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# THE μSR INVESTIGATION OF MULTI-PHASE Bi-BASED SUPERCONDUCTORS

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The muon depolarization rate $\sigma$ was measured in the ceramic Bi-Sr-Ca-Cu-O samples with various contents of the 80 K and 110 K phases. The importance of justification of the relation between $T_c$ and $\sigma$ ($T \to 0$ K) /$\propto 1/\lambda^2$/ was emphasized in / 1 /.

The values of $\sigma$ ( obtained by a standard FC technique in the external fields of 0.04, 0.08 and 0.2 T and in the temperature region 10 ÷ 300 K ) were used for estimation / 2,3 / of the magnetic penetration depths perpendicular to the basal plane:

for the 80 K-phase:

$\lambda_{\perp}(T \to 0\ K) = 2300 \pm 100$ Å ( $\lambda_{eff}(T \to 0K) = 2830 \pm 130$ Å)

for the 110 K-phase:

$\lambda_{\perp}(T \to 0\ K) = 2030 \pm 100$ Å ( $\lambda_{eff}(T \to 0K) = 2500 \pm 130$ Å).

The Bi-Sr-Ca-Cu-O family of cuprate high-$T_c$ superconductors consists of three superconductive phases: the first phase with $T_c \sim 10$ K and with c - crystallographic length of 24 Å, the second one with $T_c \sim 80$ K, c ~ 30 Å and the third one with $T_c \sim 110$ K and with c ~ 37 Å. We have determined the phase contents of all three samples by $\mu$SR technique and have estimated the dispersion $\langle \Delta B^2 \rangle$ of the distribution of the local magnetic fields for 80 K- and 110 K-phases.

## The samples

Sample I - a ceramic Bi-Sr-Ca-Cu-O tablet was prepared by the modified citrate method: citric acid was added to the solution of Bi, Ca, Cu and Sr nitrides ( ratio 1:1:2:1 ). The pH-factor was adjusted to the value of 7 with the help of aqueous solution of $NH_3$. The obtained mixture was evaporated and calcinated at 830 °C, pressed into the tablet and sintered at 860 °C for 50 hours and at 450 °C for 24 hours at the pressure of 3 MPa.



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Sample II / Bi-Sr-Ca-Cu-O / contained 46 little sintered tablets, each 16 mm in diameter and 2.5 mm thick, which were prepared as follows : After two calcinations at 810 °C for 22 h and 15 h the material was sintered at 850°C for 21 h, fast cooled in liquid nitrogen, reground and pressed into tablets which were finally sintered several times ( at 850 °C ).

Sample III / Bi-(Pb)-Sr-Ca-Cu-O / was prepared by the standard powder metallurgy method in the form of a big ceramic tablet 40 mm in diameter and 10 mm thick.

The X-ray diffraction ( XRD ) analysis was used to determine the relative population of c ~ 24 Å, c ~ 30 Å, c ~ 37 Å phases in all samples. The elimination of texture correction was controlled by measurements in two planes of Sample I with the angle 90° and 0° to the texture axis.

This analysis reveals that 35 % of Sample I are the 80 K phase, 5.2 % in the bulk and 8 ÷ 16 % near the surface of tablet are the 10 K phase with c ~ 24 Å. The rest of Sample I consists of non-superconducting impurity phases CuO, $(CaSr)_3Cu_5O_8$ and CaSrCuO. No evidence ( < 3 % ) of 110 K-phase was detected.

XRD analysis of Sample II shows that the contents of 80 K and 110 K phases were 79 % and 21 % respectively with only a small amount of nonsuperconducting phases. The X-ray analysis of Sample III reveals that the part of 80 K-phase is less than 10 % of 110 K phase.

## Experimental results.

The experiment have been performed on the muon beam of the LNP JINR ( Dubna ) phasotron using the conventional transverse field μSR-spectrometer.

The time spectrum of decay positrons in the case of a sample with n phases can be described by the formula: (1)

$$N(t) = N_0 \exp(-t/\tau_\mu) \left[ 1 + \sum_{i=1}^{n} a_i \exp(-\sigma_i^2 t^2/2) \cos \omega_i (t+t_i) \right] + N_b ,$$

where $a_i$ is the initial asymmetry; $\omega_i$ is the mean muon precession frequency connected with the mean value of the



magnetic field in the phase i by relation $B_\mu = \omega_i \cdot \gamma_\mu$ ; the muon depolarization rate $\sigma_i$ is connected with the value of the dispersion $\langle \Delta B^2 \rangle$ of the local magnetic field distribution in phase i by relation $2\sigma^2$ ; $\gamma_\mu^2 \cdot \langle \Delta B^2 \rangle$, where $\gamma_\mu$ is the muon gyromagnetic ratio.

Our interpretation of the obtained μSR spectra was performed with the following assumptions:

- muons stopped in the sample can be divided into two groups - those stopped in the region, where $B_{loc} \geq B_{ext}$ and $\sigma_i \leq 0.2\ \mu s^{-1}$ in the non-superconducting (N ) region and those stopped in the superconducting phase ( SC ), where $B_{loc} \leq B_{ext}$ and $\sigma_i > 0.2\ \mu s^{-1}$;

- the muon spin relaxation due to the distribution of local magnetic fields in both these regions can be described by the Gaussian time dependence, so the Gaussian approximation was used as a law of the local field distribution.

The μSR spectra from Sample I at temperatures above 80 K and those from Samples II and III at temperatures above 110 K were described by one ( N ) Gaussian relaxation function, the spectra at temperatures $T < T_c$ were described by two ( N and SC ) Gaussian relaxation functions.

The mean value of local magnetic fields on muons in the N-region at temperatures $T > T_c$ was equal to the value of the external magnetic field $B_{ext}$. A small excess of $B_{loc}$ over $B_{ext}$ at temperatures below $T_c$ was caused by expulsion of magnetic field from the superconducting regions into the N-regions of the sample. In the vortex state of type-II superconductors the $B_{loc}$ value is smaller than the $B_{ext}$ value.

The obtained dispersion of the local field distribution in the N-regions at temperatures 110 ÷ 150 K was practically constant: $\sigma_N = 0.20\ \mu s^{-1}$ ). A large part of this value was probably caused by dipole-dipole interaction with Cu nuclei. The increase of the dispersion from $\sigma_N = 0.13\ \mu s^{-1}$ at 250 ÷ 300 K to $\sigma_N = 0.20\ \mu s^{-1}$ at 100 ÷ 150 K is connected with muon diffusion at higher temperatures. The expulsion of the magnetic field from the SC-regions into the N-regions causes the slight increase in $\sigma_N$ at temperatures below $T_c$ ( $\sigma_N = 0.11\ \mu s^{-1}$ at 65 K, $\sigma_N = 0.25\ \mu s^{-1}$ at 10 K and $\sigma_N = 0.16\ \mu s^{-1}$ at 90 K, $\sigma_N = 0.27\ \mu s^{-1}$ at 10 K in Samples I and III respectively ).

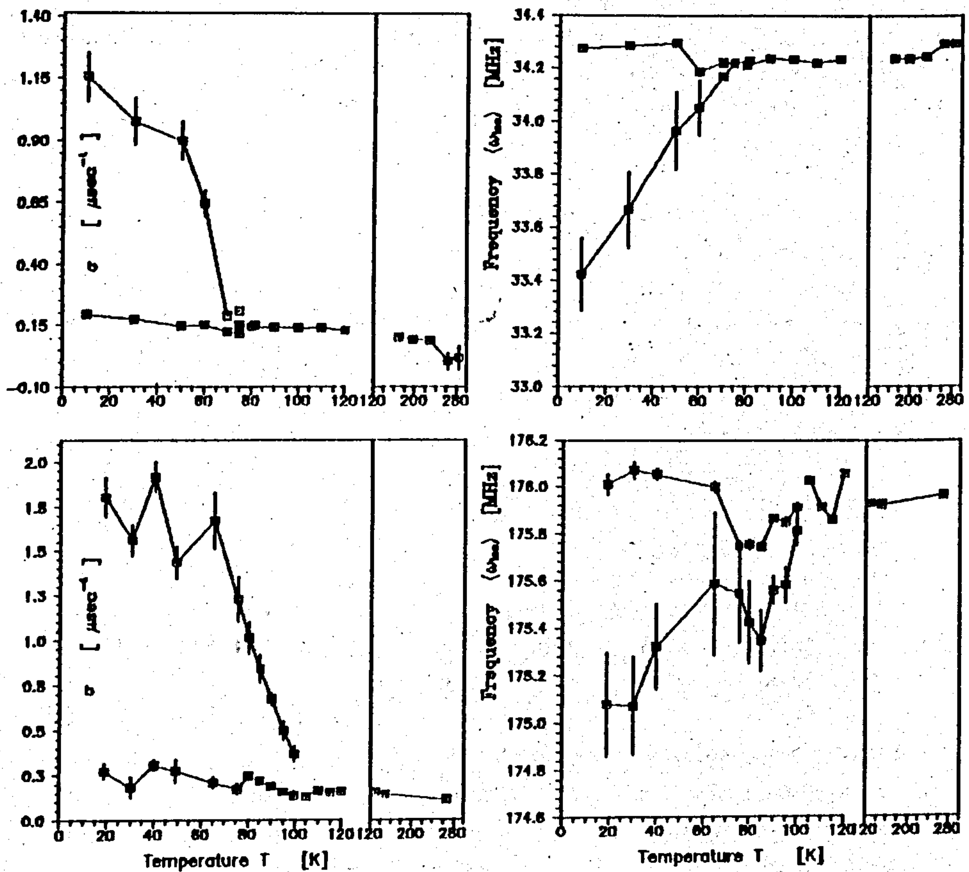


Fig. I. The temperature dependence of the muon precession frequences (right) and the temperature dependence of the depolarization rates σ (left) for Sample I (top) and Sample III (bottom). The lines are guide to the eye.

A significant part of the $\langle \Delta B^2 \rangle$ measured by μSR in the SC-region below $T_c$ is connected with the field distribution in the vortex lattice of type-II superconductors. The possible contributions to $\langle \Delta B^2 \rangle$ due to dipole-dipole interactions with Cu nuclei and due to different demagnetization factors of the grains [ 5 ] are of order of few per cent.



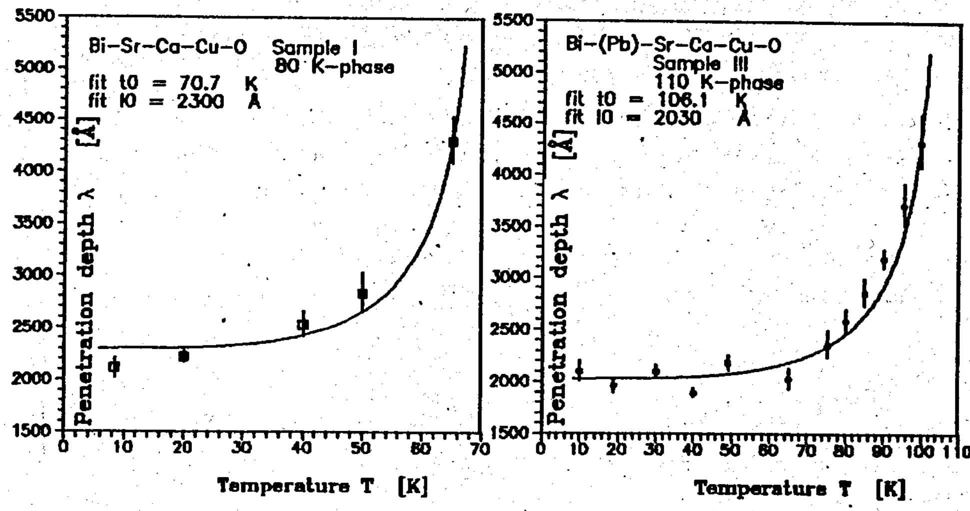


Fig. 2. The temperature dependence of the penetration depth obtained from Sample I and III muon spectra. The solid line is the fit by formula (3).

The cooling of the sample below $T_c$ leads to enhancement of inhomogeneity of the internal magnetic fields due to formation of the vortex state and hence to an increase in the depolarization rate $\sigma$ . Assuming the regular triagonal vortex lattice, one can connect the magnetic field dispersion $\langle \Delta B^2 \rangle$ with the penetration depth $\lambda_{eff}$ of type-II superconductor by the formula / 4 /:

$$\langle \Delta B^2 \rangle = 0.0037 \ \phi_0^2 \lambda_{eff}^{-4} , \qquad (2)$$

where $\phi_0$: 2 x $10^{-15}$ Wb is the magnetic flux quantum.

Formula ( 2) is valid only for the cases when the distance between vortices is less than $\lambda_{eff}$. If the ratio of the penetration depth tensor components $\lambda_{\parallel}/\lambda_{\perp}$ is greater than 5, then $\lambda_{eff}$ can be related / 3 / to the penetration depth component as $\lambda_{\perp} = \lambda_{eff}/1.23$.

The experimental temperature dependence of the penetration depth was described by the formula :

$$\lambda(T) = \lambda(0)\sqrt{1-(T/T_c)^4} \qquad (3)$$

in the temperature range 8 ÷ 75 K ( Sample I data ) and in the temperature range 10 ÷ 100 K ( Sample III data ). The results of the fitting procedure for both $\lambda(0)$ and $T_c$ parameters ( see Fig. 2 ) are as follows :

| | | | |
|---|---|---|---|
| 80 K phase | $T_c$ = 70.7 K | $\lambda_{eff}(0)$ = 2830 Å | $\lambda_{\perp}(0)$ = 2300 Å |
| 110 K phase | $T_c$ = 106.1 K | $\lambda_{eff}(0)$ = 2500 Å | $\lambda_{\perp}(0)$ = 2030 Å. |

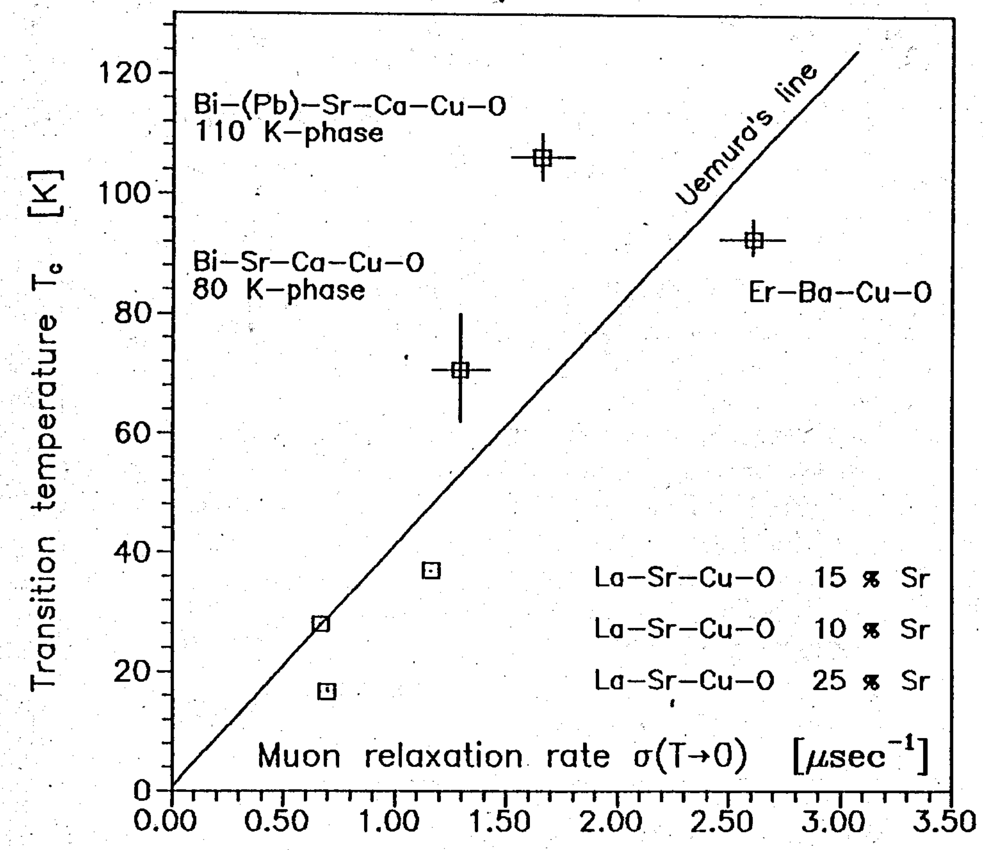


Fig. 3. The superconducting transition temperature $T_c$ plotted versus low temperature muon depolarization rate $\sigma$(T → 0 K) measured in six Dubna high-$T_c$ ceramic superconductors. The solid line is the linear relation between $T_c$ and $\sigma$(T → 0 K) from /I/.



Uemura's linear relation predicts $\lambda_{\perp}(0)$ = 1620 Å for the 110 K- phase. The fit of $\lambda_{\perp}(T)$ values taking from the temperature interval 80 K < T < 100 K yields $\lambda_{\perp}(0)$ = 2300 Å. The statistical error of the $\lambda_{\perp}(0)$ was 100 Å. The Sample II data in the temperature range 85 ÷ 110 K are consistent with the results given above.

Fig. 3. shows the depolarization rate values $\sigma$ (T → 0 K) for various high-$T_c$ superconductors obtained by the same manner from the Dubna μSR data. The monotonic increase in $\sigma$ (T → 0 K) when $T_c$ increases can be seen from Fig. 3 except for the case of 110 K phase Bi-(Pb)-Sr-Ca-Cu-O.

Now we don't know the true explanation of the remarkable divergence of Bi-(Pb)-Sr-Ca-Cu-O ( 110 K-phase ) point from Uemura's line / 1 /. In any case the presence of impurities, dislocations and another inhomogeneities can only increase the dispersion of the internal magnetic fields and hence it can only lead to the decrease of the experimental λ(T → 0 K) value. If we take Uemura's relation for granted, then the peculiarity of 110 K superconductor may be connected with another type of the vortex lattice ( e.g. non-triangular ) or with differences in anisotropy ( e.g. $\lambda_{\parallel}/\lambda_{\perp}$ < 5 ).

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