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Effect of neutron irradiation on microstructure and strength of Bi-2212 phase superconductor

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Abstract. The sustainability of superconductors when exposed to radiation environment is vital for the materials to be used as components and devices in nuclear reactors. In this investigation, TRIGA MARK II research reactor with neutron flux of 2.00×10^{11} /cm^{2}s was used as the neutron source to study the effect of neutron irradiation on microstructure and physical strength of $Bi_2Sr_2CaCu_2$ (Bi-2212) superconductor. Results between non-irradiated and irradiated samples have been analyzed with respect to phase formation, microstructure and strength of the superconductor. The bulk samples were synthesized using the conventional solid-state reaction method. Molar ratio of Bi_2O_3, Sr_2CO_3, $CaCO_3$ and CuO were mixed according to its ratio into composition of Bi:Sr:Ca:Cu = 2:2:1:2. The powder were palletized and sintered at 840°C for 48 hours. Characterization of the samples was done via X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The XRD patterns for the non-irradiated and irradiated samples show well-defined peaks of which could be indexed on the basis of a Bi-2212 phase structure. XRD patterns also indicate that irradiation did not affect the Bi-2212 superconducting phase. From observation of the XRD pattern and microstructure, there is indication that a small amount of Bi-2212 is decomposed into Cu_2O and other impurities while a significant amount of un-reacted Bi-2212 particles embedded at the grain boundaries. For the non- irradiated samples, the microstructure was found to be more textured and thus enhanced the strength of the samples. For the neutron irradiated samples, the results show disorganization of grains orientation and formation of porous structure that led to reduction in overall strength of the Bi-2212 superconductor.

1. Introduction

The sustainability and endurance high-temperature superconductor such as $Bi_2Sr_2CaCu_2O_8$ (Bi-2212) phase superconductor are very crucial before exploring their applications in radioactive environment such as research nuclear reactor. Besides, superconductor materials must be capable to be fabricated as components in various applications of engineering functions and systems. Mechanical properties of superconductor such as strength are much depending on the formation and texturing of its microstructure. Superconductors fabricated into tapes are very attractive due to its flexibility and high efficiency. In industrial applications, superconductor ceramics must display cost effective performance with sufficient mechanical properties as well as optimal electrical and magnetics characteristics [1,2]. As such, it is necessary to study the weaknesses that may hinder their applications such as low mechanical strength.

Textured microstructure plays an important role in enhancing the mechanical properties of superconductor ceramics. Ions and particles irradiation have shown to be able to influence the growth, dislocations or crystal defects in microstructure of most materials, and eventually could altered the mechanical durability of the materials [2-6]. Irradiation such as neutron irradiation is a convenient method to introduce the desired microstructure in superconductors and the correlation with basic properties of superconductors could be studied from such data [7].

In this work, Bi-2212 phase superconductor ceramics was irradiated with neutron particles. Characterization of samples was carried out through X-ray Diffraction (XRD) patterns and Scanning Electron Microscopy (SEM) micrographs. The mechanical strength was determined using compression test at room temperature. The results showed that neutron irradiation weakened the mechanical strength of Bi-2212 phase superconductor.

2. Experimental Procedure

The conventional solid-state method has been used to synthesize the samples. High purity chemical powders were acquired from Sigma-Aldrich, USA. Bi_2O_3, Sr_2CO_3, $CaCO_3$ and CuO powders were mixed according to its molar ratio of Bi: Sr: Ca: Cu = 2:2:1:2. The compounds were ground and heated at temperature of 800 °C for 24 hours to remove impurities and form the Bi-2212 oxide. The resultant powder was palletized and pressed at room temperature with pressure of 7 tons. The samples were then sintered at 840 °C for 48 hours, and eventually furnace-cooled to room temperature.

Some of the samples underwent neutron irradiation with neutron flux of 2.00×10^{11} /cm^{2}s for 6 hours, which is equivalent to fluence 4.32×10^{15} n/cm^2 at the TRIGA PUSPATI Reactor, Malaysian Nuclear Agency. Before neutron irradiation process, there were several initial preparations that need to be done such as fabrication of the aluminium sample holder, and the shielding using aluminium sheet and boron carbide powder. Estimation in details of elements' activation after completion of neutron irradition ended was also recorded. The Nuclear Malaysia Agency has being very cooperative in resolving all the issues occured throughout irradiation process.

Structural investigations of non-irradiated and neutron irradiated samples were conducted using the Bruker D8 Advanced X-Ray Diffractometer (XRD) to confirm their phase formation. Microstructure investigation was carried out using the Hitachi S3400N Scanning Electron Microscope (SEM). The compression test of the samples was conducted using Instron Material Testing System at room temperature. The maximum load was recorded along with the stress-strain data, and thus the strength of the samples was determined.

3. Results and Discussion

Figure 1 shows the XRD patterns of Bi-2212 superconductor for non-irradiated and neutron irradiated samples. The XRD patterns show well-defined peaks of which could be indexed on the basis of the Bi-2212 phase structure. A few peaks which correspond to unknown phases and other impurities are also observed. Those peaks are mainly due to an inappropriate heat treatment during the sintering process. There is indication that a small amount of Bi-2212 is decomposed into Cu_2O and other impurities while a significant amount of un-reacted Bi-2212 particles embedded at the grain boundaries. For both the non-irradiated and irradiated samples, there is no shifting of peaks happened and as such the volume fraction of the Bi-2212 phase remains unchanged. This indicates that neutron irradiation does not alter the phase formation and structure of the Bi-2212 superconductor.

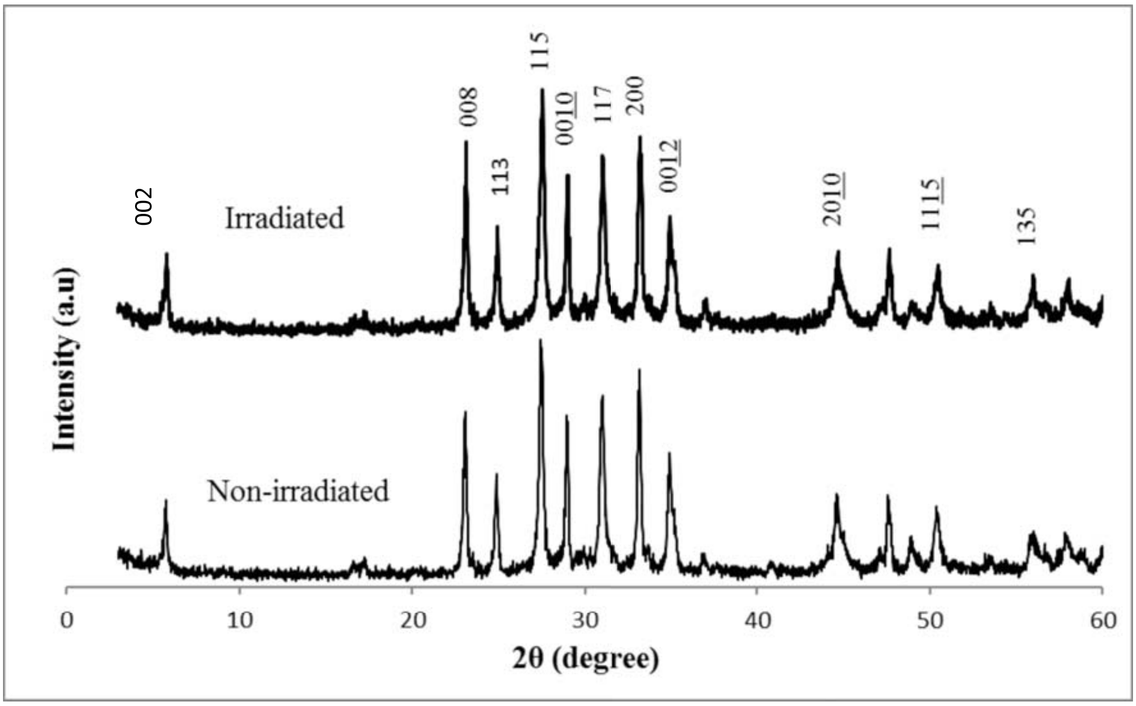


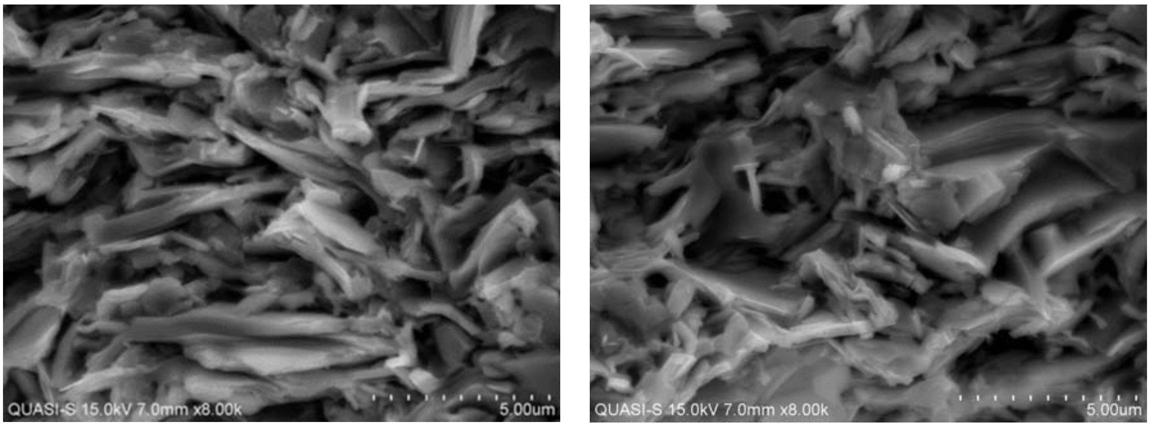
Figure 1. XRD patterns of non-irradiated and neutron irradiated Bi-2212 phase superconductor.

Table 1 shows the lattice parameters (*a*, *b*, and *c*) of the samples. Both the non-irradiated and irradiated samples exhibit orthorhombic crystalline structure in which lattice parameter $a \neq b \neq c$. There is a slight reduction in size for the irradiated sample due to re-alignment of the grains texture after subjected to neutron irradiation.

Table 1. Lattice parameter of non-irradiated and irradiated Bi-2212 phase superconductor.

Lattice parameters	*a* (Å)	*b* (Å)	*c* (Å)
Non irradiated	5.3826	4.9348	32.5208
Irradiated	5.3542	4.8941	32.3145

Figure 2(a) shows the microstructure of non-irradiated sample that has a well-distributed crystalline structure with flake-type grains of different sizes. Its microstructure consists of denser and well-structured texture. In contrast, the microstructure of the neutron irradiated sample as shown in Figure 2(b) comprises of grains that are distributed randomly and poorly connected, and oriented anisotropically. The irradiated sample shows loss of uniformity and fractured pattern with flaky disposition of the grains regularly disappeared with decreasing grain size, and thus resulted in increasing porosity. Thus, the disorganization of grains orientation and formation of porous structure resulted in the reduction of overall strength of the materials.



(a) (b)

Figure 2. SEM micrographs of (a) non-irradiated (b) neutron irradiated Bi-2212 phase superconductor.

The mechanical properties such as stiffness, strength and toughness at room temperature are able to predict such behaviors at cryogenic temperature [8]. The relationship between the microstructure and strength of the Bi-2212 superconductor is useful to assess the effect of neutron irradiation on the mechanical properties of the samples. Table 2 shows the maximum load and compressive strength of both the non-irradiated and irradiated samples at room temperature.

Table 2. Maximum load and compressive strength for non-irradiated and neutron irradiated samples.

Sample	Maximum load (kN)	Compressive strength (MPa)
Non-irradiated	21.72	163.64
Irradiated	16.02	117.41

For the neutron irradiated sample, the maximum load and strength decreases due to the disorientation and dis-alignment of the Bi-2212 superconducting grains. Disorientation and dis-alignment of the microstructure resulted in deformability in the irradiated sample. This contributed to weakening of the interface bonding between the Bi-2212 superconducting grains, and hence the deteriorating mechanical strength in the neutron irradiated samples.

4. Conclusion

Neutron irradiation does not change the phase formation of Bi-2212 superconductor. Nevertheless based on XRD patterns and SEM micrographs, there are evidences that a small amount of un-reacted Bi-2212 phase and other impurities resided at the grain boundaries of the Bi-2212 superconducting grains. Neutron irradiation does not promote the formation of textured microstructure and thus has no strengthening effect on the Bi-2212 superconductor.

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