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FABRICATION OF MgO FIBERS AND THE MECHANICAL PROPERTIES OF MgO FIBERS ADDED Bi-2212 SUPERCONDUCTOR COMPOUNDS

N.A. Hamid, N.F. Shamsudin, K.M. Chin and K.W. See

Department of Engineering Sciences & Mathematics,
College of Engineering, Universiti Tenaga Nasional,
43009 Kajang, Selangor, Malaysia

ABSTRACT

MgO fibers have been fabricated in-house using the sol-gel route. In the process, magnesium turnings were added with methanol to produce magnesium methoxide. Ethylene glycol was then added to the mixture and stirred continuously to form the desired gels. The gels were extruded and heat treated to produce the cylindrical shape MgO fibers. Small weight percentage of 3% to 8% MgO fibers were added to $Bi_2Sr_2CaCu_2O_8$ (Bi-2212) superconductor powder. The Bi-2212/MgO fibers compounds were palletized and heat treated through partial- melt process, followed by slow cooling. The samples were characterized by x-ray diffraction patterns (XRD), emission scanning electron microscopy (FESEM) with energy dispersive x-ray (EDAX), and dc electrical resistance measurements at zero magnetic fields. Compression tests were conducted to study the mechanical behavior of the samples. From the characterization results, additions of small amount of MgO fibers improved the texture of the Bi-2212/MgO fibers compounds. Significantly higher stiffness, strength and toughness were observed in the compounds with 5% MgO fibers addition.

INTRODUCTION

MgO fibers having very high melting point with lower heat capacity, are excellent candidates for reinforcement of brittle materials such as superconductor ceramics [1-3]. In addition to high melting point, the MgO fibers are extremely high corrosion resistance [1]. MgO fibers have been used to reinforce composite materials and have shown to improve the mechanical properties of superconductors [2]. In the composite reaction textured process of Bi-2212 superconductor ceramics, the MgO fibers are found to align in a planar 2D arrangement and led to a strong c-axis texture of the ceramics [3]. Strong c-axis texture is important to overcome the critical current limiting mechanism between the superconducting grains at liquid nitrogen temperature [4]. Since all high-temperature superconductors are anisotropic, the properties of the weak links could be improved by eliminating or minimizing the high angle grain boundaries in the path of transport current, which should be parallel to the ab plane [5]. Previous study by Suhara et al. [6] has proved that the grain boundary angle that is larger than 15º will limit the J_c value as weak links. However for grain boundary angle that is lower or equal to 10º, it will enhance the J_c value because of the existence of strong links between the adjacent grains. Improvement in the microstructure enhanced the connectivity of the adjacent grains, and as such promotes the establishment of strong

Corresponding Author: nasri@uniten.edu.my

links. Better texture of the microstructure in superconductor ceramics such as the Bi-2212 phase could be achieved by controlling heating and subsequent annealing processes [7].

In term of mechanical properties such as the mechanical strength, the addition of MgO fibers is expected to reinforce the superconductor compounds [1-4]. This paper reported the fabrication of MgO fibers and the effect of MgO fibers addition to the microstructure and mechanical properties of Bi-2212 superconductor compounds. The Bi-22212/MgO fibers samples were subjected to compression test where the maximum load was recorded along with stress-strain data. Significantly higher stiffness, strength and toughness were observed in the compounds with MgO fibers addition.

METHODOLOGY

In the fabrication of MgO fibers, the sol-gel route was applied. In this method, magnesium turnings, methanol with purity 99.9 % and ethylene glycol were used. In the beginning, the magnesium turnings was added with methanol and stirred continuously. After forming magnesium methoxide, ethylene glycol, was added to the mixture in a beaker and the beaker was sealed and stirred continuously. The mixture was intensely stirred until forming a gel. After 10 days, the gel was extruded using a syringe and left for a day at room temperature before thermally treated. To see the effect of heat treatment on the formation of MgO fibers, the gel was heated at 1150ºC and 1300ºC, respectively. Figure1 shows the heat treatment schedule used in the fabrication procedures.

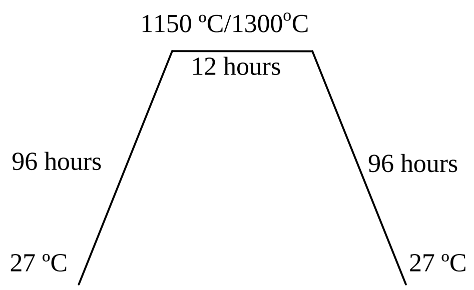

Figure 1: Heat treatment schedule for fabrication of MgO via sol-gel route

Subsequently, the MgO fibers were mixed with Bi-2212 superconductor powder (purity 99.99%) to obtain Bi-2212/MgO fibers compounds. In this study, the Bi-2212 powder was produced by Aldrich, USA. The mixed powder were used to prepare pellets with a diameter of 13-mm and a thickness of about 2-mm each after pressing under pressure of 5-6 tons. The pellets were then subjected to partial melt process as shown in Figure 2. The partial-melt process is employed to obtain the required texturing of the Bi-2212/MgO fibers compounds. Partial-melt processing of Bi-2212 is known to significantly improve the microstructure of Bi-2212 phase.

Corresponding Author: nasri@uniten.edu.my

The samples were characterized via x-ray diffraction (XRD) patterns, emission scanning electron microscopy (FESEM) with energy dispersive x-ray (EDAX), and dc electrical resistance measurements. Compression test was conducted at room temperature using the Instron Material Testing System model 5567, and the maximum load was recorded along with the stress-strain data.

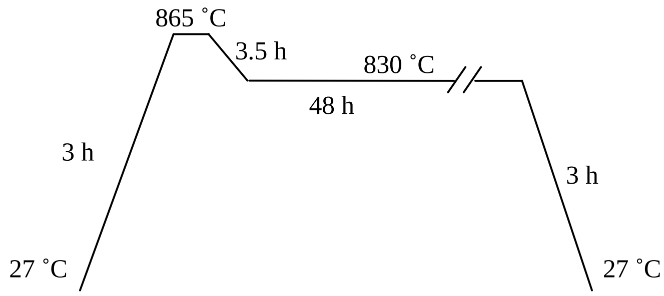

Figure 2: Heat treatment schedule in partial-melt process of Bi-2212/MgO fibers compounds

RESULTS AND DISCUSSION

Figure 3 shows the XRD patterns of the fabricated MgO fibers. The XRD patterns for MgO fibers sintered up to 1300 ºC show sharper and well-defined 2010 and 0012 reflections, indicating higher purity of MgO fibers with higher degree of crystallization. Figure 4 shows the SEM micrograph of the fabricated MgO fibers. Each strand has cylindrical shape structure with an average diameter of 400 μm to 500 μm.

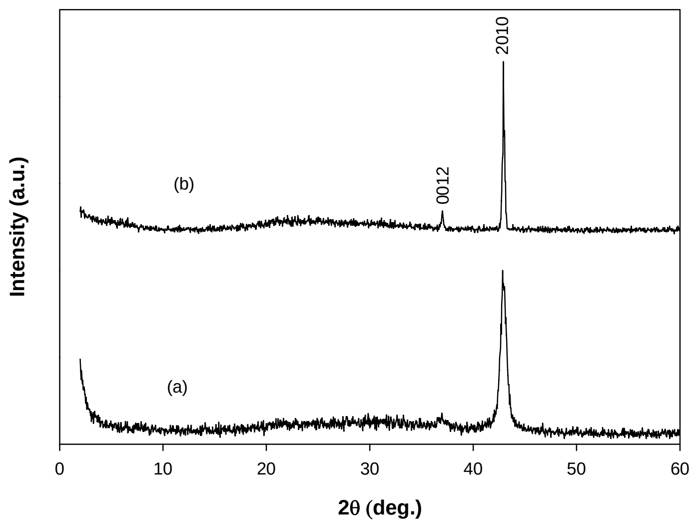

Figure 3: XRD patterns of MgO fibers heated at (a) 1150 ^{o}C, and (b) 1300 ^{o}C

Corresponding Author: nasri@uniten.edu.my

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The dc resistance measurements confirmed that all the samples are superconducting at onset temperature between 80K to 85K. Figure 5 shows the XRD patterns of the Bi-2212/MgO fibers superconductor compounds. The patterns show well defined reflections, all of which could be indexed on the basis of a Bi-2212 phase with an orthorhombic structure. Reflections corresponding to MgO can hardly be recognized due to the small amount of MgO fibers addition. By comparing with the XRD patterns of non-added sample, MgO are presence in all the added samples and confirmation were made by SEM and EDAX analysis. This indicates that the MgO fibers dispersed in the superconductor compounds without any reaction with the Bi-2212 phase during the partial-melt process.

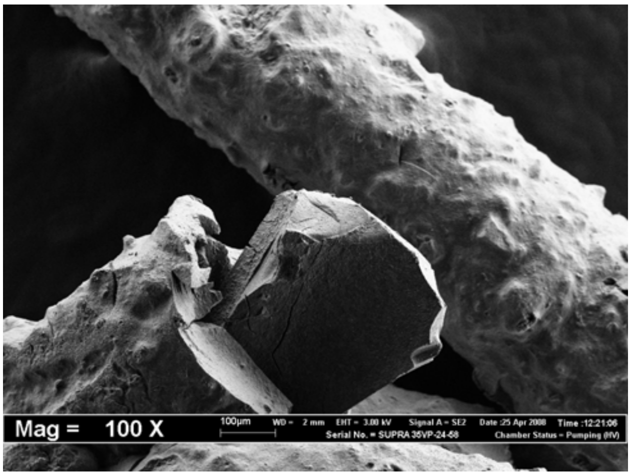

Figure 4: SEM micrograph of MgO fibers

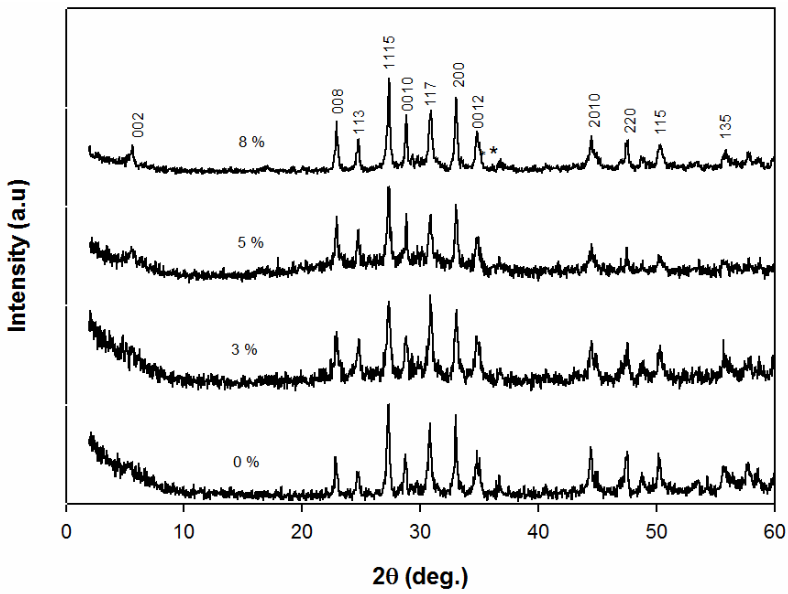

Figure 5: XRD patterns of Bi-2212 with (a) 0 %, (b) 3 %, (c) 5 %, and (d) 8 % Mg fibres addition. * indicates the peaks for MgO

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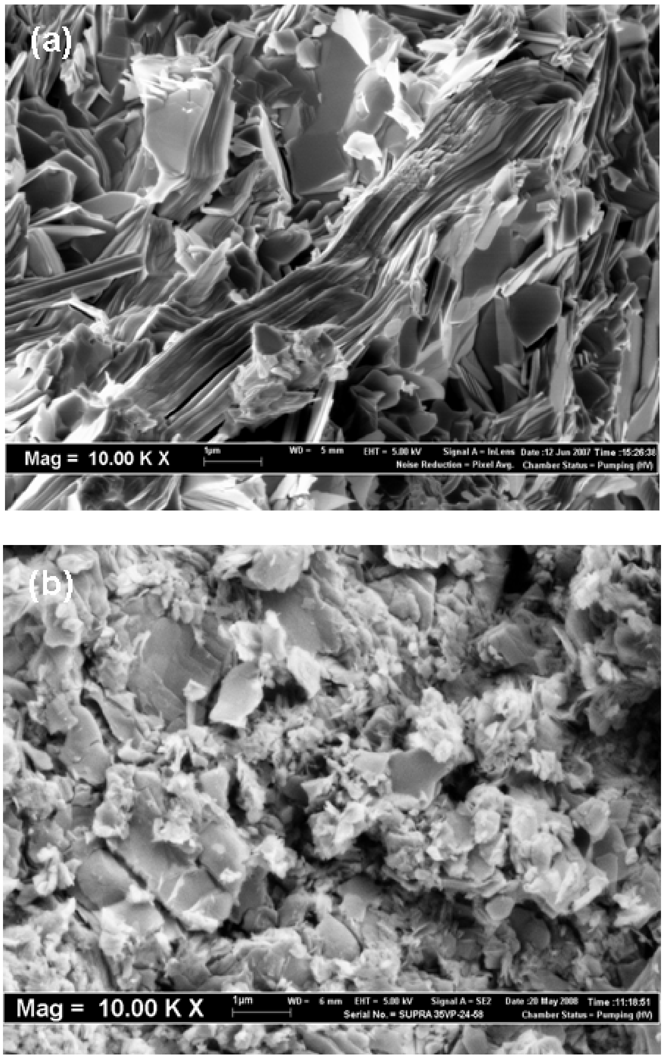

Figure 6: SEM micrographs of Bi-2212/MgO fibers compounds with (a) 0%, and (b) 5% MgO fibers addition, respectively

Figure 6 shows the SEM micrographs of Bi-2212 superconductor for non-added and MgO fibers added samples. From the SEM micrographs as illustrated in Figure 6 (b), MgO fibers are likely to reside near the grain boundaries of the Bi-2212 phase superconductor. Results of SEM micrographs and XRD patterns clearly show that a small addition of MgO fibers significantly improved the texture of the microstructure. The superconducting grains in the samples are seen to align in much lower degree of orientation. Nevertheless, for sample with 8% MgO fibers addition, the SEM microstructure shows higher porosity and subsequently resulted in microcracks between the adjacent Bi-2212 phase grains, and the orientation of the grains is much more perpendicular to the c-axis.

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The Bi-2212 phase superconductor grains are known to have a platelet-like shape and thus their morphology are able to be controlled and fabricated into well-textured microstructure. Those changes are related to the composition of the superconducting phase and the texturing of the morphology during partial-melting process [7]. Texture is an important feature of the characterization of powders utilizing the x-ray diffraction (XRD) method because it manifests itself by variation of the intensities of particular (hkl) reflections. It is possible to extract preferred orientation information from such patterns if the pattern for a randomly oriented specimen can be modeled, or simulated, from knowledge of the crystal structure parameters and various other factors which influence the pattern [8]. As such, the effect of MgO fibers addition on the texturing of the Bi-2212 compounds could be determined through the calculation of texture coefficient (TC). Based on the calculation of texture coefficient in Ni-Al_2O_3 composite as reported by Chen et al. [9], similar approach is used to calculate the texture coefficient for Bi-2212/MgO fibers compounds. As such, the texture coefficient can be expressed as

$$TC(hkl) = \frac{100 \times I_{hkl}}{\sum_{i=1}^{n} I_{hkl}}$$

where I_{hkl} is the intensity of the (hkl) reflection.

Table 1: Texture coefficient of (002) reflection and fracture limit of Bi-2212/MgO fibers compounds at room temperature

MgO FIBERS ADDITION	**TEXTURE COEFFICIENT (002)**	**FRACTURE LIMIT (kN)**
0 %	7.62	22.5
3 %	8.06	26.6
5 %	8.15	30.2
8 %	8.82	24.2

In addition, the strength of the Bi-2212/MgO fibers superconductor compounds was tested using compression test at room temperature. The test is able to measure the plastic flow behavior and ductile fracture limit of the Bi-2212/MgO fibers compounds. The results show a linear relationship between the load and the compression extension until they reached the fracture limits. Thus, at room temperature the mechanical strength of the bulk Bi-2212 superconductor is found to increase with the addition of MgO fibers. Therefore, the higher mechanical strength of the superconductor is attributed to the presence of MgO fibers. Table 1 shows the correlation between texture coefficient of (002) reflection and the fracture limit of the Bi-2212/MgO superconductor compounds. The texture coefficient shows an increasing trend with MgO fibers addition. Samples with higher texture coefficient are found to have better mechanical strength. However, the strength is deteriorating with 8% MgO fibers

Corresponding Author: nasri@uniten.edu.my

addition. As shown in the SEM micrograph, the porosity resulted in the formation of microcracks at the interface between the Bi-2212 grains and MgO fibers, and thus the decreasing trend.

The mechanical properties such as stiffness, strength and toughness at room temperature are able to predict such behaviors at cryogenic temperature. In their works on glass/epoxy reinforced Bi-2212 bulk superconductor, Tamura et al. [10] found that the compressive strength of the superconductor composites at 77 K did not change that much compared to the result obtained at room temperature. In our case, the Bi-2212/MgO fibers compounds fractured at room temperature in a brittle manner, but at 77 K the crack growth may has initiated and subsequently propagated along the interface between the superconductor matrix and the MgO fibers, before fractured. Similar behavior has been reported in the Bi-2223 reinforced compounds [11,12].

CONCLUSION

With the employment of the partial-melt processing, small addition of MgO fibers produced better texturing of the microstructure of Bi-2212 phase superconductor. The MgO fibers that resided near the grain boundaries of the Bi-2212 phase lowered the grain orientation of the Bi-2212 superconducting grains, and subsequently improved the intergranular connectivity of the grains. The texture coefficient and fracture limit of the Bi-2212 compounds were increase with MgO fibers addition. As such, small addition of MgO fibers reinforced the Bi-2212 bulk superconductor without disrupting the Bi-2212 superconductor phase structure.

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