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Characterization of ferrofluid-polymer composites on high T_c superconductor $Bi_2Sr_2Ca_2Cu_3O_y$

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Abstract

The effect of Mn–Zn–Gd ferrofluid polymer composite film on high-T_c superconducting material Bi (2 2 2 3) has been studied. These films were prepared under the influence of with and without magnetic field and were characterized by XRD, SEM and VSM techniques. The study revealed that the techniques used does not effect the superconducting properties of the material for making devices based upon this. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Composites; XRD; SEM

1. Introduction

Ferrofluid-polymer composite is a dispersion of magnetic particles in a polymer matrix. The inclusion of non-magnetic particles like high-T_c materials dispersed in magnetic fluid are called the magnetic holes [1]. Collection of particles will thus behave like a many-body system with dipole interaction which may be controlled by external magnetic field. The effective dipolar interaction may be either attractive or repulsive depending on the external magnetic field and forming an angle θ relative to the line at the center of the two magnetic particles [2]. Kalikmanov and Dyadkin [3] had carried out theoretical studies on superdiamagnetic fluid (colloidal suspension of high-T_c superconducting particles) and magnetic fluids composites has shown that it possesss very unusual properties such as high initial susceptibility, dynamic Josephson contacts and non-analytical magnetization curves and other physical characteristics. In the present investigations we have synthesized the ferrofluid-polymer composite and this was used for making two sets of experiments (i) thin film of 3 μm thickness was deposited on a high-T_c pellet Bi (2223) under the influence of with and without magnetic field and (ii) high-T_c particles were uniformally distributed in ferrofluid-polymer composite mixture and then grown as two sets of thin film on glass substrate [4]. In this paper we report and discuss the results about XRD, SEM and VSM studies carried out on the above samples. The studies will give an insight for the future experiments to understand the unusual behaviour of such composites and explore the possibility for device applications.

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2. Experimental

One gram of water-based ferrofluid (Mn–Zn–Gd) having particle size ~ 6.5 nm, a saturation magnetization of 150 G and 1 g of polyvinyl alcohol solution was used to prepare ferrofluid-polymer coating on Bi (2 2 2 3) high T_c material. The Bi(2 2 2 3) was synthesized by well-known solid-state reaction route with $T_c \sim 117.2$ K. The calcination of high purity oxides was done at 820°C and the calcined powder after ball milling was uniaxially compacted at 0.3 GPa pressure for making pellets of dia 15 mm, thickness about 2 mm. The pellets were finally sintered at 842°C in air followed by furnace cooling. Superconducting transition temperature T_c was measured by the standard four-probe DC electrical resitivity method in temperature range 77–300 K. Ultrafine particles of high-T_c material (0.2 g) was mixed into pre-prepared ferrofluid-polymer solution. The films were grown by spinning technique and dried under the influence of with and without magnetic field in the ambient and the clean environment. Basically two sets of ferrofluid polymer composite films were prepared (1) films grown on high-T_c pellet (p) under the influence of (film c) and without (film d) magnetic field, (2) homogeneous mixture of ultrafine high-T_c particles and ferrofluid polymer were used to grow on glass substrate under the influence of (film a) and without (film b) magnetic field. The applied magnetic field is of the order of 500 G and is normal to the substrate. Characterization were carried out by Siemens D-500 powder X-ray diffractometer (XRD) and DMS-880 vibrating sample magnetometer (VSM) techniques. A Jeol JSM 840 scanning electron microscope (SEM) was used to characterize the surface morphology of the films.

3. Results and discussion

The XRD study shows Bi (2 2 2 3) and Mn–Zn–Gd phases present in all the samples and a representative XRD pattern is depicted in Fig. 1. We have observed the film grown without magnetic field has the line broadening as compared to the film grown under the influence of magnetic field. This reveals that there is a increase in the crystallite size for the film grown under the magnetic field and also some reflections corresponding to high-T_c phase shows increase in their intensities. In order to see these changes precisely we have taken the slow scan of various peaks and the crystallite size was calculated by using a well-known Scherrer equation [5]. The difference in crystallite size for the film prepared with and without magnetic field on high T_c pellet is of the order of 5 nm. A similar behaviour was also observed for the film grown by mixing the ferrofluid with the ultrafine particles of the high-T_c material. The increase in crystallite size is due to crystallographically oriented individual domains forming a uniform size of agglomerates observed in the SEM micrographs.

The surface structure and morphology of high-T_c pellet, ferrofluid composite films on pellet and the homogeneous mixture of high-T_c material and ferrofluid polymer on glass substrate were recorded (for the samples p, c, d, a and b) at 15 kV with magnification 50 000. Fig. 2 shows the platelet structure of Bi (2 2 2 3) pellet. A random distribution of individual domains of ferrofluid particles in the polymer matrix have occurred in sample (d).The grain size is slightly higher than the actual

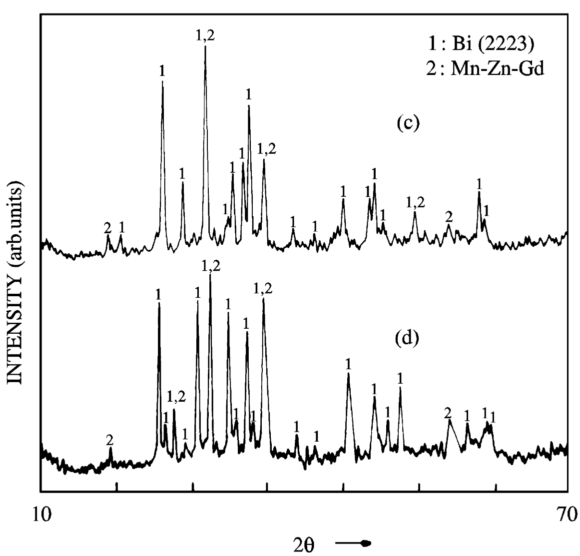

Fig. 1. XRD pattern of ferrofluid-polymer composite films grown on high-T_c Bi (2 2 2 3) pellet: (c) with field (500 G) (d) without field.

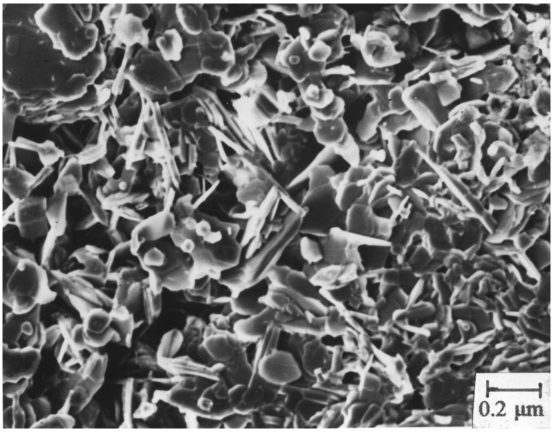

Fig. 2. SEM micrograph showing the morphology of the high-T_c pellet (p).

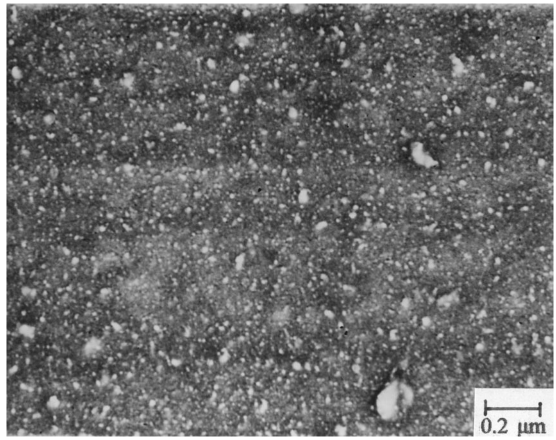

Fig. 3. SEM micrograph showing the morphology of the film (d).

particle size and is found to be in the range 8–10 nm (Fig. 3) [6]. A uniform and spherical size agglomeration of ferrofluid particles have occurred in sample (c) (Fig. 4).The size of agglomerates as measured are in the range of 30–40 nm. It is very interesting to see the localized inter-particle forces lead to the formation of uniform-sized agglomerates under the influence of the magnetic field. Also, the cross sectional morphology of the samples showed the orientation of the domains are in the field direction. This indicates the presence of superparamagnetic (SP) and non SP particles in the system which increases the interparticle interaction as also observed by Jeyadevan et al. [7] in their system on Mn–Zn ferrite and magnetite based ferrofluid. Similar observations were also observed by Yokoi et al. [8] for magnetite PVA solution and reported that the diameter of the agglomeration is of the order of 4–10 nm.

The result of studies carried on magnetic moment versus applied magnetic field (7×10^3 Oe) at room temperature of the samples using VSM are shown in Table 1. For the sake of comparison the results of only ferrofluid polymer composite films prepared with (film e) and without (film f) magnetic field are also included in the table. Films prepared under the influence of magnetic field show higher magnetization in comparison with the films prepared without the influence of magnetic field. This behaviour could be related to the presence of variation in the particle size distribution. The particle in the SP size limit may be getting aligned in the direction of the applied magnetic field leading to larger magnetic moment. Ferrofluid, as we know that, usually consist of particles below the SP size range. The SP behaviour is observed when the thermal energy kT of the particles exceeds over the magnetic energy KV. In the case of Gd ferrite used in the present study the K being relatively large (1.6×10^6 erg/cm^3), the particle size limit for SP behaviour is expected to be smaller. Therefore, a fraction of the particles are likely to be very close

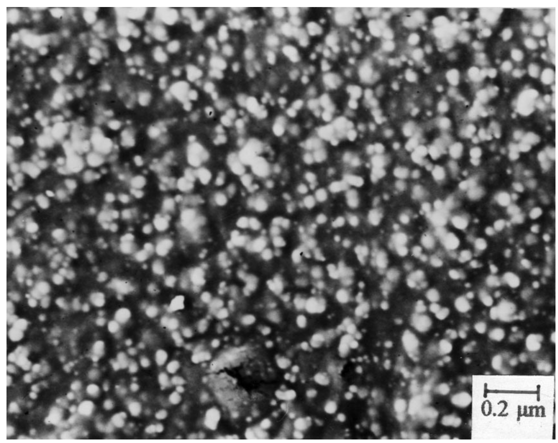

Fig. 4. SEM micrograph showing the morphology of the film (c).

Table 1
VSM data of composite films

Ferrofluid polymer composite film prepared	Magnetic moment (emu)			
Without influence of magnetic field	(p) 0.0034	(d) 0.0351	(b) 0.0252	(f) 0.0086
With influence of magnetic field	—	(c) 0.0563	(a) 0.0680	(e) 0.0129

to the SP size limit. This fraction of the particles could be getting aligned when the film is grown in the presence of applied magnetic field. A similar behaviour is observed in both the cases of films prepared on high-T_c pellet as well as in the case of high-T_c fine powder mixed with the ferrofluid particles. The high-T_c material does not seem to influence the magnetic behaviour of the ferrofluid film at room temperature because of its paramagnetic behaviour.

4. Conclusion

The study reveals that by polymerization techniques the stablized crystallographic phase of high-T_c materials retains. The uniform size agglomerate distributions are formed due to superparamagnetic behaviour of the particle under the influence of the magnetic field. This type of ferrofluid coated high-T_c pellet may have some applications in superconducting devices such as switches, sensors, etc.

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