

# Impact of Electron Irradiation on Transition Temperature of Pb-Substituted Bi-2223 Superconductor



Zaahidah Atiqah Mohiju, Nasri A. Hamid, Yusof Abdullah

**Abstract:** The synthesis of  $(\text{Bi}_{1-x}\text{Pb}_x)_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{8+\sigma}$  ( $x=0$  and  $0.4$ ) bulk samples (hereafter called as Bi-2223) were completed by using solid-state reaction method. The Bi-2223 samples were subjected to calcination for 24 hours at  $840^\circ\text{C}$ . Then, the pelletized powder for both samples were sintered at  $865^\circ\text{C}$  for 48 hours. Each sample was exposed to high energy electron irradiation with a dosage of 100 kGray. Structural examination was conducted by using the Bruker D8-Advanced X-ray powder Diffraction (XRD). According to Bi-2223 indexed phase structure, the XRD patterns obtained shows that the samples, both Bi-2223 ( $x=0$  and  $x=0.4$ ) samples showed well-defined peaks and phase structure remains unchanged after the sample being exposed to the electron source. Measurement of the critical temperature,  $T_C$  were obtained by using AC susceptibility technique and four-point probe. Bi/Pb ( $x=0.4$ ) sample showed significant  $T_C$  value compared to Bi/Pb ( $x=0$ ) sample. Grain alignment in SEM micrographs show enhancement in their grain connectivity and also layering texture in irradiated sample which might lead to has better superconducting properties. In contrast, the impact of electron irradiation on its microstructure and consequently the  $T_C$  was destructive in the Bi/Pb ( $x=0$ ) sample.

**Keywords:** Electron Irradiation, Transition Temperature and X-ray powder Diffraction

## I. INTRODUCTION

The structure's development of the BSCCO system is very important to determine its current-carrying capacities and effective pinning centers. Addition or substitution of element which has different ionic radius may alter the phase formation and stability of one material, by changing its bonding characteristics. This modification is not only affecting the density of charge carriers in the Cu-O planes [1], but also change the total density of free electron.

Thus, partial replacement small amount of Bismuth by lead in  $(\text{Bi}_{1-x}\text{Pb}_x)_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{8+\sigma}$  is expected to boost up the superconducting properties of the samples as the modulation function of these lead particles will suppress the c-axis component [2,3,4]. Furthermore, lead-substitution also turns out to be one of the ways to reduce the "structural anisotropy" of the Bi-O planes [5].

Thus, even with the small amount of lead presents in the anisotropies affected the whole physical properties of Bi-2223. Xiang et al. [6] stated in his research according to his collection of experimental data, lead-free samples were consistent with a very weak bonding between consecutive Bi-O planes. Thus, intercalation was introduced by allowing guest atomic or molecular species to be easily inserted in between adjacent Bi-O planes. After the intercalation of lead occurred, the characteristics of Bi-2223 would still be preserved. An expansion of crystal lattice also can be done by promoting in homogeneity into the material. This in homogeneity will then act as an effective pinning center. Therefore, investigation on pinning centers also important since it will determine the quality of one superconducting material.

In order to adapt to the latest technology, superconducting compounds have been placed in high radiation environments. This research work is important to explicate the impact of irradiation exposure on the material's properties and the number of lattice defects created [7-9]. This point of defects was artificially created and not only reduced  $T_C$  values of the materials, but also the critical current,  $J_C$ . These results indicated that a reduction in  $J_C$  values is also caused by the formation of grain boundaries, which are strongly related to the tilt angle between crystal axes of two grains [10]. As demonstrated by Chaudhari et al., critical current in high magnetic fields is reported to be higher when the grain size is reduced [11].

Meanwhile, Dimos et al. explored the misorientation angle effect of grain boundary [12]. A study on the transport properties of three different geometries of the grain boundaries has been covered and the results are identical. Hence, this indicates that the structural disorder at the boundary leads to poor superconducting link between grains. This research work is will clarify the consequences of electron irradiation towards superconducting material  $(\text{Bi}_{1-x}\text{Pb}_x)_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{8+\sigma}$  ( $x=0$  and  $0.4$ ). All the results are presented in the following sections.

The motivation of this work is to study the effect of partial substitution of lead in Bi-2223 on its superconducting properties and phase formation by comparing the changes in transition temperature and the scanned image of the microstructure. Starting compositions, heating temperatures and sintering time as discussed in the next section.

Manuscript published on November 30, 2019.

\* Correspondence Author

**Zaahidah Atiqah Mohiju\***, Nuclear Engineering and Energy Group, College of Engineering, Universiti Tenaga Nasional, Jalan Kajang-Puchong, 43000 Kajang, Selangor, Malaysia.

**Nasri A. Hamid**, Nuclear Engineering and Energy Group, College of Engineering, Universiti Tenaga Nasional, Jalan Kajang-Puchong, 43000 Kajang, Selangor, Malaysia.

**Yusof Abdullah**, Materials Technology Group, Industrial Technology Division, Malaysian Nuclear Agency, Bangi, 43000 Kajang, Selangor, Malaysia

© The Authors. Published by Blue Eyes Intelligence Engineering and Sciences Publication (BEIESP). This is an open access article under the CC-BY-NC-ND license <http://creativecommons.org/licenses/by-nc-nd/4.0/>

Only small concentrations are considered since large concentrations are destructive to their superconducting properties [13].

## II. EXPERIMENTAL

The Bi-2223 samples were prepared by a conventional solid-state reaction which is ideal for the preparation of polycrystalline solids. According to the nominal composition of  $(\text{Bi}_{1-x}\text{Pb}_x)_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{8+\sigma}$  (Bi-2223) where  $(x=0 \text{ and } 0.4)$ , samples were made of  $\text{Bi}_2\text{O}_3$ ,  $\text{PbO}$ ,  $\text{Sr}_2\text{CO}_3$ ,  $\text{CaCO}_3$ , and  $\text{CuO}$ . The raw chemical powders were mixed according to its ratio of 1.6, 0.4, 2, 2, 3 respectively. In order to produce fine-grained material, the mixture was ground for 2 hours and calcined in a furnace with free airflow at a temperature of  $840^\circ\text{C}$  for 24 hours. Calcination helps to remove impurities, decompose carbonates and form the oxide powder. Then, the resulting powder was ground for another hour before pressed with a pressure of 7 tonnes at room temperature with the hydraulic press. Bulk samples (dimension: 13mm diameter, 3mm thickness) were synthesized at  $850^\circ\text{C}$  for 48 hours.

Irradiation exposure of 100 kGray on the samples was carried out using the EPS-3000 electron-beam accelerator of 3 MeV and current measurement of 10 mA. The procedure was conducted at the Malaysian Nuclear Agency. Samples' phase formation was investigated by conducting a structural examination using the X-Ray Diffractometer (XRD). Meanwhile, Hitachi-S3400N Scanning Electron Microscopy (SEM) machine was used to study its microstructure. Measurement of the transition temperature,  $T_C$  for Bi/Pb ( $x=0$ ) samples was performed by using cryogenic four-point probe, while for Bi/Pb ( $x=0.4$ ) samples the measurements were done using AC susceptibility technique.

## III. RESULTS AND DISCUSSIONS

Diffraction peaks for Bi-2223 ( $x=0.4$ ) of both testing samples show sharper and well-defined (0012) and (202) peaks compared to lead-free samples. Figure 1 and figure 2 show the full results of the diffraction peaks of Bi-2223 ( $x=0$  and 0.4) for the samples. The Bi-2223 phase was confirmed by comparing peak positions and intensities of our samples with the database of Superconducting Material Database (Reference number PR0417217) in the NIMS Materials database. There is a noticeable change at the intensity of peak (0012) after irradiation in both of Bi-2223 samples. The volume fraction of Bi-2223 increased after irradiation. Thus, this will promote crystal growth while enhancing the grain sizes of the superconductor ceramics. By using Bragg's Law, the lattice parameters were calculated and defined as an orthorhombic crystal structure where  $a = 5.27 \text{ \AA}$ ,  $b = 5.47 \text{ \AA}$ , and  $c = 38.30 \text{ \AA}$ , respectively. The small amount replacement of Bi by lead effectively improved the phase formation as well as particles' distribution [14].

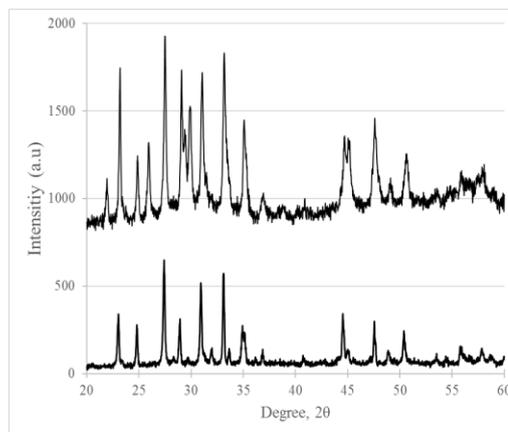


Fig. 1 XRD patterns of  $(\text{Bi}_{1-x}\text{Pb}_x)_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{8+\sigma}$  ( $x=0$  and 0.4) for non-irradiated samples.

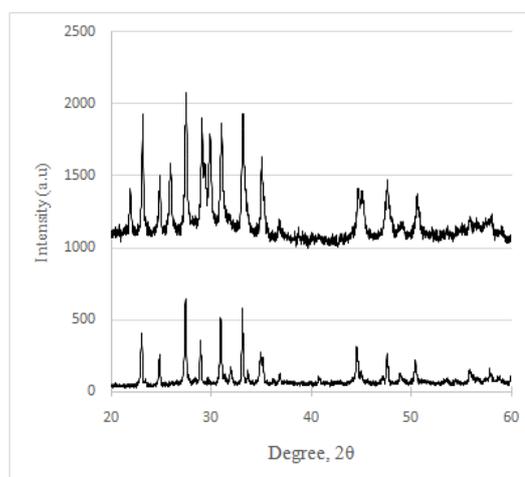
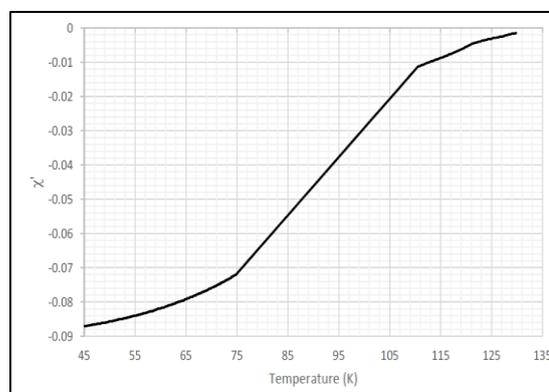
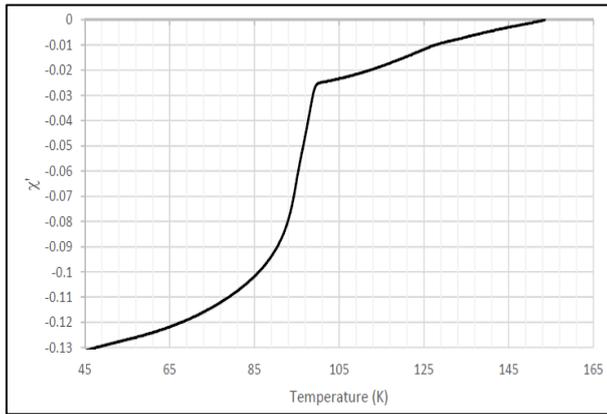


Fig. 2 XRD patterns of  $(\text{Bi}_{1-x}\text{Pb}_x)_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{8+\sigma}$  ( $x=0$  and 0.4) for irradiated samples

Measurement of the critical temperature for Bi/Pb ( $x=0.4$ ) samples has been obtained by using the AC susceptibility machine. Figure 3(a) and 3(b) show the susceptibility,  $\chi'$  (real part) of both bi-2223 samples. The inflection point for both graphs is where the superconducting coupling between the grains occurred and the points are also marked as the critical temperature,  $T_C$  for the samples [15].



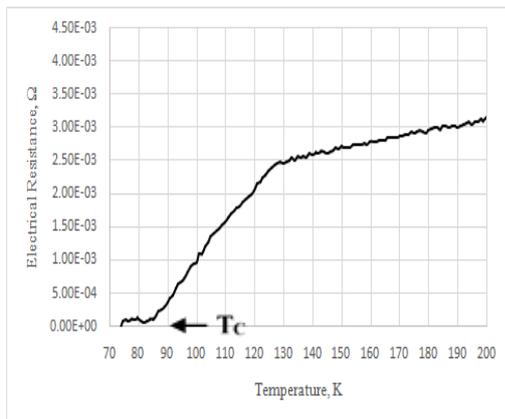
(a)



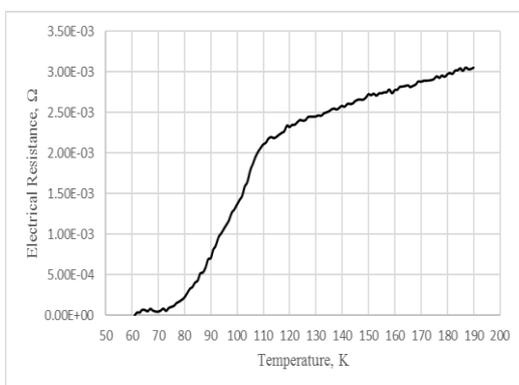
(b)

**Fig. 3 AC susceptibility, real parts ( $\chi'$ ) of for Bi/Pb (x=0.4) (a) non-irradiated sample and (b) electron irradiated sample**

The critical temperature,  $T_C$  results were plotted in figure 4(a) and 4(b) and the critical temperature points were marked on the graph line where the electrical resistance of the samples dropped to nearly zero ohm ( $\Omega$ ). Table 1 shows the  $T_C$  values of Bi/Pb (x=0 and 0.4) for both samples. Meanwhile, for Bi/ Pb (x=0) sample, there is significant degrading of  $T_C$  after irradiation when compared to Bi/ Pb (x=0.4) sample. The addition of lead in Bi-2223 increased volume fraction in Bi/ Pb (x=0.4) sample and form a continuous network within Bi-2223 phase grains to enhance the  $T_C$ .



(a)



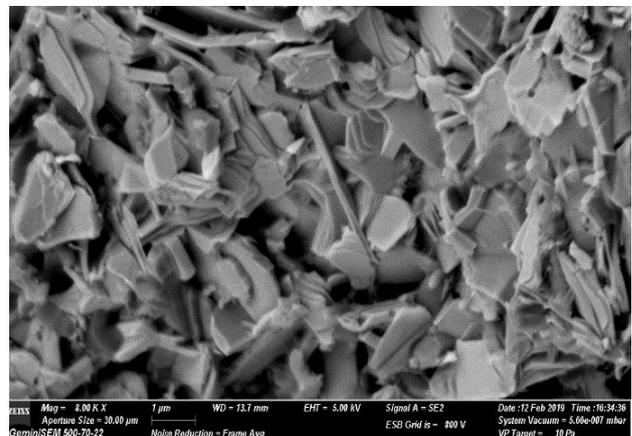
(b)

**FIG. 4 Graph of electrical resistance against temperature for Bi/ Pb (x=0) (a) non-irradiated lead-free sample, and (b) electron irradiated lead-free sample**

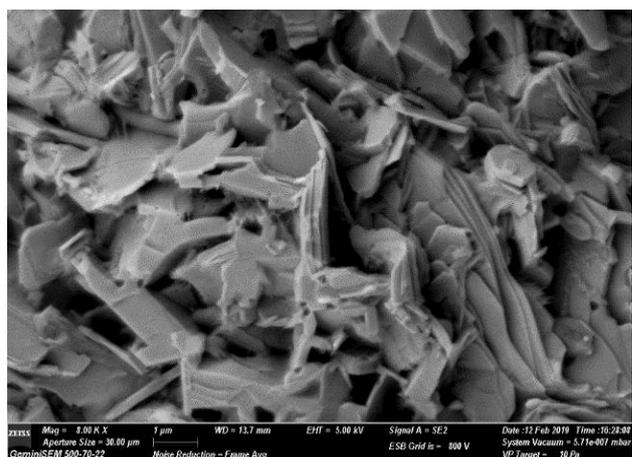
**Table. 1 Critical temperature,  $T_C$  of Bi/Pb (x=0 and 0.4) for non-irradiated and irradiated samples**

Samples	Irradiation	
	Non-irradiated (K)	Electron Irradiation (K)
Bi/ Pb (x=0)	84K	72K
Bi/ Pb (x=0.4)	97K	90K

Figure 5 and figure 6 show the micrograph results of the microstructure of Bi-2223 samples. The morphology of the microstructure of all samples consists of flaky layers with a varying degree of grain disorientation. Both Bi/Pb (x=0 and 0.4) for non-irradiated samples show lower grain alignment disorientation compared to irradiated samples. This is due to the collision of particles during the irradiation. Hence the concentration of defects is also increased. The appearance of pores and gaps is also noticeable in the irradiated sample of Bi/Pb (x=0), and this could lead to a reduction of material's strength.

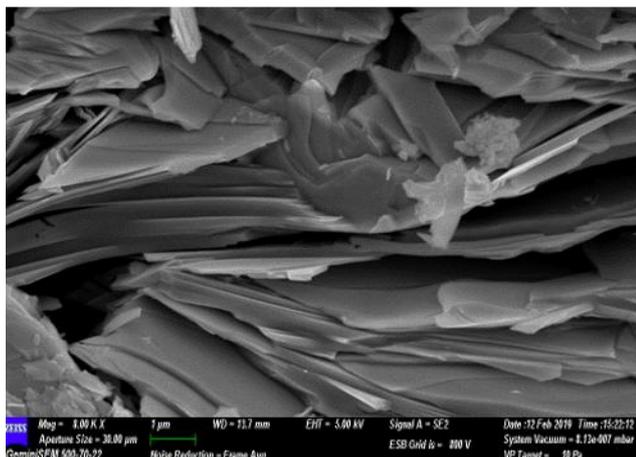


(a)

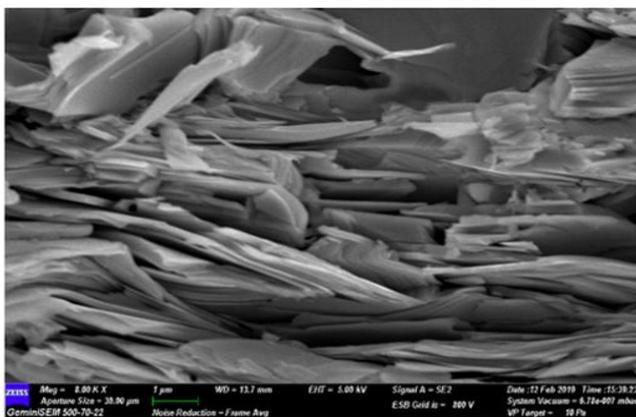


(b)

**Fig. 5 Micrographs of Bi/Pb (x=0) for (a) non-irradiated sample and (b) electron irradiated sample**



(a)



(b)

**Fig. 6 Micrographs of Bi/Pb (x=0.4) for (a) non-irradiated sample and (b) electron irradiated sample**

Small substitution of bismuth (Bi) by lead (Pb) for sample Bi/Pb (x=0.4) shows fine growth of connecting layer at grain boundaries in both samples. Furthermore, a better-textured microstructure with well-connected grains can be observed in the irradiated Bi/Pb (x=0.4) sample. The dominant Bi-2223 phase volume fraction is sufficient to form a continuous network of Bi-2223 phase grains and maintain a reasonably high  $T_C$ . In addition, Pb substitution combined with electron irradiation not only increased the volume fraction of the Bi-2223 phase but also enhanced the mechanical and transport properties in the Bi/Pb (x=0.4) sample. The microstructure of the system can be tailored and refined to yield the best superconducting properties in terms of critical current density,  $J_C$  and flux pinning properties to meet various industrial requirements.

#### IV. CONCLUSIONS

In summary, the small amount of lead (Pb) substitution in the Bi-2223 phase superconductor can promote better grain growth and also induce a qualitative change in the nature of transport properties along the c-axis. This is due to the addition of electrons' substance into the  $CuO_2$  planes. The volume fraction of the Bi-2223 phase and the degree of grain alignment in the Bi/Pb (x=0.4) sample have been maintained and improved to a certain degree after irradiation. As such Pb substitution together with electron irradiation enhances the transport and mechanical properties

of the Pb-substituted Bi-2223 superconductor while maintaining its transition temperature,  $T_C$ .

#### ACKNOWLEDGMENTS

This research work is funded by FRGS grant from the Malaysian Ministry of Higher Education, reference number FRGS/1/2017/STG02/UNITEN/02/05. In collaboration with the Malaysian Nuclear Agency. Authors would like to thank iRMC Journal Publication Fund of Universiti Tenaga Nasional for the publication sponsorship and also Prof. Roslan Abd-Shukor from the Universiti Kebangsaan Malaysia for his kindness of providing his research facilities for this project.

#### REFERENCES

1. M. Takano, J. Takada, K. Oda, H. Kitaguchi, Y. Miura, Y. Ikeda, Y. Tomii, and H. Mazaki, Japanese Journal of Applied Physics Vol.27, L1041 (1988).
2. S. Wu, J. Schwartz and G. W. Raban Jr., Physica C 246 (1995) 297.
3. S. Moehlecke, C. H. Westphal, M. S. Torikachvili, J. A. Davis and I. C. L. Torriani, *ibid.* 211 (1993) 113.
4. T. Kawai, T. Horiuchi, K. Mitsui, K. Ogura, S. Takagi and S. Kawai, *ibid.* 161 (1989) 561.
5. F.X. Regi, J. Schneck, H. Savary, C. Daguet, F. Huet, IEEE Trans. Appl. Supercond. Vol. 3, 1190 (1993).
6. X-D. Xiang, S. McKernan, W.A. Varoka, A. Zettl, J. L. Corkill, T. W. Barbee III, and M.L. Cohen, Nature Vol. 348, 145 (1990).
7. A.V. Krashennnikov and K. Nordlund, J. Appl. Phys. 10, 071301 (2010).
8. X. Zhao, J. Yu, Y. Wang, G. Yu, Y. Chen and Z. Zhang, Physica C 337, 234 (2000).
9. N.A. Hamid, Z.A. Mohiju, and Y. Abdullah, Solid St. Phenomena Vol. 280, 21 (2018).
10. T. Amrein, L. Schultz, B. Kabius, and K. Urban K, Phys. Rev. B 51, 6792 (1995).
11. S. Chaudhary, S.C. Kashyap, D.K. Pandya, and V.N. Kulkarni, Physica C 280, 37 (1997).
12. J. Dimos, P. Chaudhari, and J. Mannhart, Phys. Rev. B 41, 4038 (1990).
13. Y. F. Lu, X. L. Qiu and X. X. Chao, Solid State Communications 95 (1995) 259.
14. N. Knauf, J. Harnischmacher, R. Müller, R. Borowski, B. Roden, and D. Wohlleben, Physica C 173, 414 (1991).
15. M. Nikolo, Am. J. Phys. 63, 57 (1995).
16. Padmapriya, T., Saminadan, V. "QoE based carrier aggregation techniques in LTE-Advanced networks", Proceedings of the International Conference on Intelligent Sustainable Systems, ICISS 2017, 2018.