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Synthesis of carbon-doped MgB_2 superconducting nanowires

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ABSTRACT

A systematic study was conducted on the fabrication, structural characterization, and magnetic properties of MgB_2 wire-like nanostructures with C doping between 0% and 20%. Based on chemical vapor deposition technique, non/C-doped MgB_2 nanowires (NWs) with an average diameter of 60 nm and length up to several micrometers were produced by homemade MgB_2 nanotubes, the mixture gases ($Ar + CH_4 + H_2$), and Ni catalyst. Electron and X-ray diffraction confirm that the as-synthesized non/C-doped MgB_2 NWs are single crystalline with primitive hexagonal lattice structure. DC magnetization measurements indicate a high superconducting transition temperature (39 K) for nondoping MgB_2 NWs and that on increasing the carbon content the transition broadens and shifts toward lower temperatures. The technique is an attractive synthetic method since its flexibility allows for optimization of the doping synthesis. In particular, a comprehensive investigation of influence of NW doping on superconductivity is reported for the first time.

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1. Introduction

In the early years of discovery of renowned bulk MgB_2 superconductor, it attracted the huge interest of scientific community due to its simple chemical composition, crystal structure and highest T_c among the intermetallic non-cuprate compounds [1–14]. Recently, one dimensional nanostructures (1DNs), such as nanotubes (NTs) [4], nanowires (NWs) [5–13], nanohelices [14], and nanocables [15] can ideally be used as low-dissipation interconnects in devices, as well as it is fundamentally interesting to study the effect of dimensionality and size on superconductivity using MgB_2 1DNs [4–15]. For example, superconducting high-pure MgB_2 NWs have widely been reported [5–14]. To date; however, there has been no report on doping MgB_2 NWs although C-doped bulk MgB_2 superconductors have extensively been studied [16–22]. As far as we know, doping in superconductors with selective elements offers an effective approach to adjust the electrical and magnetic properties, which is crucial for practical applications [16–22]. Because of the same structure of the electronic shell, many physical and chemical properties of B are similar to those of C. Moreover, the ion radius of C (0.77 Å) is smaller than that of B (0.88 Å). Therefore, the B in MgB_2 can be easily substituted by C under certain conditions as reported on C-doped MgB_2 bulk materials [16–22]. Motivated by this approach, possibly, the C element from CH_4 is being substituted for some B in MgB_2 in the chemical vapor reaction, which may turn MgB_2 nanotubes into C-doped MgB_2 NWs by replacement of certain B. Previously, our group successfully synthesized MgB_2 nanotubes based on a simple thermal evaporation of MgB_2 particles precursors [4] and confined growth of superconducting F-doped SmFeAsO nanocables using ZnO NTs [15]. Here we report a chemical vapor deposition (CVD) method to fabricate single-crystal C-doped MgB_2 NWs in a bulk quantity. Magnetic measurements (Meissner effect) are used to study the influence of the carbon doping on the superconducting characteristic of MgB_2 NWs. The results indicate the as-obtained single-crystal C-doped MgB_2 NWs possess a uniform morphology, a single phase, and the same chemical composition, and a strong diamagnetic behavior (below 39 K) in zero field cooling procedure (magnetization measurements). This method can continuously be used to synthesize mass area C-doped MgB_2 NWs at friendly environment process and low cost, which is a key issue, not only for practical applications, but also for fundamental understanding.

2. Experimental details

In a typical experimental procedure, homemade MgB_2 nanotubes [4] and a mixture gases of Ar, H_2, and CH_4, were served as a source of chemical raw materials. A substrate with MgB_2 nanotubes (1.0 g) and Ni (0.2 g) powders was placed in a half of the boat, which was inserted into a half of a large quartz-tube in a horizontal tube furnace. Then, the mixture gases ($Ar + H_2 + CH_4$) was charged into the large quartz-tube, the system was heated up to 450 °C, and held the temperature for 4 h, followed by furnace cooling to room temperature, in which C acted as the dopant can be controlled by the mixture ($Ar + H_2 + CH_4$) with amounts of CH_4 (60,000–300,000 ppm). The samples were extensively characterized for morphology, phase, growth direction, and chemical composition by scanning/transmission electron microscopy (SEM/TEM), high-resolution (HR) TEM (HRTEM)/SEM(HRSEM), X-ray diffraction (XRD)/HR (HRXRD), energy dispersive X-ray spectroscopy (EDS), and selected area electron diffraction (SAED), respectively. DC magnetization measurements from

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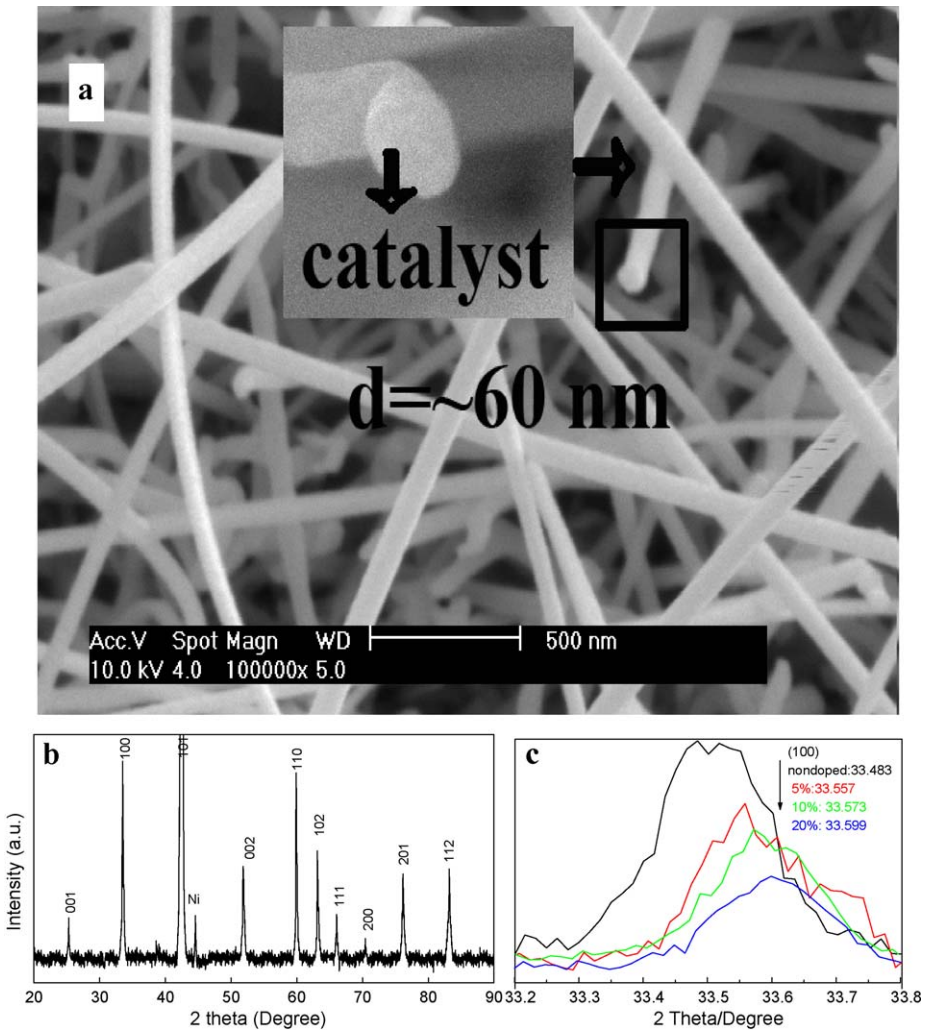

Fig. 1. (a) SEM and HRSEM (inset for the square) image of MgB_2 NWs with catalyst and diameter of ~60 nm and length up to several micrometers; (b) XRD patterns of bulk C (~20%)-doped MgB_2 NWs with Ni catalyst; (c) HRXRD pattern of (1 0 0) of MgB_2 NWs.

3 K to 50 K were performed with a superconducting quantum interference device (SQUID) magnetometer [MPMS-7, Quantum Design, zero field cooling mode (ZFC).

3. Results and discussion

Morphology/size, composition, and phase of the as-obtained MgB_2 specimens are detailedly observed by (HR)SEM/(HR)TEM, EDS, SAED, and (HR)/XRD, respectively. A representative SEM/HRSEM (inset) image is revealed in Fig. 1a, in which all MgB_2 samples have a similar morphology (wire-like nanostructures) and uniform size. From the Fig. 1(a), it's seen that individual MgB_2 NWs have a similar size with the diameter of ~60 nm and length up to several microns. In the inset (HRSEM image), a droplet can be noticed in the top of single NWs, which serves as a seed for the MgB_2 NW growth, and is most likely to be controlled by the conventional vapor–liquid–solid (VLS) mechanism [4–9,14]. A typical XRD pattern (Fig. 1b) has been shown for the as-synthesized C (20%)-doped MgB_2 NWs where the major of diffraction peaks have a slightly shift compared with nondoped MgB_2 samples [nondoped MgB_2 crystal faces with primitive hexagonal [4] (PDF: 38-1369)]. The 1 weak Ni peak occurring in the XRD pattern indicates that a small amount of Ni is used as catalyst during the growth process, which is compatible with VLS mechanism and data of SEM and TEM (Fig. 2a). Through all XRD results, no other crystalline forms are detected. Further, Fig. 1(c) shows four explored HRXRD patterns, taken from the (1 0 0) spacing of 0 (top), 5%, 10%, and 20% (bottom) -C-doped MgB_2 samples, respectively. It's much clearer that three of the (1 0 0) diffraction peaks of C-doped MgB_2 NWs have a slightly shift to high 2-theta angle compared with nondoped MgB_2. A typical TEM image of MgB_2 NWs (diameter: about 60 nm) is shown in Fig. 2a, in which the droplet (catalyst), smooth morphology, and uniform size can evidently be observed. EDS analysis shown in Fig. 2b, taken from a white circle in Fig. 2a, demonstrates that each of the NWs has the same composition and contains a small amount of Ni, and Mg, B, and C (peaks of Cu come from the Cu graticule for character of TEM). Further quantitative analysis of EDS finds that the atomic ratio of (B + C):Mg is about 2:1, indicating that a stoichiometric NW [(B + C)/Mg = 2] is synthesized. Corresponding SAED patterns from the single NW are composed of regular clear diffraction dots (a high-quality crystalline nature of MgB_2) as shown in inset of Fig. 2c, which is very good agreement with XRD and HRTEM results. As indicated in Fig. 2(c), an HRTEM image with an apparent lattice plane reveals that the MgB_2 NW is a good single crystal structure. The length is 0.35 Å between D-spaces, which is compatible with the results of XRD and SAED. According to the XRD, SAED and HRTEM, the growth direction is [1 0 0] as indicated by a large white arrow in Fig. 2(c).

A key question here is why the pure single-crystalline MgB_2 NT becomes into the form of single-crystalline C-doped MgB_2 NW. In

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