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Enhancement of Structure, T_c and Irreversibility Line in High T_c Superconductors by Heat Treatments

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ABSTRACT
AC susceptibility ($\chi_{ac}= \chi' + i\chi''$) and X ray diffraction (XRD) are very useful for characterizing high T_c superconductors. We report here on the preparation, X-ray diffraction with Rietveld refinement, resistivity ρ, AC magnetic susceptibility measurements and effect of heat treatments in $(Y_{1-x}Nd_x)SrBaCu_3O_{6+z}$. Each sample was subject to two types of heat treatment: oxygen annealing [O] and argon annealing followed by oxygen annealing [AO]. For each x, the [AO] heat treatment increases the orthorhombicity $\varepsilon = (b-a)/(b+a)$ (for $0 \le x < 1$), T_c (for x>0.2), the distance d[Cu(1)-(Sr/Ba)] (for x<0.25) and reduced the linear resistivity parameters. The irreversibility line, investigated by AC susceptibility measurements, fit well with the power law relation: $H=K'(1-T_p/T_c)^n$. Results show that the effect of annealing under argon flux leads to an enhancement of irreversibility line for low applied magnetic field which is justified by the improvement of crystallographic quality and the intergranular coupling of the samples heated under argon flux [AO]. Remarkable correlations were observed. A combination of several factors such as decrease in d[Cu(1)-(Sr/Ba)] for x>0.25; increase in cationic and chain oxygen ordering; p_{sh} and in-phase purity for the [AO] samples may account for the observed data.
***Keywords* -** Heat treatments control of T_c, irreversibility line, $(Y_{1-x}Nd_x)(SrBa)Cu_3O_{6+z}$, ceramic cuprites

I. INTRODUCTION

Since the discovery of high-temperature superconductivity in copper oxide compounds they are very attractive candidates for investigation due to their varying critical transition temperature (T_c) with oxygen content and strong flux pinning capability in high magnetic field and higher irreversibility line [1,2]. It is well-know that $YBa_2Cu_3O_{6.95}$ is superconducting below 92 K and characterized by double $Cu(2)O_2$ layers (oriented along the a-b plane) responsible for carrying the super-current and Cu(1)O chains (along the b direction) which provide a charge reservoir for these planes [3].

The effect of substitution on the structural and superconducting properties in $YBa_2Cu_3O_{6+z}$ has been extensively investigated. There are at least four distinct crystallographic sites which (excluding that of oxygen) Y, Ba, Cu plane, and Cu chain can be substituted with different elements. Concentrating on the Y site, it has been established that single-phase $LnBa_2Cu_3O_{6+z}$ (Ln= rare earth) can be prepared with the superconducting transition temperature T_c close to 92 K [4]. All these compounds show an orthorhombically distorted oxygen-deficient tripled-perovskite structure and both the orthorhombic distortion and T_c depend sensitively on the oxygen content (6+z) [4].

An extensive work on the preparation, the study of the structural and superconducting properties of $La_{1+x}Ba_{2-x}Cu_3O_y$ (with with $0 \le x \le 0.5$) was made per T. Wada et al [5], T. Izumi et al. [6]. These authors concluded that for obtaining good superconducting properties, we must obtain a triperovskite structure with an ordered arrangement of La and Ba along c axis and an occupation factor of oxygen at (1/2, 0, 0) and (0, 1/2, 0) close to 0 and 1, respectively.

Below T_c, the sharp decrease in the real part $\chi'(T)$ is a manifestation of diamagnetic shielding whereas the peak T_p in the imaginary part $\chi''(T)$ represents the A.C. losses.

It is interesting to check if an isovalent substitution of Ba by Sr would modify some of the results discussed above when Y is replaced by the rare earth Nd with bigger ionic radius. In order to study the role played by the Yttrium and Barium atomic plans and find out the factors conditions which govern the superconductivity in these compounds. We have investigated the effect of heat treatment on the structural magnetic and superconducting properties of $(Y_{1-x}Nd_x)SrBaCu_3O_{6+z}$ (x=0, 0.2, 0.4, 0.5, 0.6, 0.8, 1). Indeed we found that the influence of argon heat treatment on these properties depended on Nd content, x.

II. EXPERIMENTAL TECHNIQUES

The polycrystalline samples have been prepared by solid-state sintering of the respective oxides and carbonates. The chemicals were of 99.999% purity except in the case of $BaCO_3$ which was 99.99% pure. Y_2O_3, Nd_2O_3, $SrCO_3$, $BaCO_3$ and CuO were thoroughly mixed in required proportions and calcined at 950°C in air for a period of 12-18 h. The resulting product was ground, mixed, pelletized and heated in air at 980°C for a period of 16-24 h. This was repeated twice. The pellets were annealed in oxygen at 450°C for a period of 60-72 h and furnace cooled. This was denoted as sample [O] for each x.
X-ray diffraction (XRD) data of the samples were collected with a Philips diffractometer fitted with a secondary beam graphite monochromator and using CuKα (40 kV/20 mA) radiation. The angle 2θ was varied from 20° to 120° in steps of 0.025°, and the counting time per step was 10 sec. The XRD specters were refined with Rietveld refinement.

Superconducting transitions were checked by measuring both the real (χ') and the imaginary (χ") parts of the AC magnetic susceptibility as a function of temperature in a field of 0.11 Oe and at a frequency of 1500 Hz. In addition, χ' and χ" were measured in a static field ($0<H_{dc}<150$Oe) superimposed on the AC field H_{ac} =0.11Oe.

For each x, the same sample [O] was then heated in argon at 850°C for about 12h, cooled to 20°C and oxygen was allowed to flow instead of argon and the sample was annealed at 450°C for about 72h. This sample was denoted as [AO]. X-ray diffraction (XRD), Resistivity and AC susceptibility measurements were done on a part of this sample.

III. RESULTS

1. Real Part of the AC Magnetic Susceptibility and Crystalline Structure

We note that our XRD patterns of all the samples allowed the clear identification of the orthorhombic splitting (witch mean an increase of the orthorhombicity), as well as the observation that some weak unidentified impurity peaks are eliminated in the samples [AO]. This indicates an improvement of crystallographic quality of the samples [AO] [7].

Since the same sample was used for both heat treatments, one can compare the diamagnetic response and note that screening current of the [AO] sample increased considerably compared to that of the [O] sample for each x. Let us now look at the amplitude of the real part χ'(T) of the AC magnetic susceptibility in "Fig. 1 (a-b)"; of $NdSrBaCu_3O_{6+z}$ as a function of the temperature and heat treatment; at four fields H_{dc} ($0 < H_{dc} < 126.5$ Oe) which is nothing but the shielding effect S [8]. S represents the exclusion of magnetic flux by the sample in alternative dynamic mode. There was a remarkable improvement in the shielding effect in the case of the samples [AO] at all $T<T_c$ and for any applied field H_{dc} [9].

The effect of [AO] heat treatment on T_c was remarkable. The temperature at which the diamagnetism sets in is taken as T_c and it was found to be dependent on the heat treatment employed. As seen in "Fig. 3" when x increase from 0 to 1, T_c

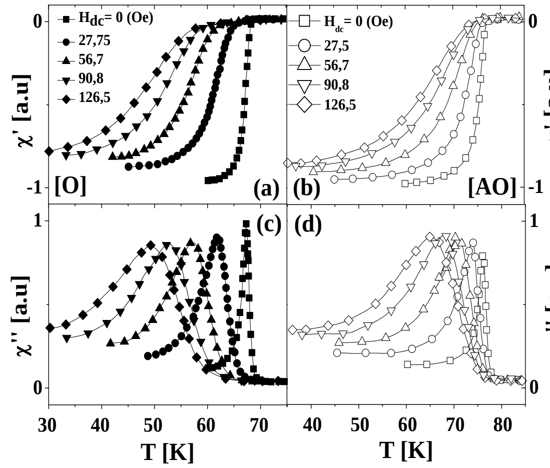


Fig. 1: "(a,b)" χ' and "(c,d)" χ" of $NdSrBaCu_3O_{6+z}$ as a function of the temperature and heat treatment at five fields H_{dc} ($0 < H_{dc} < 126.5$ Oe).

decrease and the orthorhombicity ε decreases to 0 with a transition from orthorhombic to tetragonal structure at x=1.

For each x, the [AO] heat treatment makes increase T_c (for x ≥ 0.25) and decreases it for x < 0.25 as seen in the upper of "Fig. 2". A maximum of increase in T_c of 10 K was observed for x = 1 [AO]. For each x, the [AO] heat treatment increased ε (for 0 ≤ x <1). The increase was maximum, from 2.23 10^{-3} to 5.81 10^{-3} for x=0.6.

The temperature transition in high-temperature superconductors depends strongly on the density p_{Sh} of holes by CuO_2 superconducting planes [10]. We deduced p_{Sh} from the under saturation zone of the universal relation T_c/T_c^{max} (p_{Sh}) [11]. Where T_c^{max} is taken as 92K for Y123 phase [12-15]. As seen in the table 1, the $YSrBaCu_3O_{6+z}$ for [O] and [AO] samples have the maximum hole carrier concentration value and (T_c^{max}[O]=83.06K with p_{Sh}=0.125) and (T_c^{max}[AO]=81,80K with p_{Sh}=0.123). The p_{sh} of the

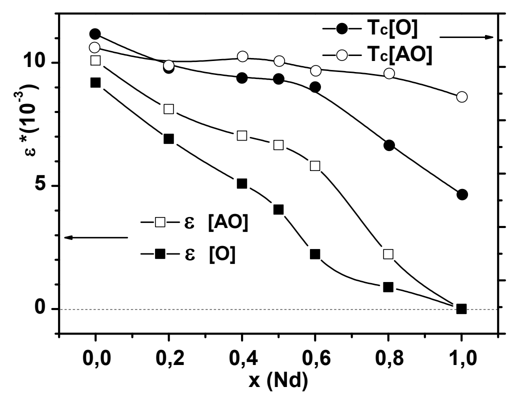


Fig. 2 Variation of the orthorhombicity ε “(a)” and T_c “(b)” as a function of x and heat treatments of $Y_{1-x}Nd_xSrBaCu_3O_{6+z}$.

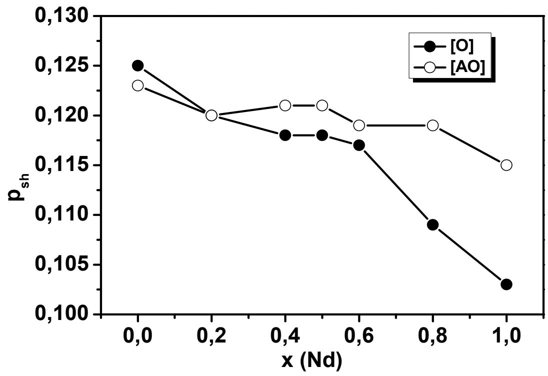


Fig. 3. The hole-carrier concentrations p_{Sh} as a function of x and heat treatment of $Y_{1-x}Nd_xSrBaCu_3O_{6+z}$.

samples studied here are illustrated in “Fig. 3”. There is a correlation between $p_{Sh}(x)$ and $T_c(x)$ in “fig 2”.

2. Imaginary Part of the AC Magnetic Susceptibility and Irreversibility Line

As example, the imaginary part χ″(T) of AC susceptibility for the sample $Nd(SrBa)Cu_3O_{6+z}$ as a function of heat treatment [O] and [AO] is shown in “Fig. 1 (c-d)”.

All samples have sharp superconducting transition width ΔTc measured between 10% and 90% of the height of the χ' signal. The changes observed in the imaginary part of ac susceptibility χ" are equally noteworthy. The full width at half maximum (FWHM) ΔT_p of the χ" transitions was smaller in the case of the sample [AO] and the peak T_p shifted less compared to that in the case of the sample [O]. An enhancement of the irreversibility line was observed due to argon treatment, when H_{dc} is plotted as a function of $t=T_p/T_c$ in “Fig. 4”. The data can be analyzed with the help of the following relation

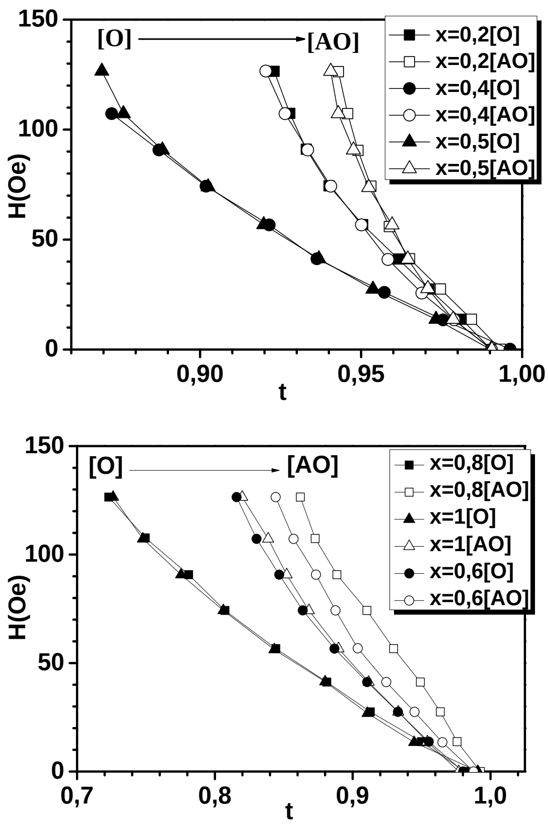
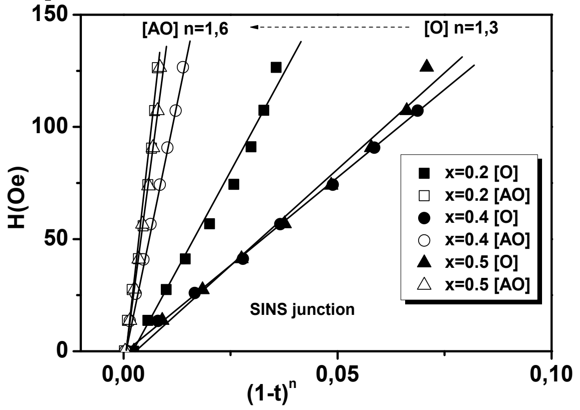


Fig. 4: H_{dc} as a function of $t=T_p/T_c$ and heat treatment of $Y_{1-x}Nd_xSrBaCu_3O_{6+z}$

$H=K'(1-t)^n$ [16]. When ln(H) was plotted against ln(1-t), straight line plots were obtained. We note that the argon treatment considerably increases the value of K’ and n in “Fig. 7” indicating an improvement in the pinning properties. K' may be interpreted as the field necessary to reduce the intergranular critical current to zero in the limit of T_p= 0 K. For example, the value of K’ was estimated to be 698.41 Oe and 1757.02 Oe respectively for the samples [O] and [AO] in $NdSrBaCu_3O_{6+z}$ (x=1). For x= 0.5 the [AO] heat treatment increased considerably K’[O]= 1.916 KOe to K’[AO]= 25.936 KOe.

“Fig. 4” can be considered as a graph of H_{dc} as a function of a temperature. As showed, our data is in good agreement with H_{dc} dependence of temperature as $H_{dc}\sim(1 - t)^{1.6}$ for sample [AO] and $H_{dc}\sim(1 - t)^{1.3}$ for sample [O] ($t=T_p/T_c$) “Fig. 5”. It is known that the polycrystalline superconductors are arrays of weak links, which behave like Josephson junctions. The temperature dependence of the field that flows through the Josephson junctions was calculated. This dependence can be written in the form $H_{dc}\sim(1-t)^n$. The value of n is 2 for superconductor–normal metal–superconductor (SNS) junctions [17], and 1 for superconductor–insulator–superconductor (SIS) junctions [18]. The values of n between 1 and 2 indicate the formation of superconductor–insulator–normal metal–superconductor (SINS) junctions.

As mentioned above for the sample [O] and [AO], the temperature dependence of $H_{dc}(T)$ is in good agreement with equation $H_{dc}(T)\sim(1-t)^{1.3<n<2}$ corresponding to weak links of SINS type “Fig. 5”. “Table 1” show the exact measured values of the superconducting and magnetic parameters of each sample as a function of the heat treatment.



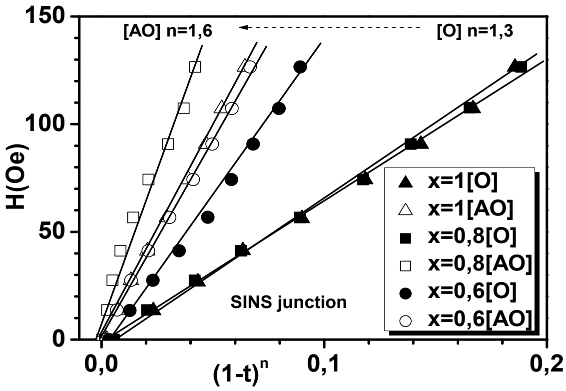


Fig. 5: H_{dc} as a function of T_p(K), x and heat treatment of $Y_{1-x}Nd_xSrBaCu_3O_{6+z}$

3. Resistivity

The superconducting critical temperature for our samples was also cheeked from resistivity measurement. For example the data points of the resistivity ρ for the sample $NdSrBaCu_3O_{6+z}$ [AO] lie clearly below those for the sample [O] in “Fig 6 (a)”. The values of T_c (ρ=0) are in good agreement with those of T_c (χ’). Note that for a given heat treatment T_c (χ’) is superior to T_c (ρ=0) by 2 to 3 K with T_p (χ″)≈ T_c(ρ=0). In the normal state, the linear part of ρ(T) obeys the relationship $\rho=\rho_0+\alpha T$, where ρ_0 is the residual resistivity extrapolated to T= 0 K and α is the slope dρ/dT. For example $NdSrBaCu_3O_{6+z}$ [O] has α =3.7 (μΩcm/K), ρ_0 =965 (μΩcm). The treatment [AO] reduced considerably these parameters; in particular α[AO] = 1.03 (μΩcm/K), ρ_0 =300 (μΩcm). This indicates a reduction of the interaction of carrier charges with phonons.

TABLE (1) Superconducting and Magnetic Parameters of $Y_{1-x}Nd_xSrBaCu_3O_{6+z}$.

x	0		0.2		0.4		0.5		0.6		0.8		1	
h Treat	[O]	[AO]	[O]	[AO]	[O]	[AO]	[O]	[AO]	[O]	[AO]	[O]	[AO]	[O]	[AO]
T_c (K)	83.06	81.80	79.87	80.12	78.95	80.96	78.86	80.54	78.10	79.62	72.64	79.36	68.02	77.18
T_p (K)	81	77.8	79.7	79.6	78.4	80.5	78.6	80	76	74.5	71.7	78.5	67.3	75.5
ε (10^{-3})	9.19	10.09	6.91	8.12	5.09	7.04	4.04	6.66	2.23	5.81	0.89	2.23	0	0
K’(Oe)	--	--	5206.69	15537.93	956.73	3013.44	1916.48	25936.07	1461.03	1638.57	649.12	1400.66	698.41	1757.02
n	--	--	1.47	1.71	1.09	1.29	1.36	1.90	1.35	1.53	1.29	1.21	1.34	1.53
p_{sh}	0.125	0.123	0.120	0.120	0.118	0.121	0.118	0.121	0.117	0.119	0.109	0.119	0.103	0.115

It is known that the high-Tc granular superconductors having a well defined superconducting transition temperature, generally display a two step resistive transition ρ(T) and correspondingly the derivative of the ρ(T) displays a peak and a tail in the lower temperature side [19]. The peak temperature marks the superconducting transition within the grains and the tail is related to the intergranular coupling.

“Fig. 6(a)” shows the resistive transition behavior of the samples [O] and [AO]. All the samples show metallic behavior in the normal state and a superconducting transition to zero resistance. It can also be seen from these curves that, the normal state resistivity regularly increases from sample [AO] to [O]. More information is gained by plotting the derivative of the resistivity curves of the samples [O] and [AO], as shown in “Fig. 6 (b)”. The transition temperature T_c taken as the maximum of the derivative of the ρ(T) curves, is almost the same for all samples [O] and [AO] (see Table 2). All samples show almost a single peak indicating a single superconducting transition in this sample.

Table (2): Results of resistivity and susceptibility measurements of $NdBaSrCu_3O_{6+z}$ for samples [O] and [AO].

Sample	T_c(ρ)(K)	T_c(χ’)(K)	T_p(χ”)(K)
[O]	67.32	68.02	67.3
[AO]	80.70	77.18	75.5

Thus we believe that the feature of the resistivity curves show that the no superconducting phase exist within the grain boundaries and plays a role of weak links and consequently reduces the intergranular coupling.

IV. DISCUSSION

In the normal state, the heat treatment [AO] reduced considerably the linear resistivity parameters indicating a diminution of the interaction of carrier charges with phonons. $T_c(\chi')$ and $T_c(\rho=0)$ were in good agreement for all the samples [9].

A magnetic field harmonically varying in time (to probe the sample) and the lock-in technique (to register the sample's induced magnetic response sensed by a pick-up coil) are often used to study the electromagnetic properties of superconductors. Measurement of the temperature dependence of the complex AC susceptibility is the most common experiment of this type. χ' reflect the shielding ability while χ" is measure of the magnetic irreversibility. Below T_c, the sharp decrease in the real part χ'(T) is a manifestation of diamagnetic shielding whereas the peak T_p in the imaginary part χ"(T) represents the A.C. losses.

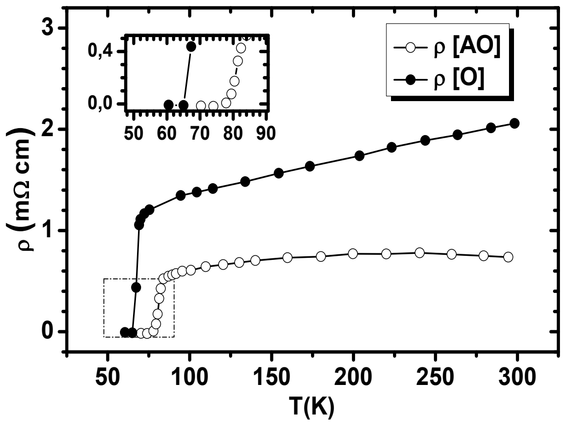


Fig 6 (a): Variation of the resistivity ρ(T) of $NdBaSrCu_3O_{6+z}$ as a function of the temperature and heat treatment.

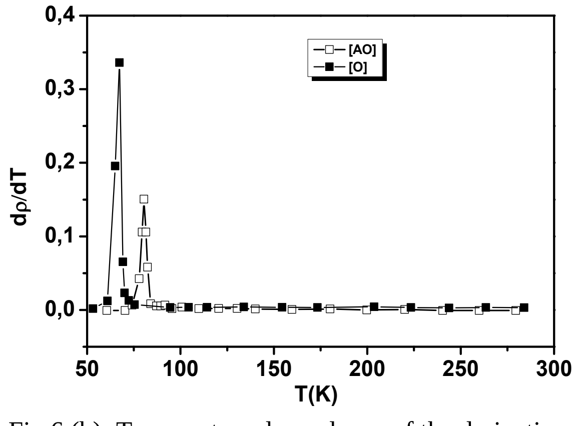


Fig 6 (b): Temperature dependence of the derivative of the resistivity of $NdBaSrCu_3O_{6+z}$ for samples [O] and [AO]

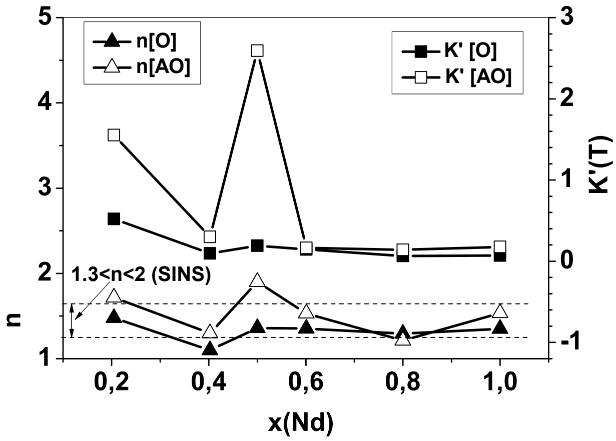


Fig 7: The field K' and the exponent n as a function of x and heat treatment of $Y_{1-x}Nd_xSrBaCu_3O_{6+z}$.

Ours measurements of H_{dc} versus $t=T_p/T_c$ give the curve $H_{dc}(t)$ called irreversibility line (IL), t decreases as H_{dc} increases according to the relationship: $H_{dc}=K'(1-t)^n$ [16].

There was a remarkable enhancement of the irreversibility line due to the argon treatment for all x. The reversible area is very wide to [O] and decline for [AO] "Fig. 4".

Here the exponent n ranges between 1.3 and 2 in "Fig. 7". Furthermore our polycrystalline samples can be modeled as superconductor—insulator—superconductor (SIS) junction consisting of superconducting grains separated by insulating grain boundaries. Any improvement in the intragranular properties will lead to the improvement in the overlapping of the superconducting wave functions across the SIS junction, which results in the improvement of the intergranular J_c as observed in the case of the [AO] samples when the reversible area decreases. A detailed mathematical description of the SIS junction and the intragranular phenomenon affecting the intergranular J_c in a polycrystalline Er-123 HTSC system is provided in [20].

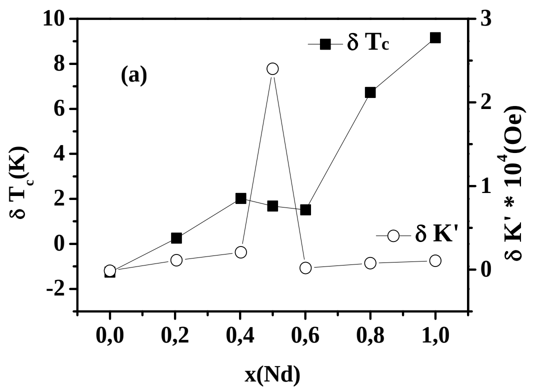
We note that the argon treatment considerably increases the value of K', in "Fig. 7", indicating an improvement in the pinning properties. These results are justified by our XRD spectra, with Rietveld refinement, that showed an improvement of crystallographic quality of the samples [AO] [8]. That means an enhancement of the quality of the grains and intergrain contacts and coupling by Josephson junctions. This argument is justified by the fact that the difference between inter- and intragrain currents vanishes and the two steps in χ'(T) (and peaks in χ"(T)) merge [21] as in our case in "Fig. 1".

When x increase from 0 to 1, ε decreases to 0 with transition from orthorhombic to tetragonal structure. ε[O] decreases with T_c[O]. However, T_c[AO] decreases with ε[AO] until x=0.2, increases for x=0.4 and after it decreases by 10 K to 77.2 K for x=1 [AO] in “Fig 3”. The crystalline parameter b is constant but a (and c) increase [8] indicating an increase of the number of oxygen atoms by chain (NOC) along a axis leading to a decrease of $\varepsilon(T_c$ [O]) from orthorhombic toward tetragonal structure for x=1 [O].

For each x, the [AO] heat treatment increases the orthorhombicity $\varepsilon= (b-a)/(b+a)$ (for $0 \le x < 1$), T_c (for x>0.2 and by 10 K for x=1) and the distance d[Cu(1)-(Sr/Ba)] (decrease T_c) for x<0.25 and decrease it (increase T_c) for x> 0.25. This enhance the transfer of the holes from chains along b axis to the superconducting plans Cu (2)-O2 via the apical oxygen O(1) along c axis and T_c. Here, the [AO] heat treatment makes decreases a and increases b. This increases the (NOC) along b axis (decrease the cationic disorder, of Y/Nd on the Ba/Sr site, along c axis and increase the anionic order in the basal plane) leading to an increase of the number $p_{sh}(x)$ of holes by Cu(2)-O_2 superconducting planes and T_c. The decrease of d[Cu(1)-(Sr/Ba)] distance for x>0.25 in [7] can be seen as an argument in the increase of the formation of the Cooper pairs and the increase in p_{sh} and T_c.

At low temperatures in the vortex state, pinning of the flux lines may be very effective; it weakens when approaching the critical temperature, where depinning may lead to dissipation. For each x, the [AO] treatment increase the number of vacant sites O(5), along a axis in the basal planes, which act like pinning centers of the vortex and increase the area under the irreversibility line in “Fig 4”.

The two arguments (cationic and anionic disorders) suggests that the superconducting coupling (i.e. the Josephson coupling) between the grains has been improved by argon heat treatment. They are justified



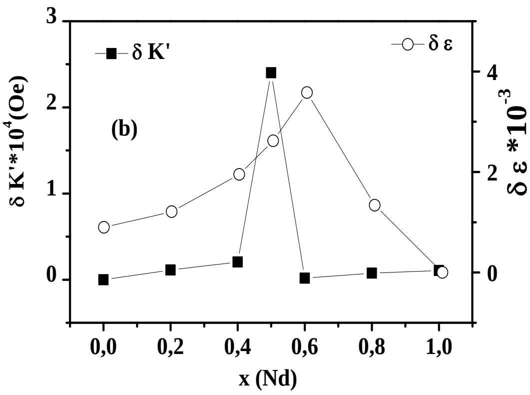


Fig. 8: Correlation between (a) $\delta T_c = T_c[AO] - T_c[O]$ and δK’ , (b) δε and δK’ as a function of x and heat treatment of $Y_{1-x}Nd_xSrBaCu_3O_{6+z}$.

here by the five remarkable correlations observed between $T_c(x)$ and the number of holes $p_{sh}(x)$, between $\delta T_c(x) = T_c[AO]-T_c[O]$ and $\delta\varepsilon(x) = \varepsilon[AO] - \varepsilon[O]$ in [7], between δK’(x) and $\delta T_c(x)$ in “Fig. 8 (a)” andbetween δK’(x) and δε(x) in “Fig 8 (b)” and on the other hand, between $T_c(x)$ and the volume of the unit cell V(x) in [7]. The later show a rearrangement of the unit cell volume witch control T_c. So the structural and superconducting properties are correlated with the effect of argon heat treatment. Hence we are tempted to believe that the changes (increase or decrease) observed in T_c, need not be related only to the ionic size of the rare earth

Nd but rather to a combination of several factors such as changes in the d[Cu(1)-(Sr/Ba)] distance, cationic and oxygen disorders, hole density etc.

V. CONCLUSION

The present studies indicate a simple heat treatment procedure to optimize superconducting properties of the high T_c superconductor $(Y_{1-x}Nd_x)SrBaCu_3O_{6+z}$. In the samples [AO], the remarkable improvement in the critical current density J_c are explained by the improvement of the quality of the grains and intergranular coupling and the pinning properties, respectively, as a result of the improvement of crystallographic quality of these samples. These results are the outcome of interplay between cationic disorder along the c axis and oxygen disorder in basal plane. In the samples [O], we are in presence of a cationic disorder of Nd on (Sr/Ba) sites that induced an anionic disorder of oxygen's chains in basal plane. Whereas anionic order dominates in the samples [AO] in agreement with the previsions of [5-6].

A combination of several factors such as decrease in d[Cu(1)-(Sr/Ba)] for x> 0.2; increase in cationic and chain oxygen ordering; p_{sh} and in-phase purity for the [AO] samples may account for the observed data. These results complete those in [9].

We believe that the present data will be useful to test or improve certain theoretical models on electronic structure and the atomic disorder.

Acknowledgements

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