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THE EFFECT OF TERNARY ADDITIONS ON THE SUPERCONDUCTING PROPERTIES OF Nb_3Al

Larry Dowd Hartsough
(M. S. Thesis)

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Larry Dowd Hartsough

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ABSTRACT

We compare the J_c vs H characteristics of Nb_3Al, with T_c of 16.8°K, with those of an alloy of nominal composition $Nb_3(Al_{0.8}Ge_{0.2})$, with T_c of 18.7°K, and find a substantial improvement for the latter. We consider the effect on T_c of As additions to Nb_3Al, but find that the solubility is less than two atomic percent.

The recently-reported high T_c (20.05°K) for well-ordered $Nb_3(Al_{0.8}Ge_{0.2})$ leads us to consider the effect of Ge additions to Nb_3Al on its upper critical field H_{c2}. Our prediction of an increase in H_{c2} is born out by the results we have obtained using pulsed magnetic fields. We further predict that the steady-field J_c vs H properties will be even better than our results.

We contend that the addition of As to Nb_3Al will improve the stoichiometry (hence the T_c) of the latter without affecting its electron/atom ratio or electron density. Unfortunately, we find that the solubility of As in Nb_3Al is too low to test our prediction.

I. INTRODUCTION

The resistivity of superconductors drops abruptly to zero ($< 10^{-22}$ ohm-cm) at a critical temperature, T_c. Furthermore, type I superconductors exhibit perfect diamagnetism up to a critical magnetic field $H_c(T)$, at which they become normal (i.e., resistivity is restored and magnetization becomes zero). While type II superconductors are also perfectly diamagnetic up to a critical field $H_{c1}(T)$, they are in a mixed superconducting state from $H_{c1}(T)$ up to a higher field, $H_{c2}(T)$, at which occurs the transition to the normal state. The mixed state of type II superconductors is characterized by decreasing diamagnetism with increasing field caused by quantized lines of flux threading the bulk of the material. Flux line density increases with field above H_{c1}. It has been shown that a transport supercurrent exerts a force on these flux lines. Motion of flux lines results in losses which show up as an effective resistance.[1-3] Thus, unless the flux lines are made immobile by pinning, a type II superconductor cannot carry the large transport current densities required of a high-field superconducting magnet.

Kunzler et al.[4] discovered, in 1961, that Nb_3Sn could support large current densities in high magnetic fields. Magnets of Nb_3Sn are now made which can produce fields of 140 kG over a 6-inch bore.[5] However, the search for materials with high critical fields continues, with much of the effort centered on alloy additions to, and pseudobinary alloys of, compounds with the A-15 structure, of which Nb_3Sn, V_3Si, Nb_3Ge and Nb_3Al are examples. As will be shown in the next section, H_{c2} increases with T_c. Until recently, efforts to increase the T_c of A-15 compounds by alloy additions have had, at best, minimal success.[6] Matthias et al.[7]

have reported a substantial increase in T_c of Nb_3Al from 18.0°K to 20.05°K by the addition of Ge to form the compound of composition $(Nb_3(Al_{0.8}Ge_{0.2})$. Their alloy was annealed for several days and was assumed to be well-ordered. They did not report any critical field data for this compound. One of the main purposes of this study is to determine the critical current as a function of field (J_c vs H) for an alloy with composition near $Nb_3(Al_{0.8}Ge_{0.2})$. The other purpose is to ascertain the influence of As additions on the superconducting properties of Nb_3Al. This will be discussed further at the end of the next section.

II. THEORY AND EXPERIMENT

A. Theories of T_c and H_{c2}

1. T_c

Extensive experimental evidence suggests that the BCS microscopic theory of superconductivity is not yet refined enough to predict the superconducting properties of intermetallic compounds, especially those containing transition elements.[6,7] However, Matthias[6,8] has developed empirical correlations which relate the occurrence of superconductivity to: 1) N, the number of valence electrons per atom, determined by counting all electrons outside a closed shell; 2) the crystal structure (especially important for intermetallic compounds); 3) the atomic volume; 4) the atomic mass; and, 5) the degree of order or stoichiometry (intermetallic compounds). Roberts[9] has applied these criteria to known superconductive A-15 intermetallic compounds and has concluded, from statistical considerations, that those with high T_c should have: 1) a transition and a nontransition element combining to give 4.7 electrons/atom; 2) atomic volume > 21 Å^3; 3) mean electron density > .25 electron/Å^3 (.26 electrons/Å^3 for Nb-based A-15 phases); and, 4) a degree of order sufficient to ensure that the nontransition metal sites are fully occupied by nontransition metal atoms.

Matthias and co-workers contend that enhancement of stoichiometry is perhaps the most important factor in raising T_c of the A-15 phases. They have demonstrated this by quenching Nb_3Ge to increase its onset T_c from about 7°K to 18°K.[10] Moreover, Matthias, et al.[7] hypothesize that the high T_c of $Nb(Al_{0.8}Ge_{0.2})$ may be due to an improvement of stoichiometry over that of its pseudobinary components, Nb_3Al and Nb_3Ge. For normal

quenching rates, Nb_3Al crystallizes at about 22.0 atomic percent Al[11], while Nb_3Ge crystallizes at about 23.2 atomic percent Ge[7]. Matthias et al.[7] do not think that N is the significant contribution to the high T_c of $Nb_3(Al_{0.8}Ge_{0.2})$. N is 4.55 for this compound, while it is 4.75 and 4.50 for stoichiometric Nb_3Ge and Nb_3Al respectively. Since the T_c of both Nb_3Ge[10] and Nb_3Al[12] increases with stoichiometry, N does not, in fact, determine T_c. On the other hand, the electron density of $Nb_3(Al_{0.8}Ge_{0.2})$ has not been determined and may correlate well with Roberts' observation.

2. H_{c2} and J_c vs H

The upper critical field of a type II superconductor, with transport current density, J, equal to zero, may be stated in terms of measurable parameters as[13]

$$H_{c2} \approx K A(t) \rho \gamma T_c[1-t^2], \qquad (1)$$

where K is a collection of physical constants, A(t) is a function which increases with decreasing reduced temperature, $t = T/T_c$, ρ(ohm-cm) is the normal state resistivity at T, and γ(erg $cm^{-3}deg^{-2}$) is the electronic specific heat coefficient. More detailed calculations, which take into account Pauli paramagnetism in the normal and superconducting states, do not affect the dependence of H_{c2} on ρ, γ, and T_c, although another parameter, the atomic number Z, is introduced.[14] The free energy difference between the normal and superconducting states, assuming that the Pauli susceptibility in the latter state is zero at T = 0, decreases with increasing magnetic field. This imposes an upper limit, H_p, on fields at which superconductivity may exist, which is given by[15]

$$H_p = 18400\, T_c[1-t^2]. \qquad (2)$$

However, it is usually found that the reduction in the superconducting state Pauli susceptibility is only a fraction of the total predicted on the basis of long spin life-times. This is due to spin flips of the superconducting electrons induced by spin-orbit coupling. Since this coupling involves lattice-atom orbital electrons, elements with high atomic number will increase the superconducting state paramagnetism. Thus, H_{c2} can be increased by maximizing ρ, γ, T_c, and Z. The values of ρ will increase with the addition of high Z elements, but this may reduce T_c, because of disorder or change in e/a.[14]

As previously mentioned, Kim et al.[3] have shown that a transport current exerts a force on the flux lines in a type II superconductor. The current density J in a magnetic field H (assuming B = H at high fields) is limited by the Lorentz force parameter,[3]

$$\alpha \sim J \times H < \alpha_c, \qquad (3)$$

where α_c is a structure sensitive free energy barrier to flux motion. Thus, a flux line or flux bundle can overcome the barrier by the combined effects of thermal activation and the Lorentz force. The critical current density, J_c, at a given H, is that which causes enough flux lines to move such that a detectable voltage drop is observed across the specimen in the direction of transport current flow. The usual specimen orientation is such that $J \perp H$, for which α is a maximum. Berlincourt and Hake[13] have discussed the meaning of this type of measurement. Figure 1 shows J_c vs H curves for various degrees of flux pinning. Since the onset of resistance is structure-sensitive, two fields are usually given: 1) that corresponding to the onset of resistance (J_{cs}) and 2) that corresponding to restoration of normal resistance (J_{cn}). At very low current densities

the former value should be approximately equal to H_{c2} (subject to the paramagnetic corrections).[13]

B. Purpose of Study

From the discussion in the previous section, we see that the H_{c2} of $Nb(Al_{0.8}Ge_{0.2})$ should be higher than that of Nb_3Al, since T_c and Z have increased. Moreover, it is argued[7] that T_c is higher because of better stoichiometry. The system $Nb_3(Al_xAs_{1-x})$ appears to provide a good check for this hypothesis. If we follow Matthias and assign an N of five (5) to As, we find that the electron/atom ratio of Nb_3Al is not changed by As additions. Furthermore, As is nearly the same size as Al and its addition to Nb_3Al should not have a large effect on lattice constant and thus the number of electrons/$Å^3$. The intermediate phases known to exist in the Nb-As system are $NbAs_2$ (monoclinic), NbAs (tetragonal)[16] and Nb_3As (Ti_3P-type structure).[17] Thus, not much As is expected to dissolve in Nb_3Al. However, it is possible that Nb_3Al would accomodate a small amount of As (up to 3 atomic percent, for instance).

C. Experimental Procedures

1. Sample Preparation and Treatment

Starting materials were elemental powders of Nb, Al, and Ge; As was added as AlAs to minimize losses. The source, nominal purity, and powder size of each of the starting materials are given in Table I. The powder samples were carefully weighed and thoroughly mixed by tumbling. Samples were then pressed at 25 ksi into 1/2-in. diameter pellets. Each 10-15 gram charge of 2-3 pellets was melted and inverted four times under a 300-350 torr argon atmosphere in a nonconsumable-electrode arc furnace with a water-cooled copper hearth. Arc currents of 150-225 amps were generally

Table I. Starting Materials

Material	Source	Nominal Purity	Powder Size
Nb	Kawecki Chemical Co.	99.9%	-325 mesh
Al	Reynolds Metals Co.	Industrial purity	Atomized powder, -400 mesh
Ge	United Mineral and Chemical Corp.	99.9%	-400 mesh
AlAs	Alpha Inorganics, Inc.	97-99%	Ground to approx. -200 mesh

used. Three ingots were selected for detailed chemical analysis, the results of which are presented in Table II.

Specimens for field measurements were spark cut from the arc melted ingots. Part of the remaining sample was crushed into lumps of suitable size for the T_c measurements and the remainder was reserved for metallography and x-ray diffraction. After the necessary data were obtained on the as-cast samples, selected specimens for field, T_c, and metallographic work were wrapped in Nb and Mo foils, sealed in evacuated and argon backfilled quartz tubes, and annealed for three days at 948±3°C in a Hevi-Duty furnace. The tubes were dropped into water at room temperature at the end of the anneal.

2. Microstructure and Composition

Metallographic specimens were ground flat with SiC paper, mounted in bakelite, ground smooth with SiC paper, and polished on a diamond-impregnated polishing wheel. An etchant of 3 parts HF, 2 parts HNO_3, and 4 parts H_2O[11] adequately brought out the microstructure.

X-ray diffraction patterns of powdered samples were taken on a Picker x-ray diffractometer. No attempt was made to obtain patterns suitable for lattice parameter measurements.

Microprobe analysis was performed on the annealed samples in an effort to determine the compositions of the phases present. A Materials Analysis Company three-counter microprobe was used. Data were reduced by computer.[18]

3. T_c Measurements

Small lumps from each sample were packed into tubes made of cellophane tape. These T_c specimens were then inserted into the center of a coil contained in a double-walled stainless steel cylinder immersed in liquid helium. The temperature of the sample could be varied quite slowly

Table II. Analyses of Selected Ingots

Ingot No.	Elements present, in weight percent											
	Nb	Al	As	Ge	Fe	Ta	Si	Cu	C	N	O	Sb
A-4	90.58	9.79	--	--	.022	ND	.005	<.0025	.018	<.001	.016	--
B-5	87.33	10.35	2.39	--	.035	ND	.005	<.0025	.043	.004	.052	<.005
C-4	90.73	6.70	--	2.58	.013	ND	.005	<.0025	.045	.002	.012	--

ND = not detected, < = less than

(about 1/2 degree/minute) by adjusting the pressure in the outer chamber. The inductance change in the coil (operated at 5 Kc) at T_c gave a strong signal. The temperature was measured with a Texas Instruments Inc. germanium thermometer which had been calibrated against another accurately calibrated thermometer of the same type. T_c was taken to be the temperature at the mid-point of the signal.

4. J_c vs H Measurements

The samples were about 10^{-2} cm^2 in cross section by 1 cm long. Measurements were done in pulsed magnetic fields up to 240 kG with $J \perp H$ and sample temperature at 4.2°K. Berlincourt and Hake[13] describe the method and discuss interpretation of the data. The magnet's rise time varied with maximum field from 11.5 msec at 30 kG to 8.8 msec at 225 kG.

D. Results

1. Composition and Microstructure

Table III presents, in abbreviated form, the compositions and microstructures of the alloys. The estimated final compositions of the ternary alloys should be used only for comparisons. Since the analyses in Table II were inconsistent (in the major components) with starting compositions, these analyses could not be used to predict final compositions. Also, material was lost by splattering during arc melting, which prevented the use of weight loss to determine composition.

The microstructures of samples B-5 and B-8 indicate that little As is soluble in Nb_3Al. Microprobe results indicated the following average compositions for the A-15 phase (in atomic %): sample B-8 -- 18.2 Al, 2.3 As; sample B-5 -- 19.0 Al, 1.1 As, sample B-3 -- 21.3 Al, 1.4 As.

Table III. Composition and microstructure.

Alloy	Composition		Microstructure	
	Starting	Final (est.)	As Cast	As Annealed
A-2	25.0 Al	21.5 Al	single phase, slight segregation	single phase
A-3	25.2 Al	22.1 Al	trace of second phase in grain boundary	---
B-3	28.4 Al 4.5 As	25 Al 3 As	cellular; second phase discontinuous	similar to as cast
B-4	25.2 Al 6.8 As	22 Al 4 As	heavily convoluted two-phase cellular	---
B-5	25.3 Al 2.7 As	22 Al 2 As	same as B-3, less second phase	---
B-6	22.2 Al 8.6 As	21 Al 5 As	three phases-peritectic reaction	---
B-7	15.0 Al 15.0 As	14 Al 13 As	same as B-6, similar to Fig. 14, ref.11	---
B-8	24.4 Al 2.1 As	21 Al 1 As	same as B-3; less second phase; primary phase cored	similar to as cast; coring more pronounced
C-1	23.4 Al 4.6 Ge	21 Al 5 Ge	two phase, primary phase continuous	---
C-2	23.8 Al 4.5 Ge	21 Al < 5 Ge	similar to C-1	similar to as cast
C-10	21.0 Al 4.7 Ge	19 Al 5 Ge	inhomogeneous; some single phase, rest two phase, almost dendritic	cellular, more homogeneous
C-11	23.0 Al 1.1 Ge	20 Al 1 Ge	small amount of second phase	---
C-12	22.8 Al 2.1 Ge	20 Al 2 Ge	two phase cellular	---

The probable accuracy and the method by which these values (and those below) were obtained is discussed in the Appendix.

Although the germanium samples are two phase, the x-ray diffraction pattern of C-10, annealed, shows only broad A-15 peaks. The microprobe analysis gave the compositions given in Table IV for two of the as-annealed germanium samples. From these results it appears that the primary phase is transformed Nb solid solution, while the secondary phase has solidified in the A-15 structure directly from the melt. Thus, C-10 is composed of two A-15 phases with similar lattice parameters.

Table IV. Composition of A-15 phases.

Sample	Phase	Atomic % Al	Atomic % Ge
C-10	Primary	12.9	2.0
	Secondary	17.0	6.2
C-2	Primary	16.7	3.1
	Secondary	18.0	6.2

2. T_c Results

The results of T_c measurements for the as-cast and annealed samples are given in Table V. The only As-bearing alloy to give an increase in T_c, compared to alloy A-2, contained an excess of Al. The start of the transition was not significantly higher.

Although significant increases in T_c were obtained for the germanium alloys, we were not able to reach 20°K. The possible reasons for this will be discussed later.

Table V. T_c Results.

Alloy No.	As Cast			As Annealed		
	Start of Transition	End of[a] Transition	T_c, °K[b]	Start of Transition	End of[a] Transition	T_c, °K[b]
A-2	18.17	16.15	17.97	17.80	~12.0	16.80
B-3	18.27	17.93	18.20	18.08	16.50	18.00
B-4	17.92	16.85	17.57	--	--	--
B-5	18.08	17.45	18.05	--	--	--
B-6	13.55	12.35	12.85	--	--	--
B-7	12.95	11.70	12.54	--	--	--
B-8	17.50	16.00	17.05	17.80	~12.0	16.84
C-1	18.35	18.04	18.23	--	--	--
C-2	18.41	18.08	18.38	19.01	16.08	18.79
C-3	18.28	17.90	18.18	--	--	--
C-10	18.50	18.30	18.45	18.92	15.45	18.66
C-11[c]	17.97	17.05	17.64	--	--	--
	14.94	14.13	14.52	--	--	--
C-12	18.43	18.00	18.27	--	--	--

(a) End of transition uncertain due to change in coil inductance with temperature.

(b) T_c determined by temperature at 50% signal change; accurate to ± 0.1°K.

(c) Two transitions.

3. J_c vs H Results

The single phase Nb_3Al alloy (A-2) and the Ge alloy with highest as-cast T_c (C-10) were chosen for detailed field study. Some discussion of the criteria used in determining the data points is required. Below 500 amp/cm^2 noise became such a problem that reliable data could not be obtained. Furthermore, the position of a J_{cs} point (chosen as 2% of full resistance) was extremely sensitive to dH/dt; i.e., J_{cs} increased with decreasing dH/dt. Thus, an additional criterion used for J_{cs} was that the field be at least one-half maximum at the 2% point.

The J_c vs H results are presented in Figs. II and III for annealed A-2 and C-10 respectively. The J_c vs H characteristic of each is not significantly different from its as-cast counterpart, except that the peak in J_{cs} for as-cast C-10 was not as pronounced. The curves for A-2 and C-10 differ in three important ways: 1) J_{cs}, and 2) the break-point in J_{cN}, have shifted to higher fields for C-10, which 3) has a peak in J_{cs} vs H. The resistivity, ρ, was calculated at two different current densities and averaged. The resistivities at 4.2°K for as-cast and annealed A-2 and C-10 are given in Table VI. The sample C-2 did not have a peak effect in the as-cast condition; it was not tested after annealing. The discontinuities in the J_{cN} curves are discussed later.

Table VI. Resistivity at 4.2°K.

Alloy No.	ρ, μ ohm-cm	
	as-cast	as-annealed
A-2	47.0	34.0
C-10	55.4	49.3

III. DISCUSSION AND CONCLUSIONS

A. Effect of As Additions on T_c of Nb_3Al

The microprobe, metallographic, and T_c results indicate that As has very limited solubility in Nb_3Al and that the stoichiometry cannot be increased by As additions. The microstructure of alloy B-7 is almost identical to that of a Nb-31 Al alloy,[11] indicating a peritectic reaction. The x-ray diffraction pattern of this alloy showed a number of extra peaks, two of which are probably from the niobium solid solution, and the rest from an as-yet unidentified phase containing Nb, Al, and As. The peaks roughly correspond to those of Nb_2Al (σ-tetragonal) but have not been checked against the structures occurring in the Nb-As system. Thus, the hypothesis that As might increase the stoichiometry of Nb_3Al (hence its T_c) without affecting the contravening variables of electron/atom ratio and electron density has not really been tested.

B. J_c vs H for Nb_3Al and $Nb(Al_{0.8}Ge_{0.2})$

The addition of Ge to Nb_3Al has a profound effect on J_c vs H. The peak effect, the exact mechanism for which is still unknown,[19] represents a substantial increase in the degree of pinning. It is unfortunate that a higher critical temperature was not attained. This is probably because the stoichiometry and/or the degree of order were not high enough. A longer anneal or one at a different temperature may have given higher T_c.

It is not clear from the article by Matthias, et al.,[7] whether or not they obtained a single phase alloy. We have shown that a reliance on x-ray diffraction alone is misleading. In any event, we contend that our samples contain two A-15 phases and hypothesize that it is the one richer in Ge

which accounts for the high T_c of these alloys.

In Figs. II and III we have shown broken lines for the J_{cN} curves. Each segment represents data taken with a different maximum field. At high current densities the misalignment of these curves becomes appreciable. The reason for this is heating of the sample which results in a decrease of upper critical field. For example, at 2.5 amp/cm^2 and a maximum field of 100 kG, the sample is partially normal for a period of about 10 msec, whereas, if we increase the maximum field to 200 kG, the sample is partially normal for only 2-3 msec. Thus, the higher of the two values for J_{cN} is the more accurate at 4.2°K. Also, in Figs. II and III we have drawn the J_{cs} curves through the highest points, since, as we observed earlier, J_{cs} is highest for dH/dt = 0, which represents steady-field conditions. Thus, even better results than reported here will probably be obtained when this alloy is tested under steady-field conditions. On the basis of the above discussion we have redrawn the J_c vs H curves for alloy C-10; the smoothed curves are presented in Fig. IV.

Finally, we note that an extrapolation of J_{cs} to low current density indicates that H_{c2} for our sample is well above 220 kG. This compares favorably with the values reported for V_3Ga[20] and Nb_3Sn,[21] which have the highest known values of H_{c2}.

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APPENDIX

Microprobe analysis on a system such as Nb-Al-As (or Ge) does not give completely accurate results, even when absorption and fluorescence are taken into account as our computer program does. This is because the atomic number effect, the correction for which is not yet well known, can be quite large. One correction for the effect of atomic number has been given as:[22]

$$k_A = \frac{\alpha_A C_A}{\Sigma \alpha_i C_i} \qquad \text{(A-1)}$$

where k_A is the ratio of intensities for element A from the specimen and standard, the experimental α-parameters may depend on accelerating voltage and concentration, and C_i is the weight fraction of element i. We set k_i equal to the concentration given by the computer program. Equation (A-1) may be re-written for a ternary alloy in a form easier for computation as:

$$C_A = \frac{k_A}{1 + (\alpha_A/\alpha_B + \alpha_A/\alpha_C - 1)(1 - k_A)} \qquad \text{(A-2)}$$

Now, we wish to take into account the fact that Σk_i may not equal 1. If we substitute $k_B + k_C$ for $(1 - k_A)$ and ignore cross product terms,

$$C_A = \frac{k_A}{1 + (\alpha_A/\alpha_B - 1) k_B + (\alpha_A/\alpha_C - 1)k_C} . \qquad \text{(A-3)}$$

The values of α_A/α_B for Nb in Al and vice-versa were calculated from sample A-2, the composition of which is known from weight loss and the phase diagram. These values were within 10% of those given in Ref. 22.

Therefore we used the α values found there to calculate the atomic number correction for the ternary alloys containing As and Ge. Admittedly, the correction given in Eq. (A-3) leaves much to be desired, but we feel that the answers are more accurate with it than without it.

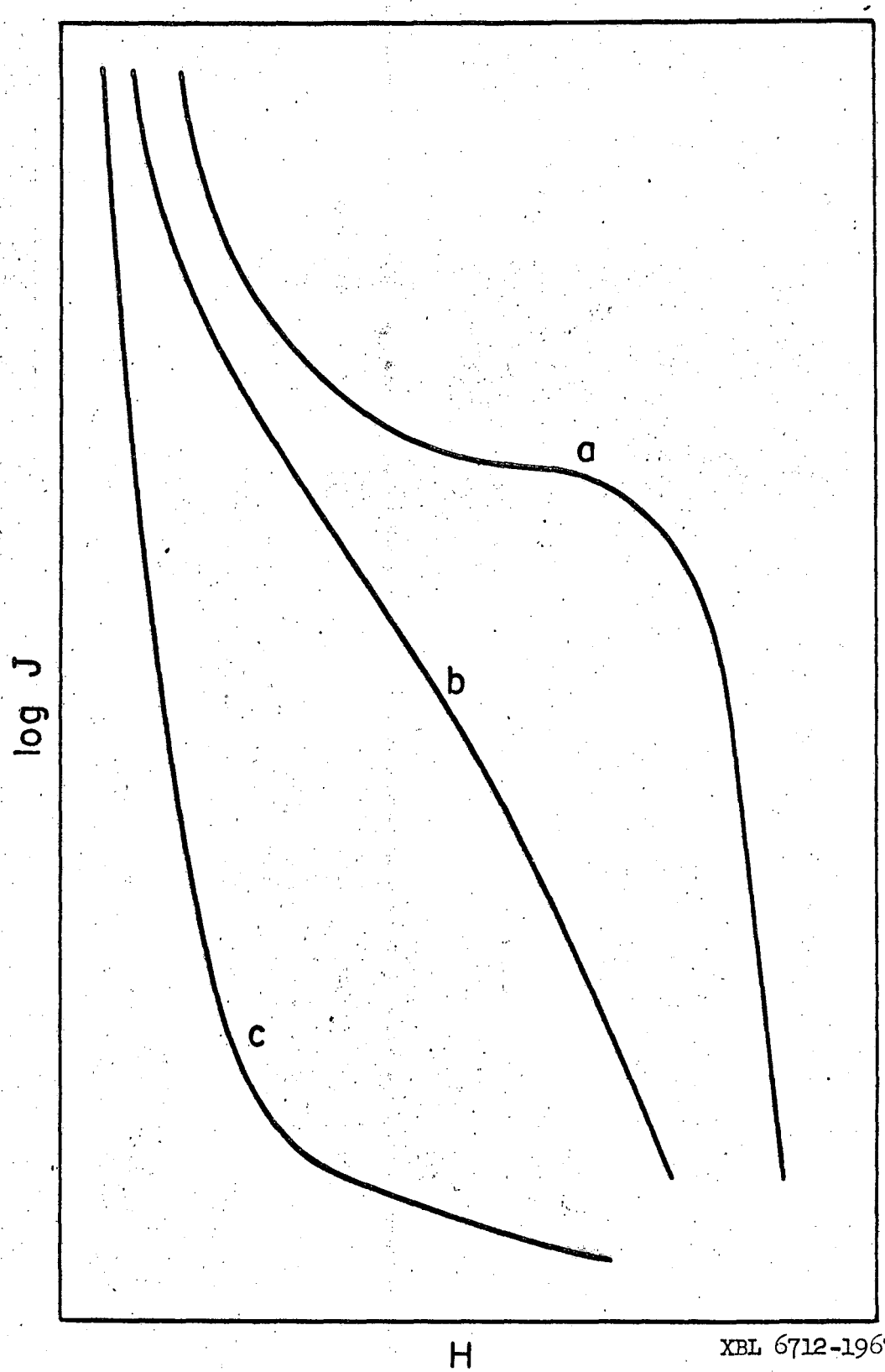

Fig. 1 Typical J_{cs} vs H curves for (a) high pinning strength, (b) moderate pinning strength, and (c) low pinning strength.

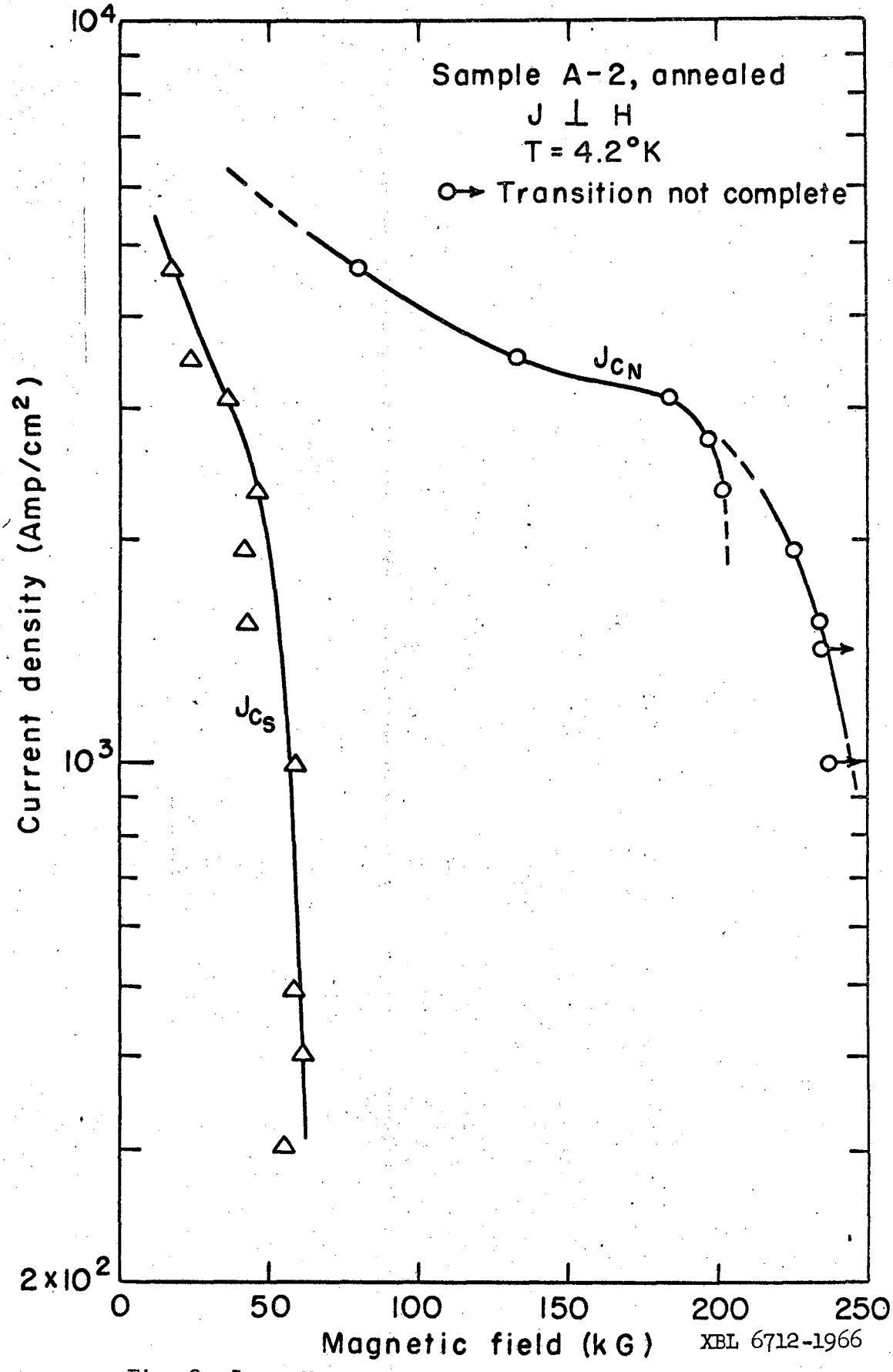

Fig. 2 J_c vs H curves for alloy A-2, single phase Nb_3Al, in a pulsed magnetic field. T_c = 16.8°K.

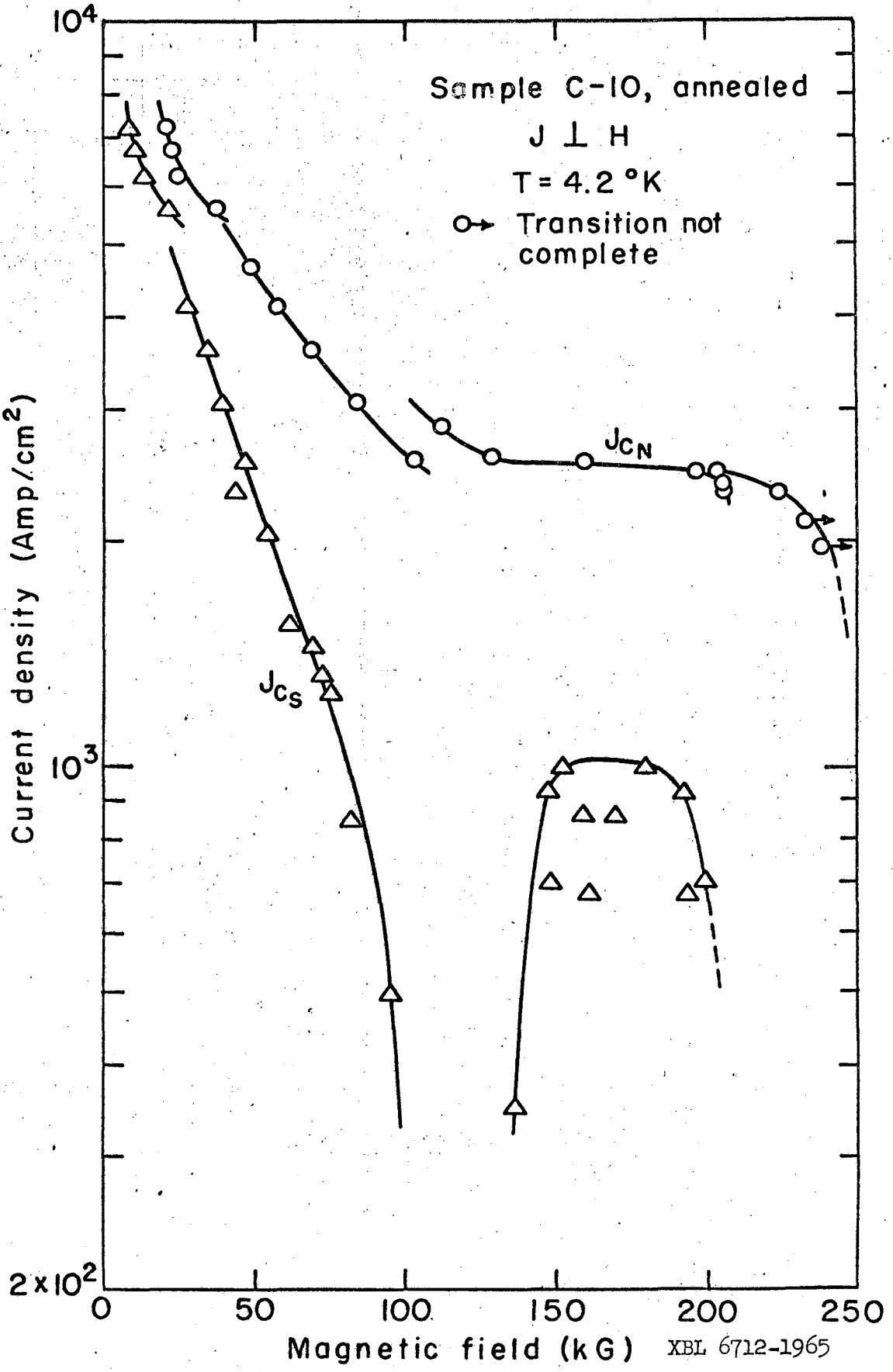

Fig. 3 J_c vs H curves for alloy C-10, two phase alloy of composition $Nb_3(Al_{0.8}Ge_{0.2})$, in a pulsed magnetic field. $T_c = 18.7°K$.

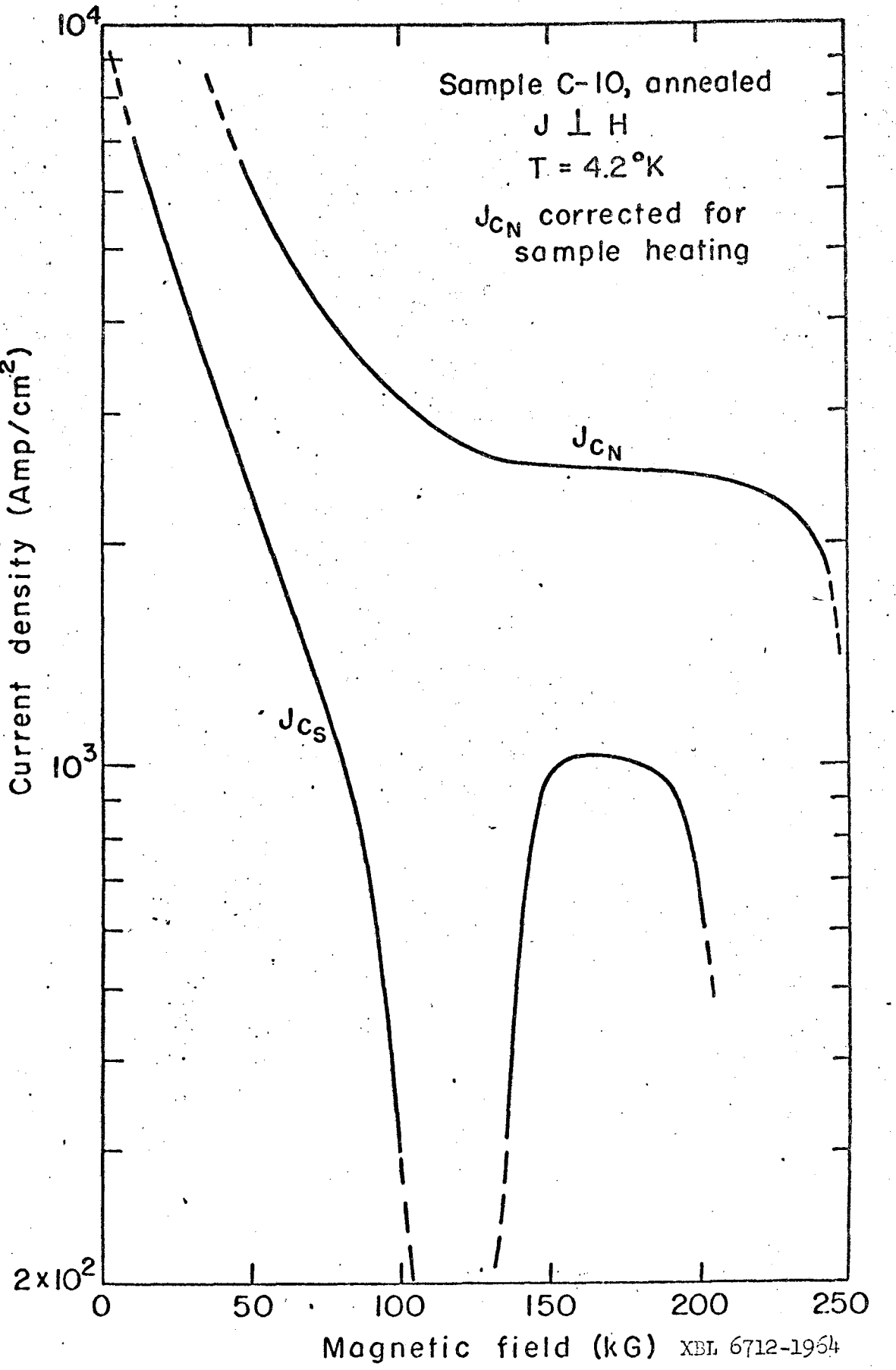

Fig. 4 Smoothed J_c vs H curves for alloy C-10. See text for discussion.

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