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# FOREIGN TECHNOLOGY DIVISION



CRITICAL CURRENT OF THE DISPERSION SUPERCONDUCTING PHASE, OBTAINED DURING AGING

by

Ye.P. Romanov, L.V. Smirnov, et al



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U. S. BOARD ON GEOGRAPHIC NAMES TRANSLITERATION SYSTEM

\*ye initially, after vowels, and after b, b; e elsewhere. When written as  $\ddot{e}$  in Russian, transliterate as y $\ddot{e}$  or  $\ddot{e}$ .

#### RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin .	sh	sinh	arc sh	$sinh_1^1$
cos	cos	ch	cosh	arc ch	cosh_1
tg	tan	th	tanh	arc th	tann_:
ಂಕ್ಷ	cot	cth	coth	arc cth	ooth];
sec	sec	sch	sech	arc sch	sech_7
cosec	csc	csch .	csch	arc csch	esch <sup>1</sup>

Russian	English
rot	curl
lg	log
GRAPHICS	DISCLAIMER

All figures, graphics, tables, equations, etc. merged into this translation were extracted from the best quality copy available.

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### CRITICAL CURRENT OF THE DISPERSION SUPERCONDUCTING PHASE, OBTAINED DURING AGING

Ye. P. Romanov, L. V. Smirnov, V. D. Sadovskiy, N. V. Volkenshteyn Institute of the Physics of Metals Submitted 19 March 1965

It is known that high critical parameters of plastic superconductors on a base of solid solutions can be obtained as a result of plastic deformation and heat treatment [1, 2]. At the same time, the critical current and field of monocrystalline samples turn out to be significantly lower [4].

It was pointed out more than once [5, 6] that the preservation of superconductivity in high magnetic fields is conditioned by the presence in rigid semiconductors of a dense network of very thin intersecting filaments, which remain superconducting during the conversion of the main mass of material into the normal state (sponge model). In confirmation of this metallographically [3] and with the help of an electron microscope [10, 11] a lamellar threadlike structure was detected which was conditioned by plastic deformation and the breakdown of a solid solution.

A. A. Abrikosov [7] demonstrated theoretically the possibility of existence of the so-called "mixed" state, capable of enduring high magnetic fields. However, the flow of current in such a superconductor was not considered. As it was cleared up later [8, 9], when a current is present a significant role for the stability of the "mixed" state should be played by the nonuniformities of the lattice

1.

(dislocations, subboundaries, dispersed phase).

Thus it is not clear whether the subboundaries and individual dislocations which are decorated with separations are those channels, over which the superconducting current flows, or do they serve only for realization of the "mixed" state with a negative surface energy between the superconducting and normal phases.

We selected an alloy, which is nonsuperconducting at 4.2°K and capable in the process of heat treatment (aging) of yielding a dispersed superconducting phase. In such an alloy, by varying the conditions of treatment ( plastic deformation, aging), it is possible to change the degree of dispersion, the amount and the distribution of the phase of separation, which makes it possible to establish the bond between the state of the separated phase and the parameters of superconductivity.

In this work some results are given which were obtained on an alloy of zirconium with an additive of niobium (4%). The alloy was prepared on a base of zirconium iodide with a purity of 99.8%, containing the following impurities (wt.%): Ti = 0.005; Hf = 0.055; Si = 0.005; N<sub>2</sub> = 0.005; Ni = 0.001; Fe = 0.03; C = 0.03; O<sub>2</sub> = 0.05; and niobium, containing (%): Ta = 0.5; Ti = 0.2; Fe = 0.06.

Melting was done in an arc furnace with a nonconsumable electrode on a copper water-cooled hearth in an atmosphere of helium, purified with activated carbon at the temperature of liquid nitrogen and by melting of a zirconium scavenger. First a master alloy containing 24 wt.% of niobium was melted out, then it was diluted with pure zirconium to the assigned composition. Weighing of the ingots snowed that the difference of their weight in comparison with the weight of the charge does not exceed 0.01%. Composition of the ingots was checked by spectral analysis. Fluctuations in the content of niobium did not exceed 0.1%.

The ingots were drawn out in square blanks and homoginized at  $1280^{\circ}$ C for 48 hours. Homoginization annealing was carried out in quartz ampoules filled with purified helium to a pressure of 300 mm Hg. The hardened samples were subjected to cold deformation (\$): 40; 93; 98 such that the final thickness of the drawn strip comprised 0.06 mm.

The strip samples were tempered in a vacuum at  $10^{-6}$  mm Hg. The measurements were made on direct current at 4.2°K. The current was introduced into the sample smoothly through copper press-contacts coated with solder. Transition into the normal state was fixed using a millivoltmeter. Simultaneously with activation of the millivoltmeter a relay which cut off the current was triggered. The critical density of the current was defined as the ratio of critical current to the transverse section of the sample.

After hardening all the samples were nonsuperconducting at a temperature of  $4.2^{\circ}$ K. At that temperature cold plastic deformation did not cause the appearance of a superconducting state. However, even short-term annealing of the deformed samples at a temperature higher than 500°C was sufficient enough so that the samples, cooled to the temperature of liquid helium, became superconducting.





Figure 1. Influence of breakdown time of a supersaturated solid solution of a Zr melt with 4% Nb at 500°C on the magnitude of critical density of current:

 $\Delta$  - without deformation; x - de-

Figure 2. Influence of breakdown time of a supersaturated solid solution of a Zr melt with 4% Nb at 550°C on the magnitude of critical density of current: ▲ - without deformation; x - deformation 40%; 0 - deformation 93%. formation 40%; 0 - deformation

93%; 🖸 - deformation 98%.

Kev: (1) Critical density of current,  $A/cm^2$ ; (2) Tempering time, hours.

The results of measurements on samples which had undergone breakdown at 500°C are shown in Figure 1. Samples which were not deformed after hardening do not produce a superconducting state even with annealing for 9 hours. However, the application of plastic deformation prior to annealing promotes the appearance of superconductivity. The critical density increases sharply in the course of the first two hours of annealing. For samples which were deformed by 93% a maximum of critical density of current was detected following annealing for 2.5 hours. At longer holdings a lowering of the density of critical current is observed. Initially the critical current density of the samples with a 40% degree of deformation exceeds that for a melt which is deformed by 93%, but already after an hour of annealing the relationship changes and the critical current of the strongly deformed alloy, increasing rapidly, exceeds the value for 40% deformation. With further holding up to 6.5 hours they are equalized again.





Figure 3. Diagram of state of the Figure 4. Microstructure of the system zirconium - niobium. Key: (1) Nb, wt.%.

alloy Zr+4% Nb, hardened from 1000°C; x6CO.

Tempering at 550°C (Figure 2) yields approximately the same regularities. However, in this case the samples which had not undergone cold deformation turn out to be superconducting. Here also a clearly expressed maximum of critical current density is observed for samples with a greater degree of deformation. The maximum value is equal to  $7 \cdot 10^4$  A.cm<sup>2</sup>. In the same manner as the foregoing, the increase of density of superconducting current in the strongly deformed samples initially lags behind the values for a lesser degree of deformation, after 1.5-2 hours exceeds them, and after 4 hours again is lowered.

Above  $865^{\circ}$ C zirconium has a bcc [body-centered cubic] lattice, which, according to the diagram of state shown in Figure 3 [12], is capable of dissolving niobium unlimitedly. Below that temperature the cubic lattice of zirconium undergoes a conversion into hexagonal close-packed, which under equilibrium conditions is capable of dissolving only 0.5% niobium. In accordance with the diagram of state, the  $\beta$ -solid solution undergoes eutectoid breakdown, so that below 560°C practically pure  $\alpha$ -zirconium and a a solid solution of zirconium in niobium (phase  $\beta$  Nb) should be found in equilibrium. The latter contains 8-10% zirconium and has a temperature of transition into the superconducting state higher than 4.2°K, while  $\alpha$ -zirconium is not a superconductor at the boiling point of helium.

Following hardening of the melt of zirconium with 4% niobium from the area of stability of the  $\beta$ -solid solution, as a result of martensite conversion a niobium-supersaturated  $\alpha$ -solid solution is obtained. A microphotograph of the hardened sample is shown in Figure 4. Following etching the microsection remains light, only a relief, corresponding to the shearing nature of the conversion, is revealed. However, this state of the alloy is unstable, following tempering there is a breakdown of the supersaturated solid solution with is accompanied by intensified etchability of the samples.



Figure 5. Microstructure of the alloy Zr+4% Nb, after annealing at 550°C for 2 hours; x600.

A microphotograph of a sample after annealing at  $550^{\circ}$ C for two hours is shown in Figure 5. The diffusion-free formation of  $\checkmark$ -crystals of zirconium, supersaturated with niobium, leads to the

creation of intragranular interfaces, over which primarily the separation of the phase  $\beta$  Nb takes place. In Figure 5 the individual "needles" of  $\alpha$ -crystals are clearly evident.

Plastic deformation raises the density of defects of the lattice considerably, correspondingly the desnity of separations increases. It can be assumed that in connection with this the increase which we observed in the critical density of current with an increase in the degree of cold deformation also takes place.

In the investigated alloy the appearance of superconductivity is conditioned only by the separating phase, since a change in the state of the matrix following tempering, as a result of its impoverishment with niobium, leads to a lowering of the already quite low temperature of conversion of the latter. The state and distribution of the separated phase and its interaction with the matrix, which is found in the normal state, determine also the critical parameters of superconductivity appearing in this alloy. Unfortunately, on the basis of only one metallographic observation it is impossible to give a complete picture of the phase state of separation. At the present time an electron-microscope study is being made of the processes which take place during tempering.

Since the phase of separation can produce a superconducting state regardless of the properties of the matrix, apparently it is right to consider that in alloys of the system Nb-Zr, used for the production of solenoids, the separation of the phase  $\mathcal{O}_{Nb}$  also takes place; dislocations, decorated with this phase, form a superconducting structure, conditioning the preservation of superconductivity following the application of large magnetic fields and high current densities. It is noteworthy that even a small amount of the separated phase is sufficient for obtaining considerable current densities.

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