Impact of gamma and electron irradiations on superconducting properties of Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_{10}$ (Bi-2223) superconductor

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Abstract. In several studies on the impact of radiation towards superconductor, there were evidences that changes may occur in superconducting properties due to alteration in lattice structure of HTS materials. In this work, the solid-state reaction method was employed in preparing the bulk samples of Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_{10}$ (hereafter Bi-2223) superconductor. The samples were then irradiated with gamma and electron sources with dosage of 100 kGray each. X-ray powder diffraction (XRD) patterns were used to determine the phases that present in the samples. Results of XRD patterns for both the non-irradiated and irradiated samples showed well-defined peaks all of which could be indexed based on the Bi-2223 phase structure. Besides, the XRD patterns indicate that gamma and electron irradiations did not affect the Bi-2223 superconducting phase structure. Measurements of the critical temperature, $T_C$ and the critical current density, $J_C$ of all the samples were conducted before and after irradiation using AC susceptibility technique. $T_C$ for non-irradiated, electron irradiated and gamma irradiated sample is 97 K, 95 K and 103 K, respectively. On the other hand the $J_C$ for non-irradiated, electron irradiated and gamma irradiated sample is 1618 A/cm$^2$, 1729 A/cm$^2$ and 1549 A/cm$^2$, respectively. Results of scanning electron microscopy (SEM) micrographs show improvement in the grain growth and disorientation in the texture of all the gamma and electron irradiated samples.

1. Introduction
Potential applications in the electricity distribution industry are more promising. Among the use of superconducting wire is to produce a transmission cable – in urban areas, where large amounts of power must be transmitted by underground cables, high current density is important and thus superconducting wires are an attractive proposition. There is also interest in the development of transformers, where superconducting materials would have several advantages over those using conventional materials. Another application of superconducting materials is the fault current limiter, which uses the resistive transition to reduce the fault current in an electrical circuit.

Radiation effects including electron irradiation and gamma irradiation are caused primarily by alteration in the structure of the lattice, and the study of radiation effects have been mainly an attempt
to resolve these alterations into a moderate number of lattice defects, and to elucidate the properties of these defects [1]. Moreover, the large difference between the critical currents achieved in textured materials and random polycrystals in early experiments on high Tc materials indicated that the grain boundaries are responsible for reducing $J_C$ [2]. This was first demonstrated directly by Chaudhari et al. [3], where the critical current inside a grain and across a grain boundary in yttrium-based superconductor (YBCO) film was studied and found that the inter-grain critical current to be more than an order of magnitude lower.

The effect of the grain boundary misorientation angle was studied by Dimos et al. [4] who fabricated thin films of YBCO on SrTiO$_3$ (STO) bicrystals. Grain boundaries with three different geometries have been studied; the transport properties of all three types of boundaries are essentially identical, which implies that the poor superconducting coupling between grains is the result of the intrinsic structural disorder at the boundary. The purpose of this research work is to study the impact of electron and gamma irradiations on Bi-2223 superconducting properties by applying the same amount of radiation exposure. All the results are presented in the following section.

2. Experimental
Specimens with nominal composition $\text{Bi}_2.4\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ were prepared via conventional solid state method. Samples were made of oxide and carbonate powders, homogeneously mixed according to its stoichiometric molar ratio of $\text{Bi}_2\text{O}_3$, $\text{PbO}$, $\text{Sr}_2\text{CO}_3$, $\text{CaCO}_3$ and $\text{CuO}$. The mixture was ground using mortar and pestle, and heated in a tube furnace with free air flow at temperature of 810°C for 30 hours to remove impurities and form the Bi-2212 oxide. Then, the oxide powder was pressed using hydraulic press machine at room temperature with pressure of 7 tonnes. Bulk samples of 13 mm in diameter and 3 mm in thickness were formed and synthesized under the same condition at 850°C for 60 hours, then furnace-cooled to room temperature.

Electron irradiation on one of the sample was carried out using a beam of 3 MeV, current measurement of 10 mA and radiation doses of 100 kGray. The irradiation was conducted using the EPS-3000 electron-beam accelerator. Meanwhile, another sample was exposed to gamma ray irradiation and it has been conducted in SINAGAMA Plant using JS10000 (IR-219) irradiator with radiation doses of 100 kGray. Both facilities are provided by Malaysian Nuclear Agency.

Structural investigations of non-irradiated and irradiated samples were conducted using a Bruker D8 Advanced X-Ray Diffractometer (XRD) to confirm their phase formation. Microstructure investigation was carried out using a Hitachi S3400N Scanning Electron Microscope (SEM). Superconducting properties for each sample, such as transition temperature, $T_C$ and the critical current density, $J_C$ were determined by using AC susceptibility technique where the magnetic field varies sinusoidally.

3. Results and analysis
Figure 1 shows the XRD patterns of Bi-2223 for both non-irradiated and irradiated samples. The XRD patterns for Bi-2223 of non-irradiated sample show sharper and well-defined (0010) and (119) peaks. A few unknown peaks and other impurities are also observed. The position of diffraction peaks of Bi-2223 phase in all patterns are identical, which suggests that no change in lattice parameters occurred within the resolution of XRD. Visible changes at the intensity of peak (1111) after being exposed with 100k Gray of electron and gamma irradiations are observed. This indicates that irradiations could increase the volume fraction of Bi-2223 phase and promotes crystal growth to the superconductor ceramics [5]. Meanwhile, lattice parameters for all prepared samples were calculated from Bragg’s law and Miller indices $hkl$. Since the values obtained for $a = 5.2662$ Å, $b = 5.4705$ Å and $c = 38.3014$ Å, so it is characterized as orthorhombic. According to previous research work, partial substitution of Bi by Pb is effectively enhanced the phase formation kinetics as well as increased the yield of Bi-2223 phase [4]. In our study, similar substitution was used in preparing our samples.
AC susceptibility has been used to measure the critical temperature and critical current of the samples. Figure 2(a), 2(b) and 2(c) show the real parts of susceptibility ($\chi'$) of non-irradiated, electron irradiated and gamma irradiated samples, respectively. In each graph, the position of inflection point on the curve is the critical temperature. This is due to the considerable difference between lower critical magnetic field of the intergranular matrix and the grains, because the flux will penetrate the intergranular matrix first before it reaches the grains [6].

Figure 1. XRD patterns of Bi-2223 non-irradiated, electron and gamma irradiated samples exposed with 100kGray, respectively.
(a) $\chi'$ vs Temperature (K) for $H_c: 5\text{oe}$ and $f: 295\text{Hz}$.

(b) $\chi'$ vs Temperature (K) for $H_c: 5\text{oe}$ and $f: 295\text{Hz}$.
Figure 2. Real ($\chi'$) parts of susceptibility for (a) non-irradiated sample, (b) electron irradiated sample, and (c) gamma irradiated sample for applied magnetic field, $H_a=5\text{oe}$ and frequency of 295Hz.

Rullier-Albenque et al. [7] stated that electron irradiation is an excellent technique to introduce homogeneously distributed point defects within the superconducting grains that produces higher $J_C$. Nevertheless, bonding between the adjacent grains within the sample is weakened after irradiation and leads to reduction in $T_C$. $J_C$ at 70 K for all samples were calculated by using this equation,

$$J_C(T_F) = \frac{400A}{a \times b} \text{ cm}^2$$  \hspace{1cm} (1)

where $a$ and $b$ are the width and height of the sample.

Based on the above results and from Table 1, $T_C$ of electron irradiated sample shows reduction of 2 K while its $J_C$ improved by 6.91\% from non-irradiated sample. Unlike gamma irradiated sample, $T_C$ value increased to 103 K but $J_C$ value dropped by 4.25\%.

Table 1, Transition temperature, $T_C$, surface area, and critical current density, $J_C$ of samples.

| Type of sample       | $T_C$, K | $a$, mm | $b$, mm | $(a \times b)$, cm$^2$ | $J_C$, A/cm$^2$ |
|----------------------|----------|---------|---------|------------------------|-----------------|
| Non-irradiated       | 97       | 2.37    | 2.58    | 6.11x10$^{-2}$         | 1617.62         |
| Electron irradiated  | 95       | 2.54    | 2.11    | 5.35x10$^{-2}$         | 1729.35         |
| Gamma irradiated     | 103      | 2.46    | 2.71    | 6.67x10$^{-2}$         | 1548.81         |

The SEM micrographs in Figure 3(a), 3(b) and 3(c) show the microstructures of Bi-2223 phase of non-irradiated and irradiated samples. The granular morphology of all Bi-2223 samples consist of flaky layers of large platelet-like structure. Non-irradiated sample shows lower grain alignment disorientation and the platelet grains are well linked when compared to gamma irradiation samples and this is in agreement with the finding reported by Mohiju et al. [8]. Nevertheless the electron irradiation
sample show better connectivity between the grain boundaries and this explained the higher $J_c$ in the sample. The disorientation of grains in the structure of irradiated samples were caused by particle collision [1]. There are increments in terms of number of layers in irradiated samples compared to non-irradiated.

The microstructures present in oxide superconductors are expected to play a prominent role in determining the current carrying capacity of these materials but the major bottleneck for the flow of the current is in the structure of grains or grain boundaries that connect these colonies. In case of Bi-2223 phase, it has been claimed that a macroscopic c-axis alignment is enough to give high critical currents [9]. Meanwhile, increases in the number of thin rectangular, indicating the decreases in $T_c$ of the Bi-2223 phase and decreasing in porosity of the microstructure.
Figure 3. SEM micrographs of (a) non-irradiated sample, (b) electron irradiated sample, and (c) gamma irradiated sample, respectively.

4. Conclusion
Samples of Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_{10}$ were successfully prepared by conventional solid state method. In the initial stage of the sample preparation, the partial substitution of Bi by Pb plays an important role to improve the structure, as well as facilitating the grain growth. Results show that electron irradiation is a noble method to further enhance the superconducting properties of Bi-2223 phase superconductor. The $J_c$ of electron irradiated sample improved by 6.91% compared to non-irradiated sample without much degradation in $T_c$.

5. References
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