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SUPERCONDUCTIVITY IN BULK, THIN AND THICK FILMS OF MAGNESIUM DIBORIDE

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Abstract- *Since the discovery of superconductivity at 39K in binary compound magnesium diboride (MgB_2), extensive research has been carried out on the fabrication of this superconductor in various form such as polycrystalline bulk sample, single crystals, thin epitaxial films and thick polycrystalline films including wires and tapes using in-situ and ex-situ methods. For fabrication various techniques are used such as sintering and infiltration techniques, pulsed laser deposition (PLD), molecular beam epitaxy (MBE), sputtering, screen printing, electrocrystallisation, hybrid chemical vapor deposition (HPCVD), electrophoretic deposition techniques etc. The preparation techniques emphasis on the critical requirements for growing high-quality MgB_2 superconductor in the form of bulk, thin and thick films. This paper describes the effect of fabrication techniques on the microstructure and the superconducting properties of bulk and thin and thick films of MgB_2.*

Keywords: thin films, thick films, bulk MgB_2, sintering, pulsed laser deposition, molecular beam epitaxy, sputtering, screen printing, hybrid chemical vapor deposition.

1. INTRODUCTION

The discovery of superconductivity at 39 K in magnesium diboride [1] which is the highest among conventional BCS binary compounds having well known AIB_2 type layered structure offers the possibility of a new class of low-cost, low weight, high-performance superconducting materials for high field magnets, superconducting electronic devices [2] and superconducting radio-frequency cavities [3] used in accelerators. MgB_2 is a phonon-mediated superconductor [4], with a relatively long coherence length [5] and larger energy gap [6]. MgB_2 has two superconducting gaps originating from two different bands. The gaps are thought to be responsible for the relatively high superconducting transition temperature of 39K.The high volatility of magnesium and low decomposition temperature of MgB_2 create difficulties for the preparation of smooth, homogeneous and epitaxial thin films for Josephson junction devices [7]. Heat treatment condition (sintering temperature) plays an important role in the development of microstructure and superconducting properties of MgB_2 . The study of MgB_2 superconductor in bulk form is important for determining the phase formation mechanism, the basic physical and electrical properties of the superconductor.

2. CRYSTAL STRUCTURE

MgB_2 has a simple hexagonal AIB_2 type crystal structure [8] with a space group P6/mmm [9]. Magnesium diboride consists of honeycomb boron layers separated by magnesium layers. Each Mg atom whose size is bigger than B is located at the center of hexagons of B atoms and donate their electrons to the boron planes. In each boron layer, each of the hexagons consists of six boron atoms giving an overall 1:2 Mg-B ratio in the unit cell. The hexagonal unit cell has the in-plane lattice parameter of a=b=3.086Å and out of plane lattice parameter c=3.524Å. In the unit cell, the atomic positions are (0, 0, 0) for Mg and (1/3, 2/3, 1/2) and (2/3,1/3,1/2) for B atoms [10]. The in-plane B-B distance is almost half that of the interplane B-B distance, resulting in anisotropy of superconducting properties. The anisotropy of MgB_2 is between 1.5 and 5 which is lower than that of the highly anisotropic high-T_c superconducting materials. At the same temperature 10.8K and in a field of 4.8mT, the measured penetration depth λ and coherence length ξ in all direction was λ_{ab}=107±8nm, λ_c=120±15nm and ξ_{ab} =39±15nm, ξ_c=35±10nm respectively [11]. The crystal structure of MgB_2 are shown in figure 1. The interaction among certain

lattice vibrations with certain electrons is strong which results from the crystal structure of MgB_2 and connectivity condition of electrons.

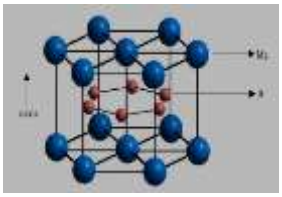

Fig. 1. Crystal structure of MgB_2[1]

3. SUPERCONDUCTING MECHANISM

An isotopic effect gives direct evidence for a phonon mediated mechanism of superconductivity. Generally, a critical temperature decreases with increasing isotopic mass of the superconductor, which indicates the involvement of the lattice in the superconductivity. The isotopic effect indicated a phonon mediated BCS superconductivity mechanism with dominant B phonon contribution to the overall pairing. The isotopic effect coefficient α for Mg ~0.02 and for B~0.30. The isotopic effect for Mg was much smaller than that for B, indicating B played the dominant role in the superconducting process [4,12].

It was theoretically predicted that MgB_2 possesses two gap superconductivity [6]. The electronic structure of MgB_2 has weakly interacting two dimensional σ and three dimensional π bands. The energy gap Δ(0K) of σ bands has the larger value~ 6.8meV and for the π bands it has smaller value ~1.8meV. Superconductivity occurs due to strong electron-phonon coupling in the two-dimensional σ bands on the boron layer and the reason of superconducting behavior of the π electron is due to their Interband coupling with σ electrons. [13]. The fermi surface consists of two tubular networks. The σ bands form cylindrical fermi surfaces whereas the π band form three dimensional tubular networks of fermi surface. The σ bands coherence length $\xi_{ab}(0K)$ ~13nm and penetration depth $\lambda_{ab}(0K)$ ~49nm, while for π band $\xi_{ab}(0K)$ ~51nm and $\lambda_{ab}(0K)$ ~34nm [14]. The two-band nature of the electronic structure enables the T_c of MgB_2 as high as 39K according to BCS theory.

The pressure effect on T_c also supports the phonon mediated superconductivity in MgB_2. MgB_2 superconductor shows decrease in T_c under pressure due to an increase in phonon frequency and decrease in coupling between phonon and charge carriers [15]. The T_c decreased monotonically with rates varying from 1 to 2 K for MgB_2. The critical current density (J_c) is rapidly degraded under applied magnetic field, which indicates poor flux pinning strength in magnetic field and low upper magnetic critical field (Hc_2). Pure MgB_2 has very lower critical field Hc_1 of less than 50mT and upper critical field Hc_2 in the range of 15-20T.To obtain high J_c in magnetic fields, it is necessary to improve the flux pinning ability. The grain size of MgB_2 in polycrystalline samples are in the range of 10nm to 10μm.

Al can substitute for Mg atoms on the Mg site to form a $Mg_{1-x}Al_xB_2$ when $0 \leq x \leq 0.5$ without losing superconductivity. The T_c as well as the out of phase lattice parameter, c, is gradually decreased with increasing Al doping level. The Al substitution for Mg and electron donation reduces the number of holes in MgB_2 thus suppresses T_c. The Hc_2 decreases with increasing Al doping concentration [16]. For Mn doping, the c-axis shows an evident decrease as the Mn content increases besides a small decrease in a-axis. The Mn substitution for Mg reduces T_c even more rapidly than Al. It was found that only 2% Mn substitution completely destroys superconductivity in MgB_2[17]. Further increasing of Al or Mn content resulted in reduction of both σ and π bands (both gaps are reduced) i.e, suppression of T_c is mainly due to magnetic breaking in MgB_2 caused by Mn doping. Table I shows the superconducting properties of MgB_2.

Table I. superconducting properties of MgB_2

S.N.	Parameters	MgB_2
1	Critical temperature (T_c)	39K
2	Lattice parameters	a=0.3084nm, c=0.3524nm
3	Anisotropy	1.5-5
4	anisotropy ratio γ	1.2-9
5	Critical current density (J_c)	~10^7Acm^{-2} (15K,0T) for thin film
6	Critical current density (J_c)	~10^5Acm^{-2} (15K,0T) for thick film

7	Critical current density (J_c)	~$9.1x10^3 Acm^{-2}$ (5K,8T) for Bulk
8	Upper critical field H_{C2} at 4.2K	15~20T
9	Lower critical field Hc_1	27-50mT
10	H_{irr} at 4.2K	6~12T
11	Coherence length ξ at 10.8K and 4.8mT	ξ_{ab}=39±15nm, ξ_c=35±10nm
12	Penetration depth λ at 10.8K and 4.8mT	λ_{ab}=107±8nm, λ_c=120±15nm
13	Resistivity $\rho(T_c)$	0.4 μΩ cm
14	Density (theoretical)	2.63 gcm^{-3}
15	Isotopic effect (α_T)	$\alpha_B + \alpha_{Mg}$=0.3+0.02
16	Carrier density	1.7-2.8$x10^{23}$ hole cm^{-3}
17	Energy gap Δ(0)	1.8-7.5meV
19	Grain size (polycrystalline Sample)	10nm to 10μm
20	Grain size (thin film sample)	120nm in the basal plane and 10nm along the c-axis
21	Surface resistance (R_s)for thin film	100μΩ(10K,18GHz)
22	GL parameter κ_{GL}	25.4

MgB_2 BULK

The solid-state sintering process is promising method for manufacturing MgB_2 bulk superconductors. High purity (99%) powders of magnesium and amorphous boron were used as starting materials in a stoichiometric composition, hand mixed by mortar and pestle then compacted to desired form and subjected to the heat treatment procedure in flowing high quality Ar gas to prevent the formation of oxides (MgO and B_2O_3). Generally, in the initial powder mixture excess Mg has been taken in order to compensate the loss of Mg and to get the stoichiometric phase-MgB_2 [18]. The melting points of magnesium and boron are 650°C and 2030°C respectively. So, the formation of MgB_2 is controlled mainly by the diffusion rate of magnesium atoms at 650°C. The heating rate was kept around 5°C/ min to attain a sintering temperature around 800°C, and then hold at this temperature for four hours. The specimen was then cooled from 800°C to 660°C with a cooling rate of 5°C /min. and kept at this temperature for 12 hours before cooling to room temperature.

The ex-situ prepared samples generally have higher density and better homogeneity as compared to the in-situ samples. However, for better superconducting properties, the ex-situ samples need much higher sintering temperatures as compared to the in-situ samples. Volatile Mg loss and Mg oxidations are two main hurdles for the in-situ synthesis of MgB_2. Mg vapor loss at elevated temperature cause non-stoichiometry or B-rich phases in the final product, and the MgO that forms at the grain boundaries acts as weak links. This affects the grain connectivity and reduces the critical current density of the superconductor.

The reactive liquid Mg infiltration method is another promising route for the fabrication of MgB_2 bulk superconductor. In this method, the B powder is packed in a container and Mg source is embedded in the B compact. The container is then evacuated, sealed and heat treated to temperatures of typically 900°C for few hours. During the heat treatment, the Mg melts, infiltrates into the B compact and reacts with B to form dense MgB_2 bulk [19].

4. MgB_2 THIN FILMS

MgB_2 thin films are successfully grown using different techniques, such as pulsed laser deposition, molecular beam epitaxy, magnetron sputtering, hybrid physical chemical vapor deposition, co-evaporation and electrophoresis. The MgB_2 films have been fabricated on different types of single crystal substrate including SiC (0001), Si (100), Al_2O_3-R, Al_2O_3-C, Sapphire, MgO (100), $LaAlO_3$, $SrTiO_3$ (100) and YSZ.

Superconducting MgB_2 thin films have been prepared by pulsed-laser ablation in two-step process [20]. The laser beam is guided by a set of optical devices into the vacuum chamber and well-focused by

a condenser lens before the laser spot strikes the target surface. The vacuum chamber was used to maintain a high vacuum up to 10^{-8} Torr. The substrates were mounted onto substrate heater, which was heated up to temperature 1050°C. The target to substrate distance can be adjusted from 20mm to 40mm in order to gain the best quality. A Mg-B target with stoichiometric ratio larger than 1:2 was used. The target composed of a mixture of Mg and MgB_2 powders compensate for the volatility of Mg and ensure a high Mg content in the film. The film formation depends on the condensation of atomic species on the substrate, followed by their surface diffusion and nucleation. The sticking coefficient is low especially if the substrate temperature is higher than 200°C. High quality, c-axis oriented MgB_2 films were successfully grown on 6H-SiC substrate followed by a low-pressure in situ annealing procedure. The T_c onset increased monotonically from 25.2 to 33.7K as the annealing temperature is increased. The J_c of the film was $10^5 Acm^{-2}$ at 5K and 7.8T [21]. Superconducting MgB_2 thin films fabricated on Al_2O_3(001) and MgO (100) substrates by a two-step method shows a T_c of 38.6 K for MgB_2/Al_2O_3 and 38.1 K for MgB_2/MgO. The MgB_2/Al_2O_3 films consist of well-crystallized grains with a highly c-axis-oriented structure. The superconducting properties depend on the annealing conditions: temperature, heating rate and time. The ex-situ annealed MgB_2 film has a T_c of 38.1 K, while the in-situ film has a suppressed T_c of 34.5 K. The ex-situ annealed MgB_2 thin films in Mg atmosphere exhibit very fine grain size (1200 Å in the basal plane and 100 Å along the c-axis). For ex-situ MgB_2 thin films on $SrTiO_3$ substrate, high T_c was obtained by magnesiating pure boron films grown by pulsed-laser ablation [22].

Superconducting thin films of MgB_2 grown in-situ by molecular beam epitaxy (MBE) with basal pressure of ~10^{-9}Torr from pure metal sources using multiple electron gun. By proper calibration and real time monitoring of the Mg and B deposition rate, growth temperature, Mg:B flux ratio and background pressure a typical film of thickness 600Å are deposited[23,24]. A very high Mg vapor pressure is required when MgB_2 films are deposited at elevated temperature. The films with preferential orientation prepared on various substrates $SrTiO_3$ (001), sapphire R, sapphire C and Si (111) have T_c onset of 36 K with a sharp transition width of ~1 K. MBE enable one step growth of epitaxial MgB_2 thin films at relatively low substrate temperature of 150-320°C. The T_c was increased with increasing annealing temperature. The maximum improvement of ~2K was achieved by annealing at ~500°C.The T_c of the as-grown superconducting MgB_2 thin films was close to the bulk value.

MgB_2 thin films grown by hybrid physical-chemical vapor deposition on 4H-SiC and 6H-SiC substrates have smooth surface with T_c ~40 K, low resistivities, high residual resistivity ratios, and high critical current densities. It shows that SiC is chemically stable and is an ideal substrate for MgB_2 thin films [25,27]. For superconducting MgB_2 thin films on single crystal $LaAlO_3$ (001) substrates, the boron precursor films grown by chemical vapor deposition uses B_2H_6 as a boron source. The Mg vapor segregates around the substrate, generating a Mg-rich environment. So, Mg react with B_2H_6 when the precursor gas flows in and magnesium was incorporated into the films during a post-annealing process in the presence of high purity magnesium bulk at 890°C for 40 min. MgB_2 films fabricated on MgO, $SrTiO_3$, and YSZ substrate have root-mean-square (RMS) roughness less than 5 nm, makes them suitable for device applications.

The thin films of MgB_2 were successfully synthesized by using electrochemical technique [26]. The film deposited from the aqueous bath of appropriate quantity of magnesium acetate and boric acid dissolved in distilled water and non-aqueous bath of dimethyl sulphoxide. The as deposited films were then heated at moderate temperature of about 450°C for 5 hour shows sharp superconducting transition at 36.4K.

Also, in situ reactive deposition thin film growth technique allows growth of double-sided, large-area films in the intermediate temperature range of 400–600°C [28]. These films were clean, well connected and consistently display T_c values of 39K with low resistivity and residual resistivity values.

MgB_2 thin films deposition by magnetron sputtering technique includes: dc and rf magnetron sputtering, which uses single MgB_2 target and co-sputtering in which two targets of Mg and B. This technique provides lower radiant heat to the substrate

and has better stability in deposition rate which result in uniformity of film in large area of the substrate.MgB_2 thin films grown using a magnetron sputtering deposition technique followed by in situ annealing at 830 °C on both sapphire and MgO substrates showed a maximum T_c of 35K (onset), a transition width of 0.5K and J_c~$10^6 Acm^{-2}$[29]. The MgB_2 thin films deposited by rf magnetron sputtering using MgB_2 or a pure B and Mg metal in a 50mTorr Ar atmosphere at room temperature followed by in-situ annealing at 650ºC for 5 to 20 minutes had T_c~27K [30]. The Mg particles from the sputtering source has difficulty in adsorbing on their heated substrate and reevaporated easily because it has higher kinetic energy (10~1000eV).

Large-area MgB_2 thin films deposited by co-deposition of Mg and B on silicon nitride and sapphire substrates after a post-annealing in Ar atmosphere at temperatures between 500ºC and 900ºC depending on the characteristics of the substrate. The films were patterned by a standard photolithographic process down to dimensions of the order of 10 µm without showing a considerable degradation of the superconducting properties. Both, ex-situ and in-situ annealed co-deposited boron and magnesium thin films on sapphire and silicon substrates give smooth nanocrystalline films. The nanocrystalline MgB_2 films co-deposited on silicon substrate have T_c ~33 K and zero resistance T_{co} ~ 27 K.

MgB_2 thin films with stoichiometric composition fabricated on sapphire substrate by using electron beam deposition and without any post-treatment at low temperature 120ºC having columnar grains aligned perpendicular to the film surface. The T_c increased to 33K with raising the deposition temperature up to 240ºC. Because of the high Mg vapor pressure at elevated temperature and strong reactivity of Mg with oxygen, the probability for Mg sticking onto substrate and reacting with B atoms drops to zero above 250ºC. Hence it is difficult to obtain high quality MgB_2 thin films via direct in situ deposition techniques.

5. MgB_2 THICK FILM

Screen printing, hybrid chemical vapor deposition, electrocrystallisation, and electrophoretic deposition techniques have been used for fabrication of thick film of MgB_2.

Polycrystalline MgB_2 thick films have been prepared at ambient pressure using screen printing technique on Al_2O_3, $LaAlO_3$, MgO and $SrTiO_3$ substrate. The boron films are first deposited via screen printing onto single-crystalline and polycrystalline Al_2O_3 followed by a reaction with Mg vapor for conversion into superconducting material. A highly viscous paste of amorphous boron was obtained using acetone and terpineol. The paste was then printed through sieve onto various substrate and dried at ~125ºC.The specimen was then put into sealed crucible together with pure magnesium as source material. The conversion into MgB_2 was carried out by heating at 850ºC for three hours in argon-hydrogen atmosphere under ambient pressure by maintaining heating and cooling rate at 5ºC.The transition temperature T_c varied from 34 and 36K with transition width smaller than 2K.The critical current densities of all MgB_2 films on either single-crystalline or polycrystalline Al_2O_3 were independent of the orientation of the film with respect to the magnetic field direction. The dense polycrystalline MgB_2 films consisted of crystallites with large dimensions of about 100nm [31].

The electrophoretic deposition technique [32] enables single- or double-sided coating on substrates. The electrophoretic deposition technique is two-step process in which boron films were deposited by electrophoretic deposition technique and further heat treated for about two hours in flowing argon atmosphere at various temperatures in excess of magnesium powders. The dc potential of 120V was applied across both the electrodes for about two minutes to achieve thick, uniform and good quality films. A stable suspension of MgB_2 powder was prepared in methanol dispersant, while iodine was used as an additive to induce positive charge on the MgB_2 particles to achieve cathodic electrophoretic deposition on to stainless steel substrates [33]. The quality of deposited film depends on the optimization parameters such as quantity of powder loading, deposition potential, deposition time, electrode distance etc. The T_c of the film was slightly less (~38.5K) due to the presence of MgO in the sample.

The electrocrystallisation process of superconducting MgB_2 film on magnesium film has been developed. It involves deposition of c-axis oriented magnesium film by vacuum evaporation technique. This magnesium films are used as electrode for reduction of boron and its subsequent electrocrystallisation into MgB_2 from non-aqueous electrolytic bath. The films deposited on Al_2O_3 substrate are further heat treated for proper superconducting phase formation, enhanced crystallinity and relatively higher T_c value[34].

MgB_2 thick films of thickness 1.5 to10 µm have been synthesized on a stainless steel and sapphire substrate by hybrid physical chemical vapor deposition technique at low temperature ranging from 520-600ºC [35]. The smaller Mg sticking coefficient at higher deposition temperatures leads to Mg-deficient films. Here, Mg ingot and B_2H_6 as the raw materials are used. MgB_2 films on three-dimensional(3D) structure using a hybrid physical chemical vapor deposition system of thickness 10µm have been fabricated on the outer surface of polycrystalline Al_2O_3 cylinder with T_c~39K and J_c~5.1x10^5Acm^{-2}.

6. APPLICATIONS

Superconductors are used in variety of commercial applications [36,7]. MgB_2 superconductors are used in magnetic resonance imaging, fault current limiters, transformers, motors, generators, adiabatic demagnetization refrigerators, magnetic separators, magnetic levitation, superconducting magnetic energy storage and high energy physics applications. For MgB_2 superconductors the operating temperature is around 30K which can be attained easily by using modern cryocooler facilities or liquid nitrogen. So, this can avoid use of very expensive liquid helium. MgB_2 is mechanically hard and brittle and therefore cannot be drawn into wires and tapes. The intergranular current flow in MgB_2 is not obstructed by weak links thus removing one serious obstacle for the fabrication of wires. The powder in tube (PIT) technique and infiltration method has been used in the fabrication of MgB_2 wires and tapes. In PIT method, the stoichiometrically weighed and uniformly mixed precursor powder is filled in metal tubes and are compacted mechanically. These tubes are then rolled into wires or tapes followed by heat treatment in inert atmosphere. The metallic tube must be chemically compatible with MgB_2 and should not degrade the superconductivity at higher temperatures. In infiltration method, reactive Mg infiltrates into B matrix to obtain MgB_2. For magnet manufacturing, kilometer long MgB_2 superconducting wires with uniform and high critical densities are fabricated. A current of 20kA at 24K in an electrical transmission line consisting of two 20-meter-long cables with a total outer diameter of only 16 cm made of MgB_2 superconductor is a promising option for the electricity grid. Low cost superconductive cable using the magnesium-diboride [37] powder is very flexible and is able to carry 500 times (3.2 GW) more electricity than copper wires. A 10MW, 8.1 rpm superconducting wind generator are designed by using MgB_2 wire for the field coils working at 20K[38]. MgB_2 cylinder of outer diameter=21.3mm, height=14.1 mm and wall thickness=3.5mm has been used for inductive fault current limiter. MgB_2 films has shown great potential in superconducting magnetic shielding for the measurement of ultra-weak magnetic field [39]. The surface resistance (R_s) value of the MgB_2 thin film at 10K and 18GHz is found to be 100µΩ, which implies that the MgB_2 is a promising material for microwave applications in Josephson junction bolometers [40] and high-power antenna.

7. CONCLUSION

MgB_2 had the highest critical temperature T_c of any non-cuprate superconductor. The conventional doping decreases the T_c of MgB_2 so to improve T_c, new dopant phases with improved characteristics are required. However, there has been no effective method developed to improve the T_c of MgB_2.There is further need to improve the ex-situ MgB_2 bulk materials by understanding of complex interaction between microstructure and superconducting properties. It is difficult to fabricate MgB_2 films due to the volatility of Mg, the low sticking coefficients of Mg at elevated temperatures, and the reactivity of Mg with oxygen. The high Mg vapor pressure is required for the phase stability which makes in-situ film growth difficult. Supercurrent flow in MgB_2 is unhindered by grain boundaries which makes it potentially attractive for technological applications in the temperature range 20–30 K.

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