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Critical current density in MgB_2 bulk samples after co-doping with nano-SiC and poly zinc acrylate complexes

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ABSTRACT

The co-doped MgB_2 bulk samples have been synthesized using an *in situ* reaction processing. The additives is 8 wt.% SiC nano powders and 10 wt.% $[(CH_2CHCOO)_2Zn]_n$ poly zinc acrylate complexes (PZA). A systematic study was performed on samples doped with SiC or PZA and samples co-doped with both of them. The effects of doping and co-doping on phase formation, microstructure, and the variation of lattice parameters were studied. The amount of substituted carbon, the critical temperature (T_c) and the critical current density (J_c) were determined. The calculated lattice parameters show the decrease of the *a*-axis, while no obvious change was detected for *c*-axis parameter in co-doped samples. This indicates that the carbon was substituted by boron in MgB_2. The amount of substituted carbon for the co-doped sample shows an enhancement compared to that of the both single doped samples. The co-doped samples perform the highest J_c values, which reaches 3.3×10^4 A/cm^2 at 5 K and 7 T. It is shown that co-doping with SiC and organic compound is an effective way to further improve the superconducting properties of MgB_2.

1. Introduction

MgB_2, discovered in 2001 [1], is considered to be a promising candidate for engineering applications operating at 20 K, thanks to its relatively high critical temperature (T_c) and low material cost. Critical current densities (J_c) of more than 10^5 A/cm^2 in self-field were measured, reflecting weak link free grain boundaries and a large coherence length. Using cryogen-free cooling, MgB_2 could be useful in the temperature range between 20 and 30 K, where the traditional low temperature superconductors such as Nb_3Sn and NbTi cannot be operated anymore. Over the last 8 years, many works have been undertaken for improving the critical current density of MgB_2 at higher magnetic fields, using techniques like chemical doping [2–10], irradiation [11] and thermo-mechanical process [12]. It was found that the doping with carbon is a simple but quite effective way: carbon particle [2–4], carbide [5–7] and organic compounds [8–10] have been successfully used for enhancing the superconducting properties of MgB_2. However, the highest critical current densities at high field achieved in SiC doped MgB_2 were compromised by the reduction at low field [13]. It is noteworthy that, although the nano-size SiC additives have been already used by many authors, it is still difficult to get a homogenous distribution of the dopants in the materials. Agglomerates of dopants in the precursors may indeed cause an unequal distribution of the fresh carbon coming from the reaction between the dopants and MgB_2.

In order to overcome these problems and to get improved superconducting properties in MgB_2, co-doping with SiC and poly zinc acrylate Complexes (PZA), of which chemical formula is $[(CH_2CHCOO)_2Zn]_n$, has been investigated and is presented in present paper. Co-doping with SiC and organic compound has two additional merits with respect to single SiC doping. Since boron powders and two dopants are dissolved in distilled water, a complex is achieved, resulting into a more homogenous distribution of additives in the precursor. Since the decomposition temperature of the PZA is different from the reaction temperature of MgB_2 in presence of SiC, a higher amount of dissolved carbon is obtained, due to the fact that there are now two temperature ranges where fresh carbon is released.

2. Experimental procedure

In this study, the MgB_2 samples co-doped with 8 wt.% SiC and 10 wt.% PZA have been prepared using the *in situ* reaction process. The PZA powder and B powder (amorphous, 99%) were put into distilled water and mixed. The mixture was dried in a vacuum oven. After which, it was mixed again with an appropriate amount of Mg powder (325 mesh, 99.8%) and SiC powder (30 nm, 99.9%) in glove box under Ar gas. The powders were pressed into pellets,

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surrounded by Nb foil and sintered at 800 °C for 1 h in flowing argon gas of high purity. Finally the samples were rapidly cooled to room temperature by shutting down the furnace. For comparison, samples with the single 8 wt.% SiC and 10 wt.% PZA doped were also fabricated using the same process.

The crystalline structure of the samples was investigated by means of a Bruker D8 Advanced X ray diffractometer using Cu Kα radiation. The microstructure of the samples was analyzed by a HITACHI S-3400 scanning electron microscope (SEM) and a JEM 2010 transmission electron microscope (TEM). The magnetic measurements were performed with a physical property measurement system (PPMS) at 5 K under magnetic field up to 7 T. The magnetic critical current densities were derived from the width of the magnetization loop based on the Bean model: $J_c = 20\Delta M/va(1-(a/3b))$ where ΔM (e.m.u) is the vertical width of the magnetization loop, v (cm^3) is the volume of the bulk samples, a and b (cm) with $b \geqslant a$ are the cross-sectional dimensions of the sample.

3. Results and discussion

Fig. 1 represents the X-ray patterns of MgB_2 samples, undoped and with the following additives: PZA, SiC and co-doping. All the doped samples and the binary sample have a good phase formation, with a well-developed MgB_2 phase and small amount of MgO. The Mg_2Si phase has been found in the SiC doped and co-doped samples. A small amount of unreacted SiC was also observed in the co-doped samples, which shows that the SiC was completely reacted in the SiC doped sample but not in the co-doped one. It is suggested that the presence of unreacted SiC in the co-doped sample is due to the carbon released from PZA decomposition between 400 °C and 600 °C. The position of both (1 0 0) and (1 1 0) peaks of MgB_2 in the doped samples were shifted to a higher angle, reflecting carbon substitution for the boron sites in the MgB_2 lattice. The (0 0 2) peaks of MgB_2 in the PZA and co-doped samples was not shifted, suggesting that the Zn did not substitute the Mg.

As shown in Table 1, the a-axis of all the doped samples decreased with respect to that of the pure one, while the c-axis was not obviously changed. Based on the lattice parameter changes, the actual carbon substitution amount x can be estimated as the formula $x = 7.5 \times \Delta(c/a)$, where the $\Delta(c/a)$ is the change in c/a compared to a pure sample [14]. The levels of PZA doped sample are nearly half of the SiC doped and co-doped one, which indicates that SiC is more helpful for the substitution of carbon than PZA doping. It was considered that all the carbon in the SiC participated to the reaction, which is shown in formula below: $Mg + SiC \rightarrow Mg_2Si + C$. The carbon in the PZA doped sample may decompose as a gas such as acetylene or carbon monoxide, carbonized polymers or fresh carbon. The x listed in Table 1 of the co-doped samples is slightly higher than that of the SiC doped one, suggesting that the carbon is released from both SiC and PZA during the MgB_2 synthesis. This means that there are two carbon sources in the co-doped sample: one is the decomposition of PZA at 400 °C to 600 °C, the other one being the reaction of SiC with Mg after 600 °C.

Table 1
Measured data for pure and doped MgB_2 samples.

Sample	Lattice		Actual C(x) in $Mg(B_{1-x}C_x)_2$
	a (Å)	c (Å)	
Pure	3.0843	3.5281	
PZA	3.0796	3.5292	0.0157
SiC	3.075	3.5305	0.0318
Co-doped	3.076	3.5321	0.0329

Fig. 2 represents the inductively measured field dependence of J_c for the PZA, SiC and co-doped samples measured at 5 K. For comparison, the curve for the undoped MgB_2 sample is also shown. It was found that the doped samples have at all fields a higher J_c value than the undoped one. The decreasing rate of the J_c vs. B curves for all doped samples was smaller than for the undoped one. Compared to the PZA doped sample, the SiC doped one shows a better performance at higher fields, which is due to an increasing carbon amount in the MgB_2 lattice. It is noticed that the J_c value of co-doped sample is slightly higher than SiC in all applied fields, indicating that, except for more substitution of carbon induced by both

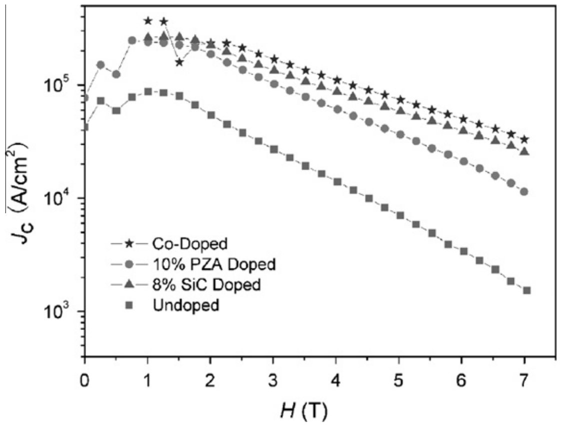

Fig. 2. Field dependence of J_c at 5 K in doped and undoped MgB_2 samples.

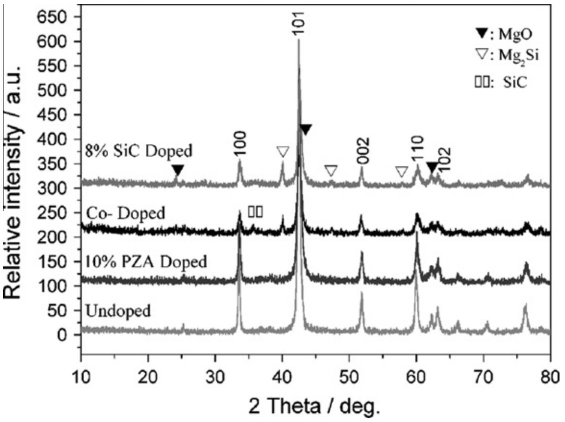

Fig. 1. XRD patterns of MgB_2, undoped and with the additives PZA, SiC and PZA + SiC.

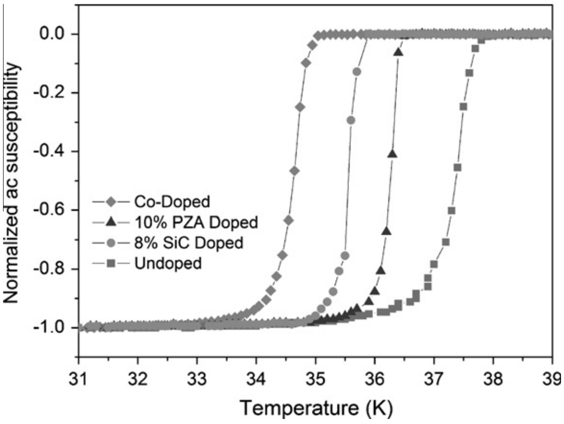

Fig. 3. Transition temperatures of pure and doped MgB_2 bulk samples, from magnetization measurements.