

DYNAMICS OF PHASE SEPARATION IN $\text{La}_2\text{CuO}_{4+\delta}$, PROBED BY MAGNETIC SUSCEPTIBILITY EXPERIMENTS

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Abstract. The dynamics of phase separation in excess-oxygen-doped $\text{La}_2\text{CuO}_{4+\delta}$ ($\delta \leq 0.04$) is investigated by magnetic susceptibility measurements with particular attention paid to the thermal history of the samples. In $\text{La}_2\text{CuO}_{4+\delta}$, phase separation forms the superconducting phase in two diffusion-controlled processes between 150 K and 180 K and between 200 K and 240 K. The processes are of significantly different efficiency and exhibit different activation energies and attempt frequencies. These results are compared with nuclear magnetic resonance data in order to relate the formation of the superconducting phase to the mobility of interstitial oxygen atoms.

Key words: superconductivity, magnetic susceptibility, phase separation, diffusion, annealing, percolation.

1. INTRODUCTION

High- T_c superconductivity appears close to the insulator-to-metal phase transition induced by the doping of nonmetallic parent compounds. The nature of this phase transition has been heatedly disputed and it is still not clear at present. What is particularly intriguing is the growing amount of the experimental evidence that doping-induced metallization, at least at small doping levels, starts as a spatially extremely inhomogeneous process in which phase separation plays the key role [1, 2].

A central problem in this matter concerns the nature of phase separation and the underlying microscopic mechanism: an electronic phase separation, in which the doping-induced quasi-particles (i.e. holes and the induced local disturbances of the antiferromagnetic arrangement of Cu momenta) agglomerate to form microscopic metallized clusters, regions or pathways which at low temperatures become superconducting, has to be closely connected to the properties of the host and of the doped counterions, e.g. their mobility. High mobility of counterions, on the other hand, opens a possibility of a chemical or macroscopic phase separation. The macroscopic phase separation can be considered as sufficient for a local adjustment of the hole concentration to achieve an optimal hole level for the maximum T_c . However, it remains unclear which microscopic mechanism is the driving force for the enrichment and concentration adjustment of oxygen atoms in certain regions of the sample.

An interplay of the chemical and electronic phase separation may come about through the Coulomb attraction between holes and counterions, which, consequently, will very sensitively depend on the (local) dielectric properties of the background accommodating quasiparticles and counterions.

The most prominent high- T_c system which has stimulated a lot of research with respect to phase separation, is excess-oxygen-doped $\text{La}_2\text{CuO}_{4+\delta}$. It was the initial discovery of high- T_c superconductivity which revealed that the replacing of about 8% of the trivalent La atoms in La_2CuO_4 by divalent Ba (or Sr), and thus the doping of the Cu-O planes with holes, leads to superconducting compounds with a transition temperature of about 40 K [3]. Immediately after this observation it was shown that a high- T_c compound can also be obtained by excess oxygen doping (i.e. $\text{O}_4 \rightarrow \text{O}_{4+\delta}$, $\delta \approx 1\%$). Despite the low doping level, superconductors (with zero resistance below T_c , but only small fractions of the ideal diamagnetic shielding) with T_c close to 40 K are generated. The critical temperature is thus surprisingly similar to T_c of optimally Sr-doped La_2CuO_4 [4]. The finding that the critical temperature in a wide range is rather insensitive to the doping level suggested an explanation in terms of a chemical phase separation into two macroscopic phases, namely an optimally doped 40 K superconducting phase and an antiferromagnetic phase with vanishing doping.

A macroscopic phase separation has been confirmed by neutron and synchrotron diffraction experiments [5]. Besides the diffraction pattern of the undoped La_2CuO_4 , the latter revealed additional Bragg reflections which could be ascribed to the oxygen-enriched phase that was found to occupy a considerable fraction of the sample volume. The diffraction experiments have enabled a crystal structure refinement and located the excess oxygen atoms on positions within the La_2O_2 block of the crystal structure [6].

The macroscopic phase separation demands for an unusually high mobility of interstitial oxygen atoms below room temperature, which is

of some principal interest by itself. According to neutron diffraction experiments, excess oxygen atoms are weakly bound to an apex oxygen atom. We have suggested that high mobility of excess oxygen atoms is due to bond breakage and reformation rather than the diffusion of O^- species [7].

Recent very enlightening ^{139}La NMR and NQR relaxation measurements have been made to investigate oxygen diffusion in $\text{La}_2\text{CuO}_{4+\delta}$ [8]. Oxygen diffusion in $\text{La}_2\text{CuO}_{4+\delta}$ has turned out to be an activated process which sizeably contributes to the ^{139}La spin-lattice relaxation rate above 200 K. Assuming a two-dimensional diffusion of excess oxygen atoms, the activation energy of the diffusion constant determined from these experiments amounts to $5000 \text{ K} \cdot k_B$ (0.43 eV) and an attempt frequency of the oxygen diffusion, to $1.7 \cdot 10^{14} \text{ s}^{-1}$. No noticeable contribution from oxygen diffusion to the relaxation rate below 200 K could be detected.

Besides the appearance of superconductivity, another phenomenon of great interest, namely the coexistence of antiferromagnetism and superconductivity over a wide range of excess oxygen doping, can be observed in $\text{La}_2\text{CuO}_{4+\delta}$ samples. In contrast to this finding, antiferromagnetic ordering of Cu momenta is very effectively suppressed when La_2CuO_4 is doped with Sr and disappears already at Sr doping levels of 2% [9].

In an early investigation we have found that the formation of the superconducting phase in 'as-prepared' $\text{La}_2\text{CuO}_{4+\delta}$ can very effectively be decreased or even completely suppressed by quenching the samples from room temperature to temperatures below about 200 K (Fig. 1) [10]. Conductivity measurements on quenched samples have proved a reduction of T_c by about 10 K, but still zero resistance at low temperatures. At very small excess oxygen doping levels the low-temperature resistance of quenched samples of $\text{La}_2\text{CuO}_{4+\delta}$, however, even remains finite, while the samples cooled very slowly from room temperature exhibit zero resistance below a T_c of about 40 K [2].

Several results of the first applications of the quenching technique to susceptibility and resistivity experiments have been summarized in a series of papers [2, 7, 10, 11]. It has become clear that quenching experiments provide a handy tool to very efficiently manipulate phase separation in $\text{La}_2\text{CuO}_{4+\delta}$ and by this the formation of the superconducting phase.

In this contribution, this tool is employed to study the dynamics of the formation of the superconducting phase as a function of various annealing procedures carried through on quenched samples of $\text{La}_2\text{CuO}_{4+\delta}$.

The results of our experiments allow the establishing of the relevant correlation times and activation energies for the phase formation and give evidence for a strong coupling of the electronic phase separation to the mobility of excess oxygen counterions.

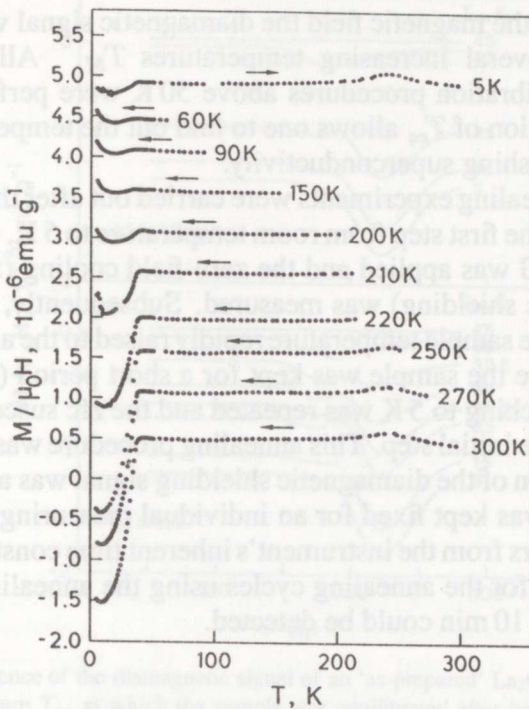


Fig. 1. Magnetization of an 'as-prepared' $\text{La}_2\text{CuO}_{4+\delta}$ sample as a function of temperature. The starting temperatures T_s to which the sample was quenched from room temperature are indicated. The uppermost curve was obtained after quenching the sample to 5 K and slowly heating it to room temperature. Beginning from the lowest data set each curve was shifted upwards by a value of $5 \times 10^{-7} \text{ emu g}^{-1}$ compared to the preceding one. The measuring field was 90 G, the arrows indicate the direction of the temperature change during the measurement.

2. EXPERIMENTAL

Ceramics and samples of $\text{La}_2\text{CuO}_{4+\delta}$ prepared as described previously [7] were doped with excess oxygen by slowly cooling them in the air after the final sintering process ('as-prepared' samples) or they were loaded with larger amounts of excess oxygen by annealing them at about 600°C in a high-pressure oxygen furnace. Although we did not determine the precise excess oxygen doping level of the samples studied, a comparison with a more recent series of similar doping experiments indicates that the samples were in the *low doping regime* with $\delta \approx 0.01$ ('as-prepared' samples) and $\delta \leq 0.04$ for the samples loaded in the high-pressure furnace.

Magnetic susceptibilities ($B = 100 \text{ G}$) were acquired with a S.H.E. VTS susceptometer or a MPMS Quantum Design magnetometer. To study the influence of thermal treatments on phase separation, two different experimental procedures were carried out with special attention paid to the thermal history of the samples.

In the so-called equilibration experiments the samples were always quenched from room temperature to T_{eq} and kept there (equilibrated) for a specified time period Δt . Subsequently they were quenched again to 5 K,

and after applying the magnetic field the diamagnetic signal was measured successively at several increasing temperatures T_M . All temperature changes and equilibration procedures above 50 K were performed in the zero field. A variation of T_{eq} allows one to find out the temperature ranges relevant for establishing superconductivity.

Isochronal annealing experiments were carried out after the sample had been quenched in the first step from room temperature to 5 K, where a magnetic field of 100 G was applied and the zero-field cooling (zfc) susceptibility (diamagnetic shielding) was measured. Subsequently, the field was switched off and the sample temperature rapidly raised to the annealing temperature T_{an} , where the sample was kept for a short period (e.g. 3 min or 10 min). The quenching to 5 K was repeated and the zfc susceptibility was determined as in the initial step. This annealing procedure was repeated until the full relaxation of the diamagnetic shielding signal was achieved. The annealing period was kept fixed for an individual measuring cycle to rule out systematic errors from the instrument's inherent time constants. No significant difference for the annealing cycles using the annealing periods of $\Delta t = 3$ min or $\Delta t = 10$ min could be detected.

3. RESULTS AND DISCUSSION

We first discuss the systematic equilibration experiments displayed in Fig. 2. Between two quenchings, from room temperature to T_{eq} and from T_{eq} to 5 K, the samples are allowed to relax for a defined period (Δt) and to build up a phase the superconducting properties of which are finally probed at low temperatures (T_M). To start each measuring cycle from the same conditions, after having measured the magnetization at several T_M 's, the samples are annealed to room temperature. Consequently, the samples lose all memory of the preceding thermal treatment.

As the most interesting result the equilibration experiments allow the distinguishing of two relevant temperature regions for the formation of the superconducting phase: already at temperatures above $T \geq 150$ K the superconducting phase starts to form and becomes rather pronounced above $T \geq 180$ K, as marked by the strong increase in the diamagnetic response, particularly for the latter process. These features in the equilibration and annealing curves are paralleled by resistivity anomalies observed at the same temperatures [11, 12]. We note a small shift of the minima at about 220 K when increasing the equilibration period from 10 min to 1 h, which becomes clearly visible when the magnetization difference for $\Delta t_2 = 1$ h and $\Delta t_1 = 10$ min is plotted (Fig. 2 for $T_M = 5$ K). A sharp cusp-like minimum at 220 K results which will be analysed quantitatively below. In the difference plot the feature between 150 K and 180 K has disappeared. This observation indicates that the time constant for establishing the superconducting phase in this temperature range is significantly reduced, compared to the time constant of the second process appearing above 180 K.

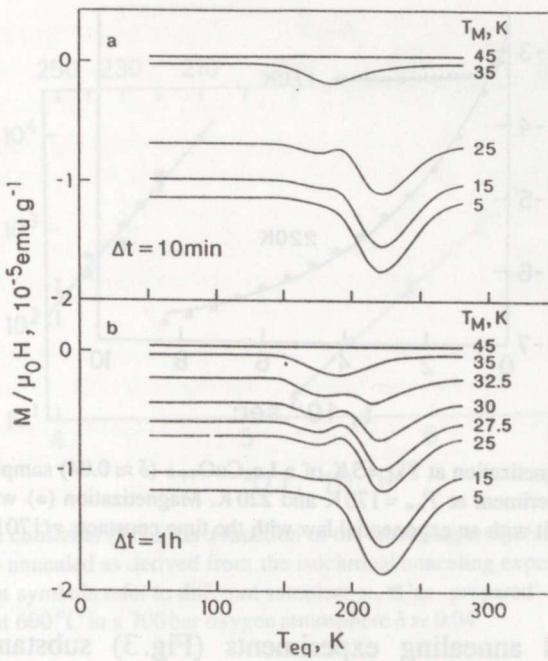


Fig. 2. Dependence of the diamagnetic signal of an 'as-prepared' $\text{La}_2\text{CuO}_{4+\delta}$ sample ($\delta \approx 0.01$) on the temperature T_{eq} at which the sample was equilibrated after having been quenched from room temperature to T_{eq} . The diamagnetic signal was probed at the indicated temperatures, T_M . Equilibration time intervals (i.e. the period for which the sample was held at T_{eq}), Δt , at T_{eq} were 10 min (a) and 1 h (b).

The data not only reveal the creation but also the destruction of the superconducting phase with increasing the temperature T_{eq} . The equilibrating of the samples at temperatures above 240 K increasingly impedes the formation of a superconducting phase. This observation has earlier been suggested to be due to a beginning oxygen motion [7], which has in fact been confirmed by recent ^{139}La nuclear relaxation experiments [8].

When comparing the diamagnetic signal obtained for the different measuring temperatures T_M (as indicated in the figures), it becomes obvious that the formation of the superconducting phase in the temperature range from 150 K to 180 K is responsible for superconductivity at high T_c 's, as can be seen e.g. from the data obtained at $T_M = 35$ K, showing the diamagnetic signal to be building up at $150 \text{ K} \leq T_{eq} \leq 180 \text{ K}$ ($T_c \geq 35$ K).

The subsystems with higher T_c 's are thermally less stable than those with lower T_c 's: some of the high- T_c subsystems start dissociating already at $T \approx 180$ K (cf. Fig. 2, $T_M = 25$ K), while low- T_c subsystems decay above $T \approx 220$ K (see Fig. 2, $T_M = 5$ K).

The annealing experiments involving a slightly varied thermal treatment of the samples reveal analogous features in the diamagnetic shielding signal and also establish two distinctly different temperature ranges as being relevant for the generation and the subsequent decay of superconductivity in the samples [7].

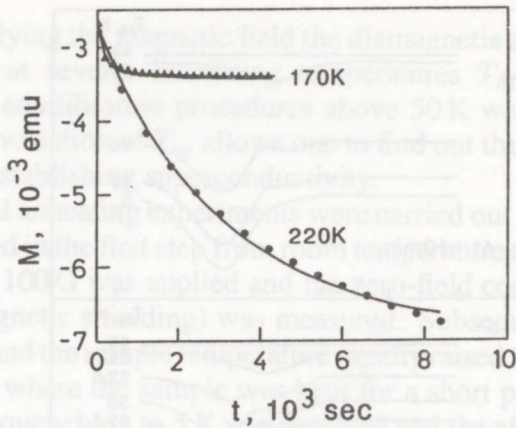


Fig. 3. Recovery of magnetization at $T_M = 5$ K of a $\text{La}_2\text{CuO}_{4+\delta}$ ($\delta \approx 0.04$) sample probed in an isochronal annealing experiment at $T_{an} = 170$ K and 220 K. Magnetization (\bullet) was measured at 100 G. The full line is a fit with an exponential law with the time constants $\tau(170 \text{ K}) = 3700$ s and $\tau(220 \text{ K}) = 180$ s.

The isochronal annealing experiments (Fig. 3) substantiate this suggestion of two different processes. The time evolution of the diamagnetic signal on annealing is significantly faster, however, with a much smaller amplitude, if the samples are annealed at 170 K, compared to 220 K. A fit with an exponential relaxation term ($\Delta t = 3$ min or 10 min),

$$M(t) - M(0) \propto \exp(-n\Delta t/\tau), \quad (n = 0, 1, 2, \dots), \quad (1)$$

results in the values for the time constant τ , which on annealing at 170 K are about an order of magnitude smaller than those observed at 220 K.

A quantitative analysis of a series of such isochronal annealing experiments using the exponential relaxation term (Eq. (1)), provides the temperature dependence of τ which is depicted for a series of different samples in Fig. 4. Within error bars the experimental data are sample-independent and fall on two straight lines in the Arrhenius representation, which points to an activated temperature dependence of the correlation time,

$$\tau_i(T) = \tau_{\infty i} \cdot \exp(Q_i/k_B T), \quad (2)$$

with the *activation energies* of the *low/high* temperature process being

$$Q_1 = 0.36(2) \text{ eV}, \quad Q_2 = 0.46(3) \text{ eV}$$

and the corresponding *attempt frequencies* ($\tau_{\infty i}^{-1}$),

$$\tau_{\infty 1} = 9(3) \cdot 10^{-9} \text{ s}, \quad \tau_{\infty 2} = 1.0(5) \cdot 10^{-7} \text{ s}.$$

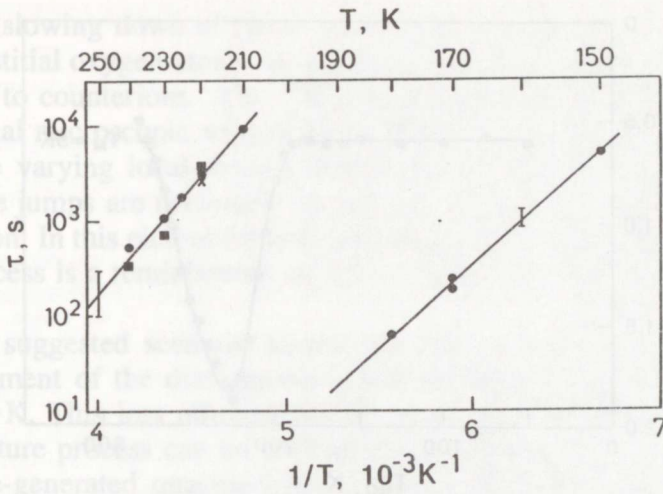


Fig. 4. The time constants $\tau(T_{an})$ as a function of the annealing temperature at which the samples were repeatedly annealed as derived from the isochronal annealing experiments (cf. examples in Fig. 3). Different symbols refer to different samples: \times , \blacksquare 'as - prepared' ($\delta \approx 0.01$); \bullet loaded with excess oxygen at 600°C in a 700 bar oxygen atmosphere $\delta \approx 0.04$.

The cusp-like minimum in the difference of the diamagnetic fractions obtained from the equilibration experiment for $\Delta t_2 = 1$ h and $\Delta t_1 = 10$ min is plotted for $T_m = 5$ K in Fig. 5 together with a theoretical fit according to

$$\Delta M(T_M = 5 \text{ K}, T_{eq}) \propto M(\Delta t_2, T_{eq}) - M(\Delta t_1, T_{eq}), \quad (3)$$

wherein

$$M(\Delta t_i, T_{eq}) \propto \exp(-\Delta t_i / \tau(T_{eq})) \quad (4)$$

and an activated behaviour for $\tau(T_{eq})$ (Eq. (2)) is assumed. A satisfying fit is obtained with an activation energy of 0.44 eV and an attempt frequency of $1/1.4 \cdot 10^{-7}$ s is in best agreement with the results of the isochronal annealing experiments.

The present results reveal clearly that the formation of the phase which carries superconductivity in $\text{La}_2\text{CuO}_{4+\delta}$ below 40 K takes place in two temperature ranges, between 150 K and 180 K, and between 200 K and 240 K. Both processes exhibit an activated behaviour, however with different activation energies and attempt frequencies.

The activation energy of the more efficient high-temperature process, measured with two independent experimental procedures, amounts to 0.45(2) eV and matches perfectly with the result of the ^{139}La nuclear relaxation measurements in this temperature region [8]. This coincidence suggests that the high-temperature formation process is closely related to oxygen diffusion. The activated temperature characteristics of phase formation is apparently determined by the activated oxygen diffusive motion.

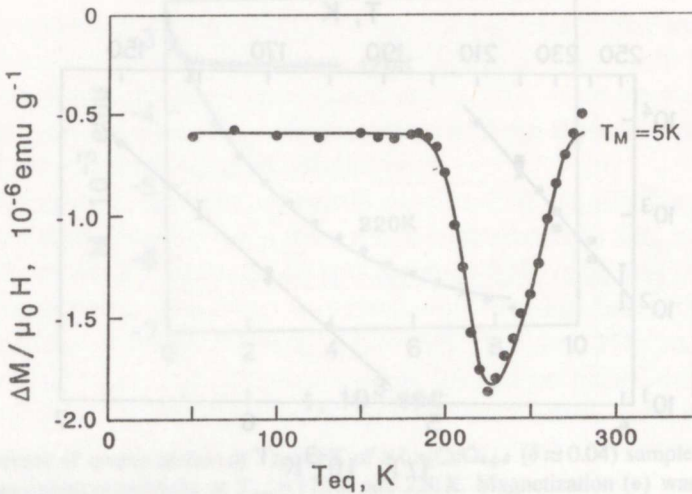


Fig. 5. The difference ΔM of the diamagnetic signals measured at $T_M = 5$ K after equilibration at T_{eq} for 1 h and 10 min, i.e. the difference of the lowermost curves of Fig. 2 ($M(\Delta t = 1 \text{ h} - \Delta t = 10 \text{ min}, T_{eq})$). The solid line is a fit with the difference of two exponentials as described in the text.

However, eye-catching is a marked, by almost seven orders of magnitude, difference in the attempt frequencies, which makes the formation of a phase becoming superconducting at low temperatures a significantly slower process than is oxygen diffusion itself. The process is slow enough for the thermal quenching with the rates of about 10 K s^{-1} to be sufficient to suppress the phase formation. We suggest that the slowing down of the phase separation process as compared to fast oxygen diffusion reflects the interaction of oxygen counterions and doped holes (and induced quasi-particles) in Cu-O planes, thus allowing conclusions about the dynamics of the electronic phase separation.

The results of our earlier experiments are largely in agreement with the predictions of the model of percolative phase separation as developed by Hizhnyakov, Sigmund, and coauthors [13]. This model predicts a hole-doping-induced generation of spin-polarized Cu-O clusters. The spin-polarized clusters, being themselves rather immobile, accommodate the doped holes. An electronic phase separation starts when at higher doping levels the number of spin-polarized aggregates increases and above the percolation threshold join to form a fractale network. In this network holes delocalize and build up metallized pathways that are embedded in an undoped antiferromagnetic matrix.

Phase separation at lower doping levels in this scheme also requires some mobility of spin-polarized clusters. Since holes are coupled to counterions by the Coulomb attraction, the mobility of holes (and spin-polarized clusters) is essentially confined to the vicinity of doping ions. The beginning diffusion of counterions, as evidenced by the ^{139}La nuclear relaxation measurements, may thus considerably promote long-range movements of holes and agglomeration of quasi-particles and by this support electronic phase separation.

The slowing down of phase formation, as compared to the diffusion of interstitial oxygen atoms, is ascribed to the coupling of spin-polarized clusters to counterions. The ^{139}La nuclear relaxation experiment senses individual microscopic oxygen atom diffusion jumps which dynamically generate varying local microscopic fields. A large number of oxygen diffusive jumps are necessary for oxygen migration to accomplish phase formation. In this picture the activated temperature dependence governing this process is a reminiscence of the activated individual oxygen diffusive jumps.

The suggested scenario allows also the understanding of the weak enhancement of the diamagnetic signal on equilibration between 150 K and 180 K. This less efficient but by an order of magnitude faster low-temperature process can be attributed to a very space-limited motion of the hole-generated quasiparticles. Since oxygen diffusion has ceased at these low temperatures, quasi-particle motion and phase growth and improvement can take place only in the close vicinity of the charge-compensating ions, thus leading but to a slight extension of the phase. A long-range motion of quasi-particles and holes is not possible any more at these low temperatures.

As can be seen from the experiments, very fast oxygen diffusion apparently counteracts the phase formation. The decrease of the diamagnetic signal above about $T_{eq} \approx 240$ K, observed in the annealing and equilibration experiments, is paralleled by a strong increase of the ^{139}La relaxation rate due to fast oxygen diffusion above this temperature.

4. SUMMARY AND CONCLUSIONS

In summary, by controlled annealing and equilibration experiments we have determined the activation energies and the relevant time constants of the diffusional reestablishment of the superconducting phase in quenched samples of excess-oxygen-doped $\text{La}_2\text{CuO}_{4+\delta}$. We have identified two temperature ranges where the superconducting phase is built up in the processes of very different efficiency and rate. From a comparison with nuclear relaxation experiments we conclude that the electronic phase separation is strongly promoted by the beginning mobility of excess oxygen atoms. Very fast oxygen diffusion above 240 K, on the other hand, generates a situation (disordered phase) which, when thermally quenched to temperatures below 100 K, exhibits but a small fraction of the superconducting phase.

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FAASIDE ERALDUMISE DÜNAAMIKA $\text{La}_2\text{CuO}_{4+\delta}$ -S MAGNETILISE VASTUVÕTLIKKUSE EKSPERIMENTIDE PÕHJAL

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On uuritud faaside eraldumise dünaamikat hapnikuga aktiveeritud $\text{La}_2\text{CuO}_{4+\delta}$ -s magnetilise vastuvõtlikkuse mõõtmiste abil sõltuvalt termilisest eeltötlusest. On näidatud, et $\text{La}_2\text{CuO}_{4+\delta}$ -s moodustub ülijuhtiv faas kahes difusiooniga reguleeritavas protsessis 150 K ja 180 K vahel ning 200 K ja 240 K vahel. Need protsessid on oluliselt erineva efektiivsusega ning neil on eri aktivatsioonienergiad ja sagedusfaktorid. Tulemusi on võrreldud magnetresonantsi andmetega eesmärgil seostada ülijuhtiva faasi formeerumist hapniku interstitsiaalide liikuvusega.

ДИНАМИКА РАЗДЕЛЕНИЯ ФАЗ В $\text{La}_2\text{CuO}_{4+\delta}$ ПО ЭКСПЕРИМЕНТАМ МАГНИТНОЙ ВОСПРИИМЧИВОСТИ

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Методом магнитной восприимчивости исследована динамика разделения фаз в активированных кислородом образцах $\text{La}_2\text{CuO}_{4+\delta}$ ($\delta < 0,04$) в зависимости от их термической предыстории. Показано, что сверхпроводящая фаза $\text{La}_2\text{CuO}_{4+\delta}$ создается при участии двух диффузионно-контролируемых процессов, происходящих соответственно между 150 К и 180 К и между 200 К и 240 К. Эти процессы имеют существенно разную эффективность и характеризуются разными активационными энергиями и частотными факторами. Полученные результаты сравниваются с данными по магнитному резонансу с целью соотнести формирование сверхпроводящей фазы и подвижность кислородных интерстициалов.