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MAGNETIZATION AND SUSCEPTIBILITY MEASUREMENTS OF POLYCRYSTALLINE NIOBIUM*

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Abstract

Magnetization and susceptibility data are presented for niobium rod samples in the temperature range of 1.2 to 9.2° K. The niobium samples tested had a range of tantalum content from 700 to 4000 ppm, and all other impurities were less than 200 ppm. The samples received were in a cold-worked condition. They successively were annealed and degassed in a controlled manner in high vacuum. The annealing temperature was varied between 2100° C and 2300° C. Values of κ_1, κ_2, H_{c1}, H_{c2}, and H_c are presented as a function of tantalum impurity in the temperature range 1.2° K to T_c. The values are compared with theory.

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Niobium is used in large quantities in superconducting linear accelerators, separators, and transmission lines in a "commercial" pure form with traces of impurities, mainly tantalum. Pure niobium with total impurity content of less than 100 parts per million by weight is obtainable in small quantities. The aim of this work was to investigate the effect of certain impurities in niobium on H_{c1}, H_{c2}, and the order parameters, and to determine the effect of annealing temperature and thermal cycling. The studies have been made on the magnetization behavior of niobium.

The niobium samples were cylinders with diameters of 0.3 cm and lengths of 6 cm. The samples were annealed in a high vacuum furnace at a temperature between 2100° K and 2300° K for 2 to 5 hours.

On analysis, Ta impurities were found to be in the range of 700 ppm to 4000 ppm. Also, O_2 and N_2 contents, after annealing, were higher than 100 ppm, which explains the non-reversible magnetization behavior of the samples.

The specimens were made from zone-refined Nb and Ta. The resistivity ratio of Nb, when pure, was 1300. The specimens were melted to alloys with a composition of ~ 700 .... 4000 ppm of Ta in an electron-beam melting furnace in a vacuum of better than 10^{-6} torr, and then rolled into cylinders of 3 mm diameter. The resistivity of these alloys varied between 80 and 135, depending on the impurity content.

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*Work supported by the U. S. Atomic Energy Commission.

The dc magnetization measurements were performed, using the circuit shown in Fig. 1. The method used is essentially similar to that described by Fietz.[1] The low frequency susceptibility circuit is shown in Fig. 2, with a detection unit consisting of a phase-sensitive lock-in amplifier. The magnetization measurements were performed with the sample axes parallel to the direction of the applied field. In both circuits, the signal-to-noise ratio was $> 10^6$. Both methods yielded values of H_{c1} within 10^{-4} T of each other. Magnetization curves of samples before annealing show fluxjumps at temperatures below 3.3^{o}K. Near H_{c2}, a peak effect was observed. At temperatures above 4^{o} K, no fluxjumps were observed and the peak effect disappeared.

When annealed, no fluxjumps were measured at any temperature in the range of 1.2 .... 8.9^{o} K. However, a slight peak effect was still observed at temperatures below 4^{o} K, as shown in Fig. 3.

Rather extensive studies have been made of the magnetization behavior of niobium.[2-5] The measurements here indicate that by adding small amounts of Ta to Nb, the variation of the upper critical field H_{c2} does not agree with the theoretical predictions as well as curves given by Finnemore,[2] and Helfand and Werthamer.[3] As seen in Fig. 4, the upper critical field is independent of Ta content in the range of 700 - 4000 ppm Ta.

The lower critical field H_{c1} is smaller than that reported by Finnemore for pure Nb. The measured lower limit (Fig. 5) corresponds to 4000 ppm Ta and the upper limit corresponds to 700 ppm Ta. Good agreement with Finnemore was obtained for the thermodynamic critical field H_c, as seen in Fig. 6.

From measured magnetization results, the two Ginzburg-Landau parameters, κ_1 and κ_2, were calculated vs reduced temperature, $t = T/T_c$. These parameters are shown in Fig. 7 and 8, and compared to the data reported for pure niobium and to the theoretical calculations by Eilenberger.[6] The experimental results deviate considerably from the theoretical calculations for the dirty limit.

CONCLUSIONS

Magnetization measurements of niobium alloyed to small amounts of tantalum illustrate that although the order parameters, κ_1 and κ_2, as well as H_c, are not greatly affected by impurities of < 4000 ppm Ta, H_{c1} and H_{c2} clearly deviate from values for pure niobium. If high values of H_{c1} are desirable, low limits for impurities should be specified. Even an impurity content as low as 700 ppm yields a substantial reduction in H_{c1}, as well as a high value of residual resistivity.

Although the samples had been repeatedly subjected to cryogenic temperatures and successive warm-ups to room temperature, no apparent effect on magnetization and susceptibility, as a function of temperature, was observed.

References

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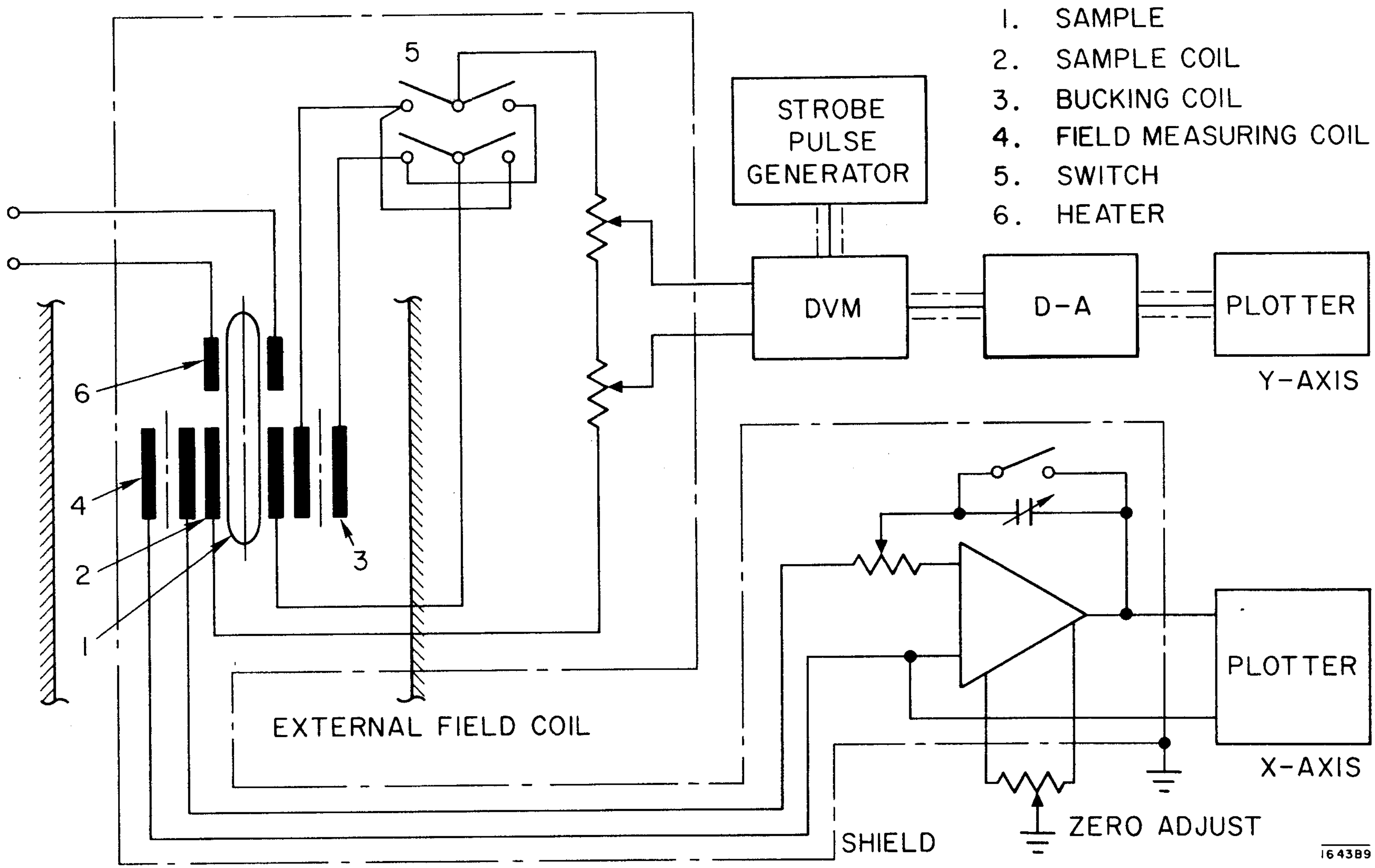
Figures



1. SAMPLE
2. SAMPLE COIL
3. BUCKING COIL
4. FIELD MEASURING COIL
5. SWITCH
6. HEATER
5
STROBE PULSE GENERATOR
DVM
D-A
PLOTTER
Y-AXIS
6
4
2
1
3
PLOTTER
X-AXIS
EXTERNAL FIELD COIL
SHIELD
ZERO ADJUST
164389

Fig. 1

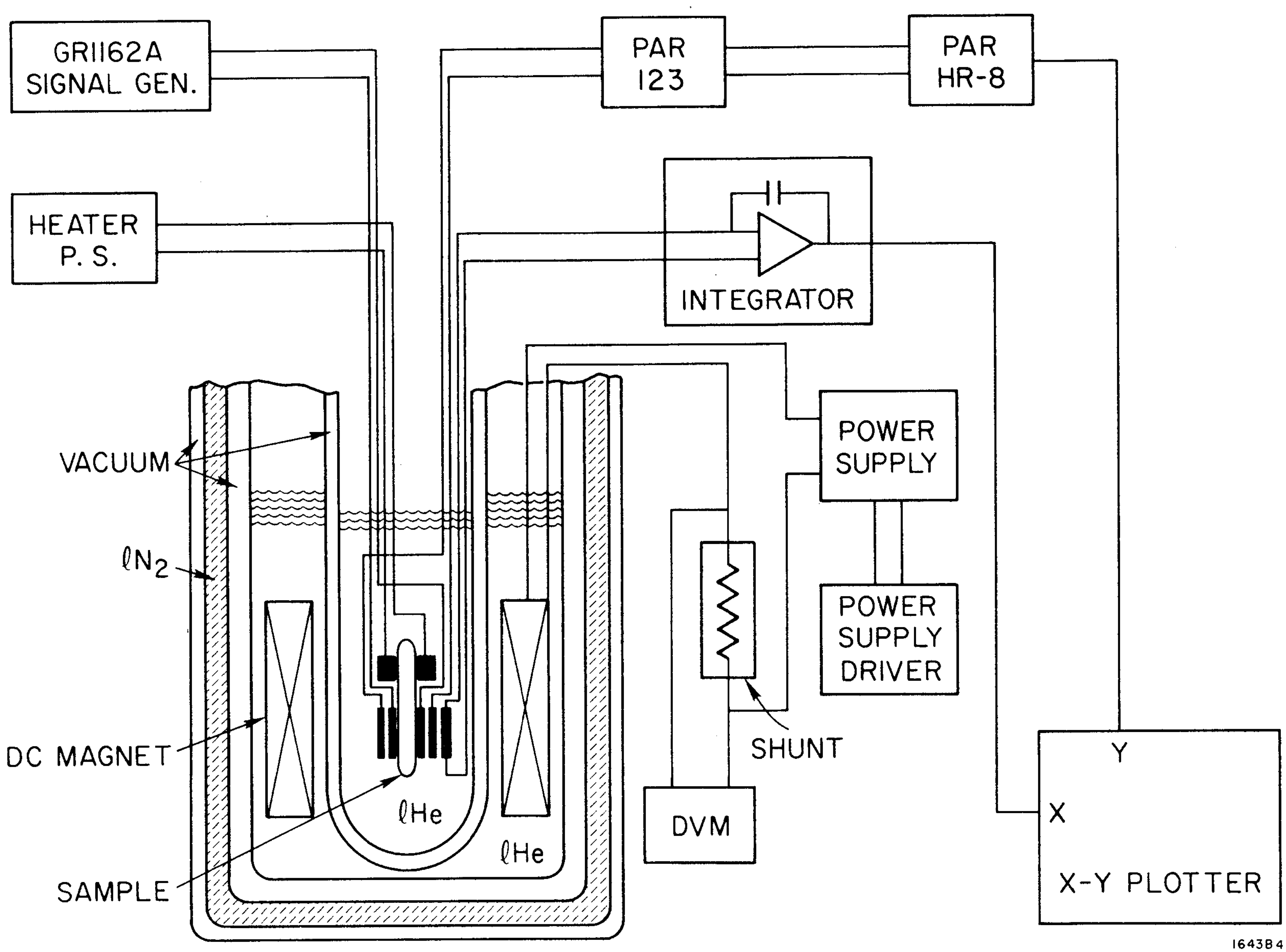
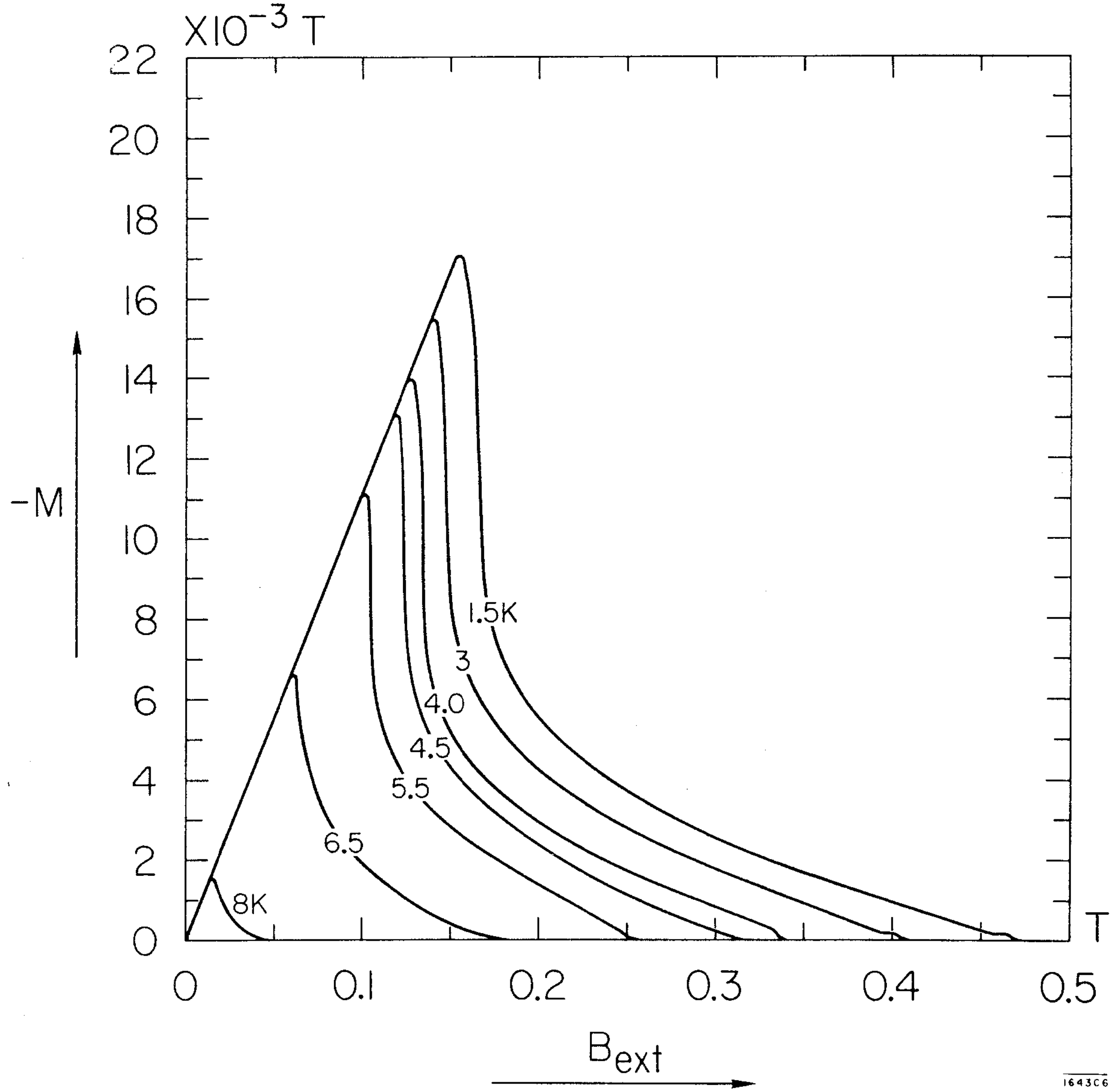
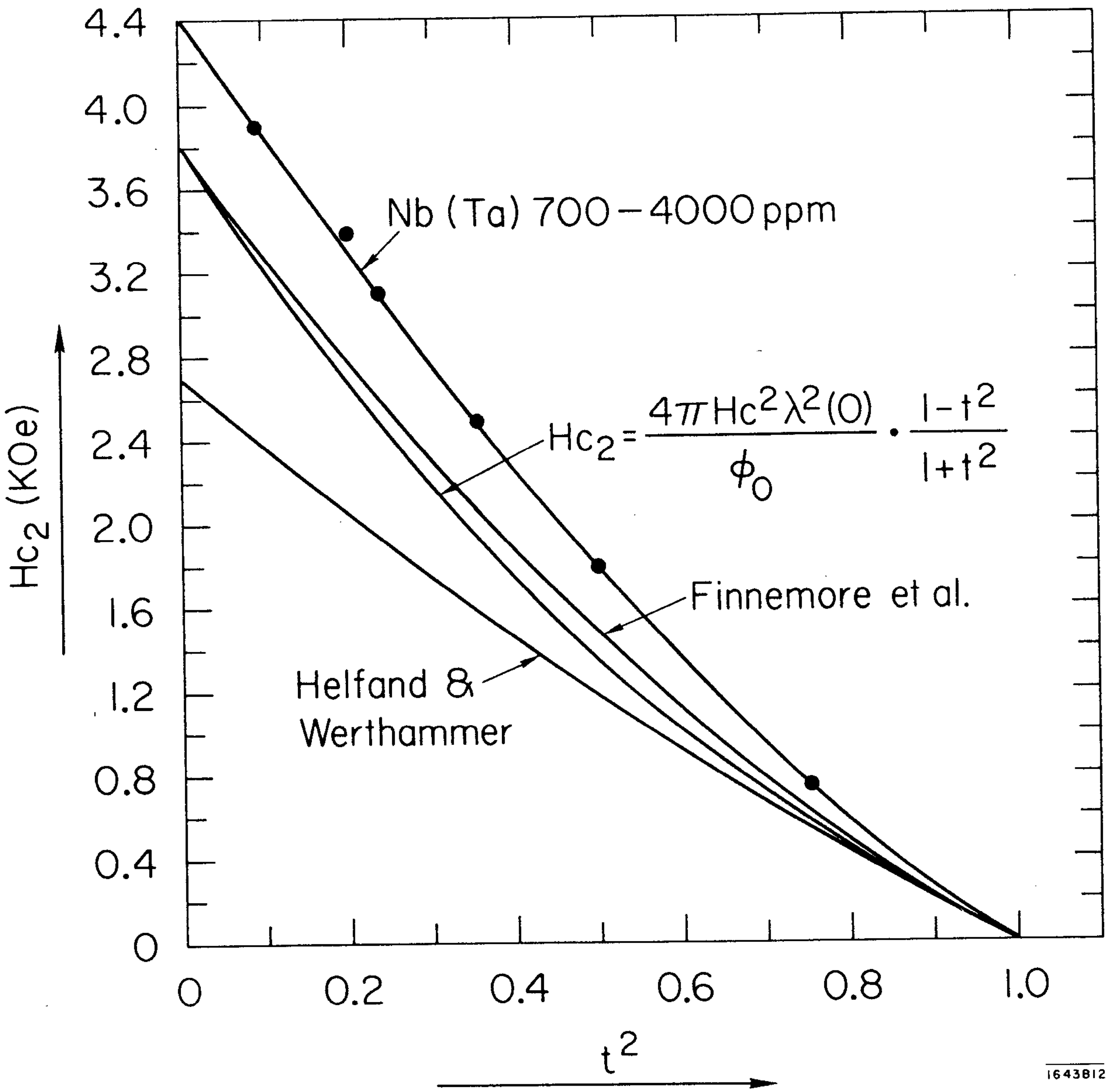

GR1162A
SIGNAL GEN.
PAR
123
PAR
HR-8
HEATER
P. S.
INTEGRATOR
POWER
SUPPLY
POWER
SUPPLY
DRIVER
VACUUM
ℓN_2
DC MAGNET
SAMPLE
ℓHe
ℓHe
SHUNT
DVM
Y
X
X-Y PLOTTER
1643B4

Fig. 2



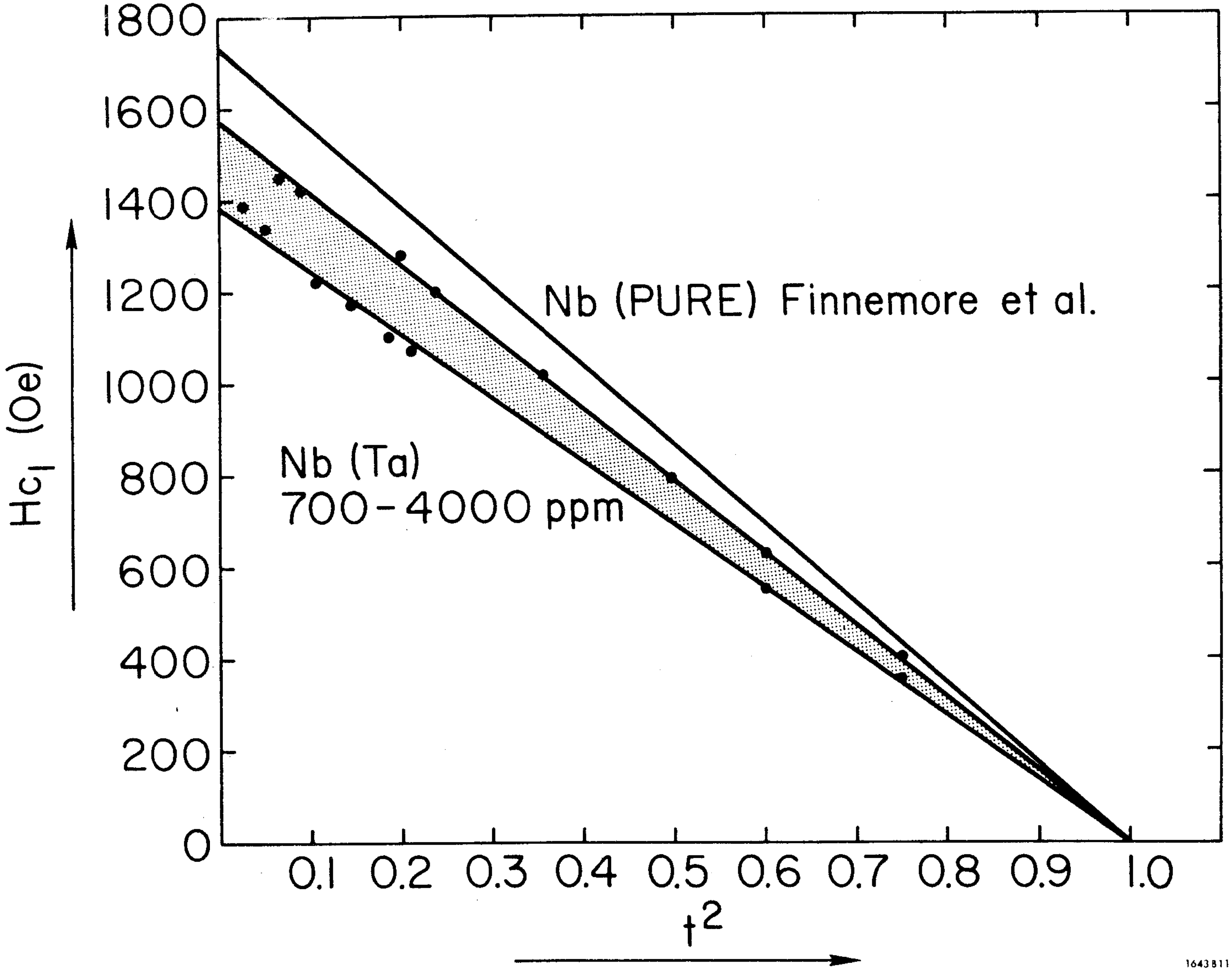
X10^{-3} T
22
20
18
16
14
12
10
8
6
4
2
0
−M
1.5K
3
4.0
4.5
5.5
6.5
8K
T
0
0.1
0.2
0.3
0.4
0.5
B_{ext}
1643C6

Fig. 3



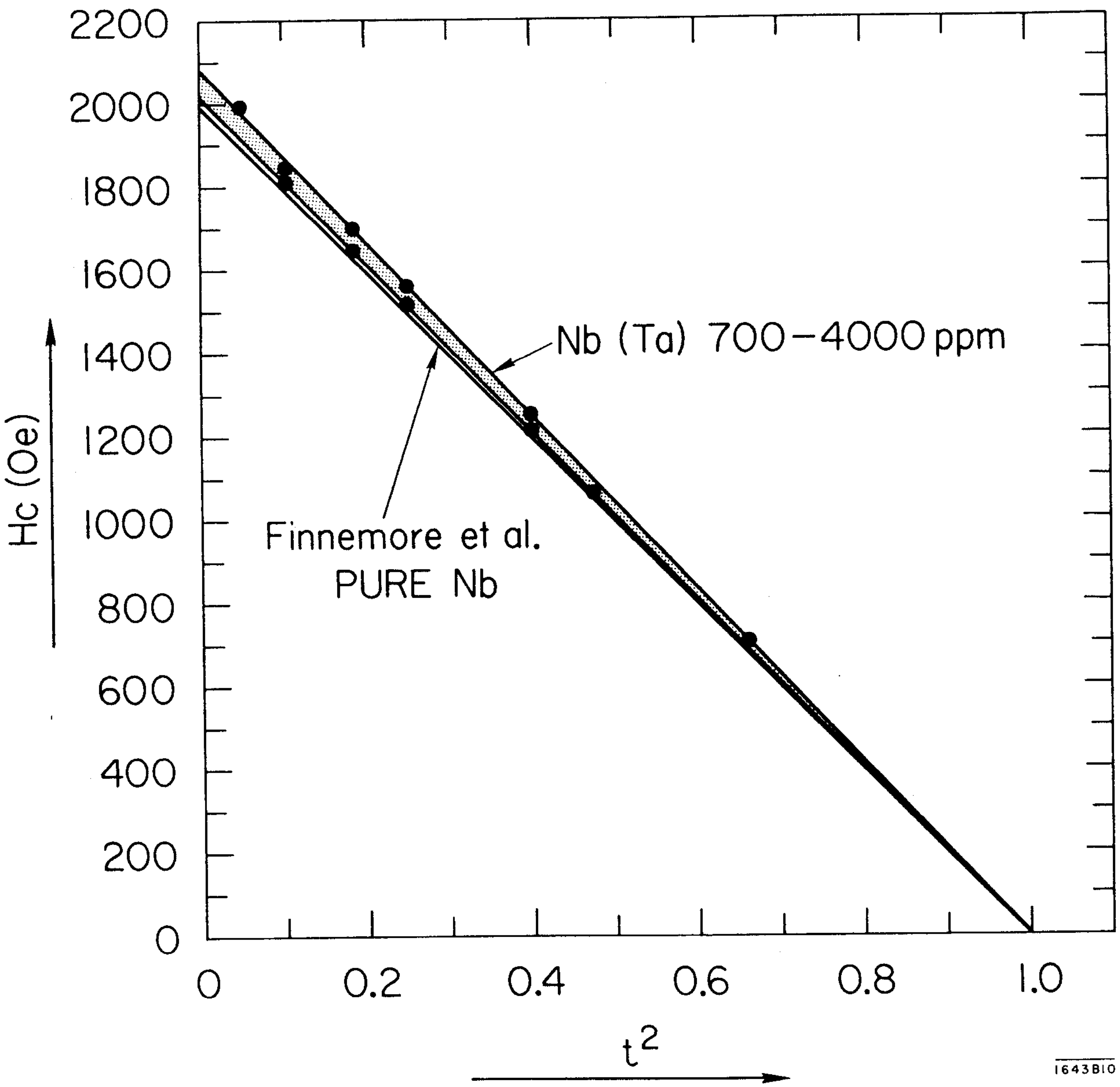
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
0.8
0.4
0
Hc2 (KOe)
0
0.2
0.4
0.6
0.8
1.0
t2
Nb (Ta) 700 – 4000 ppm
$Hc_2 = \frac{4\pi Hc^2 \lambda^2(0)}{\phi_0} \cdot \frac{1-t^2}{1+t^2}$
Finnemore et al.
Helfand & Werthammer
1643812

Fig. 4



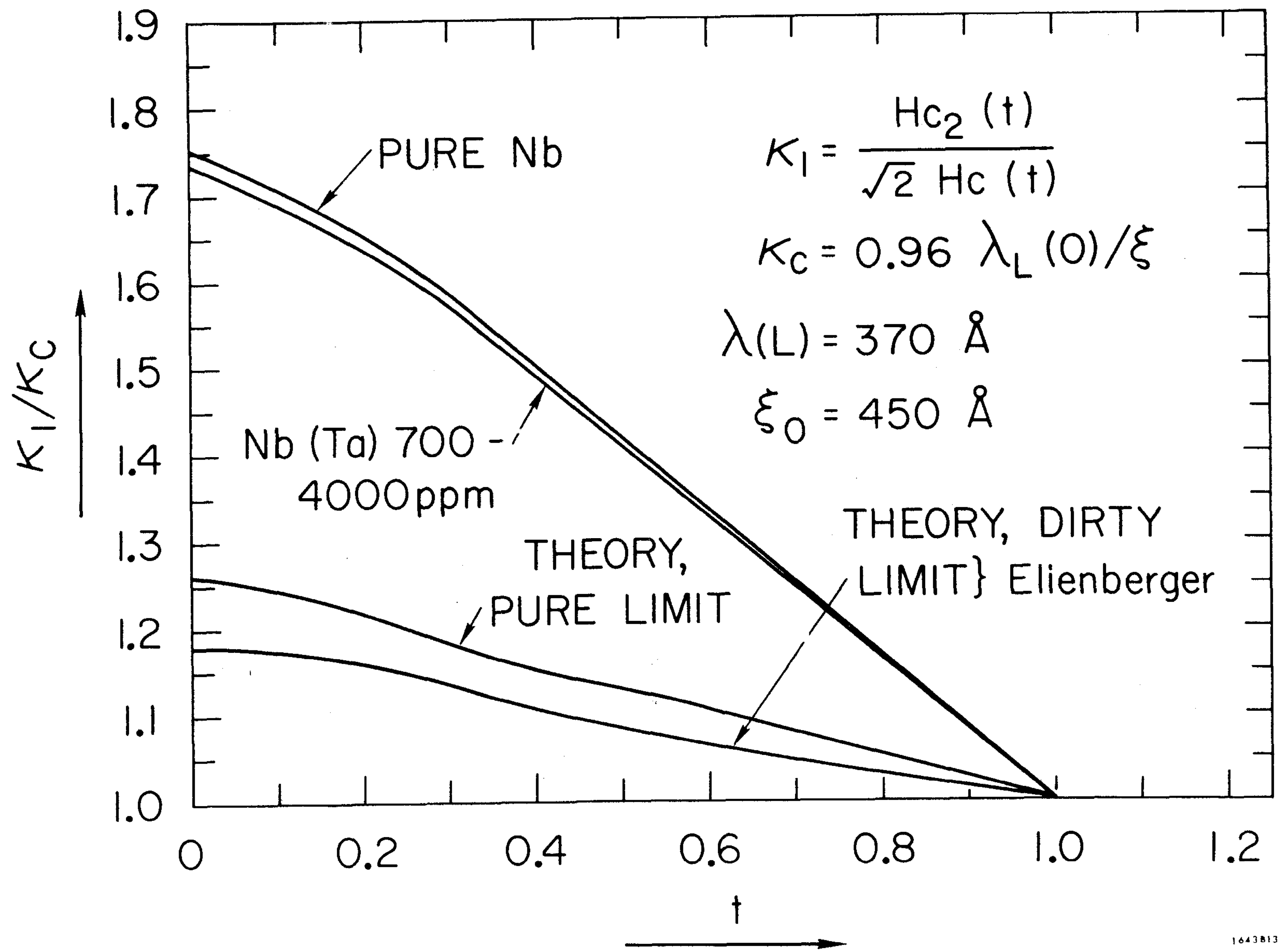
1800
1600
1400
1200
1000
800
600
400
200
0
0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0
t^2
H_{c_1} (Oe)
Nb (PURE) Finnemore et al.
Nb (Ta)
700-4000 ppm
1643B11

Fig. 5



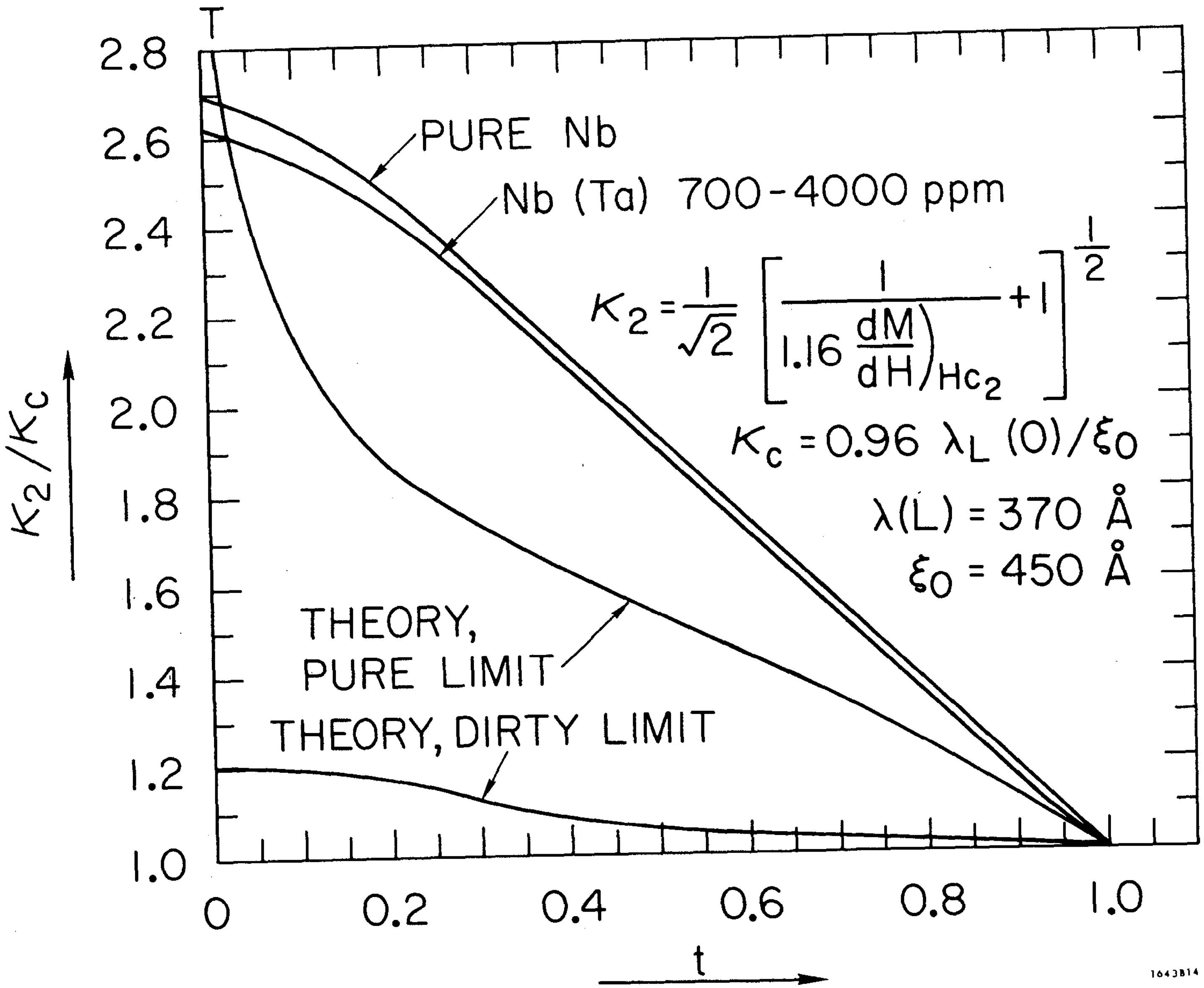
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
Hc (Oe)
Nb (Ta) 700–4000 ppm
Finnemore et al.
PURE Nb
0
0.2
0.4
0.6
0.8
1.0
t2
1643B10

Fig. 6



1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0
0.2
0.4
0.6
0.8
1.0
1.2
κ_1/κ_c
t
PURE Nb
Nb (Ta) 700 - 4000ppm
THEORY, PURE LIMIT
THEORY, DIRTY LIMIT} Elienberger
$\kappa_1 = \frac{Hc_2(t)}{\sqrt{2}\, Hc(t)}$
$\kappa_c = 0.96\ \lambda_L(0)/\xi$
$\lambda(L) = 370$ Å
$\xi_0 = 450$ Å
1643B13

Fig. 7



PURE Nb
Nb (Ta) 700-4000 ppm
$\kappa_2 = \frac{1}{\sqrt{2}}\left[\frac{1}{1.16\left(\frac{dM}{dH}\right)_{Hc_2}}+1\right]^{\frac{1}{2}}$
$\kappa_c = 0.96\ \lambda_L(0)/\xi_0$
$\lambda(L) = 370$ Å
$\xi_0 = 450$ Å
THEORY, PURE LIMIT
THEORY, DIRTY LIMIT
κ_2/κ_c
t
1643B14

Fig. 8