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SOME SUPERCONDUCTING PROPERTIES OF THE Ti-Nb-Ta ALLOYS

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ABSTRACT

Superconducting properties of a ternary system, Ti-Nb-Ta, were measured. Critical temperatures were found to decrease with increasing amounts of Ta in the alloys from those of Ti-Nb binary alloys. However, a broad plateau of high critical fields (above 120 kG) in the regions around the composition (63 at.% Ti-31 at.% Nb-6 at.% Ta) was observed. This plateau is discussed in relationship with the superconducting paramagnetism which is predicted by theories to increase critical fields. A preliminary investigation of critical current densities of some alloys is also reported.

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I. INTRODUCTION

Although superconducting critical magnetic fields of solid-solution alloys of transition elements[1] are known to be lower than those of intermetallic compounds such as Nb_3Sn[2] and V_3Ga[3], solid solution alloys are useful for many practical applications because they can easily be made into wires and wound into coils. At present the highest critical field in binary solid-solution alloys is found in the Ti-Nb alloy system at approximately 57 a/o Ti-43 a/o Nb.[1,4] The onset critical field of this alloy is approximately 115 kG[4] at 4.2°K. The maximum critical field of this alloy is limited by the Pauli paramagnetic susceptibility.[5,6] Maki and Tsuneto[7] have discussed the effect of the Pauli paramagnetism on superconductors. They also have pointed out that the spin orbit interaction possibly increases the effective paramagnetic susceptibility of the mixed state and thus enhances the upper critical fields of alloys. This prediction was further investigated theoretically[8,9] and experimentally.[10,11] Neuringer and Shapira[10] have systematically investigated the H_{c_2} of three transition element alloys, Ti-58 at.% V, Ti-44 at.% Nb, and Ti-52 at.% Ta. They found a systematic trend; the higher the atomic number of the group-V constituent.

$$[V(23) \rightarrow Nb(41) \rightarrow Ta(73)],$$

the greater the spin-orbit interaction effect, as Maki had predicted.

With these facts in mind, we have investigated the possibility of increasing critical fields of available ductile alloy superconductors. The system we have chosen is the Ti-Nb-Ta ternary alloy system in which a single body centered cubic phase exists at elevated temperatures. Over a wide range of compositions, this phase may be easily retained at room temperature and below. Of the two binary systems Ti-Ta and Ti-Nb, the latter contains the alloy having the highest critical field. It seems possible that the addition of Ta, which has a higher atomic number than Ti and Nb, but which is approximately the same size, could increase critical fields of Ti-Nb alloys, providing that the additions of Ta did not reduce the critical temperatures and normal resistivities appreciably. The effect of Ta on the critical current density is also of interest.

II. SAMPLE PREPARATION, THE PROPERTIES MEASUREMENTS, AND EXPERIMENTAL RESULTS

A. Material Preparation

All alloys were prepared in a cold mold arc furnace under an argon atmosphere and were turned over and remelted several times to insure homogeneity. To eliminate coring, all except a few high Ti alloys were homogenized at 1200°C in a vacuum better than 10^{-5} Torr for 2 hours. No coring was detectable after this treatment. The alloys were then cold rolled to approximately 30 mils in thickness (typically 90% reduction in thickness) and critical field test specimens were cut from the rolled sheets.

The starting materials were 1/8" rods of Nb (Kawecki Chemical Co.) and Ta (Fansteel Metallurgical Corp.), which were swaged to approximately 45 mils, and iodide process crystal bar Ti (Foote Mineral Co.). Interstitial impurity contents (O, N, and C) of starting materials and a typical alloy are given in Table 1.

Compositions of several alloys were measured by an electron microprobe analysis method and it was shown that nominal compositions and the compositions of alloys by a microprobe analysis were in agreement with a few percent. For the microprobe analysis, an Electron Microprobe Analyzer (Model 1400 Material Analysis Co.) was used. Computer calculations were employed to reduce the measured data following "Computer Programs for Electron Microprobe Data Processing" by Frazer, Fitzgerald and Reid.[12] This program accounts for the absorption and fluorescence corrections, but not for the atomic number correction. The atomic number effect usually makes the intensity ratios lower when a high atomic number element is determined in the presence of a lower atomic number element; the converse is also true.[13] Since we have observed that the concentrations of Ti by the microanalysis are slightly higher than the nominal concentrations, we can attribute a part of the difference between the nominal concentrations and the microanalysis results to the atomic number effect.

In the high titanium alloys, a second phase (hexagonal α titanium) was detected by x-ray diffraction. It was found that those alloys containing more than about 75 at.% Ti had detectable amounts of this hexagonal phase. These alloys are indicated by circles in the critical temperature plot of the ternary alloy system (Fig. 1).

For critical current measurements strips of cold rolled alloys were wrapped in high purity niobium foils and encapsulated in quartz tubing at a pressure lower than 2×10^{-6} Torr for heat treatment. After specimens were heated for specified periods of time, they were quenched in ice water and then the quartz capsules were broken.

B. Superconducting Property Measurements and Results

Superconducting transition temperatures, T_c, of alloys were measured by recording changes of the self-inductance of a copper wound coil on an X-Y recroder. Temperature variation was controlled by changing helium gas pressure on a double-wall stainless steel can which was placed in a liquid helium storage dewar. Temperature was varied slowly enough to avoid hysteresis in the self-inductance variation during cooling and warming about the critical temperatures. The germanium thermister used to measure temperature had an accuracy of better than ±0.1°K.

The critical temperatures of the alloys are shown in Fig. 1. In order to approximately complete the map of T_c, results from binary alloy systems[14-16] are incorporated. As is shown in the figure, the highest critical temperature of the alloy system was found to be 10.1°K for 30at.% Ti-70 at.% Nb. This value is slightly higher than that obtained by Hulm and Blaugher.[16] The difference may be attributed to possible differences in impurity contents. The general trend of decreasing critical temperatures by additions of Ta to Ti-Nb alloys was expected because Ta has a much lower critical temperature than Nb. The critical temperatures shown on the ternary diagram are the temperatures at which the self inductance of the measuring coil became half of a full transition value. The widths of the transitions were generally two to three-tenths of a degree. The effect of homogenizing the alloys was to narrow the transition widths. However, the half inductance point temperatures did not change more than one-tenth of a degree as a result of the homogenization.

Superconducting critical magnetic fields and critical current densities were measured in pulsed magnetic fields at 4.2°K. The rise time of the magnetic field was approximately 10 milliseconds for a maximum magnetic field of 130kG. The procedure is very similar to one described by Berlincourt and Hake.[1] It involved pulsing the magnetic fields while maintaining a constant current in a specimen and taking pictures of the traces of the magnetic field and the specimen voltage drop simultaneously on a dual beam oscilloscope with a highspeed Polaroid camera. The results of critical field measurements are plotted in Fig. 2. To approximately complete the plot, the critical field data for Ti-Ta alloys[17] and Ti-Nb alloys[4] are included.

A few points should be mentioned with regard to the critieria used in the reduction of data on critical field measurements. In Fig. 3, two critical fields, H_{cS} and H_{cN} are schematically illustrated as the beginning of the transition field and as the end of the transition field, respectively. In the ternary diagram of critical fields (Fig. 2), we have used H_{cN} at a current density of specimens approximately 30 amps/cm^2. It might be argued that the field which restores half of the normal state resistance or the field at the onset of the transition should be chosen as the resistively measured critical field instead of H_{cN}. However, both the onset and the half resistance critical fields will depend on pinning of magnetic flux lines since the width of the transition varies with strength of the pinning. Although H_{cN} might be slightly higher than the real critical field, $H_{c_2}(t)$ it seems to be the most consistent criterion to use in making a map of critical fields from pulsed magnetic field data for this ternary alloy system. Also, a very little heating effect on H_{cN} is observed at the measuring current density (30 amp/cm^2). A comparison of critical fields measured in a steady field* and in a pulsed field has been made with Westinghouse Ti-Nb wire (HI-120) and is shown in Fig. 4 as a part of a critical current density vs applied magnetic field plot. The steady field value is the magnetic field for the onset of the resistance. As it is shown, the agreement between the two measurements of critical fields is quite good considering the fact that the error in taking data

*The steady field results of the Westinghouse wire (HI-120) were measured in the MIT National Magnet Laboratory by one of the authors (K.M.R.) while he was at MIT.

from an oscilloscope could be as much as 3%. Also, if a criterion, which uses the half resistance transition field, is used for the steady field value of the upper critical field,[18] the choice of H_{cN} is a better agreement with the steady field value than value than is H_{cS}. However, the arbitrary nature of this choice, H_{cN} as the critical field, should be borne in mind.

Although critical current densities in pulsed magnetic fields are lower by almost an order of magnitude at high current densities than steady field values,[19] a qualitative variation of the currents can be investigated in pulsed magnetic fields.[20] In addition, having a comparison of two measurements, such as in Fig. 4, it is possible to obtain rough approximations of the possible steady field critical current densities for an alloy tested in pulsed fields.

A couple of alloys with high critical fields were heat treated at 500°C for 1, 4, 10 and 40 hours, and a variation of critical current density with heat treatment was investigated. For both of the alloys tested, the critical current densities stayed relatively constant or increased slightly for short aging times in comparison with those of the cold rolled alloys. Further increases in aging times resulted in appreciable decreases in critical current density. A typical critical current density curve for alloys which were cold rolled and aged 4 hours at 500°C is shown in Fig. 5.

III. DISCUSSION AND CONCLUSIONS

A. Critical Magnetic Fields

As indicated in Fig. 2, a relatively flat plateau of the critical field, H_{cN}, higher than 120 kG is found around an alloy composition 62 at.% Ti-31 at.% Nb-6 at.% Ta, although critical temperatures of those alloys are lower than respective Ti-Nb binary alloys with the same e/a ratio.[21] This seems to indicate that the increased critical fields by the substitution of Ta for Nb in Ti-Nb are due to the spin orbit scattering effect. To illustrate such a possible effect, parameters, which affect critical fields such as the normal resistivity and critical temperature and measured and theoretical critical fields are plotted in Fig. 6 for an approximately equi-electron to atom ratio (e/a) line or approximately constant atomic percent titanium (64 at.%) series. Full superconducting transitions are shown with transition widths as well as the half resistance fields. The critical temperature and the normal resistivity decrease monotonically from the niobium rich side to the tantalum-rich side while the critical field exhibits a peak value near a composition 64 at.% Ti-30 at.% Nb-6 at.% Ta. To attribute the observed peak to the spin orbit scattering effect, we summarize the theoretical developments on the limiting critical fields and discuss qualitatively our experimental results in comparison with the theories.

According to the GLAG theory,[22-25] as modified by Maki[9], the upper critical field of a Type II superconductor is given by

$$H^*_{c_2}(t) \cong 25,800 \frac{\kappa_1(t)}{\kappa} (1 - t^2) \rho_N \gamma T_c \qquad (1)$$

where $\kappa_1(t)/\kappa$ is a temperature dependent function derived by Maki,[9] ρ_N is a normal state resistivity, γ is the electronic specific heat coefficient, and $t = T/T_c$. This relation is applicable when normal state Bauli paramagnetism is negligible. Clogston[5] and Chandrasekhar[6] independently pointed out the importance of normal state Pauli paramagnetism on the upper critical fields. The limiting value of critical field at absolute zero due to the paramagnetic effect is given by[5]

$$H_p(0) \cong 18,400\ T_c \qquad (2)$$

The paramagnetic effect on the critical fields has been observed in many cases.[26] Various theoretical formulations combining these two limiting critical fields have been developed.[7-9,27] The critical field, including the paramagnetic effect, is expressed as[9]

$$H_{c_2}^{+}(0) = 3.1 \times 10^4\, \rho_N\ \gamma\ T_c\ (1+\alpha^2)^{-1/2} \qquad (3)$$

for t = 0 where $\alpha = \sqrt{2}\ H_{c_2}^{*}(0)/H_p(0)$.

As mentioned before, Maki and Tsuneto[7] also predicted that the effect of the spin orbit scattering might be to increase H_{c_2} by reducing the effect of normal state paramagnetism. More detailed theoretical developments which include the spin orbit scattering effect on the critical field have been presented by Werthamar, et al.[8] and Maki.[9] Maki's expression for the critical field can be rewritten as,

$$H_{c_2}(t) = 2H_{c_2}^{*}(t) \left[1 + \left(1 + \frac{2\ H_{c_2}^{*}(0)\ H_{c_2}^{*}(t)}{1.78\ \lambda_{so}\ H_p^{2}(0)}\right)^{1/2}\right]^{-1} \qquad (4)$$

or in terms of γ, ρ_N, T_c and λ_{so}

$$H_{c_2}(t) = \frac{5.16 \times 10^4\ \frac{\kappa_1(t)}{\kappa}\ (1-t^2)\ \rho_N\ \gamma\ T_c}{\left\{1 + \left[1 + \frac{1.77\ \frac{\kappa_1(t)}{\kappa}\ (1-t^2)\ \rho_N^{2}\ \gamma^2}{\lambda_{so}}\right]^{1/2}\right\}} \qquad (5)$$

where λ_{so} is Werthamer's spin orbit coupling induced spin flip scattering parameter[8] given by

$$\lambda_{so} = \frac{\hbar}{3\pi k_B\ T_c\ \tau_{so}}$$

and τ_{so} is the mean spin orbit coupling induced spin flip scattering time. Unfortunately, there is no reliable method for evaluating τ_{so} for these alloys at the present time. However, from Abrikosov and Gorkov's[28] treatment of spin orbit scattering, Neuringer and Shapira[10] expect that

$$\frac{\tau_{tr}}{\tau_{so}} \sim \frac{Ze^2}{\hbar c}^4 \tag{6}$$

where τ_{tr} is the mean transport collision time. Such a relationship is helpful in understanding of the variation of $H_{c_2}(t)$ with atomic number, Z.

In the following we compare our experimental results for constant 64 at.% Ti alloys with the theoretical developments mentioned above. It is possible, however, to make only a qualitative comparison of the experimental results and the theories since values of γ and τ_{so} for the alloys are not known and are difficult to approximate. Nevertheless, we can calculate $H_{c_2}^*(t)$, utilizing Eq. (1), and $H_{c_2}^+(0)$, utilizing Eq. (3), with measured values of ρ_N and T_c (Fig. 6) and taking $\gamma(\approx 10 \times 10^3$ erg $°K$ $cm^{-3})$ to be constant for this alloy series.[29] This value for γ is consistent with the results of Neuringer and Shapira[10] as is our assumption that it is approximately constant for the 64 at.% Ti alloys. According to Eq. (1) and (3) the decreases which are observed for ρ_n and T_c, due to substitution of Ta for Nb in a 64 at.% Ti - 36 at.% Nb alloy, should cause a similar decrease in critical field. As shown in Fig. 6, the calculated critical fields have a different compositional dependence than does the measured resistive critical field, H_r (4.2°K), which lies between the calculated fields. It is noteworthy that, for alloys containing more than 12 at.% Ta, $H_{c_2}^*$ (4.2°K) and H_r (4.2°K) are parallel and nearly equal. This behavior, interpreted in terms of Eqs. (4) and (5), indicates that λ_{so} is sufficiently large so as to reduce the effect of the Pauli paramagnetism almost to zero such that Eq. (5) reverts to Eq. (1), in agreement with the observations of Neuringer and Shapira.[10]

For Nb-rich alloys in the 64 at.% Ti series, H_r (4.2°K) falls well below the calculated $H_{c_2}^*$ (4.2°K), indicating that spin orbit scattering is less effective, as would be expected from Eq. (6), because of niobium's lower atomic number. An interesting aspect of the observed behavior is the rapid increase of H_r (4.2°K) as Ta is substituted for Nb in 64 at.% Ti - 36 at.% Nb. Relatively small amounts of Ta apparently are quite effective for spin orbit scattering. The spin orbit scattering parameter λ_{so} can be approximately evaluated with Eq. (5), although the value obtained are valid only for Nb rich alloys (Table II) where $H_{c_2}(0)$ and H_{c_2} (4.2°K) differ significantly (Fig. 6). The value of λ_{so} for 36 at.% Nb - 64 at.% Ti is smaller than the values (4.5 and 1.5) for 44 at.% Nb - 56 at.% Ti obtained by Neuringer and Shapira,[10] and Werthamen et al.,[8] respectively. However, considering the approximate nature of the equation and values used to calculate λ_{so}, it is thought that the values of λ_{so} for the present alloys are consistent with previously obtained values.

For purpose of comparison, we include values for the spatial average of Z^4 (cf. Eq. (6)) normalized to coincide with λ_{so} for 64 at.% Ti - 36 at.% Nb. The variations of λ_{so} and $\langle Z^4 \rangle$ are similar; the difference is attributed the approximate nature of the calculated λ_{so} and to the approximate validity of Eq. (6). Nonetheless, the qualitative variation of λ_{so} is in line with the above arguements concerning the effectiveness of Ta in spin orbit scattering.

From the discussion above it seems likely that the plateau observed in the critical field plot of the ternary alloy system (Ti-Nb-Ta) is attributable to the increased superconducting state paramagnetism in the alloys provided by addition of Ta to the binary Ti-Nb alloys. To confirm the effect in this alloy system, it would be necessary to make more detailed measurements of superconducting properties and other related parameters and the results should be compared with the theories of Maki[9] and Werthamer et al.[8]

B. Critical Current Densities

The critical current densities can be varied substantially by changes in the metallurgical processes of the alloys. Such processes will change the pinning conditions of magnetic flux lines. The heat treatnments used were such as to promote the formation of fine hexagonal (α) phase precipitates. However, the decrease in critical current densities indicates that the α phase precipitates formed were not as effective as dislocation networks formed during cold rolling in pinning flux lines. Also, the oxygen content of the alloys is too low to give appreciable enhancement of critical current densities with the heat treatment employed. A larger oxygen content (approx. 2000 ppm) was shown to be necessary to increase critical current densities by heat treatments for a similar alloy.[31]

Comparing these results (Fig. 5) with those shown in Fig. 4, we see that the critical current densities of the former are lower than those of the latter. However, effects of heat treatment on critical current densities in excess of 2.6×10^5 amp/cm^2 at 40 kG can be attained after suitable aging.[32]

Hence it may be possible to obtain higher critical current densities for these alloys with more suitable treatments, combining, for example, the controlled addition of oxygen and combinations of cold working and heat treatment.

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Table I. Chemical analysis of gaseous impurity contents of starting elements and a typical alloy (ppm).

	C	N	O
Nb*	45	< 10	60
Ta*	10	20	33
Ti*	10	20	20
37w/oTi-50w/oNb-13w/oTa**	60	50	240

* Chemical analyses were supplied by suppliers of those metals.

** The chemical analysis of the alloys was done by Coors Spectro-Chemical Laboratory, Golden, Colorado.

Table II. Calculated spin orbit scattering parameter, λ_{so} for niobium rich 64 at.% titanium alloys.

Composition,								
	at.% Ta	0	2	4	8	10	12	18
	at.% Nb	36	34	32	28	26	24	18
λ_{so}		0.6	1.0	1.3	2.5	4.2	7.9	18.2
Relative $\langle Z^4 \rangle$		0.6	.9	1.1	1.7	1.9	2.2	3.0

FIGURE CAPTIONS

Fig. 1. Superconducting critical temperatures (°K) of the Ti-Nb-Ta alloys (atomic per cent composition scales.)

Fig. 2. Superconducting critical magnetic fields, H_{cN}, of the Ti-Nb-Ta alloys at 4.2°K (atomic per-cent composition scales and magnetic fields in kG). X . . . W.DeSorbo, et al.[21] (pulsed fields) and □ . . . Westinghouse Nb-Ti (steady field).

Fig. 3. Schematic diagram of a superconducting to normal state transition to illustrate the criteria used to obtain experimental critical fields, H_{cS} and H_{cN}.

Fig. 4. Plots of superconducting critical current densities vs. applied magnetic field for a Ti-Nb alloy wire (Westinghouse HI-120).
●. . . steady magnetic field measurements
△,▲. . . pulsed magnetic field measurements.

Fig. 5. Plot of superconducting current densities vs. applied magnetic field for an alloy, 63.8a/o Ti - 32.5 a/o Nb - 3.7 a/o Ta, which was cold rolled and then aged for 4 hr. at 500°C.

Fig. 6. Data for Ti-Nb-Ta alloys containing 64 at.% Ti. Top: Critical temperatures, T_c, and normal resistivities, ρ_N at 4.2°K. Bottom: Experimental critical magnetic fields, H_r (4.2°K), identified with H_{c_2} (4.2°K), Eq. (4); theoretical critical magnetic fields, $H_{c_2}^*(t)$, Eq. (1) and $H_{c_2}^+(t)$ Eq. (3).

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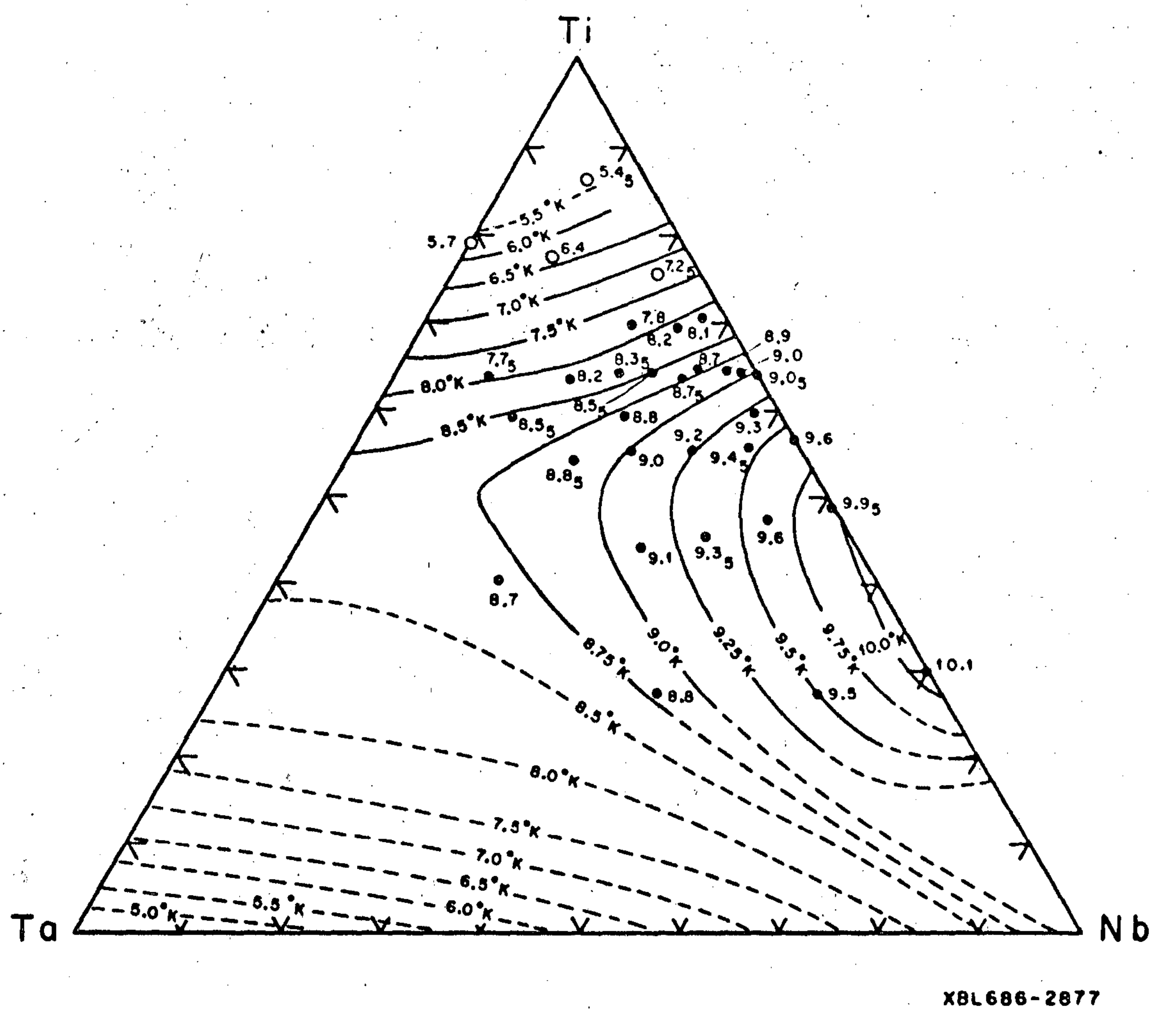

Ti
Ta
Nb
5.5°K
6.0°K
6.5°K
7.0°K
7.5°K
8.0°K
8.5°K
8.75°K
9.0°K
9.25°K
9.5°K
9.75°K
10.0°K
5.0°K
XBL686-2877

Figure 1

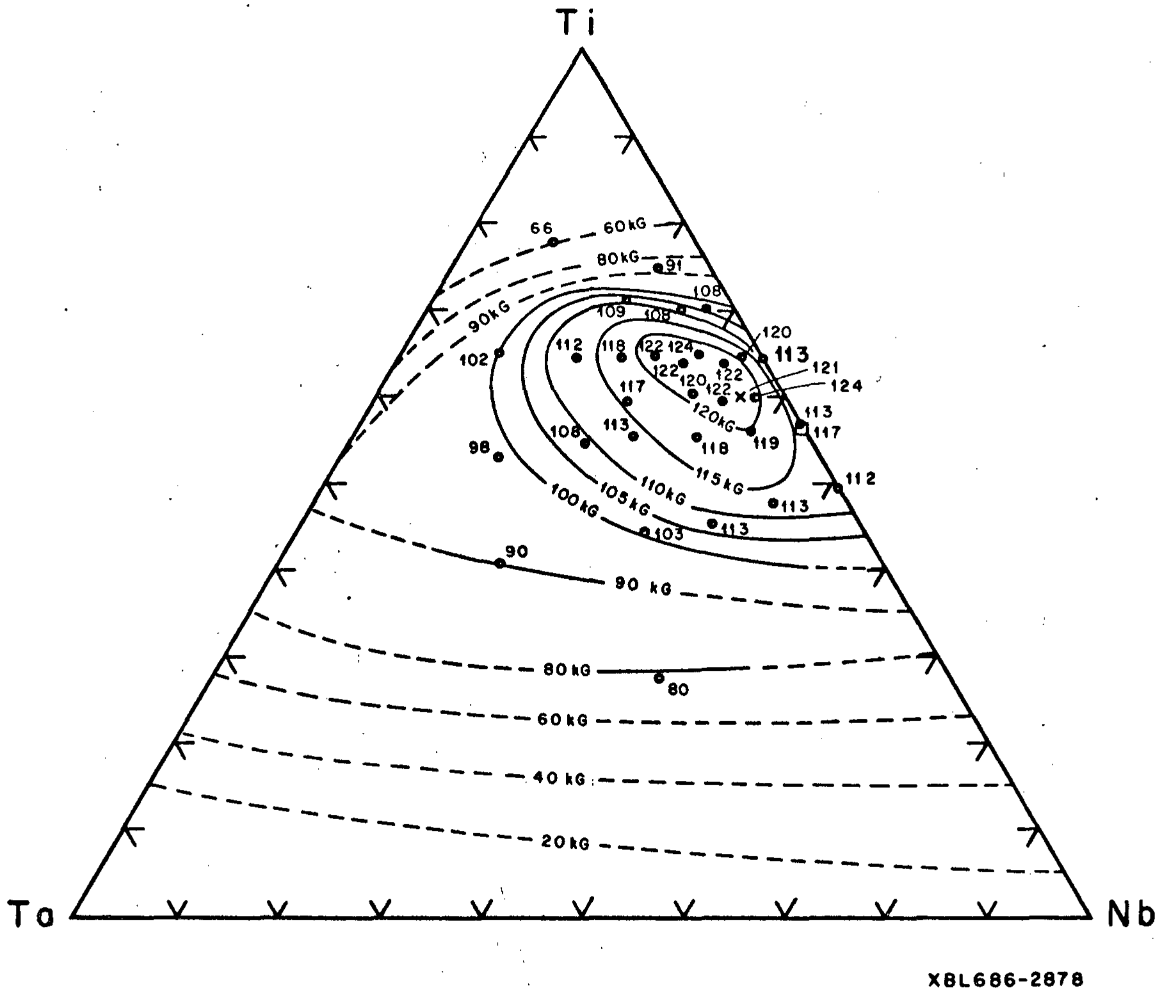
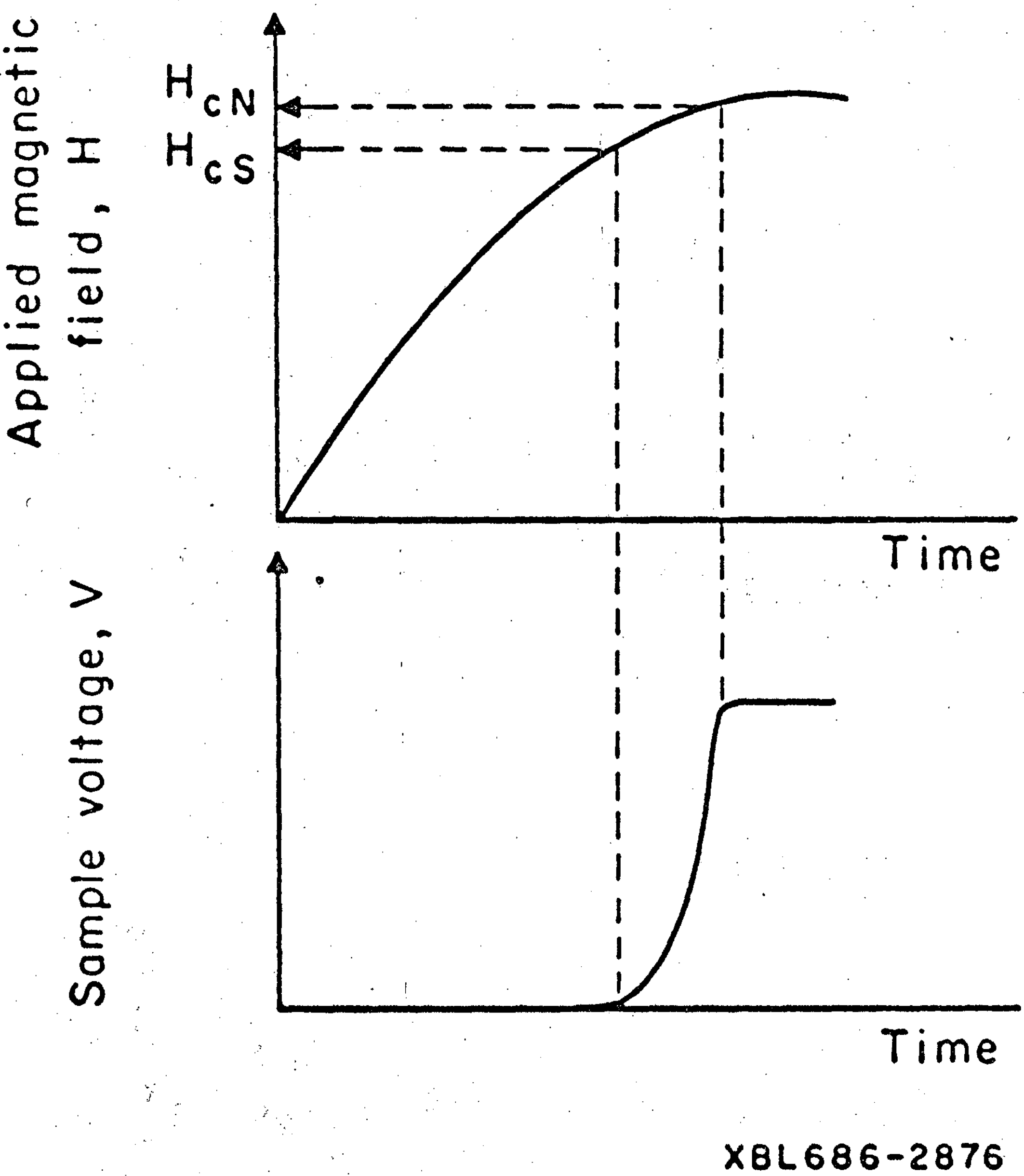

Ti
Ta
Nb
60kG
80kG
90kG
100kG
105kG
110kG
115kG
120kG
90 kG
80 kG
60 kG
40 kG
20 kG
XBL686-2878

Figure 2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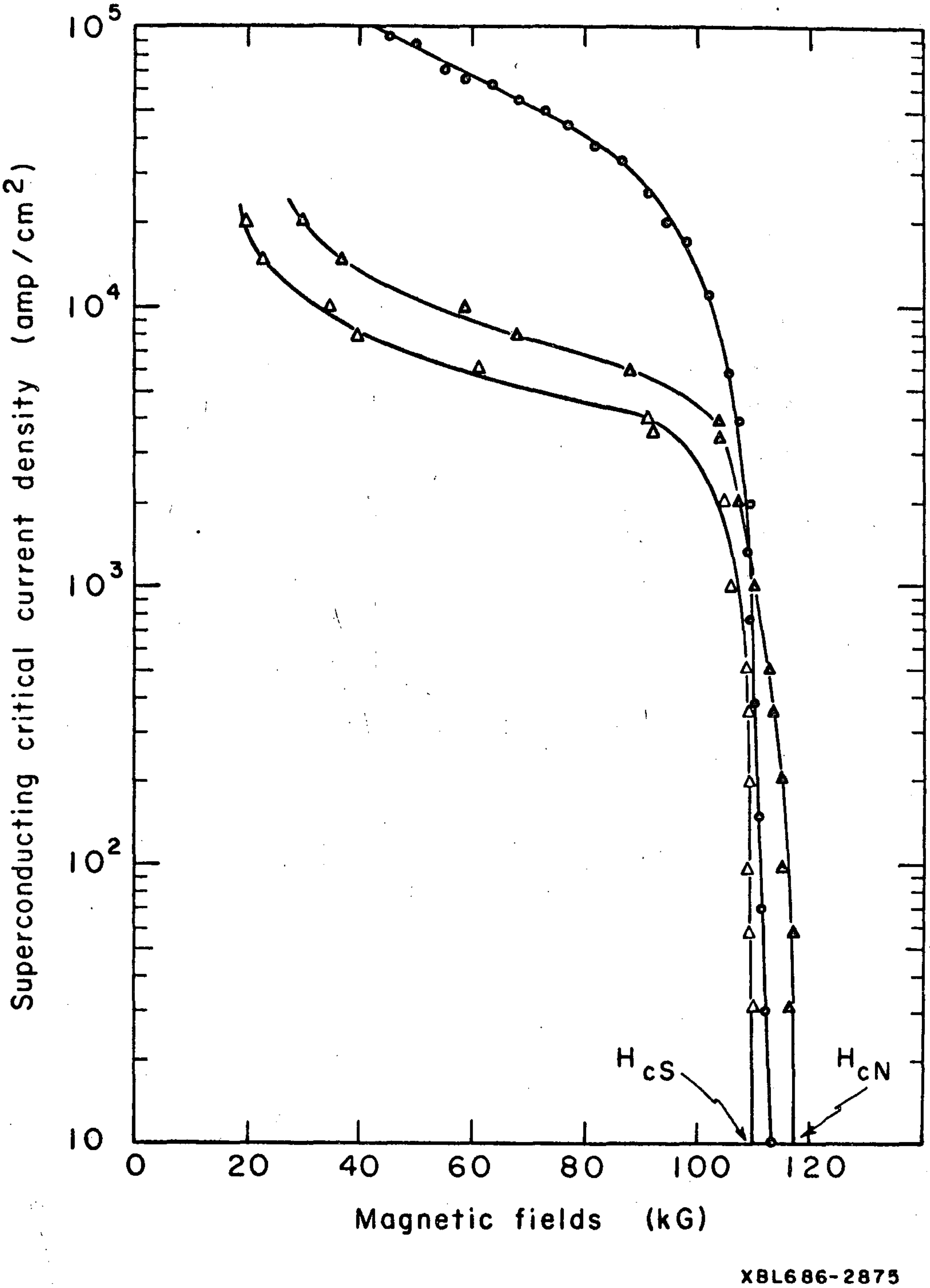
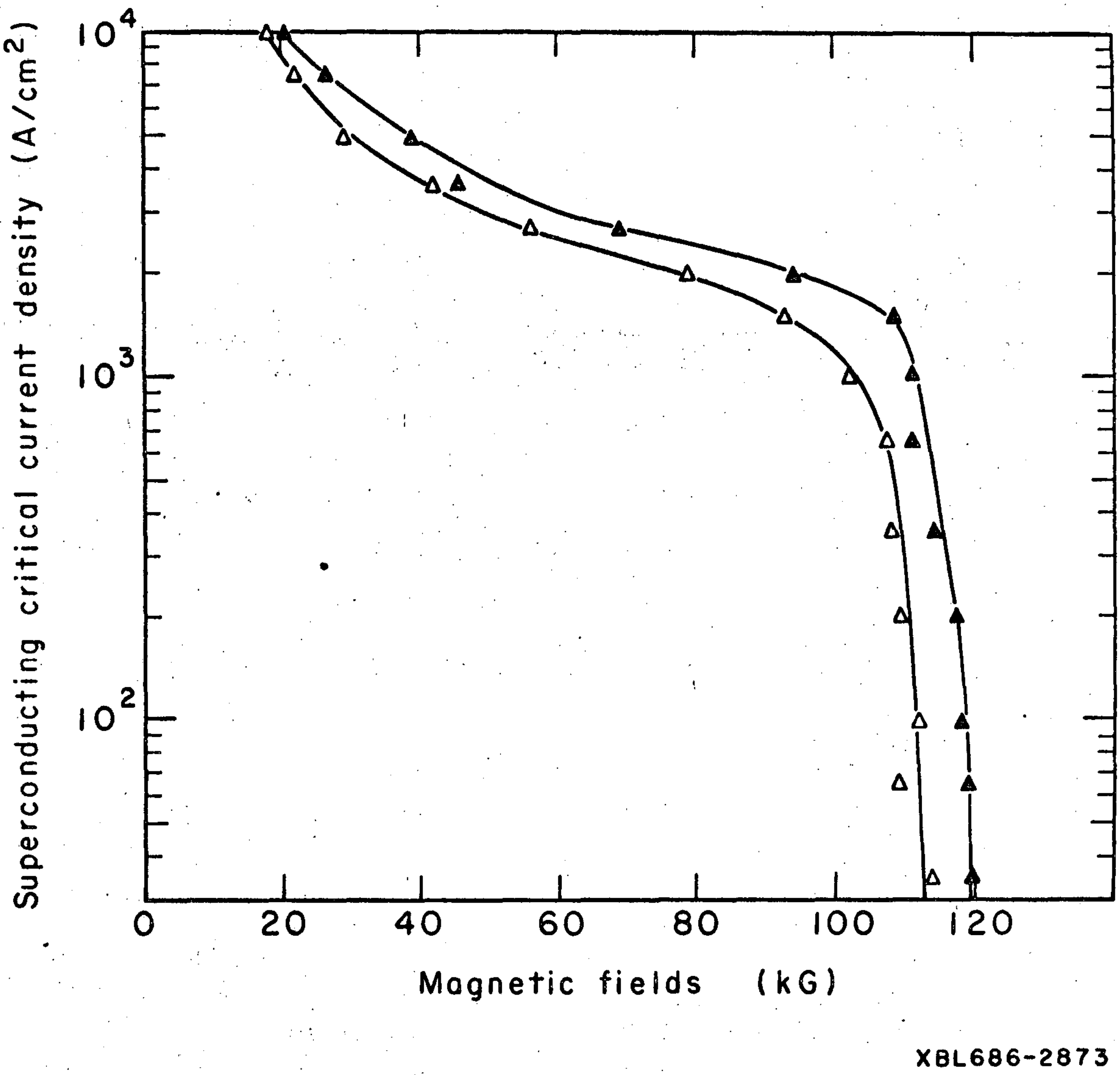
H_{cN}
H_{cS}
Time
Sample voltage, V
Time
XBL686-2876

Figure 3



10^5
10^4
10^3
10^2
10
Superconducting critical current density (amp/cm^2)
H cS
H cN
0
20
40
60
80
100
120
Magnetic fields (kG)
XBL686-2875

Figure 4



Superconducting critical current density (A/cm^2)
10^4
10^3
10^2
0
20
40
60
80
100
120
Magnetic fields (kG)
XBL686-2873

Figure 5

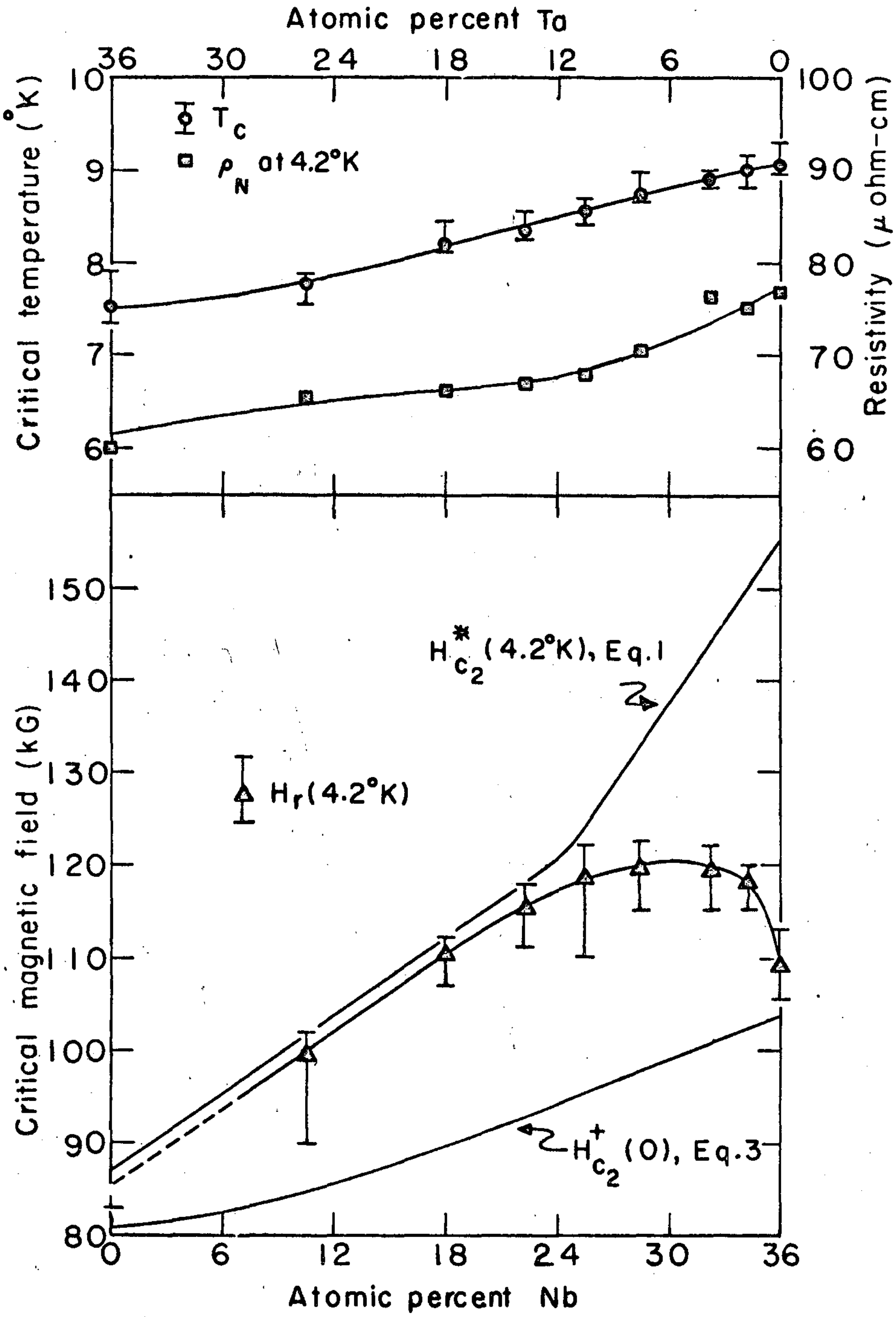


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Figure 6

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