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SUPERCONDUCTING PROPERTIES OF NANO ALUMINA AND NANO CARBON CO-DOPED $Mg_{1-x}Al_x(B_{1-y}C_y)_2$

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Abstract* - *Systematic preparation of a series of bulk MgB_2 samples of $Mg_{1-x}Al_x(B_{1-y}C_y)_2$, x=2% ; y = 0%,1%,2%, 3%, 4%,6%, 10%, were carried out on nano alumina MgB_2 co-doped with nano carbon. Bulk MgB_2 samples have prepared by standard solid-state reaction method, followed by ball milling. As the concentration of nano carbon doped MgB_2 increases their resistivity increases accordingly, results the lowering of transition temperature, T_c. Resistivity shows the systematic decreases of transition temperature with nano alumina and nano carbon doped MgB_2. Here, we report that co-doping play an important role in decreasing the lattice parameters. A systematical comparison of nano alumina and nano carbon doped MgB_2 with structure; superconducting transition, resistivity and FWHM are discussed.

Keyword - MgB_2, nano-Alumina, nano-carbon, co-doped, superconductivity,

INTRODUCTION

Since 2001, the discovery of superconductivity at 39 K in MgB_2 has initiated an interest of activity aimed to understanding the origin of the large superconducting transition temperature (T_c) [1]. MgB_2 is composed of two elements, magnesium and boron. MgB_2 have high upper critical field $H_{c2,}$ high irreversibility $H_{irr,}$ low material cost and enables to be used at the cryocooler temperature.

A number of groups undertook synthesis and characterization of $(Mg_{1-x}W_x)B_2$ or $Mg(B_{1-y}Z_y)_2$ (W = transition metal, Li, Be, Al []; Z = C, Si []) materials. Carbon substitution was reported in several reports [2–7]. Almost all substitutions have led to a decrease in T_c with an exception of Zn [8], which shows no change even for 30% substitution. As a simple and practical method, chemical doping seems to be a promising technique for improving the critical current density $J_c(H)$. Especially, carbon-containing precursors, such as SiC, graphite, nanocarbon (NC), and carbon nanotubes (CNTs), were found to be effective for the enhancement of the irreversibility field (H_{irr}) and $J_c(H)$, because the substitution of C into B site leads to intra-band scattering that increases the H_{c2}, as reported by many groups [9–12]. Among these precursors, we considered that nano carbon would be an effective dopant, because of its nano size and low decomposition temperature from 150 ˚C to 750 ˚C. Slusky et al. [13] have shown that Al can aggressively react with MgB_2 and the substitution into the Mg position leads to a loss of superconductivity.

Indeed, C as well as Al introduces extra electrons into the conduction bands of MgB_2. This leads to a reduction of the hole density-of-states of the most important σ-band, and it causes the suppression of T_c and superconducting gaps in both series of doped systems [14]. Although Al-oxide-doped MgB_2 has been studied [15] but the effect of nano-alumina oxide as well as nano carbon addition on the properties of MgB_2 has not been reported within our knowledge. In the present work, we study the effect of co-doping of nano-alumina oxide and nano carbon into bulk MgB_2 by $Mg_{1-x}Al_x(B_{1-y}C_y)_2$ formula.

EXPERIMENTAL DETAILS

Co-doped MgB_2 samples were prepared by a solid-state reaction at ambient pressure by proper mixing of starting material at stoichiometric ratio. The starting materials Mg (CERAC 99.6%) was further ground by using ball milling and amorphous B powder (Sigma-Aldrich 99.9%) were well mixed with nano-alumina powder (Sigma Aldrich, particle size < 50 nm) and nano carbon powder (Sigma Aldrich, particle size < 50 nm) with the ratio of $Mg_{1-x}Al_x(B_{1-y}C_y)_2$, x = 2%, and y = 0%, 2%,3%, 4%, 6%, 10%, respectively. Pellets of 10 mm in diameter and 2.5 mm in thickness were prepared by using a hydraulic press and encapsulated in Fe tubes by inside sealing of tantalum foil. Furthermore, Fe tubes were put inside the quartz tube and evacuated up to 18 hours in vacuum range ~10^{-7} Torr. The quartz tube was further put in preheated furnace at 770 ˚C and switched off after 2½ hours. This was followed by a furnace cooling to room temperature.

The crystalline structure analysis was investigated by powder X-ray diffraction (XRD) using an X'pert MRD diffractometer with CuKα radiation at room temperature. Resistance of the samples was measured by in-house made resistivity measurement system using the standard four probe method. Temperature dependence of resistivity was determined from 12 K to 300 K using closed cycle He

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refrigerator. The dc-susceptibility was measured by in-house made susceptometer.

RESULT AND DISCUSSION

Figure 1 show XRD pattern of $\{(Mg_{1-x}Al_x) + (B_{1-y}C_y)_2\}$, samples where x = 2%, and y = 0%, 2%,3%, 4%, 6%, 10%. Note that there are no peak related to nano carbon in XRD pattern of nano Carbon and Al oxide co-doped bulk due to the amorphous nano carbon and nano Aluminum oxide powder used. Minority phase MgO is detected. It clearly shows that there is only MgB_2 phase with minor trace of MgO and un-reacted Mg. in pure MgB2 samples the un-reacted Mg is negligible but C0% and C6% having more percentage of un-reacted Mg.

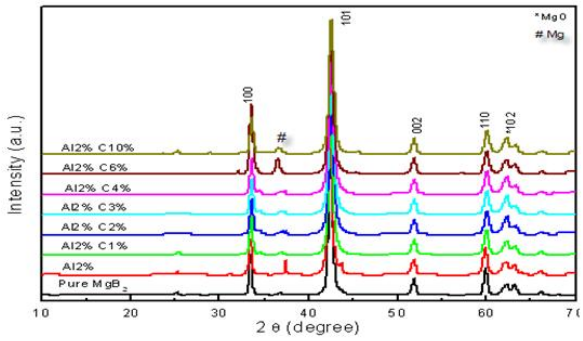

Fig 1. X-ray diffraction pattern for $\{(Mg_{1-x}Al_x) + (B_{1-y}C_y)_2\}$, with x = 2%, and y = 0%, 2%,3%, 4%, 6%, 10%.

This has been further verified by measuring the electrical resistivity of all samples as a function of temperature which is shown in Fig.2. From the figure it is clear that the normal state resistivity of pure, 0%, 2% 4% and 6% doped samples is nearly the same. There is significant change in the normal state resistivity of 10% in comparison to that of pure. This might be due to void or impurity phase MgO at grain boundary. The substitution is further confirmed from the change in the T_C of the doped samples as shown in the of Fig.2.

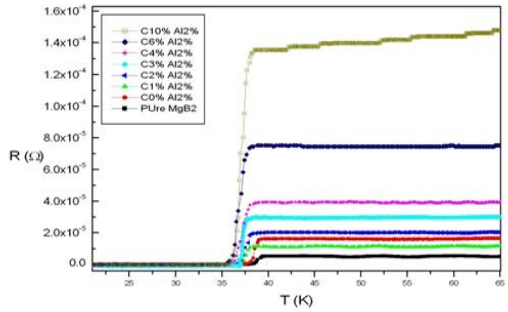

Fig2. The Temperature dependent of electrical resistivity of $\{(Mg_{1-y}Al_x) + (B_{1-y}C_y)_2\}$, with x = 2%, and y = 0%, 2%,3%, 4%, 6%, 10%.

The value of delta Tc measure from Fig 1 according to the which has been tabulated in table 1. The value of delta Tc does significantly change with doping concentration Nano carbon and nano alumina is divalent as Mg so it does not affect the carrier density in B-plane.and at the same time it is at Mg site so its interaction with the conducting B-plane is very weak. Higher delta Tc measure in C0% and Al2% doped samples and less value in C3% and Al 2% doped sample.

Samples	Onset T(K)	Tc	ΔT= T-Tc
Pure MgB_2	39.56	35.87	3.69
C0% Al2%	39.37	27.94	11.43
C1% Al2%	38.03	32.85	5.18
C2% Al2%	38.62	36.3	2.32
3% Al2%	38.28	37.03	1.25
C4% Al2%	38.83	35.53	3.3
C6% Al2%	38.43	35.29	3.14
C10% Al2%	38.3	35.66	2.64

Table1 **Resistivity comparison for** $\{(Mg_{1-y}Al_x) + (B_{1-y}C_y)_2\}$, with x = 2%, and y = 0%, 2%,3%, 4%, 6%, 10%.

The Full Widths at Half Maximum (FWHM) were determined with the program PowderX by using x-ray diffraction data. These FWHM data were used to evaluate the grain size and strain of different doped samples using the Williamson and Hall model [42]:

$$FWHM \text{ x } \cos(\theta) = \frac{0.94\lambda}{(Grain\,size)} + 4 \text{ x } Strain \text{ x } \sin(\theta)$$

where λ is the wavelength of monochromatic CuKα radiation (1.540598 Å) and θ is the angle of peak position at x-ray plot.

The above relation encompasses the combination of Scherrer equation for size broadening and Stokes and Wilson expression for strain broadening. After plotting the FWHM x cos(θ) vs. sin(θ) as shown in the figure 3 and fitting the straight line.

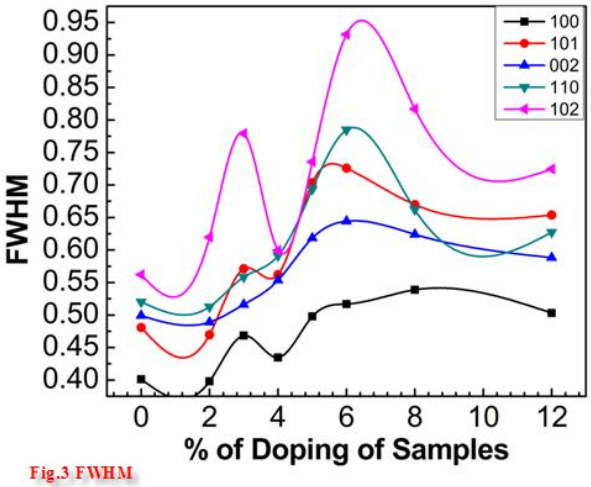

Fig.3 FWHM

CONCLUSION

In Summary, result shows the substitution of nano carbon and alumina doped in the stoichiometry of MgB_2. The impurity MgO, as seen in the XRD plot, might arise during the solid-state reaction of the starting materials. The variation in *delta Tc* value and transition temperature, T_c is perceived with the increase of doping concentration. Slight variation in T_c (onset) and T_c (ρ = 0) is observed from the temperature dependence of resistivity plot for nano carbon and alumina doped MgB_2. The Full Widths at Half Maximum (FWHM)

showing higher value in 102 peak and lower value in 100 peak.

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