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RESEARCH

Investigation of the effects of gamma radiation on microstructure, critical current density, and densification properties of lanthanum-added BSCCO bulk ceramic superconductors

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Abstract
In this study, the role of gamma irradiation on lanthanum-added $Bi_{1.85}Pb_{0.35}Sr_2Ca_2La_xCu_3O_\delta$ (BSCCO) bulk ceramic superconductors prepared by conventional solid state sintering method was examined. For this purpose, microstructure, critical current densities (J_c), and condensation properties of samples were characterized in detail before and after the application of gamma irradiation. These characteristics were analyzed by Scanning Electron Microscopy (SEM), Magnetic Hysteresis (M-H) curves, and density measurements, respectively. It was observed from SEM images that many samples had a layered and porous grain structure, characteristic of high temperature superconductors. It was found that the critical current densities calculated from the width of the M-H curves do not behave proportionally to the increasing gamma radiation intensity. It was found that the density values of the samples determined by Archimedes method were in the range of approximately 71–84% of the theoretical value.

Keywords BSCCO superconductors · Gamma irradiation · Surface morphology · Critical current density · Archimedes method

Introduction

High temperature superconducting (HTS) materials have attracted great attention since its discovery. Because according to the Bardeen-Cooper-Schrieffer (BCS) theory accepted before the discovery of these materials, it was impossible for these materials to have high transition temperatures (T_c). For this reason, many properties of these materials have been studied in detail and continue to be examined by many research groups around the world. These materials, especially $YBa_2Cu_3O_x$ (YBCO) and $Bi_2Sr_2Ca_{n-1}Cu_2O_{2n+4}$ (BSCCO), are currently used in many technological applications and a great effort is still being made to improve their transport and magnetic properties.

BSCCO compounds, whose superconductivity was first reported by Maeda et al. [1], are one of the most important families of technologically important high temperature superconductors. The general formula of Bi-based HTS can be expressed as $Bi_2Sr_2Ca_{n-1}Cu_2O_{2n+4}$. "$n$" can take different values ($n=1$, 2, 3) according to the number of Cu–O planes perpendicular to the c-axis. BSCCO is generally divided into three different structures based on these "n" values. These are $T_c=20$ K ($n=1$, 2201), $T_c=95$ K ($n=2$, 2212), and $T_c=110$ K ($n=3$, 2223) [2]. It has been reported by different groups [3] that Pb at the Bi site is effective up to an optimum concentration of 0.3–0.4 in improving the superconducting phase of 2223.

Many methods have been tried to improve the properties of Bi-based superconductors. Among them, it has become important to investigate the effects of different irradiation techniques (neutrons [4, 5], protons [6], electrons [7, 8], gamma rays [9, 10], and heavy ions [11, 12]) on these materials. In addition to these irradiation techniques, many research groups around the world aim to enhance the properties of BSCCO high temperature superconductors by making

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substitutions and additions [13–17]. The main purpose of these radiations and substitutions/additions is to create defects in the material and to enable these regions to act as pinning centers and accordingly to improve the structural, electrical, magnetic, and mechanical properties of the materials. First of all, it is necessary to mention a few important observations in order to understand the effects of radiation on high-temperature superconductors. It should be noted that the effects of stoichiometry, structural transformations, and metal-to-insulator transitions complicate the response of high-temperature superconducting materials; so much more research is needed to understand these observations. These observations can be summarized as follows [18]:

(1) Ionizing radiation has almost no effect on the superconducting phases. The absence of ionization damage classifies these ceramic superconductors as more metals than ceramics in terms of the types of radiation they respond to.
(2) The second effect is displacement damage. Displacement damage has the effect of lowering the transition temperature of the superconductor and after sufficient damage leads to the formation of an insulating amorphous phase.
(3) The third observation is the effects caused by electron radiation. Electron radiation can be used to study displacement energy. The damage effects on ceramic superconducting materials with this radiation were investigated in detail [19, 20].
(4) Displacement damage from electrons, neutrons, ions, and high-energy radiation causes changes on the critical current and superconducting transition temperature. The transition temperature is a fundamental parameter of the superconducting phase, and changes in T_c reflect damage that alters this phase. Although the critical current density is a characteristic parameter of superconductors, it is determined by parameters such as the microstructure and atomic defects of the materials.
(5) Electron or ion radiation of sufficient intensity leads to the formation of a non-superconducting amorphous phase [21–23].
(6) There are atomic location-specific and sublattice-specific damage processes [19, 24].

In this research, the effects of both lanthanum addition and gamma radiation on the density, microstructure, and critical current density of the (Bi, Pb)-2223 system were investigated together. The density of the materials was determined by the Archimedes water displacement method. The orientation and structure of the particles were analyzed by scanning electron microscopy images. Finally, the current carrying capacities of the materials are reported by calculating from the width of the magnetic hysteresis curves.

Materials and methods

In this study, the response of (Bi, Pb)-2223 bulk superconductor to gamma radiation was investigated by adding lanthanum.

Preparation of lanthanum-added BSCCO bulk superconductors

Bi_2O_3 (Alfa Asear, 99% purity), PbO (Alfa Aesar, 99.9% purity), $SrCO_3$ (Sigma Aldrich, ≥ 98% purity), $CaCO_3$ (Sigma Aldrich, ≥ 99% purity), CuO (Sigma-Aldrich nanopowder), and La_2O_3 (Alfa Aesar, 99.9% purity) were used as starting powders to produce bulk superconductors. The conventional solid state reaction method has been used as the fabrication method. Pure 1% and 5% by weight La_2O_3 addition samples were prepared. The production of the superconducting materials has been described on the basis of previous studies in detail [18]. Conditions of the samples and their names are given in Table 1.

Characterization of lanthanum-added BSCCO bulk superconductors

In our previous study, the phase analysis of these materials and their resistivity behavior under magnetic field depending on the temperature were investigated [25]. In the present study, the microstructure, density, and magnetic properties of these materials were investigated in detail before and after the application of gamma radiation. Grain structures and surface morphologies of bulk superconductors were examined by scanning electron microscopy. SEM images were taken in backscattered electron mode by using a Zeiss Supra 50VP microscope. Densities of pure and La-added samples were determined by using the Archimedes water displacement method. Magnetic properties (M-H) were analyzed

Table 1 Sample composition, names, and conditions

Composition	Sample name	Gama radiation intensity (kGy)
$Bi_{1.85}Pb_{0.35}Sr_2Ca_2Cu_3O_\delta$	L0-0	0
	L0-1	1
	L0-15	15
	L0-55	55
$Bi_{1.85}Pb_{0.35}Sr_2Ca_2La_xCu_3O_\delta$ (1% wt La_2O_3 addition)	L1-0	0
	L1-1	1
	L1-15	15
	L1-55	55
$Bi_{1.85}Pb_{0.35}Sr_2Ca_2La_xCu_3O_\delta$ (5% wt La_2O_3 addition)	L5-0	0
	L5-1	1
	L5-15	15
	L5-55	55

at 10 K by using a Quantum Design Physical Properties Measurement System. The critical current densities (J_c) are calculated from the width of the M-H curves from the Bean critical state model with the help of the following equation [26]:(Equ. 1)

$$J_c = \frac{30\Delta M}{a\left(1 - \frac{a}{3b}\right)} \tag{1}$$

Results and discussion

In this section, the analysis results regarding the effects of both lanthanum addition and gamma irradiation on (Bi, Pb)-2223 superconductor were discussed. Density analysis, scanning electron microscope images, magnetic hysteresis analysis, and critical current density of the materials were given, respectively.

Density analysis

The theoretical density of the (Bi, Pb)-2223 system calculated from the lattice parameters is approximately 6.3 g/cm^3 [27]. In this study, the density of bulk superconductors was measured by Archimedes method and the results obtained are given in Table 2. It was observed that the measured density of the materials from the density values in the table took values in the range of approximately 71 to 84% of the theoretical density. These results are also compatible with the porous grain structure which is characteristic of ceramic superconductors. It is also seen from the table that the material with the lowest density is pure and not irradiated, whereas the material with the highest density is 1% wt. La-added and subjected to gamma irradiation at 15 kGy.

Table 2 Critical parameters of all samples

Samples	Mass density (g/cm^3)	J_c (A/cm^2)	
		Self-field	8 T
L0-0	4.4728	3.7×10^4	1×10^3
L0-1	4.6221	2.3×10^4	3.5×10^3
L0-15	4.8989	3.1×10^4	4.6×10^3
L0-55	4.9665	2.1×10^4	3.1×10^3
L1-0	4.6471	2.9×10^4	3.9×10^3
L1-1	5.1474	2.3×10^4	3.1×10^3
L1-15	5.3098	1.9×10^4	2.6×10^3
L1-55	4.9346	3.7×10^4	5.6×10^3
L5-0	5.1805	2.2×10^4	3.2×10^3
L5-1	5.0553	5.9×10^4	5.5×10^3
L5-15	4.9892	2.8×10^4	3.9×10^3
L5-55	5.2372	1.9×10^4	2.7×10^3

Figure 1 shows the measured density values depending on the amount of La. It can be clearly seen from the graph that a more dense structure is obtained with the increase of La amount. In other words, the addition of La improved the density of the materials.

Scanning electron microscopy (SEM)

Figures 2, 3, and 4 show the SEM images taken at 5000× magnification in backscattered electron mode from the broken surfaces of the materials. Backscattered electrons are primary beam electrons which are inelastically scattered back from the surface of the sample. These electrons therefore possess high energy and are not easily deflected.

It is clear from SEM images that all samples have a randomly oriented layered structure. In addition, all materials draw attention to the pores between the particles. The layered and porous structure is a characteristic feature of the high temperature superconductor family [28]. Therefore, it is understood that this structure is not damaged by both the addition of lanthanum and gamma radiation.

Magnetic hysteresis (M-H) measurements

At some temperatures, this is called the critical or transition temperature; a sharp drop in resistance is one of the most basic criteria for superconductors. The resistance properties of the materials examined in this research were analyzed in detail in our previous study [25]. Another important criterion of superconductivity is the diamagnetic property that occurs during magnetization measurements. The temperature and magnetic field dependencies of magnetization provide information about the critical current density, upper critical field, irreversibility field, and pinning mechanism, which are the most important

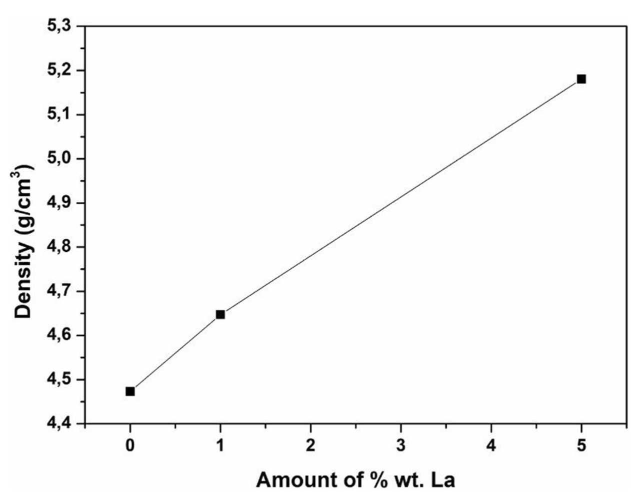

Fig. 1 Measured density values versus amount of La addition

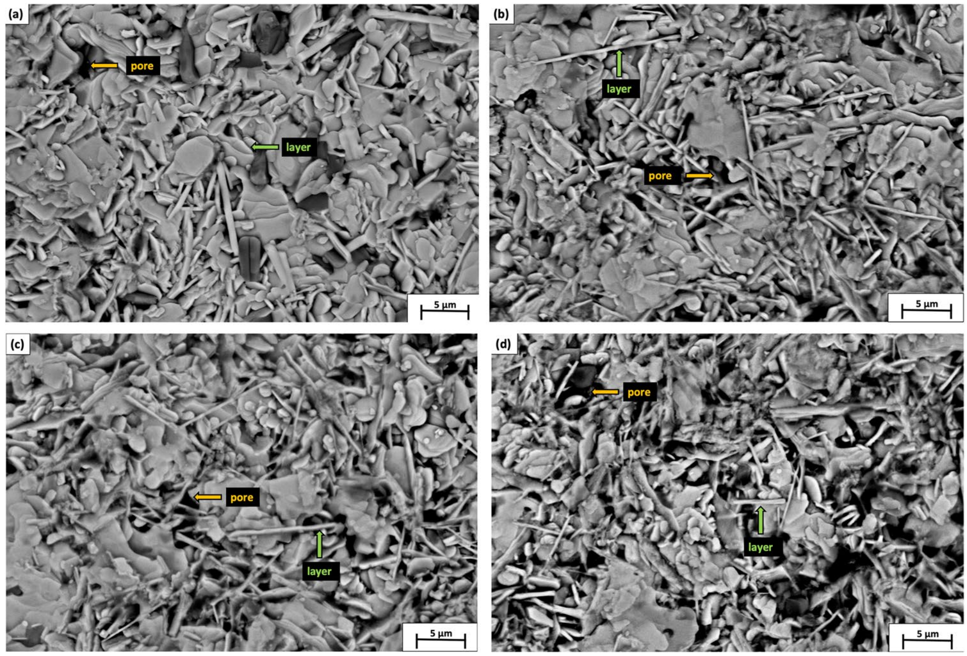

Fig. 2 SEM photographs of the broken surfaces of pure sample **a** L0-0, **b** L0-1, **c** L0-15, and **d** L0-55

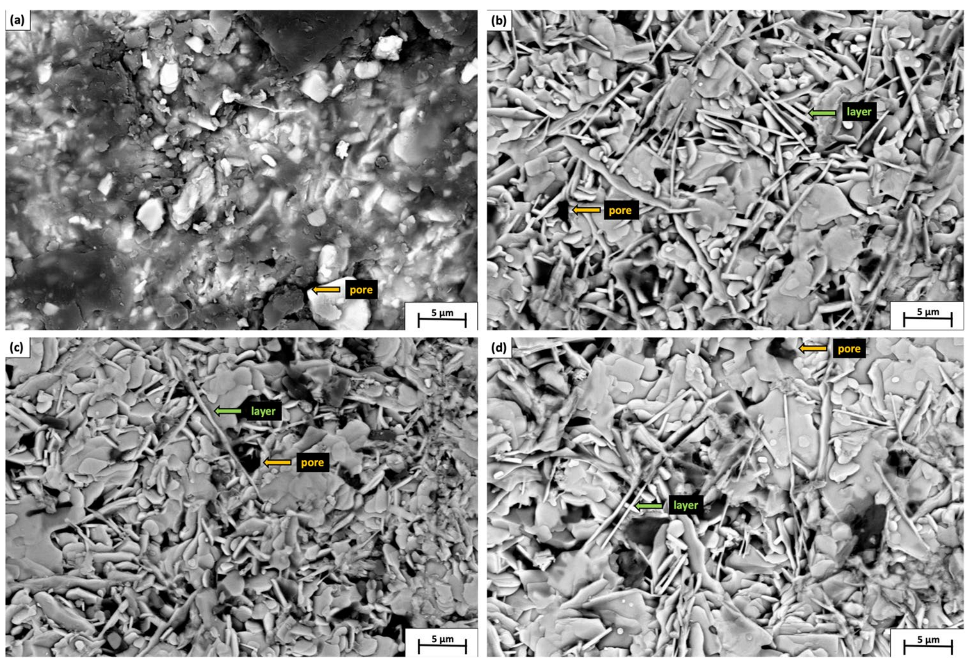

Fig. 3 SEM photographs of the broken surfaces of 1% wt. La-added sample **a** L1-0, **b** L1-1, **c** L1-15, and **d** L1-55

properties of superconducting samples. These properties reveal the potential of superconductors to be used in large-scale applications such as health, electrical, electronics, and transportation. Therefore, magnetic hysteresis measurements were performed to investigate the effects of both lanthanum addition and gamma radiation on (Bi, Pb)-2223 superconductor. These measurements were taken at 10 K by using a cooling regime under zero magnetic field (ZFC).

The magnetic hysteresis loops of the investigated samples are given in Fig. 5. It is clear from these curves that all samples give a diamagnetic response as expected. In Fig. 5(a), it was observed that the width of the curve decreases with the increase in the amount of lanthanum addition in the samples not exposed to gamma irradiation. However, as seen in Fig. 5(b) and (d), when the dose amount of gamma radiation is increased, added samples

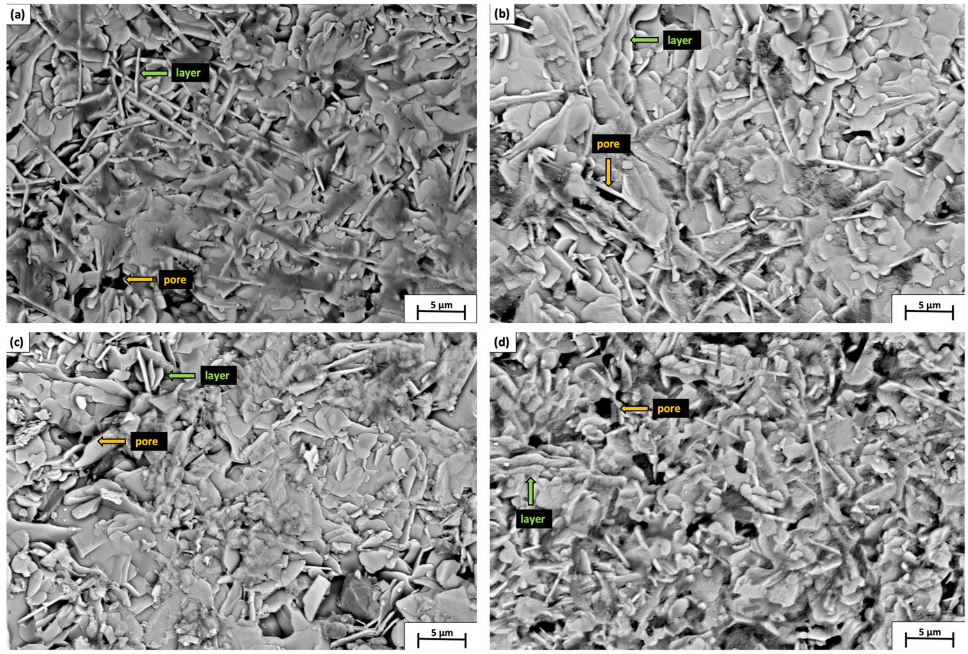

Fig. 4 SEM photographs of the broken surfaces of 5% wt. La-added sample **a** L5-0, **b** L5-1, **c** L5-15, and **d** L5-55

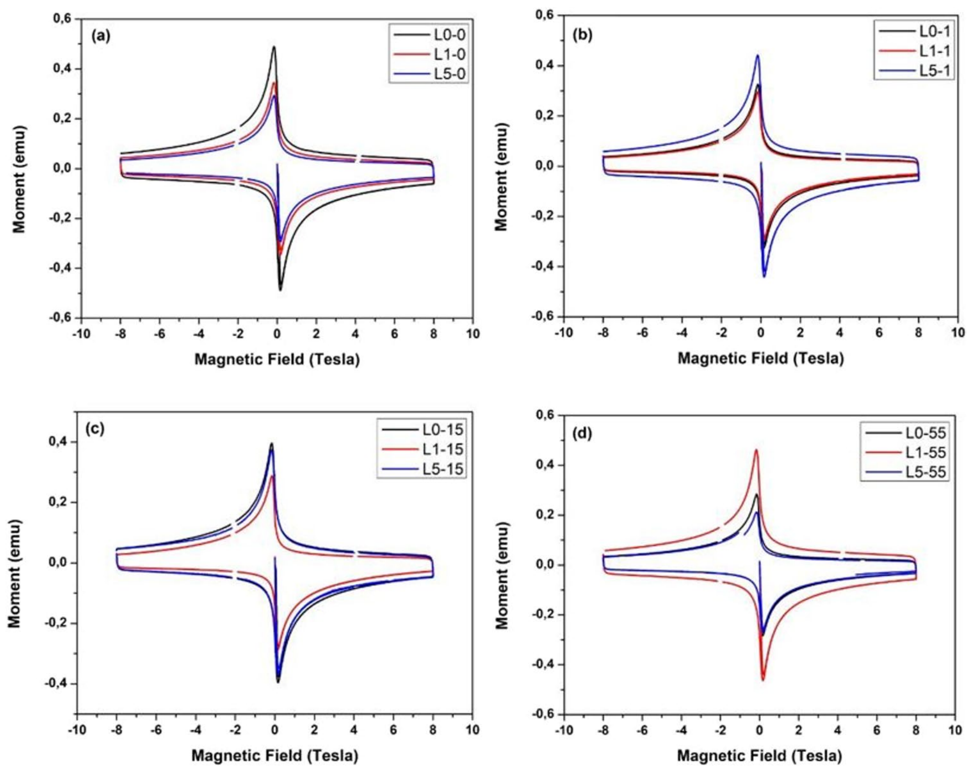

Fig. 5 Magnetic hysteresis loop of the samples **a** 0 kGy, **b** 1 kGy, **c** 15 kGy, and **d** 55 kGy

have a wider hysteresis curve compared to pure sample. While the 5% wt. lanthanum-added sample has a wider curve under 1-kGy irradiation, the curve of the 1% wt. lanthanum-added sample is wider under 55-kGy irradiation.

Critical current density (J_c)

The most important feature that distinguishes superconductors from normal conductors currently used in applications is their current carrying capacity. For this reason, many research groups around the world are still making great efforts to increase the critical current densities of superconductors [29–33].

The critical current densities at 10 K of the samples examined in the present study were calculated using the Bean critical state model from the width of the magnetic hysteresis curves. Figure 6 shows the magnetic field dependence of the critical current densities of all samples. Under 1-kGy and 55-kGy irradiation, it is seen that the critical current density values are higher than the pure sample due to the wider hysteresis curves of the samples with 5% wt. and 1% wt. lanthanum, respectively. This reveals that the critical current densities of the some samples are improved by both the addition of lanthanum and gamma irradiation. As mentioned in the previous sections, it was emphasized that the properties of superconductors can be improved by creating imperfections in materials with both doping and irradiation. This is thought to be due to the fact that these areas act as pinning centers. Therefore, it is interpreted that the increasing J_c values for added samples under irradiation result from the flux pinning mechanism. Table 2 shows the J_c values that all samples have in certain magnetic fields.

It is known that the critical current density of superconducting materials varies depending on the microstructure and defects in the materials. For this reason, it is aimed to increase the critical current density by creating defects that act as pinning centers in superconducting materials. Additions or doping is the most commonly used methods to create defects in the material. In addition, it is seen in the literature that various radiations are used for this purpose [34–36]. In this study, these defects were tried to be created by both the addition of lanthanum and gamma radiation. However, this study needs to be done separately in order to understand which effect is more dominant. In addition, there are some studies in the literature on the effects of lanthanum addition to the (Bi,Pb)-2223 system [37, 38]. We analyzed these materials both scientifically and technologically by conducting a study on whether these materials can be used in applications where ionizing radiation is present, such as gamma rays in space environments.

Conclusions

In this study, the response of pristine and lanthanum-added (Bi, Pb)-2223 superconductor prepared by conventional solid state sintering method to gamma radiation was investigated in detail by condensation, SEM images, and critical current density measurements. The results obtained with these analyses can be summarized as follows:

(1) It has been determined that many materials become denser with the increase of radiation intensity and the amount of lanthanum. In other words, it can be said that exposure to the gamma radiation and adding of La enhance the properties of the materials in terms of densification.

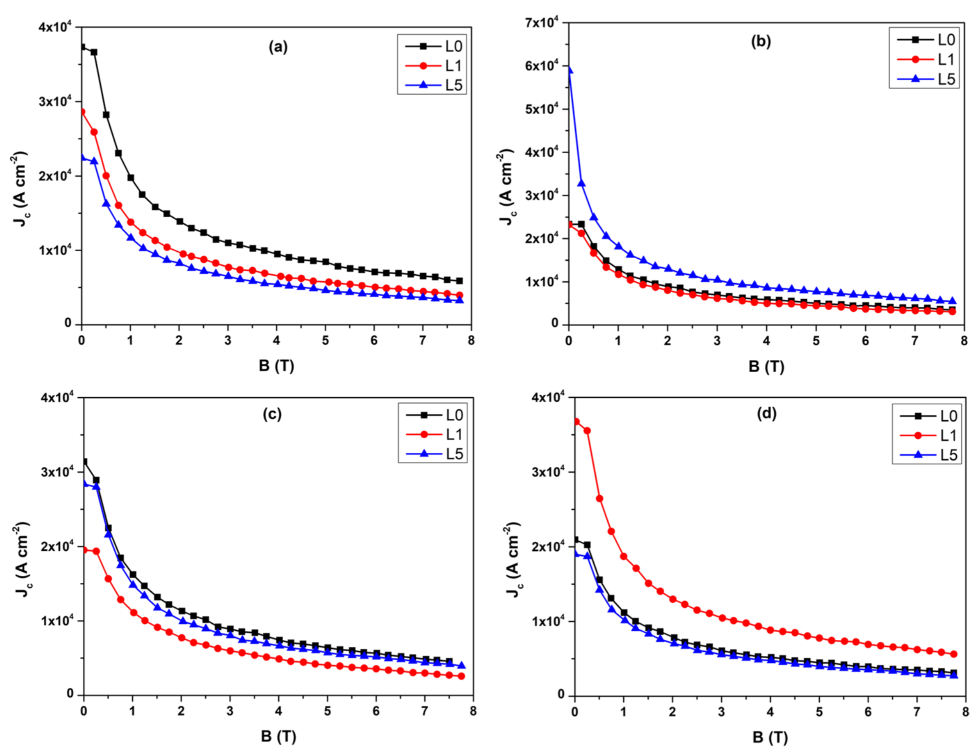

Fig. 6 Variation of critical current density with magnetic field **a** 0 kGy, **b** 1 kGy, **c** 15 kGy, and **d** 55 kGy

(2) A layered and porous microstructure has been obtained in all samples. Therefore, it was understood that both gamma radiation and lanthanum addition did not change the surface morphology of the materials.
(3) Critical current densities of some samples are enhanced by both the addition of lanthanum and gamma irradiation.

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Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Research Fund of Hakkari University, Hakkari, Turkey, under grant contract no. FM19AYP1.

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