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THE COMPARATIVE STUDY OF IRREVERSIBILITY EFFECTS IN Nb FOIL AND HIGH TEMPERATURE SUPERCONDUCTING CERAMICS BY µSR

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## I. Introduction

It is known that HTSC have irreversible and metastable magnetic properties. The dependence of the HTSC behaviour in external magnetic fields on the magnetic history of the sample is well revealed in the data of the µSR experiments. Difficulty in interpretation of the µSR data obtained in the HTSC studies induced us to study the influence of the magnetic vortex pinning in the mixed state of superconductor on the behaviour of the muon spin relaxation function, and hence on the magnetic field distribution from the point of view of the µSR experiment using well-known type II superconductor Nb.

Before the discovery of HTSC several  $\mu$ SR investigations of type II superconductors such as Nb, PbIn [1,2], V<sub>3</sub>Ga [2], V [3] were carried out. The obtained information has convincingly demonstrated the  $\mu$ SR potential in the new field of application:

### II. Samples

The difficulty of the  $\mu$ SR studies of the pinning effects when superconductor is magnetized is associated with the fact that the magnetic field inhomogeneity can be greater than 200 - 300 G, which causes high damping of the µSR signal. The field inhomogeneity inside the magnetized superconductor is proportional to the geometric dimensions. of the sample. That is why the study of the pinning in one-piece samples of several  $cm^3$  in volume usual for  $\mu SR$ experiments is extremely difficult. The niobium sample consisted of 210 round plates of the Nb 30  $\mu$ m thick and 50 mm in diameter assembled like a "sandwich" together with the mylar circles 20 µm thick. This configuration allows one to consider each foil circle as a "thin" isolated plate. The sample was packed in an aluminum container. The total target thickness was 6.4 g/cm<sup>2</sup>, 83.1 % belonging to the niobium foil, and 9.3 % + 7.6 % being the background part of the target - the mylar and the aluminum container respectively. We used the foil consisting of 99.3% -Nb, 0.5% -Ta, 0.1%-

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-Ti, 0.07% - Fe, 0.09% - Si. The superconducting transition temperature by the middle point of the electric resistance fall was  $T_c$ =9.3 K. The zero resistance was achieved at the temperature 9.1 K. The residual resistance at T = 10 K was  $\rho_n$ =2.24  $\mu$ Om·cm. The external magnetic field was applied along the plates, the muon beam was directed perpendicularly to the plates.

The ceramical granular sample of  $La_{1.9}Sr_{0.1}CuO_4$  was prepared in the form of a disk 40mm in diameter and 10mm thick by the combustion method [4] with subsequent heat. treatment at the temperatures 950°C-1050°C for 12 hours. T<sub>c</sub> for the sample was 30 K. The muon beam was directed perpendicularly to the disk plane, the magnetic field was applied perpendicularly to the disk axis.

#### III. Experimental results

The experiments have been performed on the muon beam of the phasotron LNP JINR (Dubna) using a conventional transverse field  $\mu$ SR-spectrometer.

# 3.1 Niobium

Two types of measurements were carried out : cooling in the external field starting from the temperatures above T (FC), cooling in the zero external magnetic field to the temperature below  $T_c$  with subsequent ascending and descending external field scans (ZFC). FC-measurements were carried out at two values of the external field  $H_{ext}=0.9$  KOe and 2.7 KOe. The gaussian type of the muon spin relaxation function  $\sim \exp(-\sigma^2 t^2)$  fits the experimental data well at the temperatures above T<sub>c</sub>. Fig.1 shows the temperature dependence of the relaxation rate  $\sigma(T)$ . The type of the relaxation function and invariability of the  $\sigma$  value at the temperatures above T testifies to the absence of the muon diffusion in the time scale >  $5\tau_{\mu}$ , where  $\tau_{\mu}$  is the muon life -time. The sharp increase in  $\sigma$  when temperature decreases below T\_ is connected with arising magnetic vortex structure when the sample goes over to the superconducting state.



Fig. 1. Gaussian muon spin relaxation rate  $\sigma$  as a function of temperature in the Nb foil when the sample is cooled in external field. Lines are guides to the eye.

Using the data on the temperature dependence of  $H_{c2}(T)$  for Nb from [5], which is linear near  $T_c$  one gets  $T_c(0 \text{ Oe}) =$  $9.25^{+0.12}_{-0.08}$  K,  $H_{c2}(T=0) \cong 10.5$  KOe. In the analysis of the  $\mu$ SR data obtained below  $T_c$  the contribution to the spectra from the background part of the target was excluded by processing the experimental muon polarization by two oscillating gaussian functions. One is related to the superconductor, the other, a slowly damping function, is related to the background part of the target. The obtained correlation between the function amplitudes is in good agreement with the Nb part of the target. The relaxation rate  $\sigma$  value relates to the magnetic field distribution dispersion  $\langle \Delta B^2 \rangle$ as  $2\sigma^2 = \gamma_{\mu}^2 \cdot \langle \Delta B^2 \rangle$ , where  $\gamma_{\mu} = 2\pi \cdot 13.55$  KHz/G. The magnetic field penetration depth  $\lambda$  can be estimated by the formula [6]

 $\langle \Delta B^2 \rangle_{\text{lattice}} \cong 7.5 \cdot 10^{-4} (1-b)^2 [1 + 3.9(1-b)^2] \phi_0^2 \lambda^{-4}$ , (1)

where  $\langle \Delta B^2 \rangle_{lattice}$  is the dispersion of the magnetic field distribution in the regular magnetic vortex lattice b =  $B/H_{c2}$ , B is the magnetic induction,  $\phi_0 = 2.07 \cdot 10^{-7} \text{ G/cm}^2$  is magnetic flux quantum. Extrapolating  $\lambda(T)$  by the known temperature dependence  $\lambda(T) = \lambda(0) \cdot (1 - (T/T_c)^4)^{-1/2}$  one gets  $\lambda(0) \cong 800 \text{ Å}.$ 

The ZFC measurements were carried out at the

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temperatures 6.5 K and 8 K. Fig.2 shows part of the superconductor  $n_0$  in which the magnetic induction equals to zero as a function of the external field  $H_{ext}$  at T=6.5 K.



Fig. 2. External field dependence of the part of the Nb foil volume with the zero magnetic induction when the sample is magnetized at the temperature 6.5 K. Lines are guides to the eye.

This field dependence of  $n_0$  is well explained in the frame of the critical state model (CSM) or the Bean-London model, e.g. see [7]. The external field lower than  $H_{c1} \cong 0.5$  KOe, as seen from the figure, is completely pulled out. When the field becomes greater than  $H_{c1}$ , it begins to penetrate the superconductor in the surface layer  $\delta$ , i.e. the macroscopic penetration depth  $\delta$  depended on the external field arises achieving maximum  $\delta = W/2$  at the  $H_{ext} = H^* \cong 1.9$  KOe, where W is the plate thickness. Using the field value  $H^*$  one estimates the critical current density  $J_c$  on the basis of the Bean model [8]  $J_c := (H^*-H_{c1})/(2\pi W/c) \cong 7.2 \cdot 10^5$  A/cm<sup>2</sup>. Let us consider the CSM at the  $H_{ext} > H^*$  more closely from the point of view of the  $\mu$ SR experiment. The magnetic induction B in a "thin" plate in the external field parallel to the plate obeys the critical state equation:

$$\frac{dB}{dx} = \pm k J_{c}(B), \qquad (2)$$

where  $k = 4\pi/c$ ,  $J_c$  is the critical current density. In the external field region of interest the width of the magnetic induction probability distribution in the plate  $\Delta B$  is determined by the absolute difference between the magnetic induction on the surface B(x=0) and that in the plate center

B(x=W/2). Replacing the function  $J_{C}(B)$  by its value at B = <B> - the mean induction in the plate, one can estimate the muon spin relaxation  $\sigma = \gamma_{II} \cdot \Delta B/2 = \gamma_{II} \cdot k \cdot J_{C}(\langle B \rangle) \cdot W/4$ .

At the temperature 6.5 K the experimental values of  $\sigma$ are greater than 10 - 15  $\mu s^{-1}$  at the external fields up to ~3.2 KOe due to large  $J_{c} > 2.10^{5}$ , as it follows from the above formula. At this extremely high damping of the  $\mu$ SR signal we didn't succeed in obtaining the values of the mean induction  $\langle B \rangle$  and the distribution width  $\Delta B$  with a satisfactory accuracy. At the temperature 8 K the relaxation rate is already smaller than  $\sim 10 \ \mu s^{-1}$  in the external magnetic fields greater than ~ 0.9 KOe, which is enough for determination of <B> and  $\Delta B$ . Fig.3 shows r.m.s  $\langle \Delta B^2 \rangle^{1/2}$  of the magnetic fields inside the superconductor and the difference M =  $B_{\mu} - H_{ext}$  (where  $B_{\mu}$  is the mean value of the magnetic fields on the muon, which is equal to the mean magnetic induction in the superconductor <B> ) as functions of the external field  $H_{ext}$  at T = 8 K. The insert shows the function  $J_{c}(H_{ext})$  calculated according to the curve  $M(H_{ext})$ hysteresis using the Bean model:  $J_{C} = (B_{down} - B_{up})/(kW/2)$ , where  $B_{down}$ ,  $B_{up}$  are the  $B_{\mu}$  values at the descending and



Fig. 3. The r.m.s.  $\langle \Delta B^2 \rangle^{1/2}$ and the difference  $B_{\mu} - H_{ext}$  as functions of the external field  $H_{ext}$  for the Nb sample. The solid lines in the r.m.s. plot show the half-width of the magnetic induction distribution calculated according to the CSM. The insert shows the critical current density  $J_c$ . All the plots have the same horizontal axes.

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ascending external field scans respectively. Using this function we calculated the half-width values of the magnetic field distribution by integrating equation (2). The calculation results are shown in the r.m.s. plot ( fig.3 ) by solid lines. The contribution to the r.m.s. from the regular lattice of the magnetic vortices  $\langle \Delta B^2 \rangle_{lattice}^{1/2}$ small in comparison with the contribution caused by the magnetic vortex pinning in the magnetized sample. One can estimate the  $\langle \Delta B^2 \rangle_{lattice}^{1/2}$  value from the FC experiment: in the external field 0.9 KOe r.m.s. is ~ 33 G. Following formula (1) its value has to be equal to ~17 G at  $H_{ext} \cong$ 1.2 KOe, ~6 G at  $H_{ext} \approx 1.5$  KOe which are significantly smaller than the r.m.s. values observed in the ZFC experiment. It is seen from the figure that there is good agreement between the calculated r.m.s. values and the experimental ones at the descending external field scan. For the ascending field scan the CSM describes our data in the fields lower than ~ 1.3 KOe  $\approx$  0.8H<sub>c2</sub>.

As a result of the  $\mu$ SR measurements we have obtained the following characteristics of the Nb foil with the resistance ratio  $\rho_{300K}/\rho_{10K} = 7.7$ :  $H_{c2}(0) = 10$  KOe,  $H_{c1}(0)=1$  KOe ( assuming the temperature dependence  $H_{c1}(T) = H_{c1}(0) (1-(T/T_c)^2)$ ),  $\lambda(0)=800$  Å,  $J_c \cong 1.5 \cdot 10^5$  A/cm<sup>2</sup> at 8K and the external field ~1 KOe.

# 3.2 La<sub>1.9</sub>Sr<sub>0.1</sub>CuO<sub>4</sub>

The ZFC measurements were carried out in the external field region 0 - 800 Oe. Fig.4 shows r.m.s.  $\langle \Delta B^2 \rangle^{1/2}$  of the magnetic field distribution in the superconductor and the difference  $(B_{\mu} - H_{ext})$  as functions of the external field  $H_{ext}$  at the temperatures 10, 15, and 25 K. It is known that magnetization of HTSC ceramical samples in high fields is determined by the superconducting grains because the weak links between the grains are destroyed in magnetic fields 10 - 100 Oe. As already said, the critical current density  $J_c$  in the frame of the CSM is determined by the hysteresis value  $\Delta B_{\mu} = B_{down} - B_{up}$ :  $J_c = \alpha \cdot \Delta B_{\mu}/(kW)$ , where W is the



Fig. 4. The r.m.s.  $\langle \Delta B^2 \rangle^{1/2}$ and the difference  $B_{\mu} - H_{ext}$  for the La<sub>1.9</sub>Sr<sub>0.1</sub>CuO<sub>4</sub> sample as functions of the external field  $H_{ext}$  when the sample is magnetized at the temperatures 10, 15 and 25 K. Arrows indicate how the field was changed. Lines are guides to the eye.

typical grain dimension,  $\alpha$  is the coefficient depending on the geometric shape of the grain. One can see from fig. 4 that  $\Delta B_{ij}$  very slightly depends on the external field. In the fields  $H_{ext}$  > 200 Oe the derivative  $|d(\Delta B_{\mu})/dH_{ext}| \leq 10^{-2}$ . So far as the difference between the magnetic inductions in the grains at the descending and ascending external field scans doesn't exceed 5 - 10 G the current density J determined by the magnetic induction value is changed by not more than ~1 %. Then, according to the CSM, the magnetic induction profile in the grain at the descending external field scan must practically be a mirror reflection of the magnetic induction profile at the ascending field scan, and hence the r.m.s. values must not depend on the external field change direction, which evidently disagrees with the experimental motion of the r.m.s.. It should mentioned that the r.m.s. value at the descending field scan in the ZFC experiment practically coincides with the r.m.s. value obtained in the FC experiment also performed with this sample at given temperature. The influence of the pinning on the magnetic field distribution from the regular lattice of the magnetic vortices is minimal in the FC procedure. The r.m.s. in this case is considered to be defined by the

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magnetic field penetration value. Taking this fact into account we can explain the situation observed in the ZFC experiment in the following way. At the descending external field scan the critical state in the grain is destroyed. The magnetic induction profile in the grain slightly deviates from the average value of the magnetic induction. At the ascending external field scan the grain goes over to the critical state. In this case the contribution to the r.m.s. due to the critical state should be of the order of the  $\Delta B_{\mu}$ value, which is observed in the experiment (see fig.4). The similar results were obtained for the samples with Sr content 0.15 and 0.25 [9], and in YBaCuO system in paper [10].

Thus, our experimental data allow the conclusion that at the external magnetic fields up to 800 Oe the critical state model does not describe the distribution of the magnetic induction in a high-T<sub>c</sub> superconductor adequately.

# References

- [1] A.T.Fiory, D.T.Murnick, M.Levental, W.J.Kossler, Phys.Rev.Lett, 33 (1974) 969.
- M.Gladish, D.Herlach, M.Metz, H.Orth, G.zu Putlitz, A.Seeger, H.Teichler, W.Wahl, M.Wigand, Hyper.Inter.,6 (1979) 109.
- [2] V.G.Grebinnik, I.I.Gurevich, V.A.Zhukov, A.I.Klimov,
  L.A.Levina, V.N.Majorov, A.P.Manich, E.V.Melnikov,
  B.A.Nikolsky, A.V.Pirogov, V.S.Roganov, V.I.Selivanov,
  V.A.Suetin, JETP, 52 (1980) 261.
- [3] F.N.Gygax, A.Hinterman, H.R.Ott, H.Rudigier, W.Ruegg,A.Schenck, W.Studer, Hyper.Inter., 8 (1981) 623.
- [4] A.G.Merisanov, I.P.Borovinskaya, DAN SSSR, 204(2) (1972) 366.
- [5] C.K.Jones, J.K.Hulm, B.S.Chandrasekhar, Rev.Mod.Phys., 36 (1964) 74.

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[6] E.H.Brandt, Phys.Rev., B37 (1988) 2349.

- [7] A.M.Campbell and J.E.Evetts, Adv.in Phys., 21 (1972) 199.
- [8] C.P.Bean, Phys.Rev.Lett., 8 (1962) 250.
- [9] V.G.Grebinnik, V.N.Duginov, V.A.Zhukov, S.Kapusta,
  - A.B.Lazarev, V.G.Olshevsky, V.Yu.Pomjakushin, S.N.Shilov
  - D.T.Bezhitadze, I.I.Gurevich, B.F.Kirillov,
  - E.P.Krasnoperov, B.A.Nikolsky, A.V.Pirogov,
  - A.N.Ponomarev, V.A.Suetin, G.F.Tavadze, I.P.Borovinskaya, M.D.Nersesyan, A.G.Peresada, Yu.F.Eltzev, V.R.Karasik, O.E.Omelyanovsky, Proc. 18 Int. Conf. on Hyperfine Interactions ( Prague 14-19 August 1989 ) to be published in Hyper.Inter. (1990).

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[10] B.Pumpin, H.Keller, W.Kundig, W.Odermatt, B.D.Patterson, J.W.Schneider, H.Simmler, S.Connell, K.A.Muller, J.G.Bednorz, K.W.Blazey, I.Morgenstern, C.Rossel, I.M.Savic, Z.Phys., B72 (1988) 175.

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