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FINAL TECHNICAL REPORT

STUDY OF PROPERTIES OF HIGH-FIELD SUPERCONDUCTORS AT ELEVATED TEMPERATURES

7 OCTOBER 1966

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By
D. A. Gandolfo
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FOREWORD

This report was prepared by the Radio Corporation of America, Applied Research Department, Camden, N. J. , under NASA Contract No. NAS 8-11272. Mr. E. W. Urban was the NASA Project Engineer.

This is the final technical report on this contract. Progress is described in the period from 27 April 1966 through 26 August 1966. The research work was done by D. A. Gandolfo and C. M. Harper of Applied Research. The report was prepared by D. A. Gandolfo who was the Principal Investigator. G. D. Cody of RCA's David Sarnoff Research Center, Princeton, N. J. , served as consultant.

ABSTRACT

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Magnetization experiments on specimens of Nb_3Sn , Nb Ti, and Nb are reported. The Nb_3Sn specimens were pressure sintered, and one of them contained a ferromagnetic inclusion. To our knowledge results on such a specimen have not been reported before. Values of the critical state parameter, α , as a function of temperature were obtained. The Nb Ti specimen was cast and cold worked. Values of α and the threshold for ac field-induced flux jumps were obtained before and after a 400°C heat treatment. An improvement in both quantities by a factor of more than 2 was observed. The Nb sample was also cast and cold worked. For H near H_{c2} the supercurrent in this specimen was very stable against ac field-induced flux jumps, while for H somewhat less than H_{c2} , behavior in superimposed ac and dc fields resembled that of Nb Ti. Recommendations for further effort are given.

Author

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SECTION I

INTRODUCTION

A. REVIEW OF PAST WORK

Initial work done on this contract was concentrated on the study of the critical-current density and flux jumping in hard superconducting tubes and solenoids. These quantities were measured as functions of applied magnetic fields from 0 to 30 kG and at temperatures from 4.2°K to T_C . Hollow cylinders of Nb₃Sn, NbZr, and NbTi, and solenoids of NbZr wire and Nb₃Sn ribbon were investigated.¹

At any fixed temperature below T_C , the critical-current density J_C , in the wall of one of the hollow cylinders was found to be described by Kim's equation, as long as critical-state data points could be obtained from magnetization curves. In Nb₃Sn samples at temperatures around 4.2°K, where J_C was large and flux jumps were numerous, a different approach was used to derive J_C . The values of magnetization at a flux jump were plotted against the applied field, H , and a "practical J_C " was derived. When these values of J_C were plotted against temperature, a maximum occurred near 8°K. This suggested that different mechanisms may trigger flux jumps at different temperatures. At $T = 14°K$, flux jumps gave way to pure critical-state performance.

The behavior of solenoids of Nb₃Sn ribbon and NbZr wire varied markedly. In the NbZr solenoids, flux jumps never occurred when flux was moving freely across the windings; i. e., when the sample was in the critical state. On the other hand, in the solenoids made of 0.090-inch wide Nb₃Sn ribbon, flux jumps occurred only after the sample had entered the critical state. These facts may indicate that different mechanisms trigger flux jumps in each type of solenoid. In both types of solenoids the field interval between flux jumps decreased if either magnetic field or temperature was

increased. At higher temperatures, the flux jumps were incomplete and were reduced to small excursions away from the critical state at higher values of magnetic field. At very high temperatures (12° through 16°K), flux jumps were eliminated completely in the solenoid made of 0.090-inch wide Nb₃Sn ribbon. In contrast, NbZr solenoids exhibited some flux jumps even at temperatures near T_c.

Flux jumps were sensitive to the magnetic field sweep rate in the Nb₃Sn solenoids and could even be eliminated at low values of temperature and field by employing quasistatic field sweep rates (≈ 1 gauss/sec). The flux jumps in NbZr solenoids, on the other hand, showed little dependence on the field sweep rate and persisted for all rates used (1 to 100 gauss/sec).

More recently our interest has centered on the manner in which an alternating field penetrates a hard superconductor in which the mixed state has been established by the application of a dc field.² Since the most interesting properties of hard superconductors are associated with the motion of flux lines (and the pinning of these lines by structural defects), we felt that much could be learned from the study of a situation in which the lines are constantly excited, as by an alternating field. To this end, we performed magnetization experiments on tubular specimens of NbTi using superimposed ac and dc fields.

Before the ac experiments were performed, the NbTi tubes were determined to be "well-behaved" hard superconductors through dc magnetization experiments. These tubes exhibited a field-dependent, critical-current density that was consistent with Kim's semi-empirical expression $J_c = \alpha / (H^* + B_0)$. Values of α and B_0 for the different samples were in reasonably good agreement. When exposed to an ac field, a sample shielded its interior as long as the ac field amplitude, h , remained below a certain threshold. When this threshold (which was smaller than the dc field which could be shielded) was exceeded, flux rushed into (and out of) the sample in a precipitous manner identified as flux jumping. At low frequencies ($f < \text{several hundred c/s}$), no flux entered or left the specimen interior during any portion of the cycle other than

the instant at which the flux jumps occurred. Square waves caused by two flux jumps per cycle were observed by means of a Hall probe within the sample, and were displayed on an oscilloscope. At higher frequencies ($f >$ several hundred c/s), flux entered and left the specimen throughout the cycle and a sine wave, similar in amplitude to the applied signal, was detected by the Hall probe. At low frequencies a multitude of well defined flux jumps per cycle could be observed by increasing the amplitude of h . At low frequencies the duration of the flux jumps was estimated at 2×10^{-4} sec. This was believed to be a fairly general property of the specimen. The flux jumps were strikingly similar in amplitude over a range of frequencies (6.3 to 208 c/s) and amplitudes (430 to 2200 gauss) of the ac field; the most frequently observed values were in the interval from 400 to 500 gauss. The value of $h(f)$ at the onset of flux jumping varied inversely with frequency from 6.3 to 2080 c/s with the dependence being stronger at lower frequencies. The value of $h(H_{dc})$ at the onset of flux jumping varied inversely with the dc field ($2,000 < H < 25,000$ G) with the dependence being stronger at lower fields. The practical critical-current density in the superimposed ac and dc fields was smaller than the true value in the dc field alone by a factor between 1.2 (low frequency) and about 4 (high frequency). Several mechanisms by which the ac field might dissipate power in the specimens were considered, and the computed hysteresis losses associated with the field-dependent penetration depth were far greater than the normal eddy current losses. Temperature increases associated with ac power losses were computed (for a much simplified situation), and these average temperature increases were not great enough to bring the temperature of any part of the sample up to T_c . The result of this computation was in qualitative agreement with temperature measurements made with a carbon resistor. The measurements indicated that no significant temperature rise at the inner surface occurred prior to penetration of the ac field to the interior of the sample. This observation did not rule out the possibility of a highly localized temperature increase which might be revealed by a more realistic calculation.

B. SUMMARY OF PRESENT REPORT

During the period covered by the present report we have been concerned with the phenomenon of flux pinning as manifested by the critical currents which flow in dc fields and superimposed ac and dc fields. We have examined samples of Nb_3Sn , NbTi and pure Nb. The Nb_3Sn samples were pressure sintered and one of them contained a ferrimagnetic second phase. The inclusion of the second phase represents an attempt to control the size, strength, and distribution of pinning centers. To our knowledge this is the first time results have been obtained on a composite structure such as this. These results are discussed in Section IIA. Section IIB contains results obtained when a well characterized sample of NbTi was exposed to a heat treatment in an attempt to alter its flux pinning characteristics. Finally, by way of contrast to hard superconductors, we observed magnetization and flux jumps in an alternating field in a sample of pure (but cold worked) niobium. These results are shown in Section IIC. Conclusions based on our study and recommendations for future effort are given in Section III.

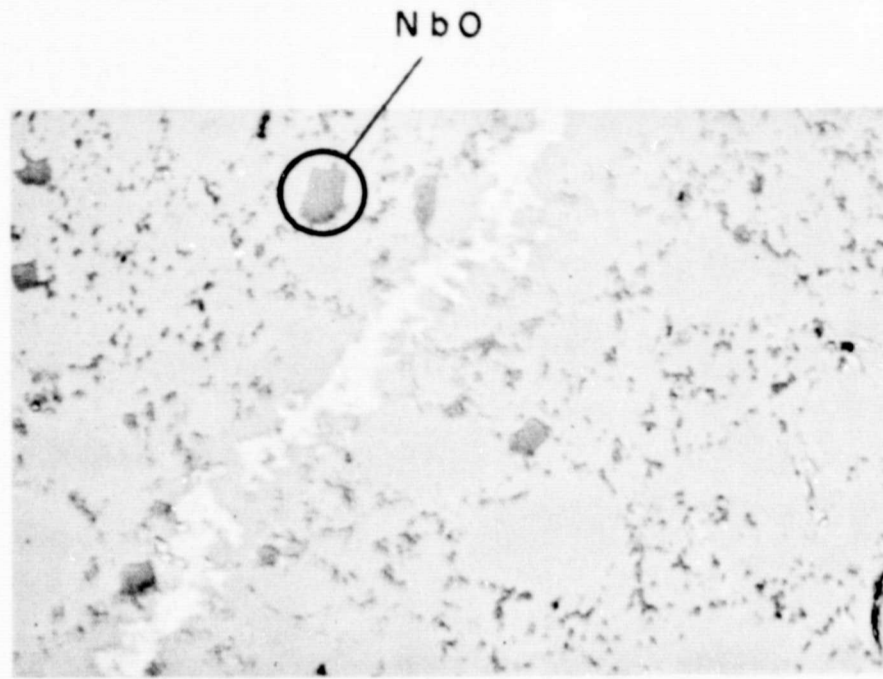
SECTION II

EXPERIMENTAL RESULTS

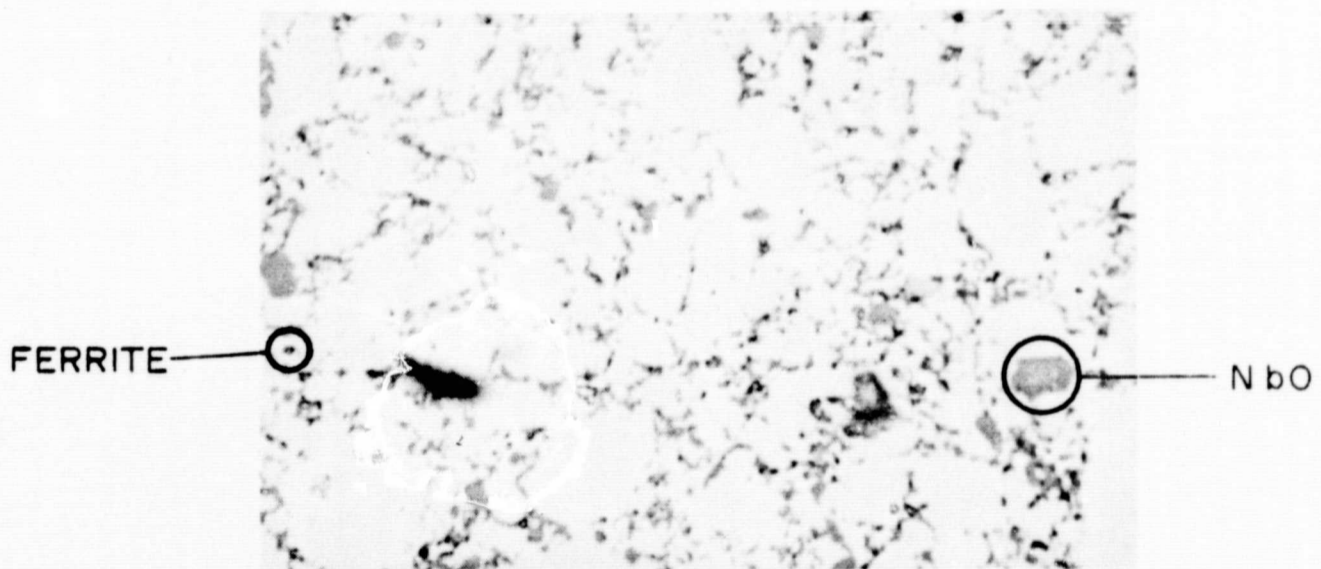
A. EXPERIMENTS ON PRESSURE SINTERED Nb_3Sn

1. Fabrication and Characterization of Samples

Two samples of Nb_3Sn , prepared by pressure sintering,³ were studied. See Fig. 1a, b. These samples were fabricated in the following manner. First, Nb_3Sn was made by sintering powders of pure Nb and Sn. Next the Nb_3Sn was reduced to a powder with an average particle size of 10 microns. For sample Nb_3Sn No. P. S. 5, this powder was mixed with a zinc-manganese ferrite ($\text{Zn}_{.5}\text{Mn}_{.5}\text{Fe}_2\text{O}_4$) such that the ferrite constituted 5%, by volume, of the mixture. This particular ferrite was chosen for two reasons. The first is that it has a large saturation moment. The moment is about 7 kG at room temperature and increases with decreasing temperature. While the moment was not measured at He temperatures, it is believed to be about 10 kG at 4.2°K. The second reason is that it was thought to be chemically stable at the high temperatures experienced during the sintering process. The average size of the ferrite particles was 1 micron. This mixture was then subjected to a pressure of 37,500 psi (2.6×10^3 bar) at a temperature of 1200°C for one-half hour under vacuum conditions. A qualitative experiment in which the sample was suspended between the poles of a large electromagnet indicated that the sample as a whole has a weak magnetic moment. This experiment was conducted at room temperature, and we expect that this moment would be larger at 4.2°K than at room temperature. Sample Nb_3Sn No. P. S. 3, which was to be a control sample, experienced the same fabrication sequence except that it did not contain the ferrite powder. The transition temperature of sample No. P. S. 3 was 17.59°K, while that of the ferrite-loaded sample No. P. S. 5 was 14.29°K. This decrease in T_c correlated with a decrease in the lattice constant⁴



(a)



(b)

Fig. 1. Photomicrographs of pressure sintered Nb_3Sn samples (1140X)
a) Sample No. P. S. 3
b) Sample No. P. S. 5

from 5.288 Å (for sample No. P. S. 3) to 5.279 Å (for No. P. S. 5). It appears that at the temperatures reached during the fabrication process a portion of the ferrite decomposed and reacted with NbO already present. The structures of these samples are shown in the photomicrographs in Fig. 1a and b. The chemical changes in samples No. P. S. 5 and the concomitant changes in physical properties prevent one from making a precise, quantitative comparison between the two samples; yet it is instructive to compare the critical-current density for the two samples as a function of temperature. This will be done in subsequent paragraphs.

2. Measurement of J_c as a Function of H and T

The critical current density, J_c , was measured by means of the Kim-⁵ type, tube magnetization experiment described in previous reports. J_c was measured in applied fields up to 30 kG and at temperatures ranging from 4.2°K to within a few degrees of T_c . The data are summarized in Figs. 2 and 3 for samples No. P. S. 3 and No. P. S. 5 respectively. Sample No. P. S. 5 exhibits unusual behavior at low values of applied field (e. g. , $H^* < 10$ kG at 4.2°K), namely J_c increasing with increasing H^* . At present we are not sure whether this is a pinning effect or a magnetization effect, i. e. , a result of the magnetizing of the ferrite particles by the applied field penetrating the sample. (The data for sample No. P. S. 5 shown in Fig. 3 are for H increasing. For H decreasing J_c exhibits conventional critical state behavior.) For $H^* > 10$ kG, sample No. P. S. 5 exhibits the expected inverse proportionality between J_c and H^* . Nearly all the data may be represented by an expression of the form $J_c H^* = \alpha$. The only exceptions are the data at 12.2°K for sample No. 5 where J_c falls off more rapidly than $1/H$. These data may be described by $J_c(H^* + B_0) = \alpha$.

3. Temperature Dependence of Pinning

The proportionality constant between the critical current density and the average field in the specimen is the critical state parameter, α . Anderson^{6, 7} has given the following expression relating α to more fundamental quantities

$$\alpha = \frac{JH}{c} = \frac{1}{l} \left\{ p \frac{H_{cB}^2 \xi_0^2}{8\pi \lambda^2} + \frac{kT}{\lambda^2 \xi_0} \log_e \left(\frac{R_c}{\omega_0} \right) \right\}$$

Where λ = the penetration depth (considered to be equal to the size of a flux bundle)

l = the average distance between pinning sites

p = the average fractional pinning

H_{cB} = the thermodynamic critical field

ξ_0 = the coherence length

R_c = the "hop rate" of the flux lines

ω_0 = the natural vibration frequency of the flux lines

α is strongly temperature dependent, through λ , H_{cB} and the explicit appearance of T in the second term of the above expression. Using values of α derived from our critical current data we have plotted α against the reduced temperature, t (Fig. 4), where

$$t = T/T_c.$$

Curves based on Anderson's expression have been fitted to the data for $t \lesssim 0.5$. These curves predict a near-linear approach of α to zero for $t \approx 0.7$.

Our data show a non-zero α at $t = 0.85$ and a much slower approach to zero. Anderson⁷ has stated that as H_{c2} is approached the lattice of flux lines may become more rigid, so that flux bundles may not be able to slip past each other. This would result in a decrease in the rate at which lines move past the barriers and a slower approach of α to zero. Cody⁸ and Cullen have also observed large critical currents in Nb₃Sn for $t > 0.8$.

We note in Fig. 4 that the curves $\alpha(t)$ of the two samples are similar, differing by a multiplicative constant. Since both samples are Nb₃Sn we are tempted to assume that the terms inside the bracket in Eq. 1 are the same for both samples and that α (sample No. P. S. 3) differs from α (sample No. P. S. 5) only because of the term, l . This is equivalent to saying that the pinning centers have about the same strength

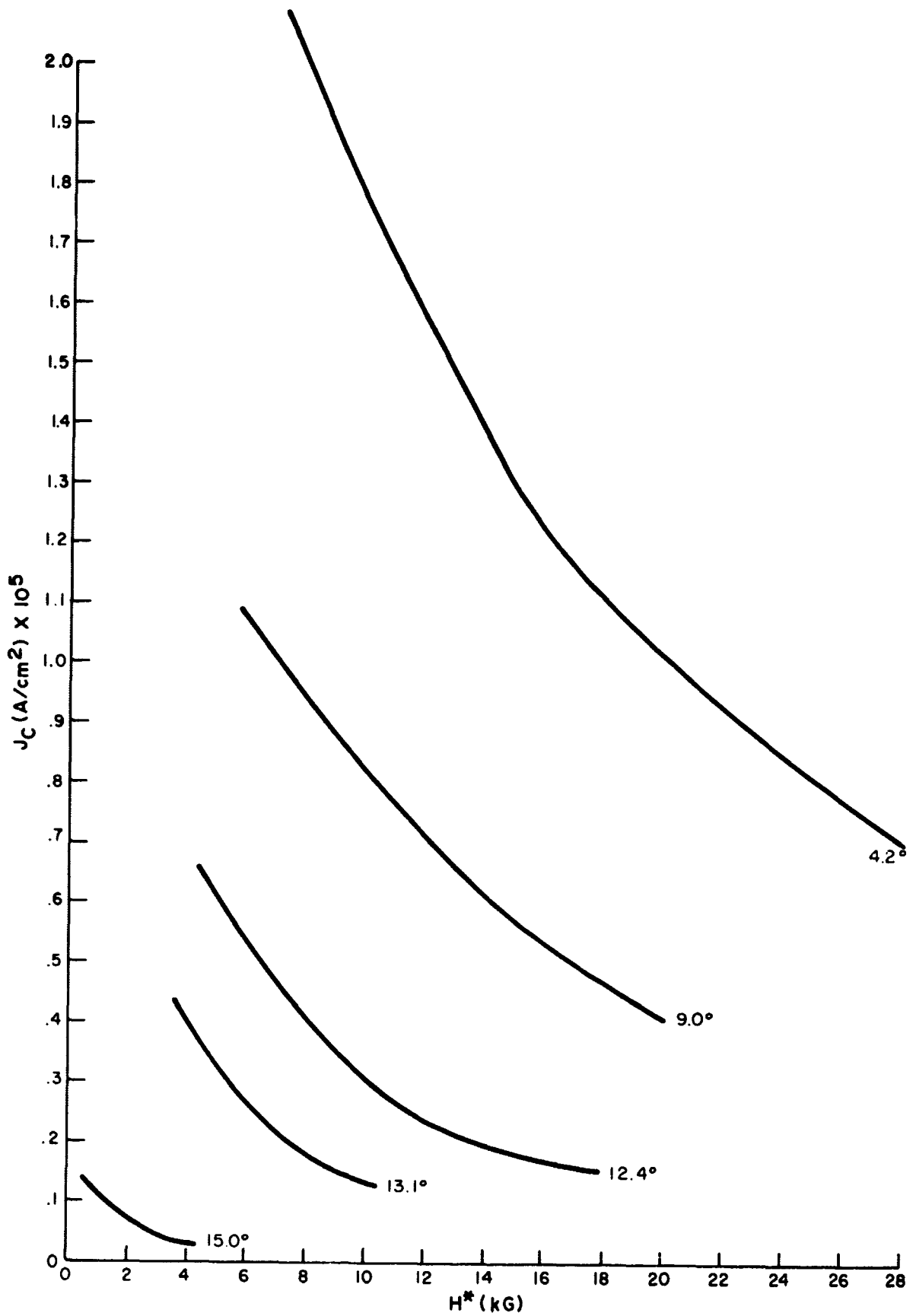


Fig. 2. Critical-current density, J_C , as a function of average magnetic field, H^* , and temperature. Sample No. P.S. 3.

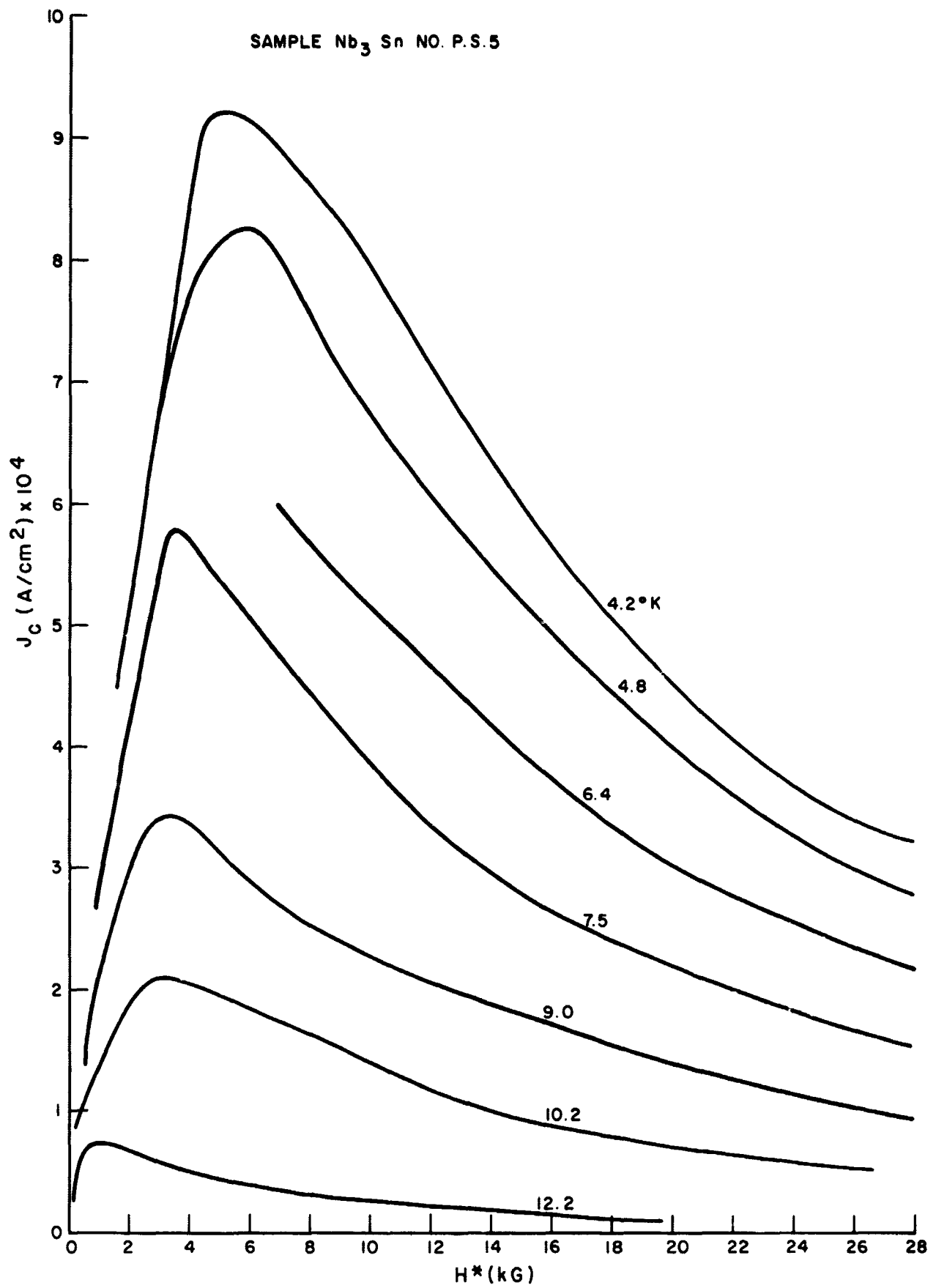


Fig. 3. Critical-current density as a function of average magnetic field and temperature. Sample No. P.S.5.

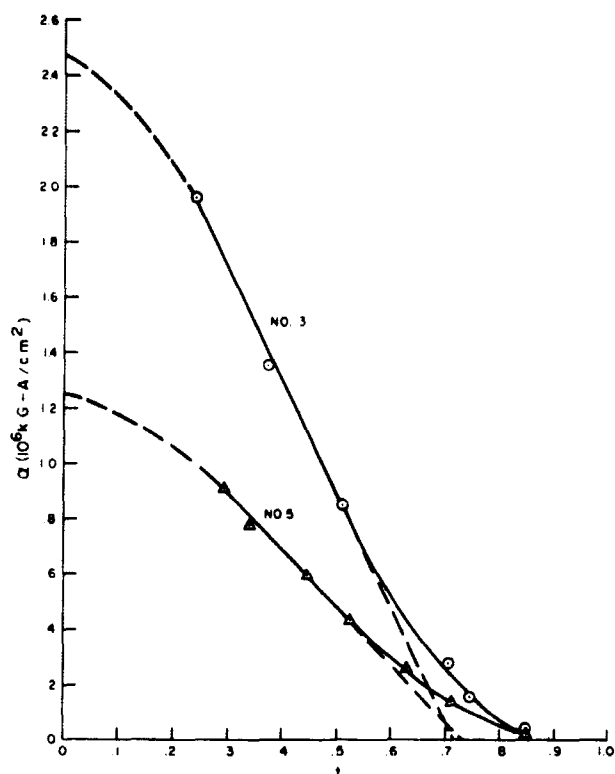


Fig. 4. Critical state parameter, α , as a function of the reduced temperature, t , for samples No. P. S. 3 and P. S. 5.

in each sample but in sample No. P. S. 5 they are farther apart by a factor of about 2. This is, of course, only approximate since the samples have different values of T_c and therefore of H_{cB} . Therefore pinning centers in sample No. P. S. 3 are almost certainly stronger, than those in sample No. P. S. 5.

This and the non-zero values of α for $0.7 < t < 0.85$ are worthy of further investigation.

B. EXPERIMENTS ON NbTi SPECIMENS

1. Results of Heat Treatment

We have been concerned with the manner in which heat treatment alters pinning sites in NbTi, as evidenced by the critical-current density and the threshold for ac

field-induced flux jumps. A sample of NbTi (No. 5a) which had been investigated thoroughly in the "as received" condition; i. e. , 70% cold worked, was subjected to a temperature of 400°C in a vacuum of 10^{-6} mmHg for 2.5 hours. The magnetization experiments in dc fields and in superimposed ac and dc fields were then repeated. Figure 5 shows the critical-current density as a function of the applied field for this specimen before and after the heat treatment, and it is apparent that the critical current density has been significantly increased by this treatment. Heat treatment is believed to cause titanium atoms to migrate and form small titanium particles of the proper size to serve as effective pinning sites.⁹ The α parameter, which describes the pinning strength, was 0.46×10^6 kG-A/cm² before heat treatment and 1.10×10^6 kG-A/cm² after, an increase by a factor of 2.4. This additional pinning strength is reflected in the threshold for the ac field-induced flux jumps. These thresholds are shown in Fig. 6. Note that, for a given magnitude of the dc field, the threshold after heat treatment is greater than that before heat treatment by a factor of slightly more than 2 for frequencies from 5 to 500 Hz. This is consistent with the increase in critical-current density. Since the flux jumps are caused by internal heating of a specimen by the dissipative motion of the flux lines, we expect that a mechanism which impedes the motion of the lines will reduce the power dissipation caused by a periodic field. Further, because of the higher critical-current density the field changes occur in a region closer to the surface of the specimen. Thus the power dissipated may be more easily conducted to the He bath.

2. Results of Copper Plating

We wished to observe the effects of a slightly modified surface on the magnitude and stability of the critical current density in NbTi. The modification was accomplished by means of a copper plating approximately 2×10^{-3} cm thick on both the interior and exterior surfaces of the samples. Two samples were studied - one in the "as received" condition and one which had been heat treated. Magnetization experiments were then performed in dc fields and in superimposed dc and ac fields.

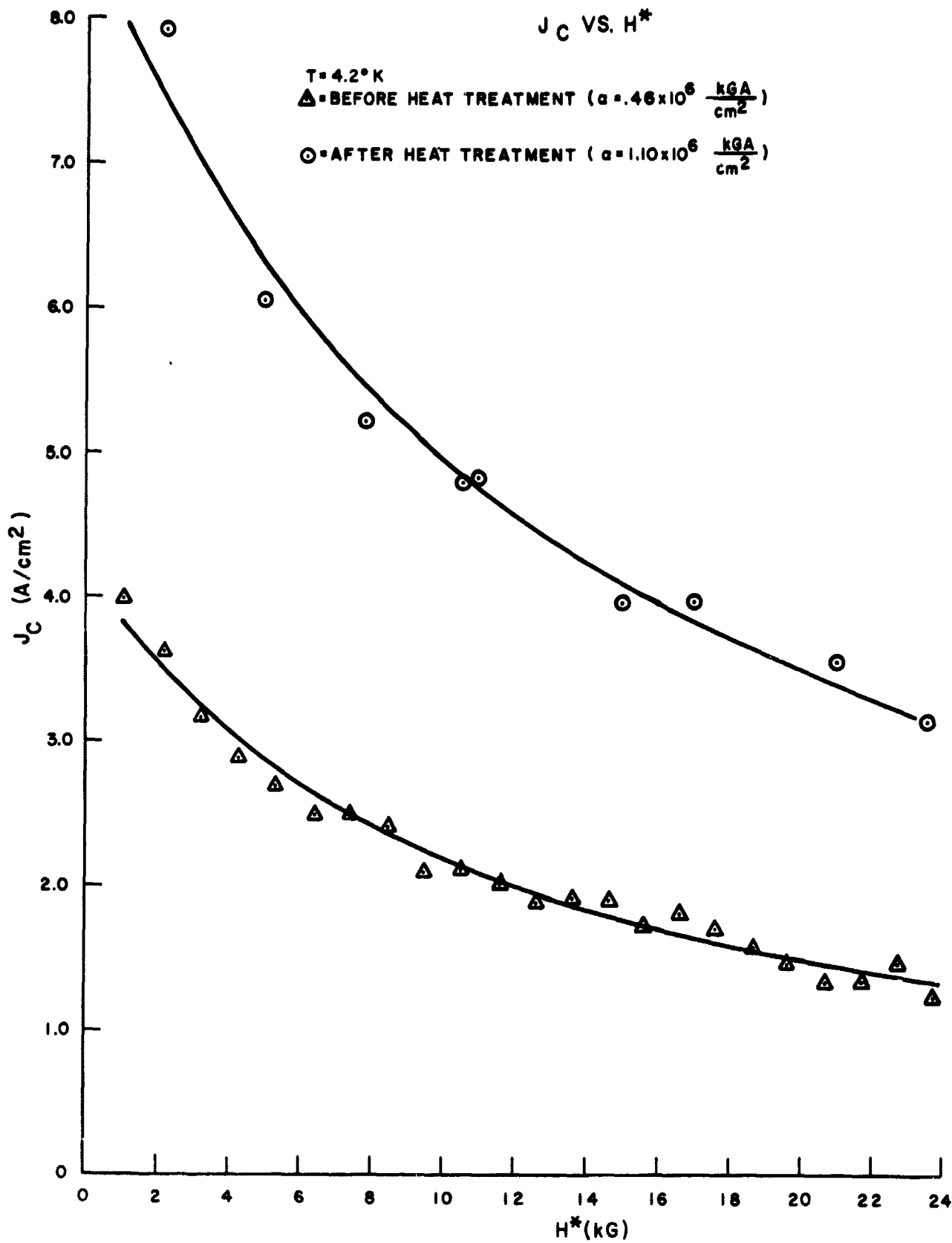


Fig. 5. Critical current density as a function of average magnetic field. NbTi Sample No. 5a.

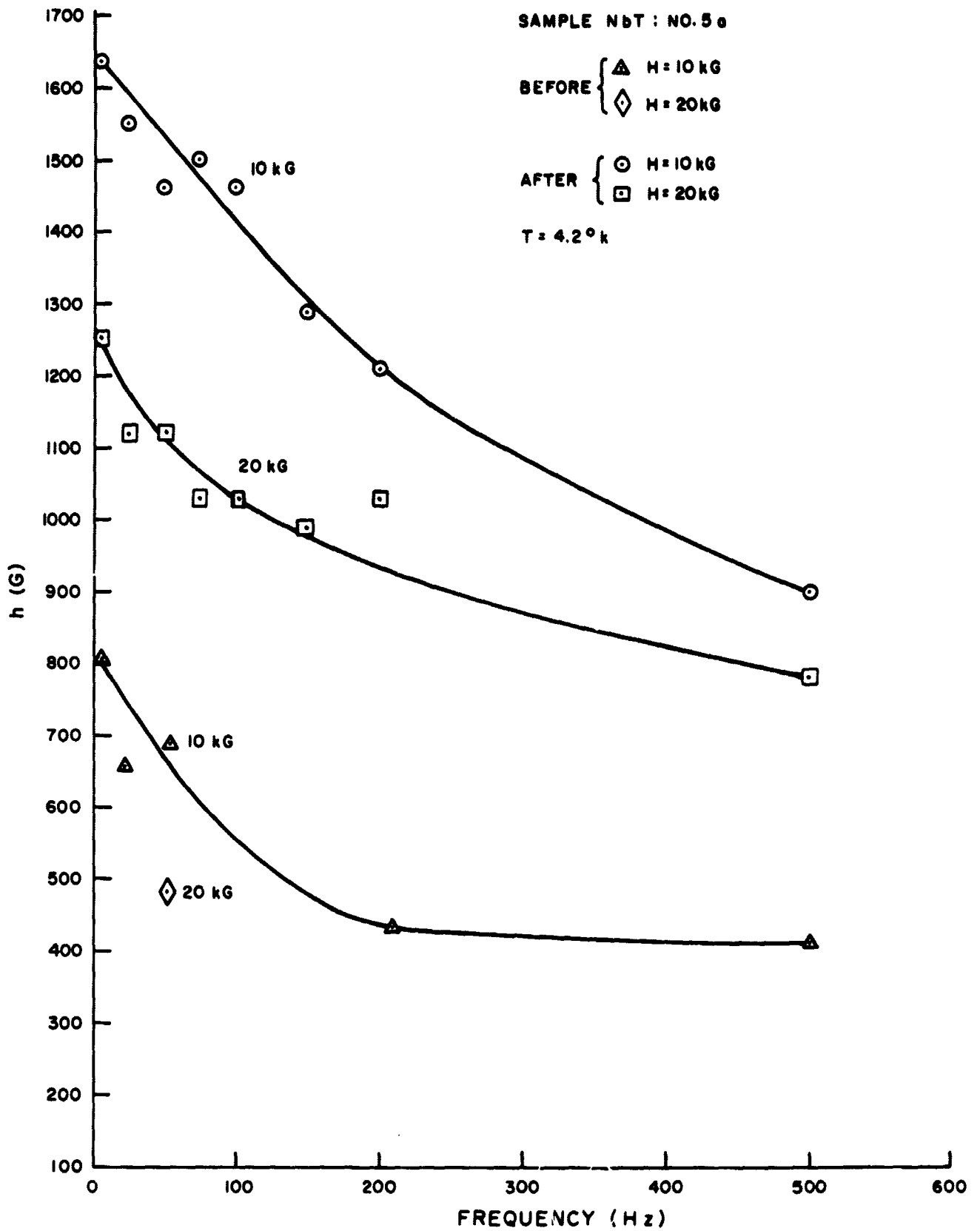


Fig. 6. Thresholds for ac field induced flux jumps. NbTi Sample No. 5a.

Analysis of the result indicated that neither sample underwent any change as a result of the copper plating. The magnitude and stability of the critical currents remained as they had been before the plating.

C. EXPERIMENTS ON NIOBIUM

We have investigated the manner in which an ac field causes flux jumps in a sample of type II material which is not a hard superconductor. The specimen was niobium,¹⁰ and it was measured in the "as received" condition, i. e. , cast then cold worked. The magnetization curve for the sample is shown in Fig. 7, and some critical-current densities derived from the magnetization curve are shown in Fig. 8. (H is the applied field and H' is the field in the interior of the specimen as measured by a Hall probe.) These critical-current densities do not follow a Kim-Anderson formula as do the critical currents in hard superconductors. In Nb the critical-current densities decrease much more rapidly with increasing H field.

The response of this sample to an alternating field is shown in the sequence of photographs in Fig. 9. In these photographs the upper trace represents the signal applied to the specimen while the lower trace is the signal received by the Hall probe in the interior of the specimen. As seen from Fig. 9a, when the sample is in the normal state ($H = 12$ kG and $>H_{C2}$), the ac field is transmitted through it unchanged. The magnitude of the signal reaching the Hall probe is identical (within the experimental error) to that created by the tickler coil, and the form of the signal is a pure sinusoid. No shielding action is occurring. The same conclusions may be drawn from Fig. 9b where $H = 10$ kG, which is still greater than H_{C2} . In Fig. 9c, taken at $H = 8$ kG, note that the signal received by the Hall probe is slightly flattened at the ends. Although the amplitude of this signal is equal (within experimental error) to that generated by the tickler coil, the flattening at the extremities indicates that some shielding is occurring. When the dc field is reduced somewhat below H_{C2} , appreciable shielding currents are possible as indicated in Fig. 9d (taken with $H = 6$ kG), where there is a pronounced flattening at the extremities of the signal. Note that during portions of the cycle, when the field

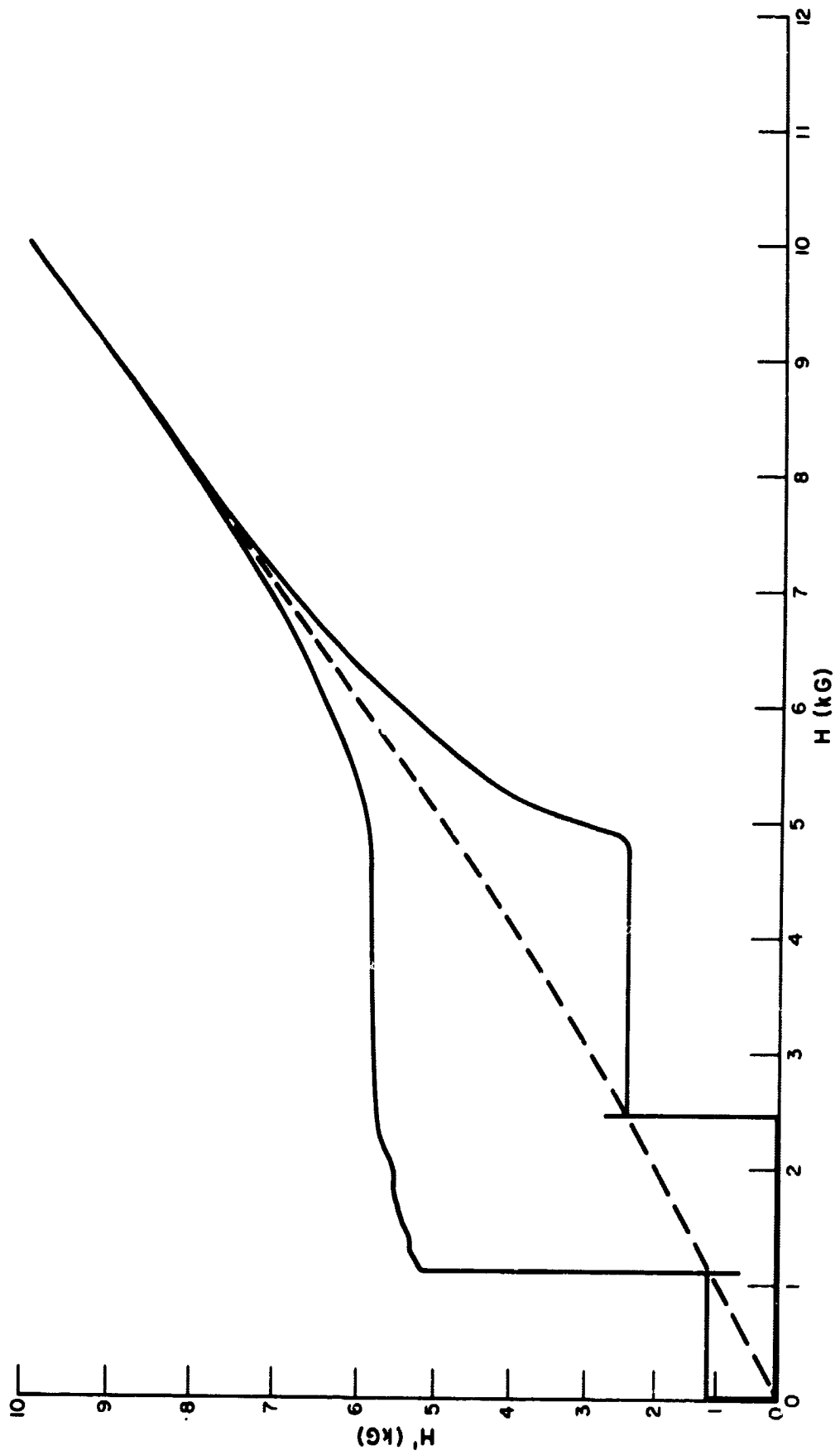


Fig. 7. Magnetization curve for pure niobium.

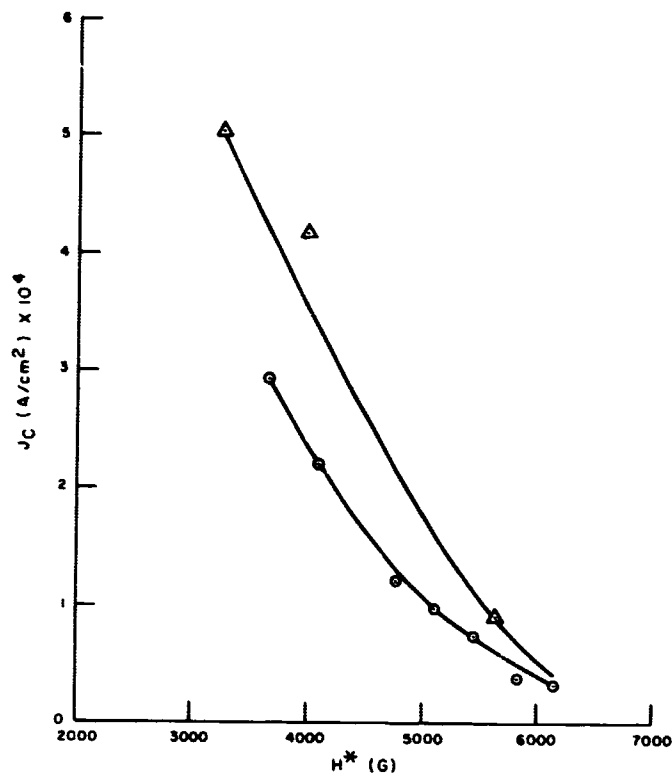


Fig. 8. Critical-current density as a function of average magnetic field for pure niobium.

within the sample is increasing or decreasing, $\frac{dh'}{dt}$ is equal to $\frac{dh}{dt}$ (see Reference 2 for a description of the manner in which the field inside the sample follows the critical-state curves during portions of the ac signal). The differing lengths of the plateau regions correspond to the differing intervals between the trapping and shielding curves at opposite extremities of the ac signal. Figure 9e shows that a signal equal in amplitude to that illustrated can cause one flux jump per cycle. Flux enters the specimen along the critical-state curve during the increasing portion of the ac cycle, but leaves by means of a flux jump during the decreasing portion. The smooth entrance of flux into the sample is occurring at a point where a relatively small shielding current can flow, while the flux jump occurs where a relatively much larger trapping current is possible. Apparently, during the increasing field portion of the cycle, the field is close enough to H_{c2} that little or no pinning occurs. If this is so then the pre-condition for a flux jump, viz. large numbers of flux lines prevented from moving by strong

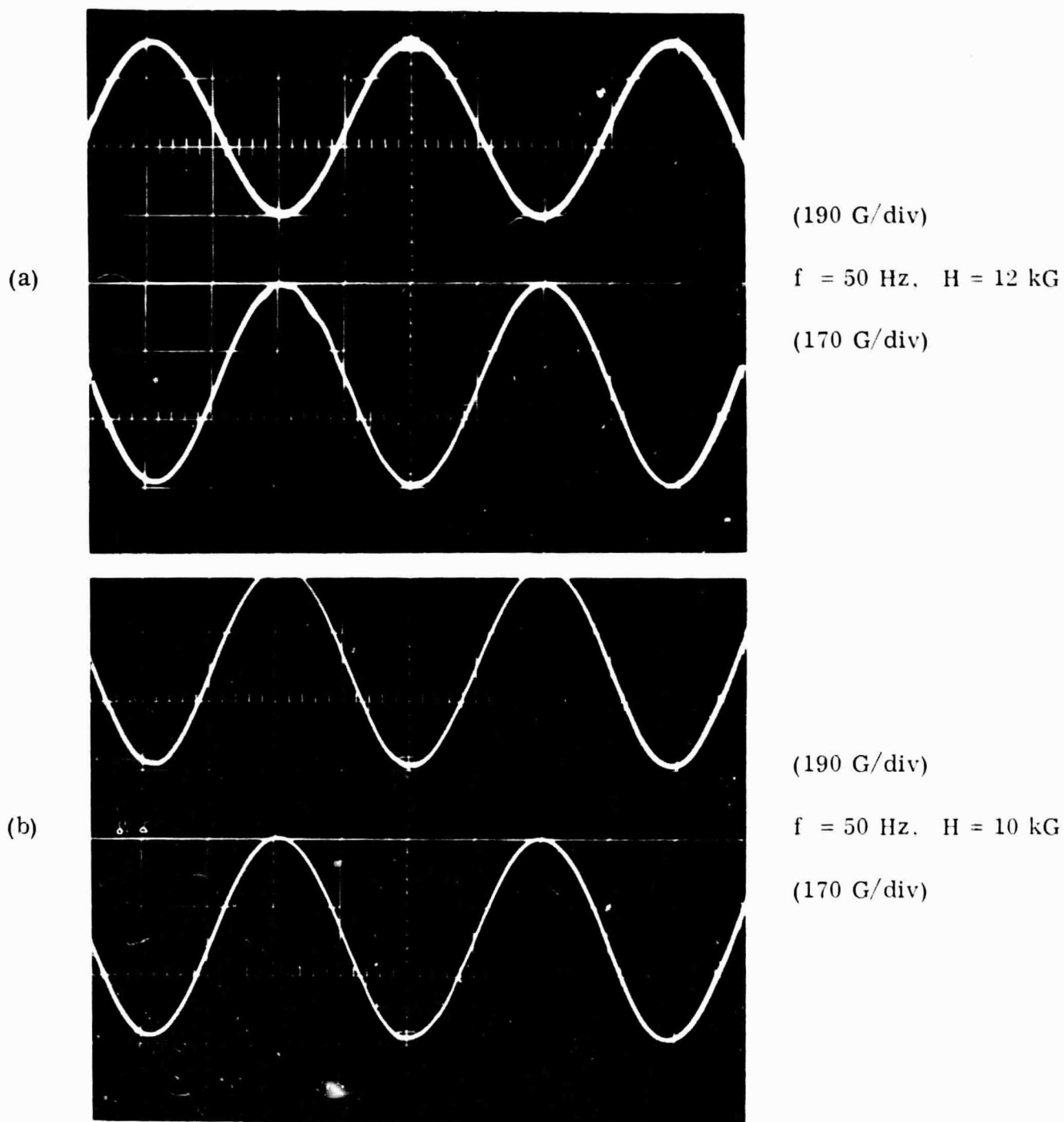


Fig. 9. Oscilloscope traces of the response of the pure niobium sample to an alternating field. ($T = 4.2^{\circ}\text{K}$)

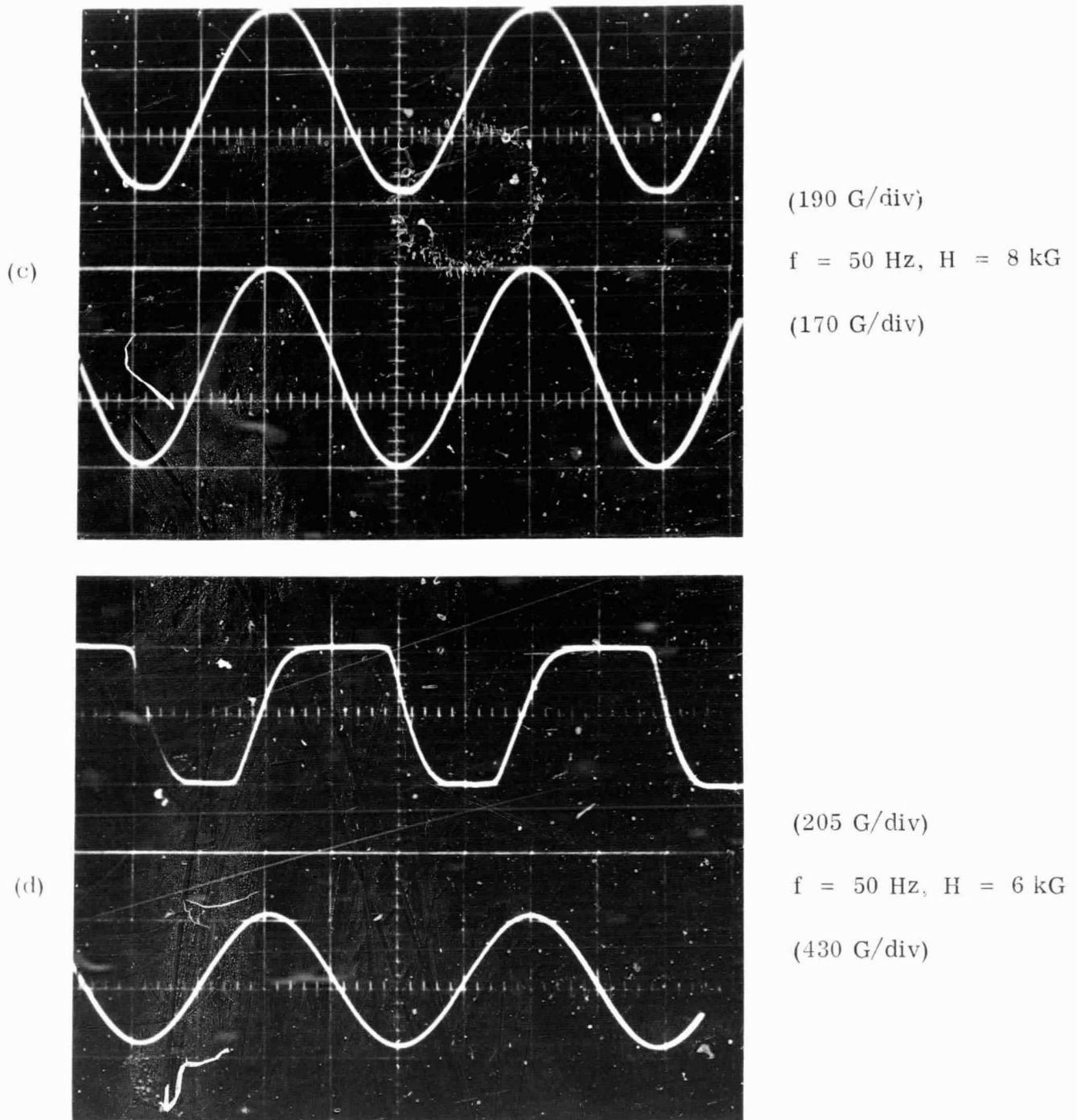


Fig. 9, (cont.) Oscilloscope traces of the response of the pure niobium sample to an alternating field. ($T = 4.2^\circ\text{K}$)

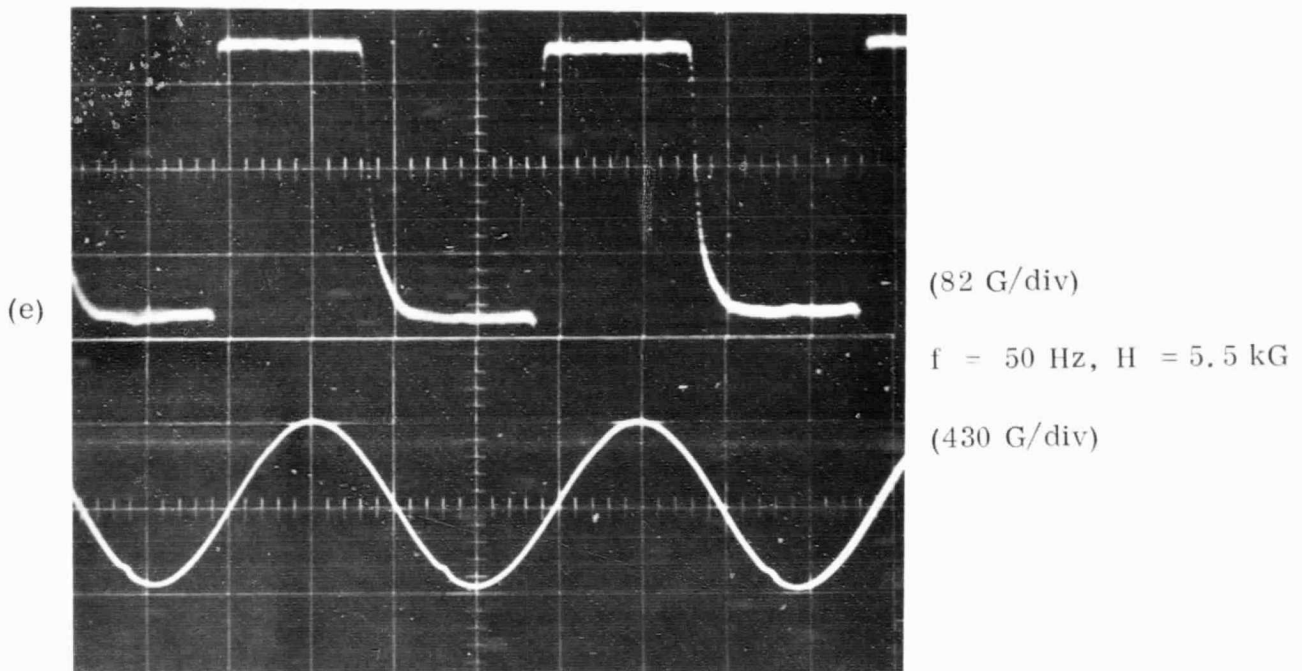


Fig. 9. (cont.) Oscilloscope traces of the response of the pure niobium sample to an alternating field. ($T = 4.2^\circ\text{K}$)

barriers, is not present. During the decreasing field portion of the cycle, H is sufficiently smaller than H_{c2} to permit appreciable pinning. Since large trapping currents can flow in this regime, an appreciable Lorentz force can build up. Another factor to be considered is the thermal conductivity which goes through a minimum for H slightly larger than H_{c1} , then increases as H approaches H_{c2} .¹¹ During the decreasing portion of the cycle the thermal conductivity of the sample is being reduced, thus making dissipation of heat generated by the motion of flux lines more difficult.

From a practical point of view the situation in this specimen is roughly comparable to that which exists in a hard superconductor at a temperature near T_c . Under these conditions the supercurrents are quite stable against flux jumps. Note that in Fig. 9d flux is reaching the interior of the specimen at a rate of about 10^5 G/sec, yet no flux jumps are occurring.

A plot of h , the threshold for ac field-induced flux jumps, as a function of frequency, is shown in Fig. 10. It is seen that $h(f)$ for Nb is similar to that for NbTi.

SAMPLE Nb NO. 2 (UNANNEALED)
T = 4.2°K
H = 5 kG

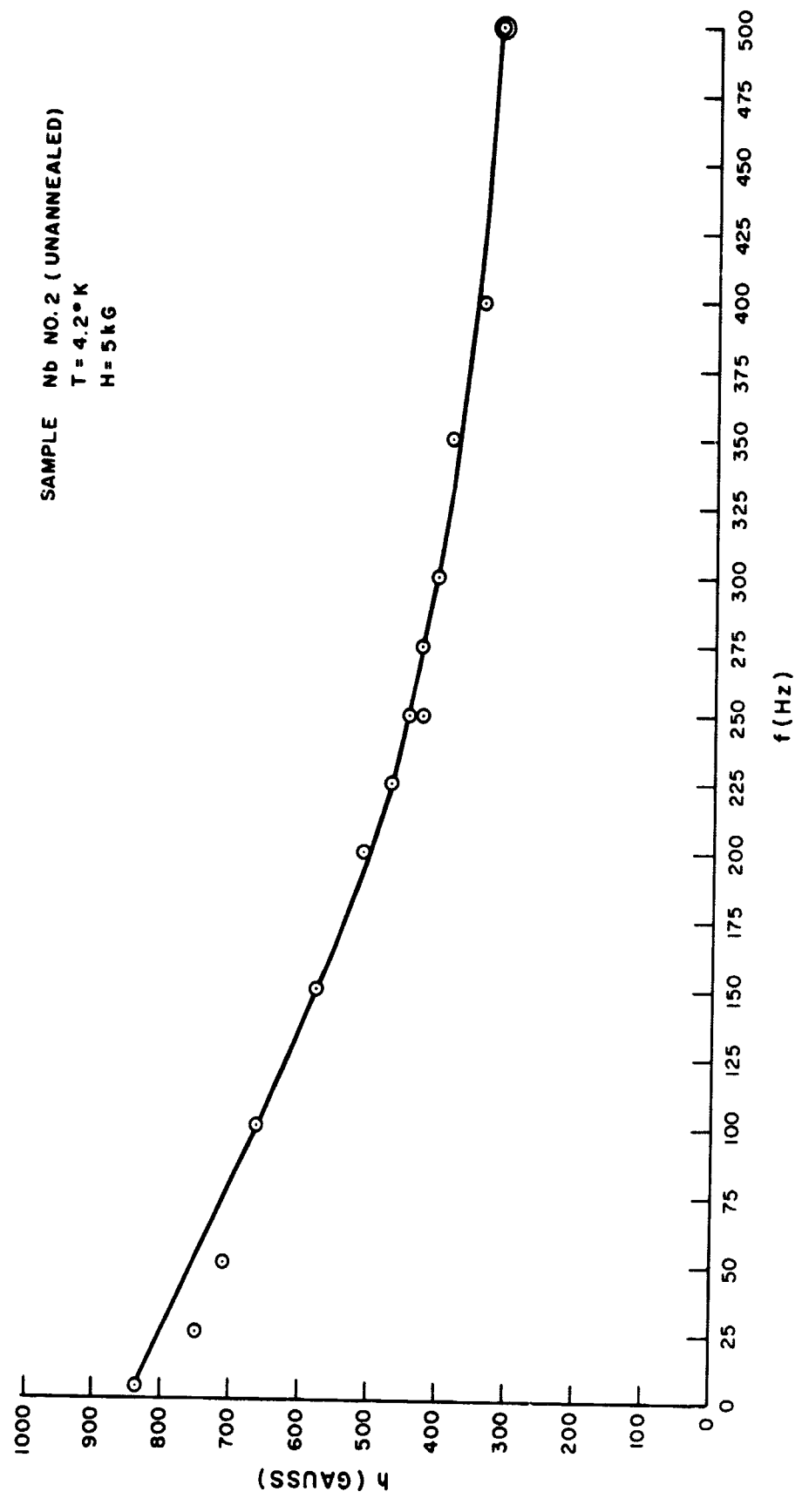


Fig. 10. Thresholds for ac field-induced flux jumps. Pure Nb.

SECTION III

CONCLUSIONS AND RECOMMENDATIONS

A. SUMMARY OF FINDINGS

Magnetization experiments on pressure sintered Nb_3Sn yielded critical state data for two samples, one of which contained 5% $\text{Zn}_{.5}\text{Mn}_{.5}\text{Fe}_2\text{O}_4$ as a "second phase" distributed throughout the Nb_3Sn . Values of the critical state constant, α , were obtained for both samples. For the pure sample, which had a transition temperature of 17.59°K, α at 4.2°K was 1.9×10^6 kG-A/cm². For the sample with the ferrite inclusions, which had a transition temperature of 14.29°K, α at 4.2° was 0.91×10^6 kG-A/cm². Chemical interaction between the ferrite and the Nb_3Sn lowered the transition temperature of the latter sample, thus preventing a quantitative comparison of the results for the two samples. Values of α were determined for several values of $4.2^\circ < T < T_c$. For both samples the dependence of α on the reduced temperature was similar. From the temperature dependence we inferred that the strengths of the pinning sites in the two samples were approximately equal, but that the sites were more numerous in the sample which did not contain the ferrite. In both samples, $\alpha(t)$ for large t approached zero more slowly than the theoretical curves fitted to the low t data. This may be of practical interest since it indicates that substantial current densities are possible at higher temperatures than expected.

Magnetization experiments were performed on samples of NbTi which were heat treated and copper plated. It was found that heat treatment (at 400°C for 2-1/2 hrs in a vacuum) increased α at 4.2°K by a factor of about 2.4. The heat treatment also increased the stability of the critical currents in the presence of an alternating field so that the threshold for ac field induced flux jumps was approximately doubled. A coating of copper 2×10^{-3} cm thick on the interior and exterior surfaces had no effect on the

magnitude or stability of the critical currents in either the heat-treated or cold-worked specimens.

Magnetization experiments in superimposed fields were performed on specimens of pure, cold-worked Nb. For H slightly less than H_{c2} the supercurrents were very stable against ac field-induced flux jumps. The specimen remained in the "critical state" while flux flowed into and out of it at a rate of about 10^5 G/sec. For H somewhat lower than H_{c2} , the same alternating field caused flux jumps. From this we might infer that pinning, as we usually understand it, does not occur close to H_{c2} . Thus the critical currents are not susceptible to the instabilities associated with flux bundles breaking loose from pinning sites. A similar situation possibly exists in hard superconductors at elevated temperatures ($t \gtrsim .7$), and this may account for the observations we discussed earlier, viz. appreciable critical currents at values of t where we expect α to be zero.

B. RECOMMENDATIONS FOR FURTHER INVESTIGATION

Our investigations have indicated a number of areas in which further expenditure of effort will lead to a better understanding of the physics of hard superconductors and to the improvement of these materials for device applications. Further experiments on specimens with composite structures (such as the Nb_3Sn sample discussed earlier) will enable us to determine the effectiveness of different types and concentrations of defect sites. High temperature studies, particularly for $0.7 < t < 1.0$, will shed light on the interaction between flux lines, a problem of fundamental significance in the understanding of type II superconductors. Studies in superimposed ac and dc fields also continue to be of interest. Experiments on samples with very thin walls may permit the measurement of a true critical-current density in alternating fields, which in turn would permit deeper understanding of the strength of pinning sites and the behavior of flux bundles in their presence. A variation on the ac field experiments we have performed, in which the sample is placed in the critical state, then exposed to small-amplitude fields of various frequencies and wave shapes, will help us to estimate the sizes of flux bundles.

We believe that further investigation in these areas will result in a better understanding of the relationship between the macroscopic critical-current density and the microscopic condition of the specimen. From this understanding should result hard superconductors with critical currents enhanced in magnitude and stability, and of greater utility in such applications as high-field magnets.

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