

Ting LUO, Yi ZHANG, Xing-guo LI, Li LIN,
Yue-yang ZHANG, Qing-rong FENG

Studies of synthesizing behaviors and superconductivity of sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples in flowing oxygen atmosphere

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Abstract Systematic studies of synthesizing behaviors of sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples in flowing oxygen atmosphere and their superconductivity have been performed. A set of high temperature ρ - T curves has been obtained for the whole synthesizing process. After four rounds of synthesizing, the resistivity of the sample was around $\rho=1.00\times 10^{-3}\Omega\cdot\text{cm}$ at room temperature. The ρ - T curve of the fourth round shows that the orthorhombic to tetragonal phase transformation of the sample occurs around 600 °C, which is lower than that of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ sample prepared by conventional solid-state reaction method. Other measurements, such as X-ray diffraction, SEM measurement and low temperature R - T and M - T measurement, were also performed. And the R - T and M - T measurement results suggest that during the synthesizing process, there exist some state at which the sample has better superconductivity than the other states. Moreover, we found screw dislocations presenting on the sample broken surface from the SEM images. This will change the concept that the screw dislocations can only grow on the surface of the YBCO thin films and single crystals.

Keywords sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_y$ sample, high temperature

Ting LUO¹, Yi ZHANG¹, Xing-guo LI², Li LIN², Yue-yang ZHANG¹,
Qing-rong FENG¹ (✉)

¹ School of Physics and State Key Laboratory for Artificial Micro-structure and Mesoscopic Physics, Peking University, Beijing 100871, China

² School of Chemistry and Molecular Engineering, Peking University, Beijing 100871, China
E-mail: qrfeng@pku.edu.cn

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1 Introduction

It is well known that the oxygen deficiency in the material $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO-123) is very important to its crystal structure, electron transport property, superconductivity, and many other properties [1–3]. In the past decades, many researches have been done on this. Most measurements indicated that the compound has a superconducting transition temperature T_c around 90 K with x near 0.1 and becomes semiconducting with x near 1, and the orthorhombic to tetragonal (O-T) phase transition happens when the value of x is close to 0.5 [4], or near 0.6 [5].

However, there were also samples with an x -value of 0.7 that had an orthorhombic symmetry [6], and samples with an x -value of 0.3 that had a tetragonal symmetry [7]. That is to say, the superconducting transition temperature of the material $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ is determined not only by the average concentration of oxygen atoms, but also by their spatial arrangement [6].

People already know the optimum synthesizing conditions of the compound $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ very well after years of research. However, as the preparation conditions can greatly affect the properties of the samples, it is necessary to study the whole synthesizing process to get detailed information about the relationship between their behaviors during synthesizing and the low temperature properties. This work could also improve our understanding of the mechanism of superconductivity of these kind of materials.

Studies have been done with $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples prepared by conventional solid-state reaction technique in our earlier work [8,9] (this kind of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples will be referred to as A-YBCO samples latter). In this paper we will present the results of our recent studies on the relationship between the resistivity of sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples and the synthesizing temperature while they are being synthesized in flowing oxygen atmosphere. A set of high temperature ρ - T curves was obtained. Other measurements, such as the X-ray diffraction, the low temperature R - T and M - T measurement, and the SEM measurement, were also taken. When investigating YBCO-123 thin films by atomic force microscopy (AFM) [13, 16, 17, 22], scanning tunneling microscopy (STM) [14, 15, 17, 20, 21, 23–25], and scanning electron microscopy (SEM) [16,18], researchers found that the surface morphology of YBCO-123 thin films and single crystals [26] differs from that of the bulk samples. The screw dislocations were only presented on the surface of the YBCO-123 thin films and single crystals. However, until now, no paper has reported that the screw dislocations could be presented on the surface of powder bulk samples. For the first time, we observed the screw dislocations present on the broken surfaces of the sol-gel YBCO-123 powder bulk samples.

2 Experiments

In this experiment, the samples were synthesized from YBCO-123 powder prepared by sol-gel method. The powder was first ground and then pressed in a special mold into rectangular samples having the size of $2.50 \times 0.60 \times 0.10 \text{ cm}^3$. Four gold wires of $\phi=0.40 \text{ mm}$ were pressed into one of the samples in order to measure the resistance of the sample by DC four-probe method in the synthesizing process. All the samples were then heated in a quartz tube under flowing oxygen atmosphere, and the furnace temperature was controlled by a programmable temperature controlling system. There were four rounds of synthesizing, each with three stages: the heating stage (the temperature of the furnace rose from room temperature to $920 \text{ }^\circ\text{C}$ with a rate of $7.5 \text{ }^\circ\text{C}$ per minute), the holding stage (the temperature held at $920 \text{ }^\circ\text{C}$ for 5 hours), and the cooling stage (the temperature controlling system stopped heating and the rate of cooling was determined by the cooling rate of the furnace). A computer collected the measured data, including the resistances and the temperatures of the sample. After each round, the samples (one piece was kept for later measurements) were re-ground and pressed. For convenience of later discussion, we will refer to the samples, which have gone through the first

round, the second round, the third round, and the fourth round of synthesizing as sample *a*, *b*, *c*, and *d*, respectively. The X-ray diffraction patterns were obtained from a Philip X' diffractometer with Cu K_α radiation. The low temperature resistances of all the samples were measured on PPMS. The magnetic measurements were carried out on a MPMSXL-7 SQUID magnetometer, and the SEM photomicrographs were taken by a QUANTR-200-F scanning electron microscope and FEI STRATA DB 235 focus ions beam (FIB).

3 Results and discussion

Figure 1(a), (b) and (c) show the high temperature (HT) ρ - T curves of the sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples *a*, *b*, *c*, and *d* in the heating stage, the holding stage and the cooling stage, respectively. There are two special temperatures, T_V and T_P , where the samples change their conductivity for every HT ρ - T curve (except for sample *b*, whose resistivity drops monotonically with the rising of synthesizing temperature) in the heating stage. In the cooling stage, there is another special temperature, T_{ms} , at which the samples change from metallic to semiconductive. Table 1 is the summary of the characteristic resistivities and special temperatures of all these samples.

The curves in Fig. 1(a), (b) and (c) are similar to what were reported in Ref. [8] and Ref. [9]. However, since the holding time in our experiment is 5 hours, only half of that in Ref. [9], and since we have taken more measurements, our results can give more a detailed information about these samples. The final sample *d* has a resistivity of $1.00 \times 10^{-3} \Omega \cdot \text{cm}$ at room temperature, and its T_c is around 90 K . Comparing our results with those of Cava [6], we can conclude that the oxygen deficiency of our sample is very close to zero.

From these results listed above, we can conclude that: (1) It is typical of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples to have special temperatures, T_V and T_P , at which the samples change their conductivity in the heating stage. The more times the sample is synthesized, the lower T_V it has and the larger the ΔT is, which indicates that the phase formation of the sample becomes easier and easier, and the amount of the metallic phase becomes larger and larger. Finally, if the sample is not reground, it is metallic in the whole heating stage. (2) The lowering of the Semi-M phase transition temperature and its final disappearance could be a good guideline for judging the completion of the superconducting phase formation. (3) The formation of the superconducting phase in sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples is much quicker and easier than in A-YBCO samples [8].

Table 1 The comparison of resistivity of the sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples in different stages as prepared in flowing oxygen atmosphere.

Sample name	<i>a</i>	<i>b</i>	<i>c</i>	<i>d</i>	
Heating stage	$\rho_{iS}/(\Omega \cdot \text{cm})$	1.49×10^3 (50.9 °C)	8.57×10^3 (46.0 °C)	4.55×10^{-3} (47.8 °C)	6.69×10^{-3} (60.6 °C)
	$\rho_{tE}/(\Omega \cdot \text{cm})$	2.98×10^{-2} (926.8 °C)	2.40×10^{-2} (927.5 °C)	2.76×10^{-3} (927.2 °C)	5.00×10^{-3} (923.9 °C)
	$\rho_{pI}/(\Omega \cdot \text{cm})$	1.01×10^1 (791.1 °C)	No	3.61×10^{-3} (803.0 °C)	1.02×10^{-2} (792.8 °C)
	$\rho_{vI}/(\Omega \cdot \text{cm})$	1.66 (567.4 °C)	No	2.08×10^{-3} (384.0 °C)	3.35×10^{-3} (345.0 °C)
	$(\rho_p - \rho_v)/\rho_v$	5.1	No	0.7	2.0
Holding stage	$\rho_{hS}/(\Omega \cdot \text{cm})$	2.72×10^{-2} (916.1 °C)	8.40×10^{-3} (916.6 °C)	2.66×10^{-3} (917.3 °C)	5.40×10^{-3} (916.1 °C)
	$\rho_{hE}/(\Omega \cdot \text{cm})$	2.13×10^{-2} (916.7 °C)	3.53×10^{-3} (916.4 °C)	3.55×10^{-3} (916.5 °C)	4.12×10^{-3} (916.4 °C)
	Holding time/min	290	280	290	328
Cooling stage	$\rho_{cS}/(\Omega \cdot \text{cm})$	2.13×10^1 (916.6 °C)	3.53×10^{-2} (916.6 °C)	3.78×10^{-2} (916.7 °C)	4.12×10^{-2} (916.4 °C)
	$\rho_{cE}/(\Omega \cdot \text{cm})$	1.93×10^1 (55.3 °C)	0.70×10^{-3} (54.1 °C)	1.19×10^{-3} (49.1 °C)	1.02×10^{-3} (49.9 °C)
	$T_{ms}/\text{°C}$	500.1	351.7	149.7	No

Note: ρ_S , ρ_E , ρ_P and ρ_V refer to the starting resistivity, the ending resistivity, the peak value of the resistivity, and the valley value of the resistivity respectively. T_{ms} refers to the metal to semiconductor transition temperature.

The inserted figure in Fig. 1(c) indicates that the O-T phase transition in our sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ sample *d* occurs between 550 °C and 600 °C, which is much lower than 690 °C [8] and a little lower than 613 °C [9], and the transition takes place in a wider temperature range. This is quite understandable considering that the transition temperature could be decreased dramatically by lowering the partial pressure of oxygen from 690 °C in 100 % O_2 to 470 °C in 0.034 % O_2 , and that the O-T phase transition is completed in temperature ranges spanning nearly 100 °C [10]. Our sample having a little lower transition temperature than 613 °C must be caused by the inadequateness of oxygen during synthesizing. As to the difference between the sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ sample and the A-YBCO sample, the raw material of the former,

having a grain size on the nanometer scale, makes it behave much differently from the latter, which has a grain size of several micrometers.

Figure 2 gives the X-ray diffraction patterns of all our samples. Most of the observed lines could be indexed with the orthorhombic cell [3], and the lattice constants of sample *d* are $a=3.826 \text{ \AA}$, $b=3.883 \text{ \AA}$, $c=11.710 \text{ \AA}$, respectively. All the samples have strongly preferred orientations. This might be caused by the unreleased stresses introduced by the pressing treatment.

From the X-ray diffraction patterns, we cannot see the difference between sample *a* and sample *b*. However, the SEM images indicate that the grains in sample *a* are smaller than that of sample *b*, and that they have no distinct boundaries. We can also see that the grain size of sample *c* and *d* is

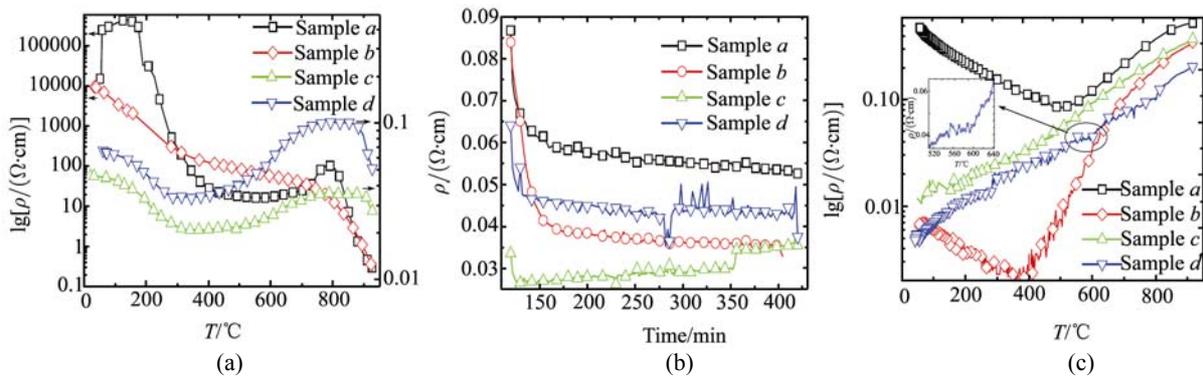


Fig. 1 (a), (b), and (c) are the H - T ρ - T curves of sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples in the heating, holding, cooling stage respectively. The samples, which have gone through the first round, the second round, the third round, and the fourth round of synthesizing, are referred to as sample *a*, *b*, *c*, and *d*, respectively. (a) The arrows refer to the vertical axes. (b) The resistivity of sample *a* is divided by four. The sudden jump in sample *c* might be caused by the lack of oxygen. (c) The resistivity of sample *a* and *d* is divided by four and two respectively. The inserted figure indicates that the O-T phase transition occurred between 550 °C and 600 °C.

more uniform than that of sample *b*, which agrees with the X-ray diffraction results.

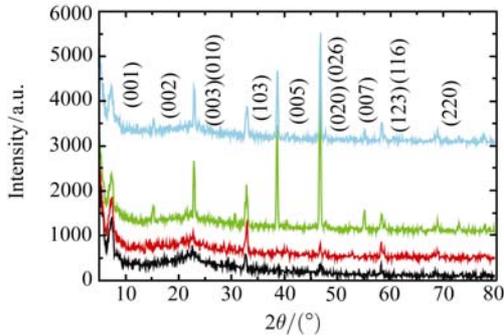


Fig. 2 The X-ray diffraction patterns of the sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ sample *a*, *b*, *c*, and *d*, respectively, from the bottom to the top. In sample *c* and *d*, not only the number of the peaks is larger, but also the intensities of all peaks are stronger than those of sample *a* and *b*. This indicates the gradual formation of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ phase.

From the SEM images showed in Fig. 3, there are distinct grain boundaries in sample *b*, while the grains in sample *c* and *d* are a little melting. Will this affect the superconductivity of the samples? As we come to the low temperature R - T and M - T measurement results, we find it quite strange that sample *b* seems to be a better superconductor than sample *c* and *d*. We will discuss this phenomenon later. Moreover, if we pay more attention to the SEM images in Fig. 3(b), (c) and (d), screw dislocations can be seen on their surfaces.

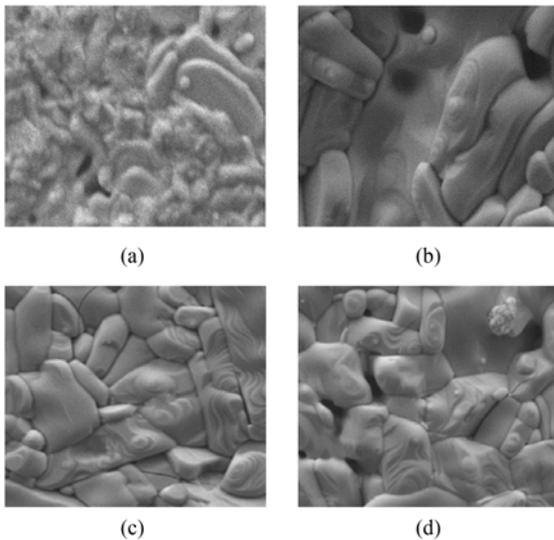


Fig. 3 The SEM images of the sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ sample *a*, *b*, *c*, and *d*. They are in the same magnification 3000 \times . The grain size of sample *a* is about 1 μm and the grain size of sample *b* ranges from several micrometers to more than 20 μm . The images show little difference in sample *c* and *d*. Both of them have grains several micrometers big.

From the results of low temperature R - T and M - T

measurements showed in Fig. 4 and Fig. 5, we can conclude that the sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ sample synthesized for 5 hours at 920 $^\circ\text{C}$ in flowing oxygen atmosphere is semi-conductive at room temperature, which can also find proof in the H - T ρ - T curve, and has no superconductivity. However, after being synthesized for 10 hours or more, the samples become superconductors with T_c higher than 91 K. Detailed superconducting transition temperatures are listed in Table 2. The width of transition of our samples is small ($\Delta T_c < 2$ K), which shows that our samples are well qualified.

Figure 4(a) and Fig. 5(a) show that the samples *b* and *c* have little difference in low temperature behaviors, except that sample *b* has higher resistivity at room temperature, which may have arose from the incomplete formation of the superconducting phase. The X-ray diffraction results also suggest that the amount of grains in the superconducting phase in sample *c* is much larger than that in sample *b*. From Fig. 4(b) and Fig. 5(b), we can see that sample *b* has a much higher transition temperature than that of sample *d*. We also note that sample *b* has a much better diamagnetic property.

From the above results, we can say that sample *b* has better superconductivity than both sample *c* and *d*. However, the H - T ρ - T curve suggest that sample *b* is still semiconductive at room temperature and that the X-ray diffraction pattern shows that the peaks of that typical orthorhombic cell, which are supposed to be correlated to the superconducting phase, are far too weak. This kind of phenomenon has never been reported before since only few measurements have been taken and little comparison has been made on samples which underwent different rounds of synthesizing. Ginley [11] once reported that in the material $\text{YBa}_2\text{Cu}_3\text{O}_7$, superconductivity occurs only in thin shells surrounding normal grains which are metallic. The SEM images suggest that some of the grains in sample *c* and *d* are melting. So, we may deduce that some of the superconducting shells in our sample *c* and *d* have been destroyed. More work needs to be done to show if this is a reason for the lowering of the superconducting transition temperature.

Figure 6 shows the screw dislocations on the broken surface of our sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ bulk sample. There are a number of screw dislocations of different sizes on the cross section of this sample, and many white dots, which are the grains of the Y_2BaCuO_y (YBCO-211) phase, can be seen around the screw dislocations. Some green materials mixed with the YBCO-123 phase or grown on the YBCO-123 bulk sample were observed after the first round of sample sintering. This is the evidence of the existence of YBCO-211 phase. However, some grains of the YBCO-211 phase have been separated out of the YBCO-123 during the sintering

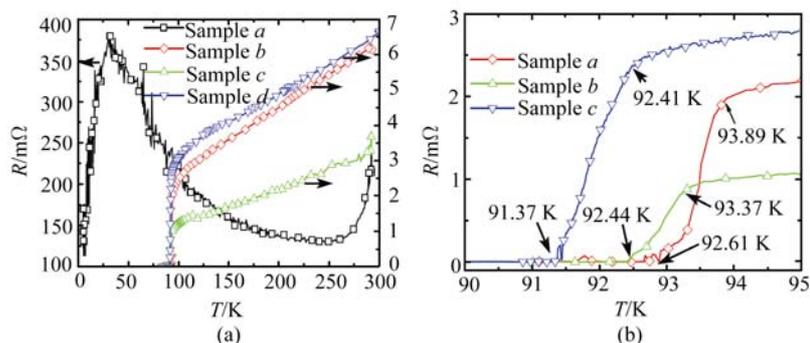


Fig. 4 The low temperature R - T curves of sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples. (a) shows the low temperature resistance of all the samples. The arrows refer to the vertical axes. Obviously, sample a is not superconductive. (b) shows behaviors of the superconducting samples near the superconducting transition.

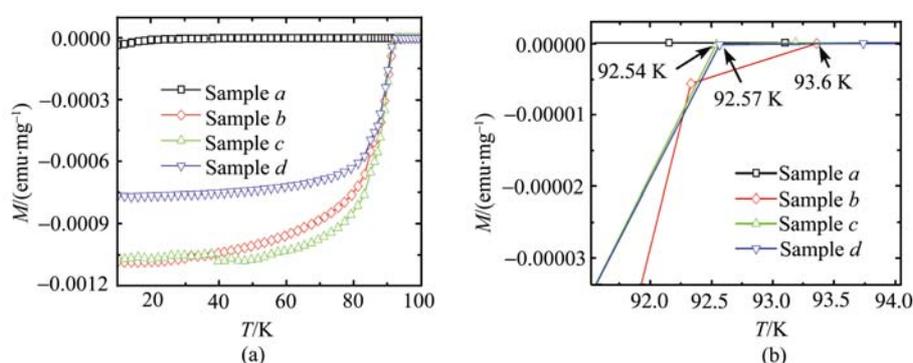


Fig. 5 The low temperature M - T curves of sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples. Samples were first cooled in zero field and then the magnetization of these samples while temperature increased were measured in a field $H=50$ Oe. (b) shows the properties of the superconducting samples near the superconducting transition. There is a small packet between the temperature 80 K and 90 K. This could be explained by the fact that our samples are not good single phase, with an additional superconducting phase $T_c\sim 85$ K.

Table 2 The superconducting phase transition temperatures of all the sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples.

	Sample a	Sample b	Sample c	Sample d
T_c^{onset}/K	No	93.89	93.37	92.41
T_c^{zero}/K	No	92.61	92.44	91.37
$\Delta T_c/K$	No	1.28	0.93	1.04

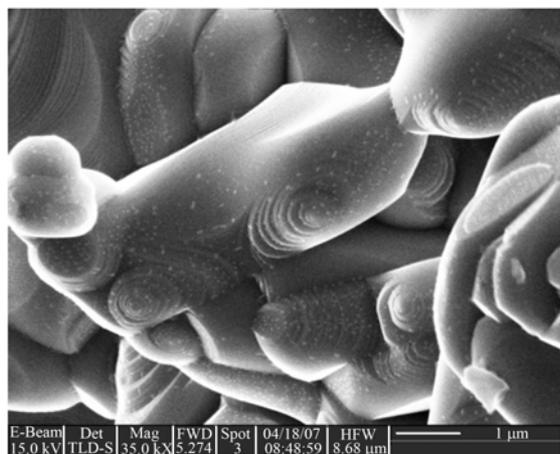


Fig. 6 The SEM images of the screw dislocations grown on the cross section of sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples. The magnification-times is thirty-five thousand-fold and there are 11 screw dislocations.

process. Moreover, it can be known that there are 11 screw dislocations on this sol-gel YBCO-123 surface. The density of screw dislocations of our sample can be calculated as $2.2\times 10^7/\text{cm}^2$.

It is well known that the screw dislocations can strongly affect the YBCO-123 thin films in different aspects: they are the strong vortex pinning sites potentially, which are directly related to the critical current density J_c values, and have a dramatic effect on the film growth [13–21]. Hawley *et al.* [17] pointed out that the significant difference in superconductivities, such as critical current, between YBCO-123 thin films and its bulk materials is due to their different microstructures. Therefore, a lot of research about screw dislocations on YBCO-123 thin films has been carried out, and many papers on this topic have been published [13–26]. However, only papers discussing the screw dislocations

present on the surfaces of YBCO-123 thin films and single crystals can be found. No one has reported screw dislocations present in the bulk YBCO-123 samples. It is generally believed that screw dislocations usually are presented in the layer structure materials. This report about screw dislocations appearing in the bulk YBCO-1123 sample is a first one. We will be discussing the mechanism of the screw dislocations in sol-gel YBCO bulk sample in another paper.

4 Conclusions

We have obtained HT ρ - T curves of the sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples synthesized in flowing oxygen atmosphere. They give typical behaviors of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples while synthesizing. They have T_V and T_P in the heating stage, and T_{ms} gradually reduces, as the rounds of synthesizing increases, and then disappears eventually. Our sample has an O-T phase transformation temperature around 600 °C, which is lower than 690 °C [8] and 613 °C [9], this can be caused by the nanometer particle size of the raw material used and the inadequateness of oxygen during the synthesizing process. The results of the X-ray diffraction, the SEM measurement, the low temperature R - T and M - T measurement suggest that our samples are well qualified. However, there seems to be some state during the preparation at which the samples are better superconductors than the final sample. This phenomenon has never been reported before. We believe that revealing the cause of this phenomenon can help synthesize better superconductors in the compound $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$. Moreover, our group is the first to find that the bulk sol-gel $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples can have screw dislocations on their cross sections. The density of screw dislocation in our sample is $2.2 \times 10^7 / \text{cm}^2$.

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