

Bi_{2-x}Hg_xSr_{2-y}Ba_yCaCu₂O₈/Ag Sheath HTSC Wires, (Hg, Ba) Substitution Effect on the Critical Temperature

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ABSTRACT

Bi_{2-x}Hg_xSr_{2-y}Ba_yCaCu₂O₈ High Temperature Superconductor (HTSC) has been prepared as a pellet by solid state reaction with a certain substitution percentages (0 ,0.05 ,0.1) of Hg and Ba substitution instead of Bi and Sr respectively . Then, HTSC wires were fabricated from the prepared superconductor pellets using powder in tube (PIT) method utilizing silver as the tube material. The prepared wires are of three types; with monofilament MOF, 9 multifilament core 9MF and 81 filaments core(81MF). Several cycles of mechanical drawing and rolling process performed to the starting silver tube of 0.4 cm diameter and 5 cm length to minimize the filament diameter. The average filament diameter with 81MFC wire was about 25μm measured with an optical microscope.

T_c critical temperature for superconductivity is measured for the pellets and wires using four point probes techniques. These results show that the substitution Bi by Hg give a rise to the superconductor to improve highly T_c , while substitution Sr with Ba lowers T_c .substitution of 0.05 , 0.1 Hg to the composition Bi_{2-x}Hg_xSr_{2-y}Ba_yCa₁Cu₂O₈ will raise the transition temperature (T_c) . Also substitution of Sr by Ba decreases the transition temperature (T_c), Hg(0.05 – 0.1) substitution still raise T_c after substitution of Ba with (0.05 – 0.1). low – T_c phase (2212) , 2201 phase in B-2212 system and the addition of Ag to silver sheaths and a small amount of impurity phases appear in the result of XRD analysis.

Keywords: Component; Superconductor, Wire ,Filament ,Cryogenic ,Current Density, Critical Temperature ,Powder In Tube .

تأثير أستبدال (Hg, Ba) على درجة الحرارة الحرجة لأسلاك مفرطة التوصيل عالية الحرارة لمركبات $\text{Bi}_{2-x}\text{Hg}_x\text{Sr}_{2-y}\text{Ba}_y\text{CaCu}_2\text{O}_8$ المغلفة بالفضة

حضرت مركبات $\text{Bi}_{2-x}\text{Hg}_x\text{Sr}_{2-y}\text{Ba}_y\text{CaCu}_2\text{O}_8$ فائقة التوصيل عاليه الحرارة بشكل حبيبات باسلوب تفاعل الحاله الصلبه وبقيم استبدال مئوية محددة وهي (0,0.05, 0.1) من Hg و Ba تعويضيا عن Bi و Sr على التوالي. اسلاك فائقة التوصيل عالية الحرارة صنعت من الحبيبات فائقة التوصيل باستخدام طريقة المسحوق داخل الانبوب (PIT) بالاستعانة بالفض كماءة للانبوب. الاسلاك المحضرة كان بثلاثه انواع : بشعيه مفردة MOF و 9 شعيرات 9MF و 81 شعيره (81MF) . عديد من عمليات السحب والبثق الميكانيكي متعاقبه للانبوب الفضة الاولى ذو القطر 0.4 سم وبطول 5 سم لتصغير قطر الشعيرة. متوسط قطر الشعيرة للسلك 81MF هو حوالي 25 مايكرون حسبت بالمجهر الضوئي.

درجة الحرارة الحرجة T_c للموصلات الفائقة حسبت للحبيبات وللأسلاك باستخدام تقنية ماخذ الاربع نقاط. اظهرت هذه النتائج ان استبدال Bi و Hg تزيد من T_c بينما استبدال Sr ب Ba تخفض منها, الاستبدال $\text{Hg}(0.05,0.1)$ للمركب يزيد من T_c , كذلك فان استبدال Sr ب Ba تخفض T_c , ان نسب الاستبدال $\text{Hg}(0.05,0.1)$ تبقى مرتفعة مع وجود استبدال Ba بنسب (0.05, 0.1) , الطور الواطي (2212) والطور 2201 في نظام الطور B-2212 مع الاضافة من الفضة ونسب ضئيلة من اطوار الشوائب ظهرت في نتائج تحليل طيف الاشعة السينية المستطارة XRD.

INTRODUCTION

Essentially every application of superconductivity to electric power technology, especially with regard to transmission and distribution cables, depends on the successful development of suitable wire or tape in long lengths. Progress toward this end using the new HTSC materials has moved much more rapidly in the first 20 years since their discovery than what at first appeared to be likely given the universally poor ductility (one might say nonductility) of ceramic materials. An added factor, the high degree of crystalline, and therefore electronic, anisotropy of the HTSC compounds. The two decades since their discovery have seen more than two dozen successful prototype and demonstration projects worldwide involving the new discoveries—ac cables, motors, generators, current limiters, power leads, small superconducting magnetic energy storage units, transformers, reactive power controllers, flywheels, and more[1] .

Anderson –Kim model of flux creep relates the measured critical current density (J_c) in type-II superconductors to the pinning strength (F_0) and voltage criterion (E_c) in the following manner [2]:

$$J_{c(T)} = J_{c(0)} [1 - (k_B T / F_0) \ln(Bd\Omega / E_c)] \quad \dots (1)$$

where $J_c(0)$ is J_c at absolute zero temperature, Ω is the frequency of a flux-hopping event, d is the hopping distance, B is the magnetic induction, and E_c is the voltage criterion (usually the smallest discernible electric field, V/m). It is clear that for any volt-per-meter criterion E_c , there will be either a given magnetic field or a temperature for which a certain value of J gives a discernible voltage drop per unit length of the

superconducting sample [2]. Therefore, the concept of critical current (J_c) for type-II superconductors is no longer well defined, and there is a significant discrepancy of the J_c values obtained with the different voltage criteria. A less sensitive E-criterion, typical of transport current measurements will give correspondingly a higher value of J_c ; the $1\mu\text{V}/\text{cm}$ is the most commonly used voltage criterion.

In this power-law model the electric field E is proportional to the current I of power n in the following way[3]:

$$E = E_c (I / I_c)^n \quad \dots(2)$$

where (E_c) is the voltage criterion, I_c is the corresponding critical current, and the n -value defines the steepness of the transition curve.

The I-V characteristics of HTS wires can be expressed using the power law [4]

$$V = k I^n \quad \dots (3)$$

Where V is the voltage rise across the voltage tap, I is the current flowing in the wire, n is a positive number indicating the index of transition or a measure of the sharpness of transition, and k is a constant of proportionality.

Within the region where the current I is less than I_{max} , the voltage V increases non-linearly. This is due to the flux creep phenomenon in the superconductor. Afterwards, the voltage V increases linearly with the current I because of the homogeneous flux-flow of the wire. The slope corresponds to the full flux-flow resistivity.

Typical electric field and resistivity criteria are $1\mu\text{V}/\text{cm}$ and $2 \times 10^{-13} \Omega \text{ m}$, respectively. I_c is determined as the current corresponding to the point on the I-V curve where the voltage is V_c measured relative to the baseline voltage.

$$V_c = L E_c = I_c \rho_c L / A \quad \dots (4)$$

where E_c and ρ_c are the electric field criterion and resistivity criterion, respectively; L is the voltage tape or wire separation along the sample; A is the total cross sectional area [5].

Bi-Sr-Ca-Cu-O System(BSCCO) conductors represent the basis for all present large-scale applications of HTS. They are called conductors of 1st generation. BSCCO has a well-established manufacturing technology, and commercial conductors are available in long lengths (up to 1 km). Bi – based superconductors have three superconducting phases described by a general formula $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_8$ ($n = 1, 2, 3$).

The superconducting BSCCO has an orthorhombic structure[6], they consist of perovskite where copper oxide planes sandwiched between double bismuth oxide layers with rock salt coordination, and have been compared to Aurivillins phase[7,8]. This body – centered orthorhombic Braveries lattice is only an average structure, however as the compounds display incommensurate modulation [9].

Bi-2212 tape conductors with a HTS fill factor of up to 60 % can be produced by simple surface coating of Ag bands. By improved processing (“Pre-Annealing and Intermediate Rolling“, “PAIR”) tapes with J_c (4.2 K°, 10 T) > 500 kA/cm² have been fabricated [7] [9].

The aim of the work is to fabricate superconductor Bi_{2-x}Hg_xSr_{2-y}Ba_yCaCu₂O₈/Ag Sheath HTSC Wires using powder in tube technology and to determine the (Hg, Ba) substitution effect on the Critical Temperature(T_c).

Experimental

The samples were prepared by solid state reaction using appropriate weights of pure materials Bi(NO₃)₂, Sr(NO₃)₂, BaCO₃,CaCO₃ and CuO, and in proportion to their molecular weights.

The weight of each reactant was measured by using a sensitive balance type [Sartorius]. The powders were mixed using agate mortar; 2- propane was added to homogenize the mixture during grinding for 40-60 minutes. The mixtures were dried in an oven at 150 °C. The mixtures were calcined in programmable tube furnace and the powder was heated to 800 °C for 24 hours. The calcined powder was then reground and mixed with the appropriate weight of HgO, then pressed into disc-shaped pellets 1.3 cm in diameter and 0.1-0.2 cm thick, under a pressure of 0.370 MPa. The pellets were presintered under O₂ gas flow at 840 °C for 72 hr with 1 °C/min heating rate and then cooled at rate of 60 °C/ min to room temperature[11,12].

The presintered pellets were reground, repressed and resintered in O₂ at the same range of temperature for further 24 h. 10 mAmp. current was supplied to the sample by a current source D.C power supply (Electronica - Veneta DV 30/V3); the voltage drop was measured by a Keithley model 180 nanovoltmeter with sensitivity of about ± 0.01 nanovolt was used for voltage measurements; Edward pirani 12 a gage was used to determine the pressure inside the cryostat. The resistivity (ρ) could be found from the relation[13]:

$$\rho = Vwt/IL \quad \dots (5)$$

for pellet of 1.3 cm diameter^[14] where : I is the current passing through the sample, V is the voltage drop across the electrodes. w is the width of the sample. L is the effective length between the electrodes, t is the thickness of the sample. All measurements of L, t and w were made by using digital vernier.

Critical temperature could be found from the curve of resistivity versus temperature. T_c is the temperature at which the resistivity drop to 50 % of its extrapolated normal state value at room temperature, or is the temperature at the midpoint between the resistivity at the onset of the transition T_{c2} and the zero resistivity point (T_{c1}), as given by the following relation[15]:

$$T_{C3} = \frac{1}{2} [T_{C1} + T_{C2}] \quad \dots (6)$$

To fabricate silver tubes in order to use it in later, these steps were followed : Starting with commercial silver powder of 90% purity we made several steps for purification,

firstly washing by water. Immersing silver powder in delicate HCL solution for 24 hr. Melting silver powder in ceramic boat. Drawing the solid pellet to long tapes of about 15 mm width and 0.4mm thick. Drawing the Ag tape through circle hole of about 0.45 cm dim., using drawing bench shown in Fig.2-a. In this step tube will be with open side surface. Welding the open side surface of the tube using a special Ag-Mo welding material. Again drawing tube in 0.45 cm hole. Cutting the drawing tube to shorter tubes of an 4-5 cm length. To make more purification, Ag tubes are immerse in delicate HCL solution, each tube joined with Copper wire whose its second terminal will be free in air out from the HCL solution, to make electrolyte reaction remove impurities as Cu from Ag tubes[16].

Technical success with the BSCCO wires has been greatest with the powder-in-tube method. In this method, BSCCO powder is packed in a silver tube and sealed. The tube is then subjected to a series of mechanical deformations, such as drawing, rolling, pressing or swaging, and heat treatments as showing in Fig.1.

To fabricate BSCCO/Ag wires, using the powder in tube method [17,18] as shown in fig.1, Pellets that passed the resistivity measurement, as a superconductor and, having well T_c temperature, were crushed and reground into fine powder by hand in an a gate mortar and a pestle in air for 40 minutes[16].

Superconductor powder was packed in prepared silver tubes enclosed in 0.45 cm outer diameter, 0.37cm inner diameter and 0.035cm wall thickness, with 5-10 cm length. One end of the tubes was closed, and other remain open, some tubes were internally inscribed with a thread so that they could be capped at a latter stage. Filling each these tubes was performed by adding approximately 50 mg of powder at a time. To complete filling, repeated the filling for 5-10 times to get rid complete roides or gaseous gap. In this step the ratio powder/Ag approached 70%. The tubes were lightly capped and degassed in alumina boats in air at 800 °C for 3 hours with 3 °C/min, Degassing allowed adsorbed gasses to be driven off prior to sintering in order to reduce defects such as blistering, in order to get the best continuous wire. Before drawing, degassed packed tubes were then capped by plugging the open end of a tube with coil of silver tapes or with an a appropriately threaded cylindrical cap [16] .

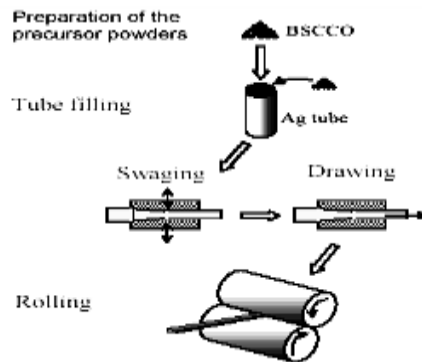


Figure (1) Schematic presentation of the OPIT technique [15].

In order to fabricate the wire, attempted to get wires with multifilament. By drawing and rolling the starting packed tubes in three steps. The packed tubes were drawn from 0.45 cm to 0.09 cm diameter wire in 19 step drawing process. Figure(2) shows the drawing bench and rolling machine that were used for drawing operation with Lanolin grease as the die lubricant, Drawing Bench contains many holes of different diameter from several cm. to about 0.15 cm and produce crucial cross Section wire while rolling machine which contains also different gaps between each two rolls, producing square cross section.

An intermediate heat treatment at about 500 °C for 15 minutes in air was carried out after 7th and 16th steps. This annealing operation was above the recrystallization temperature of silver which is 200 °C and served only to the sheath material, many steps of drawing can lead to significant work hardening of the silver sheath which increases the probability of fracture of the sheath and failure by breakage of wire composite. The result at this first step of drawing was producing wire of 0.09 cm outer diameter with more than 600 μm net diameter of single core superconductor.

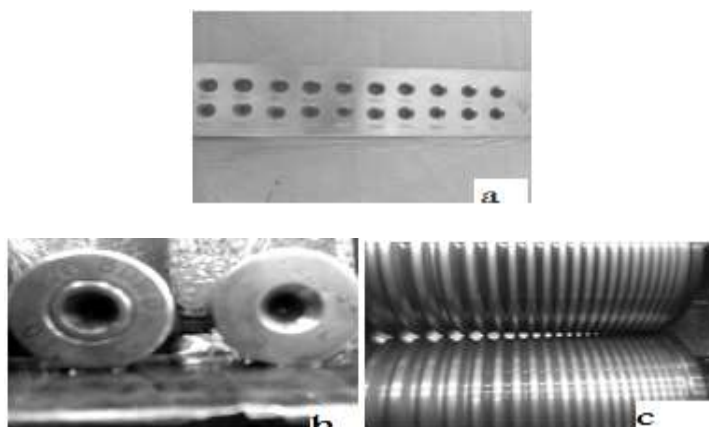


Figure (2) a- Drawing bench b- Drawing pieces, and c- Rolling machine.

Wires fabricated in first drawing step of about 30-50 cm length, were cut to short pieces of 4 cm length, 0.09 cm diameter and 9 pieces of these were packed as a beam (bundle) inside other empty silver tube of 0.45 cm outer diameter and 0.37 cm inner diameter ; at this point SC/Ag ratio was about 45% by weight. Repeat steps of drawing and rolling as in first step of drawing. The above drawing produced wire of 0.09 cm outer diameter with 9 superconductor filaments, each filament was of about 150-250 μm diameter.

Wires fabricated in second drawing step of about 30-50 cm length, were cut to 9 short pieces of 4 cm length, 0.09 cm diameter, each having 9 filaments of wires packed as a bundle inside another silver tube of 0.45 cm outer diameter and 0.37 cm inner diameter; with about 30% SC /Ag ratio in this step, an outer tube containing 81 fine filaments. Repeat all drawing steps in first and second drawing steps [16].

These steps of drawing produced wire of 0.09 cm diameter with tubes of 9 filaments at each beam which means 81 filament of superconductor wire with 30-50 cm length. Cut many sample from these mono core MOC, 9 multifilament (9MF) and 81 multifilament

(81MF) wires to the desired length for all test Critical temperature ,X-ray diffraction XRD, Optical Microscopy OM, and I- V Characterizations tests.

Samples of wires of three types (1, 9 and 81 filaments), were cut and subjected to one processing treatment. These samples underwent sintering operation on alumina trays in O₂ gas flow for 24 hours at (840 °C for B2212 with 1 °C /min. heating rate and fast cooling rate of 10 °C /min.

The four–point probe DC is used to measure the resistivity ρ , at temperature range 77-300 K⁰, and to determine the critical temperature T_c. The sample is fixed in the cryostat instrument which is joined to a rotary pump to get a pressure of 10⁻² mbar inside the cryostat, and also joined to a sensor of digital thermometer (type Pt 100 resistance to temperature detection RTD) near the sample position. Fine copper wires attached to the sample by furnace-dried silver paste served as the current and voltage leads [16]. After thermal treatment, critical temperature T_c measurement can be tried or tested. It was carried out under the same cryogenic system with 4cm length of wires, No external field was applied so the measurement was at self-field only (s.f), Resistivity measurement of the standard 4-point direct current DC contact method, using equation (5) were L=1 cm the distance between the two inner voltage points [16,17].

RESULTS AND DISCUSSION

Figure (3) show the DC Resistivity temperature diagrams to obtain T_c for the HTSC Pellet of Bi_{2-x}Hg_xSr_{2-y}Ba_yCaCu₂O₈ compounds with Hg and Ba substitution with ratio (0,0.05,0.10). Figuer (4) Shows resistivity –temperatures to obtaine T_c. Results are shown in Table (1) ,the T_c results for each compound . These results show that the substation Bi by Hg give a rise to the superconductor wires to improve highly T_c , but only at these ratio of substation , while substation Sr with Ba lowers T_c of the superconductor , G. Hermiz [19] and K.A. Jassim [20] proved that more than 0.1 of Ba lost the superconductivity state of pellets samples.

Behavior of resistivity with temperature of Bi_{2-x}Hg_xSr_{2-y}Ba_yCaCu₂O₈ samples for the different value of Hg ,suggests that the substitution of Hg has effect on the reaction to form the high-temperature phase. A certain amount of Hg (x=0.1, 0.05) is necessary for the occurrence of this reaction. Indeed the amount of Hg suitable for the formation of the high-T_c phase is determined by the competition between these reactions, increases with increasing the addition of Hg as shown in Figure(5) for Bi_{1.95}Hg_{0.05}Sr_{2-y}Ba_yCaCu₂O₈ .The conduction path in Bi-base and Hg-base are holes in the Cu-O₂ layers which is enhanced by the Bi-O and Hg-O layer. The deformation in the c-axis adjusts the amount of charge transfer from Bi layer to Cu layer , this will force the generation of hole pairing in the Cu (3d)-O(2p) band[18].

Substitution Bi by Hg will raise the transition temperature (T_c), It was found from Figure (4) that the substitution of 0.05 , 0.1 Hg to the composition Bi_{2-x}Hg_xSr_{2-y}Ba_yCaCu₂O₈ of different Hg content(x=0.05,0.1) will raise the transition temperature (T_c), but more of Hg content (x=0.25) yields semiconductor [19] . Also substitution of Sr by Ba decreases the transition temperature (T_c), Hg(0.05 – 0.1) substitution still raise T_c after substitution of Ba with (0.05 – 0.1) . This behavior is due to the fluctuation of

oxygen excess and the increase in Ba, while it may lead to metastable structure ,that decreases T_c , thus more T_c .

XRD diagrams for wires fig.(5)show that all samples have reflection intensity of the low- T_c phase reflections (peaks labeled L), (006), (008), (0012), (103), (110), and (0010). And 2201 - T_c phase reflections (peaks labeled *), (311), (315), (545), (220) and (103). The Low- T_c phase reflections of the free sample ($x=y=0$) have lower intensity than samples which have Ba . A small amount of Ba addition is quite effective in decomposing the low- T_c phase (2212) of Bi-Sr-Ca-Cu-O superconductor systems by producing BaBiO₃ and BaCuO₂ accompanied by high- T_c phase formation [18]. It has been reported that the low- T_c phase of double CuO layers strongly prohibits the formation of high- T_c phase.Low - T_c phase (2212) and 2201 phase and the addition of Ag to silver sheaths and a small amount of impurity phases of (Ca, Sr)₂CuO₃ and CuO. The appearance of more than two phases could be related to the stacking faults along the c - axis.

The lattice constants evaluated from 2θ of major peaks are also listed in Table (2). Thus, the above results suggest that the growth of the high - T_c phase is promoted by Ba - doping, similar to Hg substitution of the Bi-Sr-Ca-Cu-O system. This may be attributed to the ordered growth under the partial melting point and/or the Ba substitution for Sr. Table (2) and figure (4) show an increase in the c - axis lattice constant for Ba - doped samples as comparable with the B2212 - free samples, the reason is due to the substitution of Ba for Sr where the ionic radii of Ba⁺² (1.35 Å) is longer than that of Sr⁺² (1.13 Å) which renders c-parameter to be longer or deformed .

Indeed the deformation in the c-axis , as a result of substitution or deficiency of some atoms, adjusts the amount of charge transfer from Bi layer to Cu layer ,this will be a driving force to the pairing generation of superconductor holes forming bosons [155] which are the current carriers in our superconductor.

B2212 compounds show behavior exhibiting a decrease in the high - T_c phase and an enhancement of the peaks due to the low - T_c phase.

From Optical Microscopy pictures for the transfer cross sections of the three different types of wires MoC , 9 MF and 81 MF the filament diameter is measured ;and the HTSC is real area in comparison to total cross section (HTSC + Ag sheath) . We can see from figure 6 and table 2 that 81 MF wire was drawn in three processes in different steps of drawing and rolling , which make it possible to fabricate wires with multifilament of each one with average diameter of 25 µm or less which is near the penetration depth of 500 Å for Bi_{2-x}Hg_xSr_{2-y}BayCa1Cu2O8 [13].

Since the median particle size for the two types of HTSC compounds is very fine ,then HTSC particles flow easily in the silver tubes through drawing and rolling process and in all types of wires MoC,9 MF and 81 MF as shown in Figure (7). Small diameter wires of 81 MF (25 µm) and the sintering process controlled the HTSC grains from misalignment in MoC and 9MF wires to more alignment in 81 MF wire to the very thin platelets or plate like grains [14] .

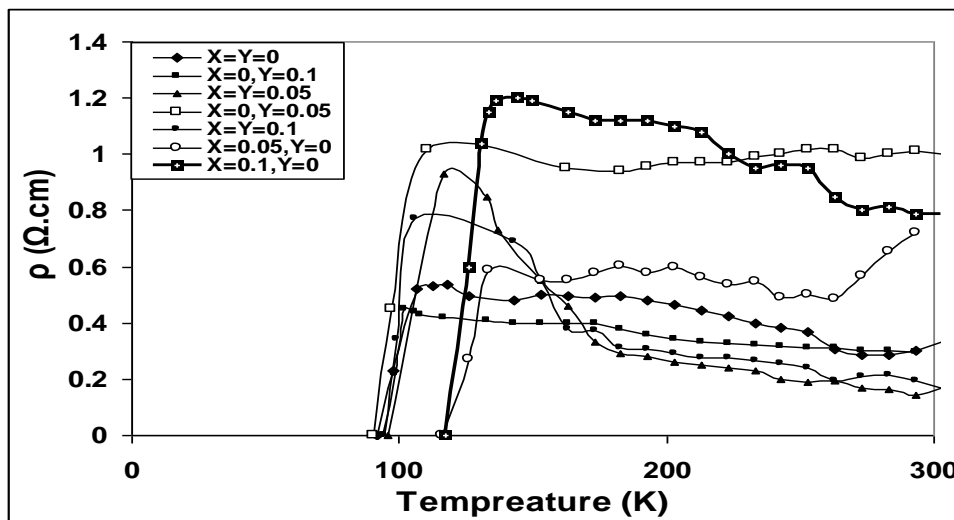
The smooth interaction between Ag matrix and the HTSC ceramic as shown in Figure (7) is an important property to produce fine filament which is necessary to obtain wire with high critical current and gives rise to Ag to decrease the sintering temperature of the

HTSC . Generally ,it is considered that BSCCO grains are aligned more strongly near the silver / core interface .

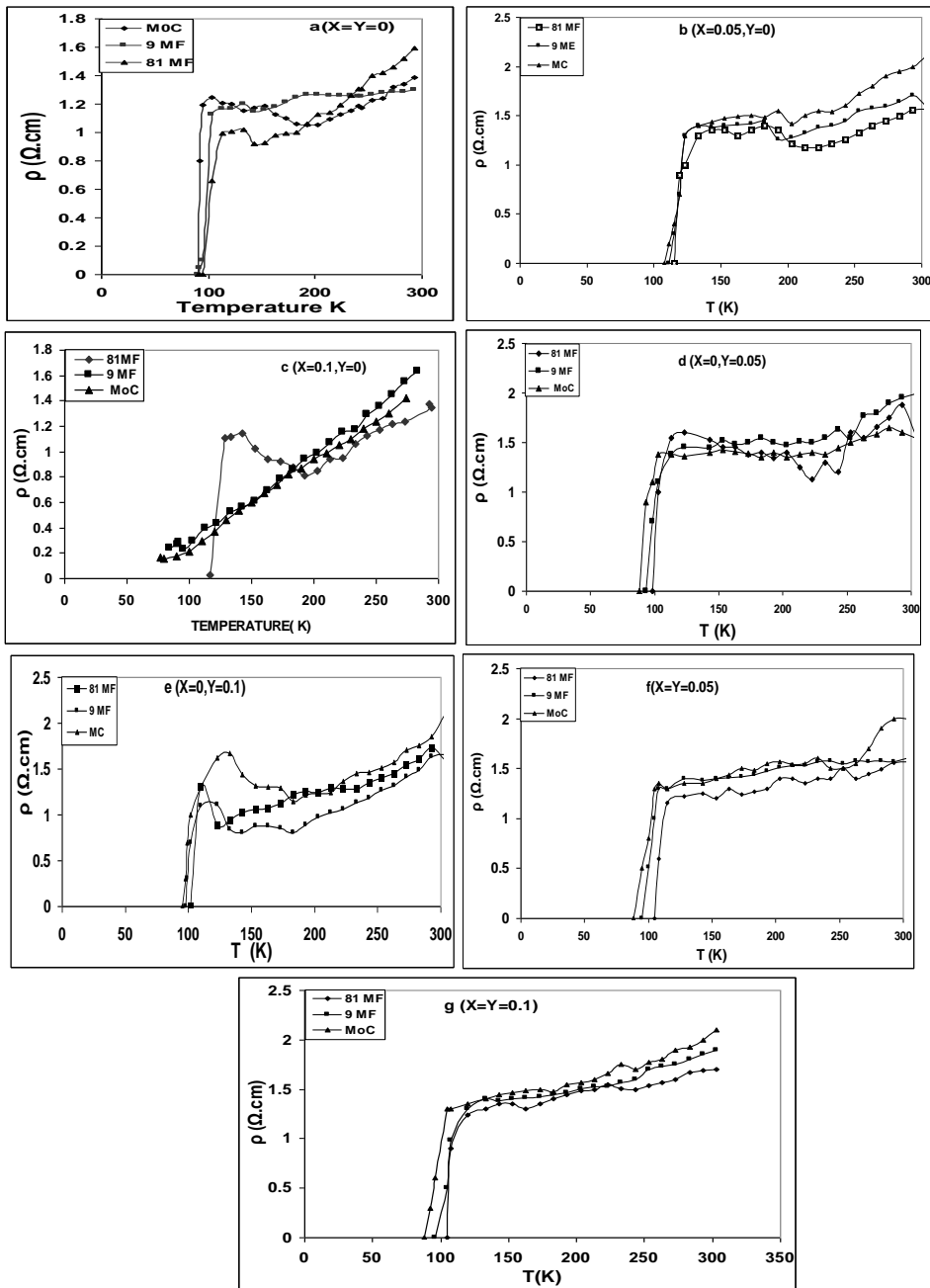
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Figure(3) Temperature dependence of resistivity for Bi_{2-x}Hg_xSr_{2-y}Ba_yCa₁Cu₂O₈ pellet compounds.



Figure(4) Temperature dependence of resistivity for the sample $\text{Bi}_{2-x}\text{Hg}_x\text{Sr}_{2-y}\text{Ba}_y\text{CaCu}_2\text{O}_8$ for the three types of HTSC wires to obtained T_c .

Table(1) B2212 Compounds wires critical Temperature .

Wire sample	X	Y	T _{c1} (K)	T _{c2} (K)	T _c (K)
MoC	0	0	91	92	91.5
9 MF			92	99	95.5
81 MF			91	103	97
MoC	0.05	0	109	119	113.5
9 MF			111	115	113
81 MF			115	119	117
MoC	0.1	0	----	---	---
9 MF			---	---	---
81 MF			110	117	113.5
MoC	0	0.05	88	93	90.5
9 MF			93	95	94
81 MF			98	103	95.5
MoC	0	0.1	95	99	97
9 MF			98	101	99.5
81 MF			101.7	106	103.8
MoC	0.05	0.05	88	94	91
9 MF			92	100	96
81 MF			96.6	102	99.3
MoC	0.1	0.1	88	97	92.5
9 MF			95	102	98.5
81 MF			105	105	106.5

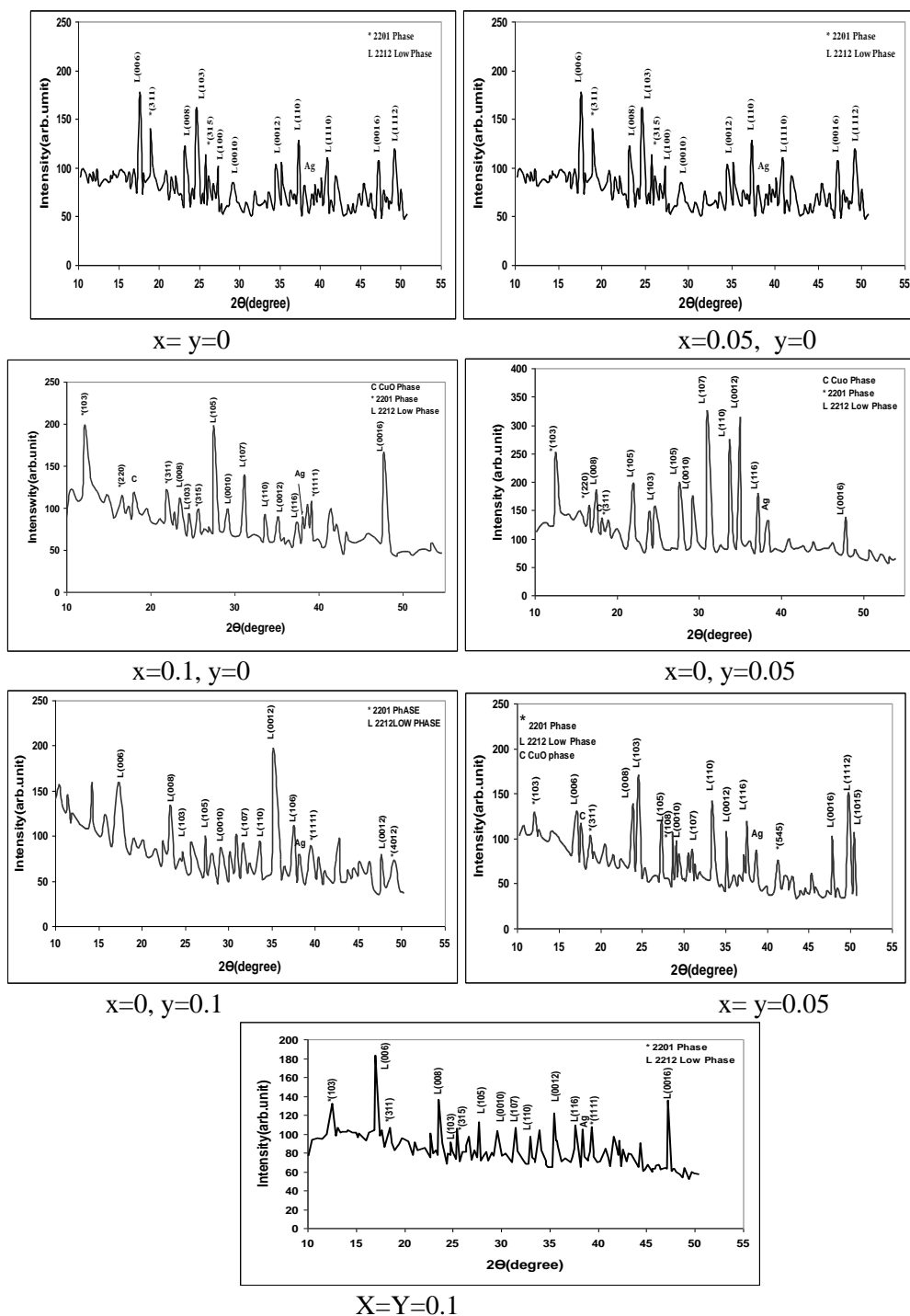


Figure (5) XRD patterns for the sample Bi_{2-x}Hg_xSr_{2-y}Ba_yCaCu₂O₈ Compounds wires.

Table(2)Phase intensity and lattice parameter for Bi_{2-x}Hg_xSr_{2-y}Ba_yCaCu₂O₈compounds wires.

X	Y	2212 Low phase %	Other phases%	a (Å°)	b (Å°)	c (Å°)
0.00	0.00	85.8	14.2	3.82	3.86	30.02
0.05	0.00	80.75	19,25	3.99	3.90	30.1
0.10	0.00	82.1	17.9	3.8	3.86	30.00
0.00	0.05	87.5	12.5	3.76	3.87	30.46
0.00	0.10	89.9	10.1	3.86	3.73	30.65
0.05	0.05	78.5	21.5	3.95	3.90	30.56
0.10	0.10	76.2	23.8	3.82	4.00	30.6

Table (3)Measurement of filament diameter obtained from optical microscopy images.

Wire sample	Maximum filament diameter(μm)	Minimum filament diameter (μm)	Average filament diameter(μm)	Area of once filament(cm²)	Area of total filaments in wire(cm²)	Fill factor % #
MoC	750	650	700	3.85×10^{-3}	3.85×10^{-3}	65
9 MF	225	125	175	2.37×10^{-4}	2.14×10^{-3}	33
81 MF	40	10	25	4.5×10^{-6}	3.6×10^{-4}	5.6

fill factor =total S.C cross section Area /total wire cross section Area .

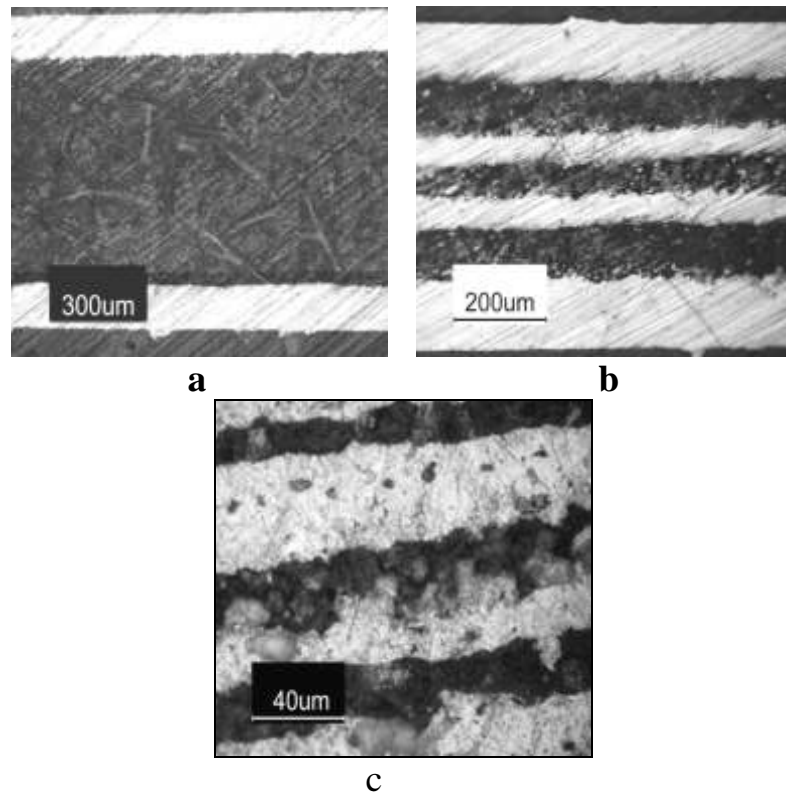


Figure (6) Optical Microscopy images of the longitudinal cross section a- MoC b-9 MF c- 81 MF wires .

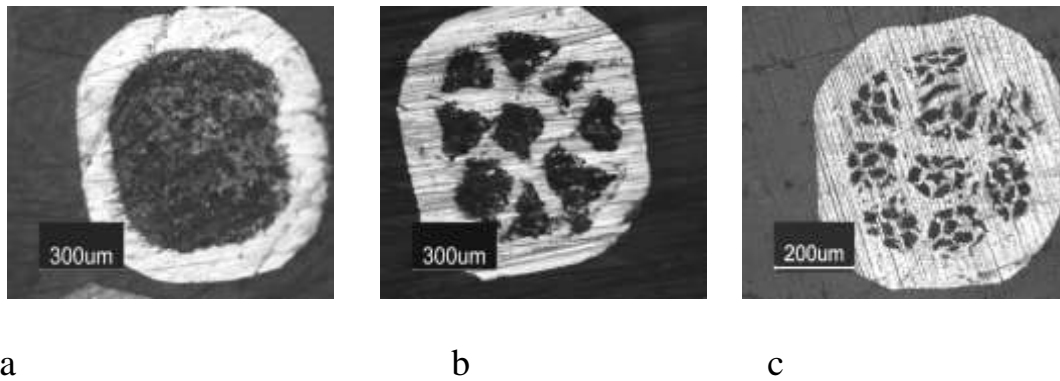


Figure (7) Optical Microscopy Images Of HTSC wires showing transfer cross section area of a- MoC , b- 9MF and c- 81 MF .