

# Observation of branching of domains of the Shubnikov phase in a niobium single crystal

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The structure of the intermediate-mixed state is observed directly in niobium single crystals with high resolution. Multiple branching of domains of the Shubnikov phase is observed.

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In Ref. 1, Landau examined the model of an intermediate state with multiple branching of the domains of the normal phase at the surface of a specimen. In Ref. 2, within the scope of the multiple branching model, the expansion of the normal domains at the surface of the specimen was taken into account, and it was shown that the minimum possible width of the domains at the surface must be  $\sim 200 \Delta$  ( $\Delta$  is the surface tension constant at the boundary of the  $n$  and  $s$  phases, determined from  $a = (H_c^2/8\pi)\Delta$ , where  $a$  is the coefficient of surface tension and  $H_c$  is the critical magnetic field). We note that multiple branching effects, as indicated in Ref. 2, should not occur in clean type I superconductors, which are of reasonable thickness, due to an insufficiently low surface energy of the  $n$ - $s$  boundary.

In Ref. 3, it was demonstrated experimentally that domains of the Meisner and Shubnikov phases can coexist in type II superconductors with a Ginzburg-Landau parameter  $\kappa \sim 1$  and nonvanishing demagnetization factor. Such a state is called an intermediate-mixed state. The existence of an intermediate-mixed state is related to the appearance of attraction between vortices in type II superconductors ( $\kappa \sim 1$ ); as a result, an equilibrium induction  $B_0$  is established in the domains of the Shubnikov phase ( $B_0 = n\Phi_0$ , where  $n$  is the equilibrium density of vortices, and  $\Phi_0$  is the magnetic flux quantum equal to  $2.07 \times 10^{-7}$  G cm<sup>2</sup>). In Ref. 3, the structure of the intermediate-mixed state was described within the scope of the intermediate-state theory without branching,<sup>4</sup> replacing  $H_c$  by  $B_0$ . It is clear that with this approach there should be a rather large number of vortices in the domains of the Shubnikov phase, while the distance between the vortices should not be large compared to their dimensions. Since the energy concentrated at the boundary between the Meisner and Shubnikov domains can be rather low,<sup>3</sup> branching of the domains of the Shubnikov phase in the case of the intermediate-mixed state can be manifested more strongly compared to analogous effects in type I superconductors.

In the present work, with the help of the high-resolution decoration technique in Ref. 5, we observed the magnetic structure at the surface of a niobium single crystal at  $T = 4.2$  K [ $B_0 \approx 800$  G (Ref. 6)]. The specimens were cut out from a single crystal of niobium by electron-beam melting, mechanically ground, and chemically polished in the mixture HF (40%) + HNO<sub>3</sub> (60%). Then, the specimens were annealed in a vacuum  $\sim 5 \times 10^{-9}$  mm Hg at  $T = 2200$  °C for  $\sim 5$  hours. The decoration was carried out

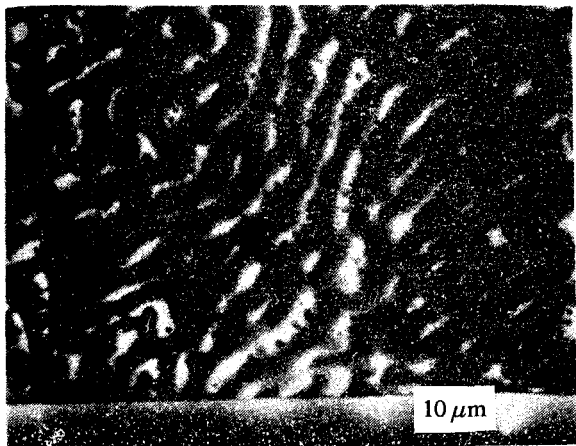


FIG. 1.

with particles of iron with dimensions  $\sim 50 \text{ \AA}$ . The Bitter patterns obtained were studied in a scanning electron microscope in the secondary electron regime. The light-colored sections in the photographs are the domains of the Shubnikov phase.

Figure 1 shows the structure of the intermediate-mixed state, formed on the surface of a plate with thickness  $\approx 100 \text{ }\mu\text{m}$  in a perpendicular magnetic field  $\approx 400 \text{ Oe}$ . It is evident that the characteristic period of the structure  $a$  turns out to be equal to  $\sim 10 \text{ }\mu\text{m}$ . After determining the characteristic period, it is possible to estimate the parameter  $\Delta$ . From Ref. 4, we have

$$\Delta = a^2 \varphi(\eta) L^{-1},$$

where  $L$  is the thickness of the specimen,  $\eta = H/H_c$  ( $H$  is the external field), and  $\varphi(\eta)$  is a tabulated function.<sup>2</sup> Replacing  $H_c$  by  $B_0$  and substituting  $L \approx 10^{-2} \text{ cm}$ ,  $\eta = 0.5$ ,  $\varphi(\eta) = 2 \times 10^{-2}$ , and  $a = 10 \text{ }\mu\text{m}$ , we obtain  $\Delta \approx 200 \text{ \AA}$ . We note that inside the domains of the Shubnikov phase a vortex lattice with induction  $B'_0 \approx 500 \text{ G}$  was resolved, consistent with the model in which the normal domains expand at the surface of the specimen.<sup>4</sup> Figure 2 shows the structure of the intermediate-mixed state which forms on the surface of a cylinder with height  $\approx 1.2 \text{ cm}$  and  $\phi \approx 0.6 \text{ cm}$  in a field  $\approx 250 \text{ Oe}$ , perpendicular to the end face. It is evident that the characteristic period of the structure turned out to be of the order of  $1 \text{ }\mu\text{m}$ . The induction in the domains of the Shubnikov phase was estimated from photographs on which separate vortices were clearly resolved. It turned out to be equal to  $\approx 800 \text{ G}$ , in agreement with the data in the literature for equilibrium volume induction in pure niobium single crystals. In contrast to a thin specimen, in a thick specimen, a vortex fluid was observed in the domains of the Shubnikov phase. If  $\Delta$  is known, it is possible to estimate the characteristic period of the intermediate-mixed state structure at the surface and in the bulk of the specimen. From Ref. 2, we have

$$a_v = 0.882 \eta^{-1/3} (1 - \eta)^{-2/3} L^{2/3} \Delta^{1/3} \text{ and } a_s = 1.46 \eta (1 - \eta)^2 \Delta \varphi^{-2}(\eta),$$

where  $a_v$  and  $a_s$  are the periods in the bulk and on the surface, respectively. Substituting  $\eta \approx 0.3$ ,  $\Delta \approx 200 \text{ \AA}$ , and  $\varphi \approx 2 \times 10^{-2}$ , we obtain  $a_v \approx 250 \text{ }\mu\text{m}$  and  $a_s \approx 10 \text{ }\mu\text{m}$ .

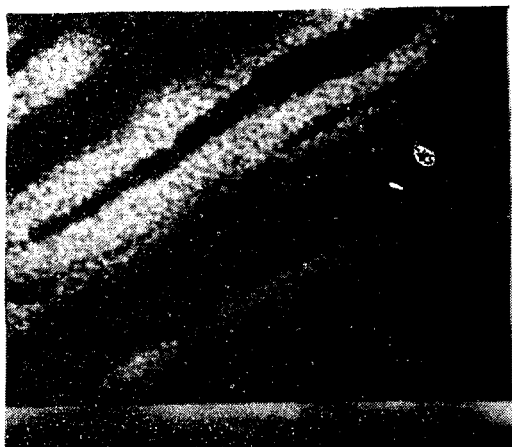


FIG. 2.

Thus, the intermediate-mixed state structure observed in Fig. 2 generally corresponds to a surface structure formed as a result of multiple branching of domains of the Shubnikov phase, but the characteristic scale of the surface structure turns out to be slightly smaller than the scale predicted in Ref. 2. In this connection, we note that as a result of multiple branching, domains of the Shubnikov phase, which contain a small number of vortices, approach the surface of the specimen; in this case, the width of the domains is comparable to the distance between the vortices. Apparently, in calculating the scale of the surface structure of the intermediate-mixed state, it is necessary to take into account more precisely the fact that the domains of the Shubnikov phase consist of separate vortices that carry magnetic flux quanta, in contrast to the uniform field distribution in the domains of the normal phase in the case of the intermediate structure of type I superconductors.

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