sr) shrink because the extra oxygen resides in the Bi-O double layers. The extra oxygen balances the increased valency due to the replacement of Sr2⁺ by Dy3⁺. Consequently the net positive charge in the Bi-O layers reduces. Hence, the repulsion between them is reduced and the distance between all the layers along the c-axis in the structure contracts [14]. Again, the Lotgering index calculated from XRD data (Table 1) is found to be dependent on the sintering temperature. As the sintering temperature increases, the F value of the doped samples increases monotonically up to a temperature of 856 °C (F = 0.90), which indicates the increase of texturing with increase of sintering temperature. It is also noted that the undoped sample shows the best F value (0.92).

![Fig. 2.](image)

Table 1

<table>
<thead>
<tr>
<th>Samples</th>
<th>Tc (K)</th>
<th>Lotgering index (F)</th>
<th>Fpmax (kN m⁻³)</th>
<th>Field at which Fpmax occurs (T)</th>
<th>U₀ at 0.28 T (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P48</td>
<td>80.6</td>
<td>0.92</td>
<td>14</td>
<td>0.12</td>
<td>0.072</td>
</tr>
<tr>
<td>D46</td>
<td>92.2</td>
<td>0.70</td>
<td>2228</td>
<td>0.06</td>
<td>0.731</td>
</tr>
<tr>
<td>D48</td>
<td>92.2</td>
<td>0.75</td>
<td>1874</td>
<td>0.84</td>
<td>0.550</td>
</tr>
<tr>
<td>D50</td>
<td>92.2</td>
<td>0.77</td>
<td>1707</td>
<td>0.76</td>
<td>0.483</td>
</tr>
<tr>
<td>D52</td>
<td>92.2</td>
<td>0.82</td>
<td>1465</td>
<td>0.68</td>
<td>0.415</td>
</tr>
<tr>
<td>D54</td>
<td>92.2</td>
<td>0.88</td>
<td>1199</td>
<td>0.56</td>
<td>0.318</td>
</tr>
<tr>
<td>D56</td>
<td>92.2</td>
<td>0.90</td>
<td>1029</td>
<td>0.52</td>
<td>0.285</td>
</tr>
<tr>
<td>D58</td>
<td>92.2</td>
<td>0.89</td>
<td>1403</td>
<td>0.60</td>
<td>0.354</td>
</tr>
<tr>
<td>D60</td>
<td>92.2</td>
<td>0.86</td>
<td>1523</td>
<td>0.64</td>
<td>0.409</td>
</tr>
</tbody>
</table>

Fig. 2. The SEM images of the fractured surfaces of pure (P48) and Dy-doped (Bi,Pb)-2212 pellets after the last stage heat treatment.
temperature is observed. For example, in D46 the characteristic flaky morphology of pure (Bi, Pb)-2212 grains transformed into round grains with reduced texture. As we go from D46 to D56, the flaky morphology and texture reappear with gradual improvement in microstructure with respect to increase in sintering temperature. For D58 and D60 the microstructure again deteriorates which shows that the optimum sintering temperature for obtaining the best microstructure for the Dy-doped samples is <858°C. Also this improved microstructure plays an important role in increasing the self-field $J_c$ of the system. Because the weak links associated with the grain boundaries are known to limit the $J_c$ values of superconductors and the possible reasons for the formation of these weak links are the misorientation of grain boundaries and the compositional variations at the grain boundaries. In our case the maximum misorientation is observed for D46 and it gradually decreases with increase of sintering temperature and it is least for sample sintered at 856°C (D56) as shown in Fig. 2. Here among the Dy-doped samples D56 has least number of grain boundaries and hence have minimum amount of weak links. Thus by tuning the sintering temperature, the effect of weak links in the system can be minimized. Compositional analysis has performed by EDAX using spot mode and the EDAX spectra of the pure (P48) and Dy-doped (Bi, Pb)-2212 (D56) grains are shown in Fig. 3. The presence of Dy is detected in the Dy-doped (Bi, Pb)-2212 grains with a corresponding reduction in Sr. This supports that the Dy atoms are successfully doped into the (Bi, Pb)-2212 system.

The variation of $T_c$ with Dy content and sintering temperature is given in Table 1 and it shows that the Dy-doped samples have a much higher $T_c$ values (92.2 K) compared to the undoped sample (80.6 K). It is found that all the Dy-doped samples show same $T_c$ values (Table 1) irrespective of the sintering temperature and this is because the same amount of Dy is present in all of the doped samples. Again there is a systematic trend observed between the sintering temperature and self-field $J_c$ values (Table 1). In the case of Dy-doped samples, the lower sintering temperature (846°C) resulted in lower $J_c$ values (9100 kA m$^{-2}$) at 64 K. The $J_c$ value increase with increase of sintering temperature as shown in Fig. 4(a) and finally the sample D56 show maximum $J_c$ (27600 kA m$^{-2}$). The enhancement of self-field $J_c$ with respect to the sintering temperature is obviously due to the improvement in microstructure of the sample. The sample D58 and D60 show lesser $J_c$ than D56 (Table 1) because of reduced texturing [Fig. 2]. The $J_c$ value of an undoped sample (P48) which was sintered at its optimum, sintering temperature (848°C) is also included as a standard and it show a $J_c$ value of only (1290 kA m$^{-2}$). The doping of each Dy atom in the Sr site of (Bi, Pb)-2212 supplies one electron to the system, which reduces the number of holes and thus shifts the system from the ‘over doped’ to ‘optimally doped condition’ as far as the hole density is concerned [15]. The attainment of optimum hole concentration is responsible for the enhancement of $T_c$.
a cross-over from 3D flux lines to 2D pancake vortices at higher fields and temperatures. The 2D pancake vortices are mainly confined in the CuO$_2$ layers. Pb in the system improves the Josephson coupling between the CuO$_2$ layers by reducing the anisotropy and increasing the c-axis conductivity [16]. Dy doping in (Bi, Pb)-2212 decreases the c-axis length, and hence increases the Josephson coupling strength between the CuO$_2$ layers across the blocking layers (Sr--O/Bi--O/Sr--O). This improves the self-field and in-field $J_C$ of the optimized Dy-doped samples. The crystal defects created due to the Pb doping is mainly confined in the Bi-layer, but the defects produced by Dy doping are mainly in the Sr-layer. The vortices are confined in the CuO$_2$ layers, which are closer to the Sr-layer than the Bi-layers. Thus it is likely that the strongly coupled vortices in the CuO$_2$ layer are effectively pinned by the defects in the nearer Sr-layer than the defects produced in the distant Bi-layer. This is attributed to the enhanced pinning and the unusually high $J_C(B)$ performance of (Bi, Pb)-2212 doped with Dy. Among the Dy-doped samples, the $J_C(B)$ characteristics are found to be much better at the sintering temperature of 846°C. The ratio, $J_C(B)/J_C(0)$ of D46 is higher than that of undoped sample (P48) by a factor of ~11 at 0.16 T and ~61 at 0.32 T and the decrease of $J_C$ with increasing magnetic field is slower for D46 than that of P48 and all other Dy-doped samples. That is the deterioration of $J_C$ due to the magnetic field which is significantly reduced for D46. In order to bring out the increase in $J_C(B)$ performance of D46, the $J_C(B)/J_C(0)$ values of all samples at 0.16, 0.32, 0.64 and 1.20 T are separately given in Fig. 4(c). This figure clearly shows that the in-field performance and the flux pinning strength are very much improved for D46. When the sintering temperature increases, the $J_C(B)$ performance of Dy-doped samples gradually decreases and the sample sintered at 856°C shows the least $J_C(B)$ performance. The result can be understood again from the microstructure of the samples (Fig. 2). In sample D46, the grains are smaller, more rounded, less textured with maximum number of grain boundaries and hence the pinning due to the grain boundary is more effective leading to the best $J_C(B)$ performance. However, more grain boundary weak links in the sample reduces the self-field $J_C$. In D56, the flaky grains are highly textured and have lesser grain boundary and hence lesser weak links due to its optimum sintering temperature, which leads to lesser pinning and higher, self-field $J_C$. The samples D58 and D60 have better pinning than D56 which is obvious from the earlier observations on the deterioration of microstructure (Fig. 2).

It is widely accepted that a very useful tool with which one can investigate the flux pinning properties of superconductors is the determination of pinning force density $F_P = J_C \times B$. The normalized pinning force density ($F_P/F_{P_{\text{max}}}$) as a function of applied field is shown in Fig. 5. It is seen that the maximum value of $F_P$ is shifted to much higher values for Dy-doped samples. For example, for the Dy-doped samples D46 and D48, the $F_P$ values are 1697 and 1191 kN m$^{-2}$ as against 616 kN m$^{-2}$ for the D56 sample (Table 1). Similarly, for D46 and D48 the peak positions of $F_P/F_{P_{\text{max}}}$ appear beyond 0.96 T and 0.84 T, respectively, as against 0.60 T for D56. This supports that the flux pinning strength of Bi$_{1.6}$Pb$_{0.5}$Sr$_{1.8}$Dy$_{0.2}$Ca$_{4.1}$Cu$_{2.1}$O$_{8+\delta}$ significantly increases with increase of sintering temperature. Again the pinning strength of D58 and D60 slightly increases the deteriorated microstructure.

The thermally activated flux creep model proposed by Anderson [17,18] can be used to calculate the pinning potential (activation energy) of the superconductors. The activation energy is the height of the potential barrier, which is a measure of the flux pinning ability of the superconductor. According to this model,

$$\rho(T, B) = \rho_0 e^{\left(\frac{U_0}{k_B T}\right)}$$

(2)
where \( \rho(T, B) \) is the resistivity as a function of temperature \( T \) and magnetic field \( B \), \( \rho_0 \) is the normal state resistivity, \( U_0(B) \) is the pinning potential for flux creep and \( k_B \) is the Boltzmann’s constant.

The value of \( U_0(B) \) for the pure and Dy-doped samples can be calculated from the slopes of the linear part of the ln \( \rho \) versus \( T^{-1} \) curve.

Fig. 6(a) shows the temperature dependence of the resistivity of pure and Dy-doped samples at the magnetic field of 0.28 T. The plots are almost linear in a wide range, which indicates that the resistance tails are caused by a thermally activated process. The calculated \( U_0(B) \) as a function of sintering temperature and Dy content is shown in Fig. 6(b), which shows that the pinning potential of D46 increases noticeably when compared to P48 and other Dy-doped samples. Our results show that under 0.28 T \( U_0(B) \) of D46 is 0.731 \( \pm 0.001 \) eV, which is \( \sim 10 \) times larger than that of P48 (0.072 \( \pm 0.002 \) eV) and \( \sim 2.5 \) times larger than that of D56 (0.285 \( \pm 0.001 \) eV). The calculated \( U_0(B) \) is given in Table 1, which shows that \( U_0(B) \) of D46 increases noticeably as compared with that of other samples, i.e., the flux pinning strength is maximum for D46 sample. Also this is the reason for the very large pinning force density for this sample.

The higher \( U_0(B) \) value and the shifting of peak position of \( F_{\text{max}} \) towards higher fields for Dy-doped samples supports that the irreversibility line of these samples shifts to higher temperatures and magnetic fields compared to the undoped sample (P48).

4. Conclusion

In this paper we report a systematic study on the effect of sintering temperature on the microstructure, critical current density, flux pinning properties and pinning potential values of the Bi$_{12}$Pb$_{0.5}$Sr$_{1.5}$Y$_{0.2}$Ca$_{2}$Cu$_{2}$O$_{8+\delta}$ superconductor sintered between 846 and 860 °C. It is found that critical current density \( (J_C) \) and \( J_C(B) \) characteristics of Dy-doped samples sintered at different temperatures are highly sensitive to the sintering temperature. Among the Dy-doped samples the D46 shows the best \( J_C(B) \) performance and least \( J_C \), while D56 shows the least \( J_C(B) \) performance and best \( J_C \). These changes are explained on the basis of the microstructural changes brought about in the samples by varying the sintering temperature from 846 to 860 °C. The higher pinning potential values and the shifting of peak value of the bulk pinning forces density \( (F_{\text{max}}) \) towards the higher fields for Dy-doped samples show a shifting of the irreversibility line (IL) towards the higher fields. The enhancement of critical current density and flux pinning properties are discussed based on the microstructural variation, changes in the chemical as well as electronic inhomogeneities due to the doping of Dy atoms in (Bi, Pb)-2212 system.

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Reference