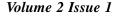
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Nd-laser irradiation effects on nano-synthetized Sb-doped-123-cuprate composite

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ABSTRACT

The nano-structured Sb-doped-YBCO (YSb_{0.1}Ba₂Cu₃O₇) pellet which has Tc (Tc-onset = 91.7K and Tc-offset = 84.45K) was selected to be the target for Nd-pulsed laser irradiation source. The pellet was exposured for two different doses of laser beam irradiation the 1st 15 W/cm² for 100min. and 2nd 30 W/cm² for 200min. The irradiation was carried out in air without any external heating. The energies of pulsed Nd-laser were sufficient to melt homogeneously the surface and near surface layers. SEM Investigations was used for monetoring the nano/microstructural changements as a function of laser irradiation dose. The structural and superconducting properties were investigated carefully as a function of Nd-laser irradiation doses. © 2008 Trade Science Inc. - INDIA

INTRODUCTION

123-YBCO superconducting regime is considered the most interesting superconducting materials for various reasons, in particular for their rather high critical temperature Tc and high critical current density Jc. Many researchers have investigated the effect of metal cation dopants on the123-YBCO superconducting system^[1-8]. Others like^[9] have studied the effect of CeO₂ and PtO₄ mixed oxide additives on the microstructural and critical current density Jc. They reported that (Ce + Pt) oxides added to the melt- textured YBCO have significantly improved on the value of Jc ~4.3×10⁴ A/cm². The role of additives as impurity phases like (silver, silver oxide,.) to improve processing, magnetization and microstructure of YBCO system were studied by many authors^[10-16].

KEYWORDS

Nano-synthesis; Doping; Laser irradiation; SE-microscopy; X-ray; Superconductors.

The influence of laser radiations on the characteristics of HTSC was studied in a some previous publications such as^[17-24]. However their results are ambiguous and inconsistent. In general, degradation of the HTSC-material is observed, but there is also data showing an increase in the critical current^[17-22].

Thin ceramic films or powder-sintered ceramic surface recrystallized with precise control over thickness, structure, orientation of grains, and stoichiometry are highly desirable for applications in electronic devices. Surface treatment by infrared laser beam is effected instantaneously via sequential thermal processes such as heating, melting, vaporization, and molten zone solidification during rapid cooling^[19-21]. Under such thermal conditions, the surface is expected to experience non-equilibrium state, thereby acquiring totally new functional properties. The surface of oxide material con-

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taining many pores, micro-cracks, insulating phases and impurities at the grain boundaries have modified to achieve significant increase in the critical current density.

A few numbers of authors were found in literature such as^[25,26] who investigated the effect of Laser-irradiations on HTc's superconductors. Luctiv et al.^[25] have studied the effect of different kind of laser (Nd,CO₂ and ruby) laser irradiations on three families of high-Tc's superconductors namely 123-YBCO,Bi-2212 and Bi2223 and they reported that laser irradiations cause an enhancement of critical current denisty J_c but supresses T_c because of fraction volume of the diamagnetism inside the bulk of superconducting sample decreases.

The major goal in the present article is to investigate the effects of Nd-laser irradiations on;

- a. Structural and nano/micro-structural properties of optimally Sb-doped YBCO sample.
- b. Superconducting properties of optimally Sb-doped YBCO sample (Tc-onset 91.7 K)

EXPERIMENTAL

Samples preparation

The best antimony containing composite with general formula; $Y_{1+x}Sb_xBa_2Cu_3O_z$, where x = 0.1 mole was selected from another study for author^[27] to be the target for Nd-Laser irradiation.

Nano- synthesis was attempted by using solvo thermal route and sintering procedure using the appropriate amounts of Sb_2O_3 , Y_2O_3 , BaCO₃ and CuO each of highly pure chemical grade purity.

Precursor synthesis

The mixtures oxides $(Sb_2O_3, Y_2O_3, BaCO_3 and CuO)$ were ground carefully and sieved through 50µm mesh,10ml of conc HNO3 was added to the fine powders with stirring till complete miscibility.

The resultant nitrates solution was diluted to 100ml by distill water then sprayed into liquid nitrogen medium and finally the powders forwarded to the freeze dry machine.

The dry powders were calcined at 800°C under a compressed O_2 atmosphere for 20hrs then reground and pressed into pellets (thickness 0.2cm and diameter 1.2cm) under 6 Ton/cm². Sintering was carried out un-

Aano Solence and Aano Technology An Indian Journal der oxygen stream at 920°C for 200hrs. The samples were slowly cooled down (20°C /hr) till 500°C and annealed there for 20hrs under oxygen stream. The furnace is shut off and cooled slowly down to room temperature. Finally the materials are kept in vacuum desiccator over silica gel dryer

Laser irradiation source

The pellet of the best Tc (Tc-onset= 91.7K Tcoffset = 84.45K) $Y_{1+x}Sb_xBa_2Cu_3O_z$, where x = 0.1 mole was selected and cut into square with dimensions 0.4×0.4cm then polished carefully and forwarded to be the target for Nd-pulsed laser which has the following parameters : wavelength λ max =1.06µm, pulsed rate $\alpha = 10^{-3}$ s. The target was exposured for two different doses of laser beam irradiation the 1st 15W/cm² for 100min. and 2nd 30W/cm² for 200 min. The irradiation was carried out in air without any external heating.The energies of pulsed Nd-laser were sufficient to melt homogeneously the surface and near surface layers.SEM was used for monetoring the morphological changements.

Phase identification

The X-ray diffraction (XRD) measurements were carried out at room temperature on the fine ground samples using Cu-K_a radiation source,Ni-filter and a computerized STOE diffractometer/Germany with two theta step scan technique.

Scannig Electron Microscopy (SEM) measurements were carried out at different sectors in the prepared samples by using a computerized SEM camera with elemental analyzer unit (PHILIPS-XL 30 ESEM / USA).

Superconducting measurements

The cryogenic AC-susceptibility of the prepared materials was undertaken as a function of temperature recorded in the cryogenic temperature zone down to 30K using liquid helium refrigerator.

RESULTS AND DISCUSSION

Phase identification

Figure(1a-c): displays the X-ray powder diffractometry patterns for non-irradiated optimally Sb-doped YBCO (YSb_{0.1}Ba₂Cu₃O₂) and after 1st and 2nd

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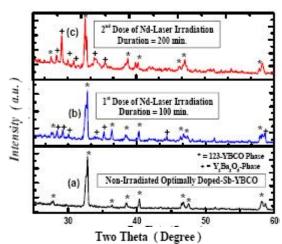


Figure (1a-c): X-ray diffraction patterns recorded for (a):Non irradiated optimaly Sb-doped YBCO; (b): After 1st Nd-Laser irradiation dose 15W/cm²,100min; (c): After 2nd Nd-Laser irradiation dose 30 W/cm²,for 200min

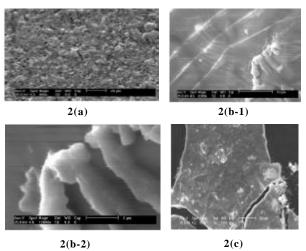


Figure (2a-c): SE-micrographs recorded for :(a) Non irradiated optimaly Sb-doped YBCO with M.F.= 10 μ m; (b) After 1st Nd-Laser irradiation dose 15 W/cm², 100min. 1.M.F. = 20 μ m and 2. M.F. = 2 μ m; (c) After 2nd Nd-Laser irradiation dose 30 W/cm², for 200 min. with. M.F. = 20 μ m M.F.= Magnification Factor

Nd-laser-irradiation dose respectively. Analysis of the corresponding 20 values and the interplanar spacings d (Å) were carried out and indicated that, the X-ray crystalline structure mainly belongs to a single superconductive orthohombic phase 123-YBCO in major besides few peaks of Sb_2O_3 as secondary phase in minor. The unit cell dimensions were calculated using the most intense X-ray reflection peaks to be a = 3.8178Å, b = 3.8163Å and c = 11.6131Å for the optimally Sb-doped

123-YBCO phase which is fully agreement with those mentioned in the literature^[4-8].

It is obviously that, the additions of Sb_2O_3 has a negligible effect on the main crystalline structure 123-YBCO by increasing Sb-content (x=0.1) as shown in figure (1a-c).

From figure 1a-c one can indicate that the two different doses of laser beam irradiation the 1^{st} 15 W/cm² for 100min. and 2^{nd} 30W/cm² for 200min.have a slight effect on the internal structure of the optimally Sb-doped YBCO sample as expected but in our view it will give a remarkable changements on the morphological structure.

SE-microscopy measurements

Figure (2a-c) show the SEM-micrographs recorded for non-irradiated optimally Sb-doped YBCO (YSb $_{0.1}$ Ba₂Cu₃O_z) and after 1st and 2nd Nd-laser-irradiations doses respectively. The estimated average of grain size was calculated and found in between 0.11 and 0.60µm (100-160nm) supporting the data reported in^[27].

The EDX examinations for random spots in the same sample confirmed and are consistent with our XRD analysis for polycrystalline doped-YBCO composites, such that the differences in the molar ratios EDX estimated for the same sample is emphasized and an evidence for the existence of 123-YBCO superconductive phase with good approximate molar ratios.

From figure (2a-c), it is so difficult to observe inhomogeneity within the micrograph due to that the powders used are very fine and the particle size estimated is too small.

The grain size for 123-YBCO-phase was calculated according to;

Scherrer's formula^[28],

 $\mathbf{B} = 0.87 \ \lambda/\mathbf{D} \cos \theta$

(1)

where D is the crystalline grain size in nm, θ , half of the diffraction angle in degree, λ is the wavelength of X-ray source (Cu-K α) in nm, and B, degree of widening of diffraction peak which is equal to the difference of full width at half maximum (FWHM) of the peak at the same diffraction angle between the measured sample and standard one.

From SEM-maping, the estimated average grain size was found to be (0.33-0.79µm~330-780nm) which is relatively large in comparison with that calcu-



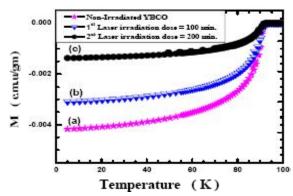


Figure (3a-c): AC-magnetic susceptibility curves recorded for; (a):Non irradiated optimaly Sb-doped YBCO; (b): After 1st Nd-Laser irradiation dose 15W/cm², 100min.; (c): After 2nd Nd-Laser irradiation dose 30 W/cm², for 200min.

lated applying Scherrer's formula for pure 123-phase (D~0.58µm=580nm).

This indicates that, the actual grain size in the material bulk is smaller than that detected on the surface morphology.Furthermore, in our EDX (energy disperse X-ray) analysis,Sb³⁺ was detected qualitatively with good approximate to the actual molar ratio but not observed at 123-YBCO grain boundaries which confirm that, antimony (III) has diffused regularly into material bulk of superconducting 123-YBCO-phase and Sbion induces in the crystalline structure through solid state reaction by some extent.The inclusion of Sb-ion is confirmed also by the depression in T_c of Sb-added YBCO.

Superconducting properties

Figure (3a-c) shows the AC-magnetic susceptibility curves for non-irradiated optimally nano-sructured Sb-doped YBCO (YSb $_{0.1}$ Ba₂Cu₃O_z) and after 1st and 2nd Nd-laser-irradiations doses respectively.

It is clear that, optimally nano-sructured Sb-doped YBCO exhibits HTc-offset ~91.56 K corresponding to 123-phase which is annealed in oxygen and is noticeable clearly in our XRD as major phase. This confirmed magnetically the existence of 123-YBCO in highly pure phase.

From magnetization curves (meissner lines) one can notify an important observation which is the amount of diamagnetic ratio interior material bulk represented by ΔM (difference between Meissner and shielded lines) increases (shifted up) with increasing laser irradiationdose from 1st 15W/cm² for 100 min. and 2nd 30W/cm²

Aano Solence and Aano Technology An Indian Journal for 200 min respectively referring to that laser-irradiation has no effect on the Sb-(III) ion oxidation number and consequently the conduction mechanism and magnetic order still as it is without noticeable change.

REFERENCES

- [1] G.Xiao, F.H.Streitz, A.Gavrin, Y.W.Du, C.L.Chien; Phys.Rev.B, **35**, 8782 (**1987**).
- [2] Y.Maeno, T.Tomita, M.Kyogoku, S.Awaji, Y.Aoki, K.Hoshino, A.Minami, T.Fujita; Nature, 328, 512 (1987).
- [3] J.M.Tarascon, P.Barboux, P.F.Maceli, L.H.Greene, G.W.Hull; Phys.Rev., B37, 7458 (1988).
- [4] H.Renevier, J.L.Hodeau, M.Marezio, A.Santoro; Pysica., C220, 143 (1994).
- [5] R.G.Kulkarni, D.G.Kuberkar, G. J.Baldaha, G.K. Bichile; Physica., C217, 175 (1993).
- [6] J.F.Bringley, T.M.Chen, B.A.Averill, K.M.Wong, S.J.Poon; Phys.Rev., B38, 2432 (1988).
- [7] Y.Shimakawa, Y.Kubo, K.Utsumi, Y.Takeda, M.Takano; Jpn.J.Appl.Phys., 27, L1071 (1988).
- [8] Z.Hiroi, M.Takano, Y.Takeda, R.Kanno, Y.Bando; Jpn.J.Appl.Phys., 27, L580 (1988).
- [9] M.P.Delamare, M.Hervieu, I.Monot, K.Verbist, G. Tendeloo; Physica, C262, 220 (1996).
- [10] P.N.peters, R.C.Sisk, E.Ubran, C.Y.Huang, M.K. Wu; Appl.Phys.Lett., 52, 2066 (1988).
- [11] C.Y.Huang, Y.Shapiro, E.J.McNiff, P.N.Peters, B.B.Shwatrz, M.K.Wu, R.D.Shull, C.K.Chiang; Mod.Phys.Lett., 2, 869 (1988).
- [12] J.P.Singh, H.L.Leu, R.B.Poepple, E.Voorhees, G.T. Goudery, K.Winsley, D.Shi; J.Appl.Phys., 66, 3154 (1989).
- [13] B.Dwir, M.Affronte, D.Pavuna; Appl.Phys.Lett., 55, 399 (1989).
- [14] J.Jung, M.A.Mohammed, S.C.Cheng, J.P.Frank; Phys.Rev., B42, 6181 (1990).
- [15] J.Joo, J.P.Singh, R.B.Poepple, A.K.Gangopadhyay, T.O.Mason; J.Appl.Phys., **71**, 2351 (**1992**).
- [16] H.R.Khan, T.L.Fancavilla, R.A.Hein, C.S.Pande, S.B.Quadri, R.J.Soulen, S.A.Wolf; J.Supercond., 3,189 (1990).
- [17] M.Okutomi, H.Nomura, T.Tsukamoto, N.Dahorte, H.Shen; Nucl.Instr.Meth.Res., B169, 6 (2000).
- [18] H.Dyck, N.Munser, W.Jaszczuk, C.Seega, H. Altenburg, J.Plewa, I.Kononyuk, V.Vashook, S. Tolochko, A.Shkadarevitch, A.Sabasnov; In: '6.Statussem.: Supraleitung and Tieftemperatur technik, Gelsenkirchen', Tagungsband, Okt., 497

29

(1998).

- [19] V.A.Lomonosov, I.F.Kononyuk, S.P.Tolochko, V.V.Vashook, H.Dyck, H.Altenburg; In: 'Second International Conference on Inorganic Materials', Santa Barbara, USA, September, 44, (2000).
- [20] M.M.Vasyuk, R.V.Lutciv; In: 'Second International Symposium on High T_c Superconduction and Tunnel Phenomena', Slavyanogorsk, Donetsk, Ukraine, 169, (1995).
- [21] R.Lutciv, J.Plewa, I.Solski, V.Vashook, O.Tolochko, N.Munser, H.Altenburg; In: '7.Statussem.; Supraleitung and Tieftemperaturtechnik', Garmischparten-Kirchen, Tagungsband, V.Dez., 542 (2000).
- [22] A.L.Mikhailchenko, G.N.Michailova, A.M. Prokhorov, A.S.Seferov, A.V.Trocki, A.O.Mednikov, B.L.Mikhailov, G.S.Burshanov, I.E.Lapshina; Quant. Electron., 23, 715 (1996).

- [23] M.M.Vasyuk, I.V.Lazaryuk, M.V.Matviyiv, H. Altenburg, J.Plewv Visnyk Lviv; Univ.Ser.Phys., 33, 173 (2000).
- [24] J.C.Diez, L.A.Angurel, H.Miao, J.M.Fernandez, G.F.De La Fuente; Supercond.Sci.Technol., 11, 101 (1998).
- [25] R.Luctiv, J.Plewa, M.Vasyuk, I.Solski, VV.Vashook, O.Toloch, N.Munser, H.Ltenburg; Physica C, 372, 1195 (2002).
- [26] K.Hotta, H.Miyazawa, T.Ogue, K.Nihei, P.Wu, H.Hirose; Physica, C237, 654 (2001).
- [27] Khaled M.Elsabawy; Physica, C432, 263-269 (2005).
- [28] L.D.Zang, J.M.Mu; 'Nanomaterial Science', Liaoning Science and Technology Press, Shengyan, China, 92 (1994).