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Phase formation and microstructure of gamma irradiated Bi-2223 Superconductor

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Abstract. The Bi-2223 superconductor has been synthesized using the conventional solid state reaction method. The effect of gamma irradiation on phase formation and microstructure of high-temperature Bi-2223 superconductor ceramic was investigated. The bulk samples sample were palletized with 7 tons pressure of hydraulic press machine and sintered at 840°C for 48 hours. The gamma irradiation was performed at the Nuclear Malaysian Agency with dose of 50 kGray at room temperature. Structure characterization using X-ray diffraction (XRD) showed that the patterns for all the samples demonstrate well-defined peaks all of which could be indexed on the basis of a Bi-2223 phase structure. However, for irradiated sample, it showed reduction in the peak intensity indicating a decrease in the content of the Bi-2223 superconducting phase. The effect of gamma (γ) irradiation on surface morphology and its composites has also been investigated by scanning electron microscope (SEM) and the micrograph shows that the grains are distributed randomly with poorly connected inter and intra-grain microstructure. This shows that the morphology of the Bi-2223 superconductor is very sensitive to gamma irradiation. The effect on the phase formation and microstructure of non-irradiated and gamma irradiated of Bi-2223 superconductor is compared and evaluated.

1. Introduction

Highly textured bulk superconductors have a high potential for electric and magnetic applications such as transmission cables, transformers, current limiters, motors and generators. This is due to its ability to carry high transport critical current densities, J_c. However, the performance of Bi-2223 bulk superconductor is influenced by the density and homogeneity of the microstructure [1-3]. Low electrical current densities, J_c in superconductor are usually associated with grain boundaries weak coupling problem. Since grain boundaries of these materials act as weak links, it is important to produce a single phase material of high transition temperature, T_c (high-T_c) oxide superconductors. Nevertheless due to the high complexity of the reaction and to the small difference in their thermodynamic stabilities, it will be intensely difficult to prepare the (Bi, Pb)-2223 phase as a single phase since it usually intergrowths with the (Bi, Pb)-2212 phase.

There are several methods used to synthesize the high-quality Bi-2223 precursor powder. The most recommended methods are solid-state reaction, oxalate co-precipitation, spray drying and spray pyrolysis [4-6].

It is believed that the formation of Bi-2223 phase is significantly improved through the partial substitution of Bi by Pb. Thus, it is proven that this elemental substitution is effective in stabilizing the formation process of a high-T_c Bi-2223 phase [7-10].

The purpose of this work is to focus on understanding the phase formation and microstructure of gamma irradiated Bi-2223 superconductor. The phase development, microstructure and mechanical properties of gamma (γ) irradiated Bi-2223 sample are reported in this paper.

2. Experimental Procedure

The high-T_c superconducting samples of composition $(Bi_{1.6}Pb_{0.4})\ Sr_2Ca_2Cu_2O_x$ were prepared by the conventional solid-state reaction method. After the initial grounding process, the compound went through annealing process at temperature of 800°C for 36 hours. In order to develop the homogeneity of powder, the resulting oxide powder was reground in agate mortar before pelletized into bulk form. The powder was then pressed into pellets under hydrostatic pressure around 7 tons or 70 000 psi. The pellets were then sintered at 840°C for 48 hours and then furnace-cooled down to room temperature.

Gamma irradiation on the samples was carried out using JS10000 (IR-219) irradiator with radiation dose of 50 kGray. The irradiation was conducted at SINAGAMA Plant of the Malaysian Nuclear Agency. Structural investigations of non-irradiated and irradiated samples were conducted using the Bruker D8 Advanced X-Ray Diffractometer (XRD) to verify their phase formation. Microstructure investigation was carried out using the Hitachi S3400N Scanning Electron Microscope (SEM). Compression tests on the samples were done at room temperature in order to obtain the plot of stress-strain curve, and thus the mechanical properties of the superconductor ceramics.

3. Results and Discussion

The structure of samples was characterized by X-ray diffraction. In figure 1, it shows that both samples are multiphase where Bi-2223 phase co-existed with Bi-2212 phase. Well resolved sharp peaks corresponding to the Bi-2223 phase are observed and indicates a high fraction of textured Bi-2223 phase. The grain alignment is indicated by the predominance of the three main Bi-2223 peaks, while the other reflections of this phase are less obvious. A few peaks that correspond to secondary phase and other impurities are also observed. After the sample is exposed to gamma irradiation, the volume fraction of Bi-2223 phase reduced. This indicates that gamma irradiation diminished the presence of Bi-2223 phase in the superconductor ceramics.

Figure 2(a) shows a well-distributed crystalline structure with flake-type grains and some needle-type grains of different sizes. The grains are distributed randomly and poorly connected, and oriented anisotropically. This growing feature promotes a volume increase, thus the bulk density which resulting in the expansion of the sample.

Meanwhile gamma irradiated sample in figure 2(b) shows that the loss of uniformity and fractured pattern with flaky disposition of the grains regularly disappeared with decreasing grain size, and thus resulted in increasing porosity. These grains are disconnected due to the presence of minor impurities and caused by the energy deposition and thus validates to the decreasing superconductor properties of the ceramics. The microstructure exposes a significant amount of small crystals in the order of 1–2 μm when comparing the morphology of the samples before and after gamma irradiation.

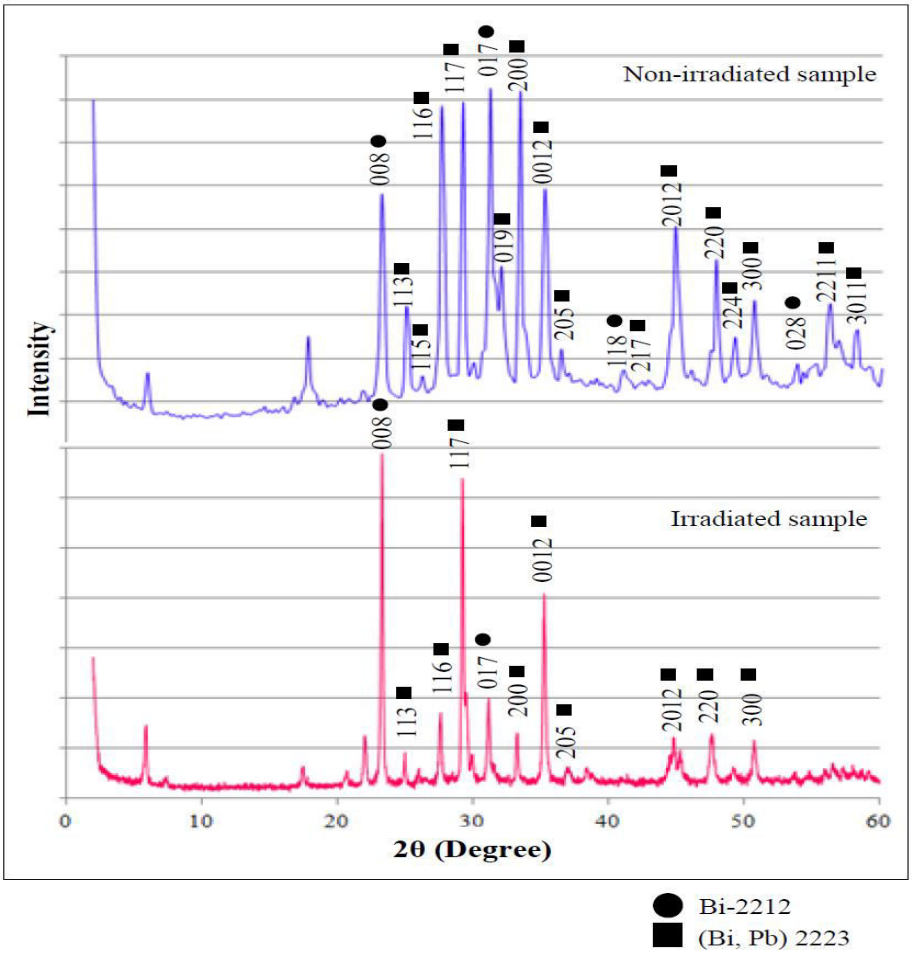
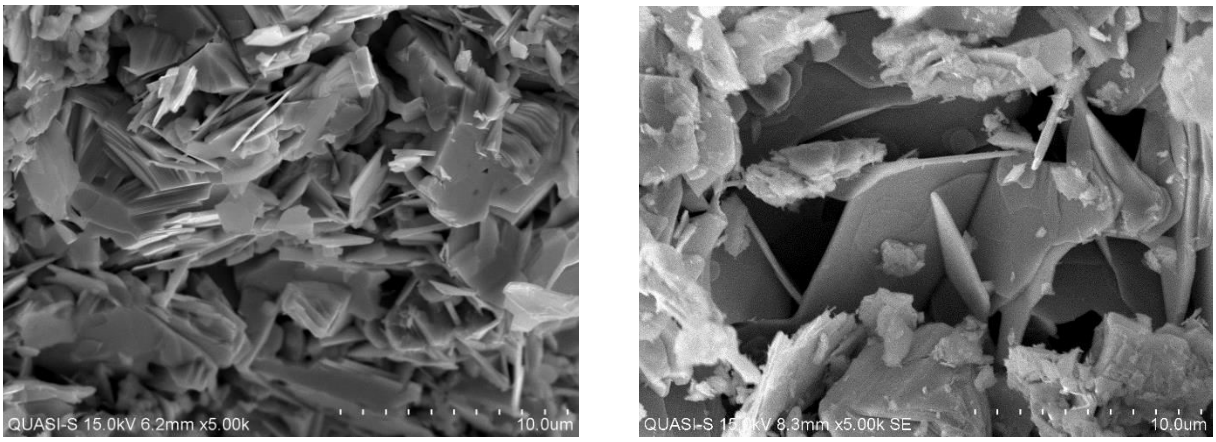


Figure 1. XRD patterns of Bi-2223 for non-irradiated and gamma irradiated sample exposed to 50 kGray.



(a) (b)

Figure 2. SEM micrographs of (a) non-irradiated samples (b) gamma irradiated samples with 50 kGray dose**.**

The energy deposition to local position such as lattice and/or atoms at lattice sites may lead to this rare occurrence of small crystals since the dose rate was not high enough to heat up the samples. Decreasing in grain size would resulted in a decrease in T_c, because of discretization of the energy levels that has been shown to decrease the effective density of states. This phenomena is in accordance with the Bardeen-Cooper-Schrieffer (BCS) relation. For the whole concentration, the transition temperature of the sample decreases proportionally with increasing irradiation dose. Nevertheless further investigation is needed to understand the mechanism better.

4. Conclusion

Irradiation of 50 kGy of gamma ray does not change the phase formation of Bi-2223. However, the intensity of peaks in irradiated sample of Bi-2223 superconductor decreases, and thus reduces the flux pinning and superconducting properties of the sample. SEM analysis was used to view sample's surface topography and composition by creating various images. The non-irradiated micrograph showed a randomly distributed crystalline structure with flake-type grains mixed with needle type-grains. However, the gamma irradiated micrograph showed that the uniformity has lost and the structure is fractured with flaky disposition of the grains that regularly disappeared with decreasing grain size. This resulted in increasing porosity of the microstructure.

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