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Effect of high-intensity ultrasound on superconducting properties of polycrystalline $YBa_2Cu_3O_{7-x}$

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Abstract. High intensity ultrasonic irradiation (sonication) of alkane slurries of polycrystalline $YBa_2Cu_3O_{7-x}$ leads to a significant modification of the grain morphology and, if performed with enforced oxygen flow, results in the increase of the superconducting transition temperature. Sonication with added $Fe(CO)_5$ produces magnetic Fe_2O_3 nanoparticles deposited on the surface of $YBa_2Cu_3O_{7-x}$ (YBCO) granules. Upon sintering these nanoparticles should act as efficient pinning centers utilizing both condensation and magnetic contributions to the free energy. The developed method could become a major technique to produce practically useful high-pinning nanocomposite materials based on $YBa_2Cu_3O_{7-x}$ superconductor.

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The major challenge of applied superconductivity is to find a delicate balance between strong pinning and yet retain basic superconducting properties, such as transition temperature, T_c, and intergrain connectivity. Best candidates for applications are nanocomposite superconductors where pinning centers are introduced in a controlled way. There is a great deal of activity in this field. Tapes and films is a large and promising area of research [1]. Others concentrate on improving bulk superconductors. In a typical scenario, additives (dopants, nanoparticles) are mixed into the pre-cursors or simply into powdered initial material. The composite is then sintered at high temperature and/or pressure to produce the final strong-pinning superconductor [2, 3]. Our research shows that high-intensity ultrasound can be efficiently used to produce nanocomposite superconducting materials [4]. Enhanced pinning properties of MgB_2 [5] and $Bi_2Sr_2CaCu_2O_{8+x}$ [6] superconductors have been reported. Recently, high - temperature isostatic pressure treatment of sonicated precursors resulting in a dense nanocomposite MgB_2 superconductors has been demonstrated [7].

In this contribution we show that sonochemically treated $YBa_2Cu_3O_{7-x}$ (YBCO) is a promising main component for such superconducting nanocomposite. Successful deposition of Fe_2O_3 nanoparticles on the surfaces of individual YBCO granules is demonstrated. Importantly,

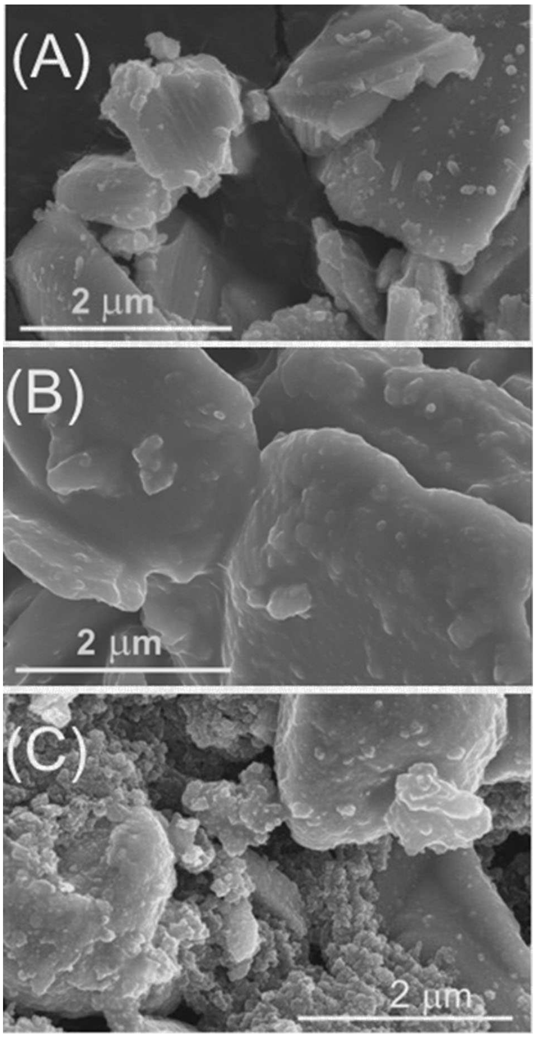

Figure 1. $YBa_2Cu_3O_{7-x}$ 1-6 μm powder from Alfa Aesar) was used as received. Pentane and decane (anhydrous, 99+%, Aldrich) were distilled under argon prior to use. Polycrystalline $YBa_2Cu_3O_{7-x}$ was ultrasonically irradiated for 120 min at 263 K in 20 mL of decane in an open flask under moderate argon flow (20 mL/min), using direct immersion ultrasonic horn (Sonics VCX-750 at 20 kHz and 50 W/cm^2). Various slurry loadings were used. A similar set of slurries was sonicated under ambient atmosphere with the addition of different amounts of $Fe(CO)_5$. Oxygen content in the starting material was probed by the iodometric titration. To maintain the necessary oxygen content during the sonochemical irradiation of YBCO powder, and to explore effects of sonochemical conditions on T_c, sonication was performed in 2% (w/w) slurry in ethylene glycol, under 40:20 Ar:O_2 flow. All ultrasonically treated materials were collected by filtration, washed with dry pentane, and air-dried overnight. The resulting dry powders were pelletized at room temperature at a pressure of 2 GPa for 24 hours. The figure shows: (A) Commercial powder. (B) YBCO sonicated in 2% (w/w) decane slurry at 263 K. (C) Fe_2O_3 nanoparticles, distributed over the grains of irradiated YBCO after sonication with 180 μmol of $Fe(CO)_5$.

the process does not result in the deterioration of the basic superconducting properties. Moreover, when sonication was performed under partial oxygen flow, the superconducting transition temperature of the resulting material had increased.

Powder morphology was studied by using Hitachi S-4700 and FEI Quanta 200 SEMs. Particle size was analyzed on a Phillips CM12 TEM equipped with EDX. Surface chemical composition of the modified powders was monitored by using XPS and microprobe EDX. XPS analysis was conducted on a Physical Electronics PHI 5400 X-Ray Photoelectron Spectrometer. Powder X-ray Diffraction study was performed by using a Rigaku D/MAX diffractometer. Magnetic measurements were conducted using a Quantum Design MPMS-5 magnetometer.

SEM images of sonochemically treated YBCO powders shown in Fig. 1 (B)(C) reveal substantial fusion, smoothing, and improved interconnecting of individual grains, as compared to the starting material, Fig. 1(C). Figure 2 shows powder XRD spectra of several powders used in this study. Noticeably, material that spent 6 months on the shelf shows significant degradation. However, when sonicated with partial O_2 flow, it was not only fully restored, but became better indicating that such treatment is similar to the annealing in oxygen atmosphere.

To further explore this observation and examine the effect on superconducting properties, figure 3 shows the M vs. T curves of sonicated YBCO. Compared to co-sonicated with O_2, as received powder shows smaller T_c and superconducting screening.

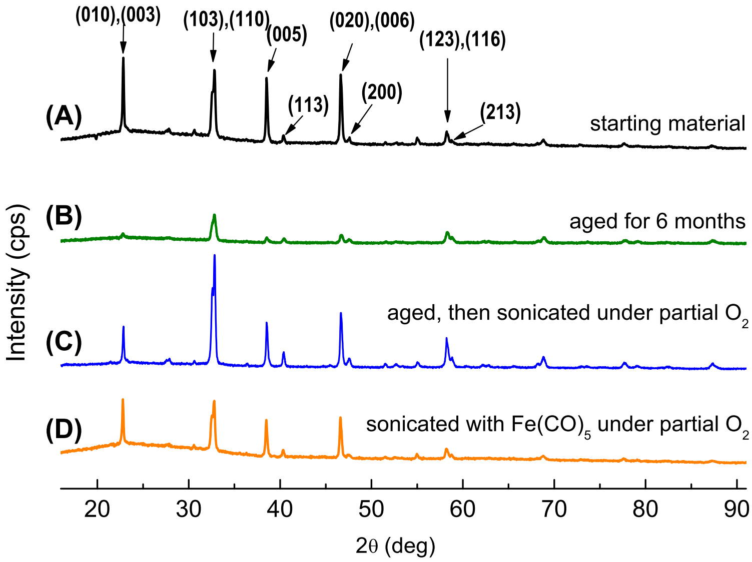

Figure 2. Powder x-ray diffractograms (XRD) of polycrystalline YBCO. (A) Starting (as received) material. (B) XRD pattern of YBCO aged for 6 months under ambient conditions. (C) Aged YBCO sonicated under partial O_2 flow. (D) YBCO powder co-sonicated with $Fe(CO)_5$.

In Fig. 4, XPS O^{1s} spectra for several $YBa_2Cu_3O_{7-x}$ samples are compared. The starting material

In conclusion, irradiation with high-intensity ultrasound of alkane slurries containing granular superconductors results in a substantial change of powders' morphology without affecting its bulk chemical composition. Sonochemical treatment of polycrystalline $YBa_2Cu_3O_{7-x}$ high-Tc superconductor showed a dramatic effect on morphology of materials and their superconducting properties. Decane slurries of polycrystalline superconducting materials were ultrasonically irradiated with different slurry loadings and duration of the sonochemical treatment. To maintain the oxygen content in sonochemically treated $YBa_2Cu_3O_{7-x}$, sonochemical irradiation

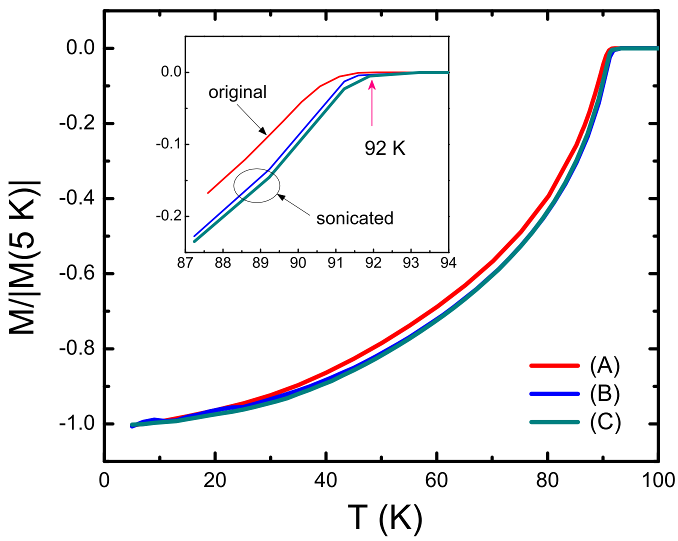

Figure 3. Effect of oxygen flow during sonication on the $M(T)$ curves measured in resulting YBCO powder. (A) starting material. (B) YBCO sonicated under partial oxygen pressure. (C) YBCO sonicated under partial oxygen pressure with 9 mmol $Fe(CO)_5$. Sonication of 2 % (w/w) ethylene glycol slurry at 263 K, 20 kHz, 50 W/cm^2. Inset: vicinity of T_c.

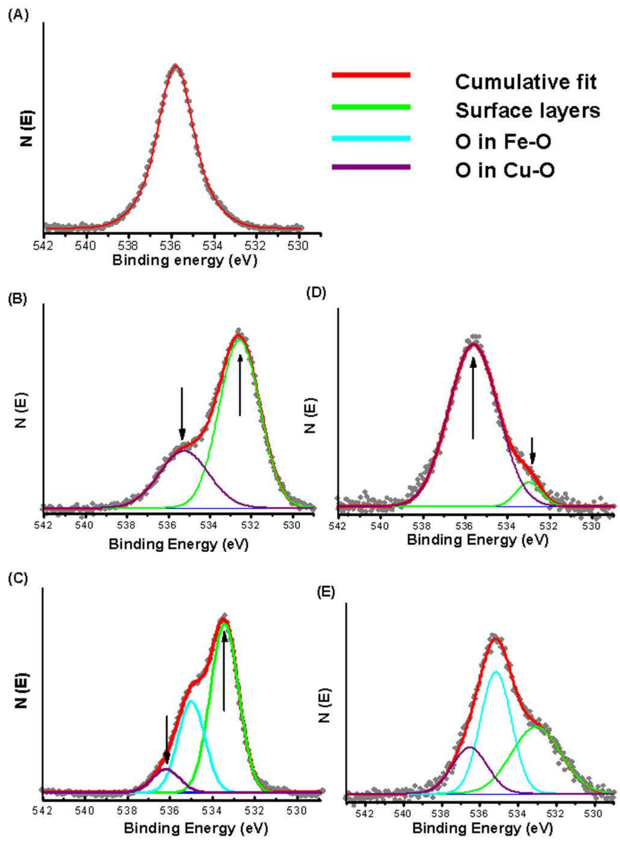

Figure 4. Comparison of XPS peaks on O^{1s} site in several YBCO samples. (A) Starting material shows relatively narrow single oxygen peak. (B) Low-energy O^{1s} peak appears in the sample sonicated under a partial oxygen flow. The position of the peak matched that of samples sonicated under the Ar. (C) YBCO sonicated under air/Ar flow with 4.5 mmol $Fe(CO)_5$. (D) YBCO sonicated under O_2/Ar flow; (E) aged YBCO sonicated in ethylene glycol under O_2/Ar flow with 4.5 mmol of $Fe(CO)_5$. Evidently, sonication under partial oxygen flow with small amount of $Fe(CO)_5$ leads to disruption of oxygen surface layer, as indicated by the appearance of the intermediate peak. These observations indicate that sonication in oxygen allowed control over the surface oxygen concentration.

of slurries was performed under a partial oxygen flow in decane and in ethylene glycol. Ultrasonic irradiation of decane slurries leads to significant modification of morphology and improved intergrain coupling in polycrystalline superconductors. Effectiveness of sonication increases with the decrease of slurry loading, due to more effective interparticle collisions. Novel composite materials with enhanced pinning properties and increased critical current have been successfully prepared. Materials show enhanced Meissner screening and larger magnetic irreversibility. Critical temperature Tc remains intact, while the pinning properties are improved. The sonochemical method and post-sonochemical annealing are to be further optimized to achieve maximal pinning enhancement in granular YBa2Cu3O7- .

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