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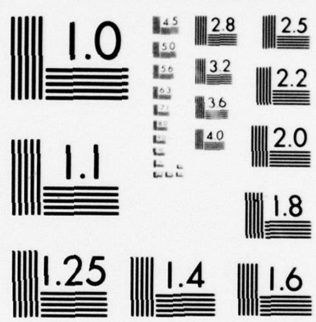

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Very pure annealed Nb samples were irradiated with 3.2 and 5 McV protons to fluences of about 6.8 x 10(16) p/cm(2). Local magnetic induction profiles, when the samples were in the superconducting mixed state, were obtained using an ac technique. Electron micrographs of the same irradiated samples show dislocation loops in the damaged region of the samples. The critical current and the volume flux pinning force were determined from the slope of the measured field profiles. Assuming pinning due to the observed dislocation loops, the pinning force

next page

calculated from a direct summation law is an order of magnitude higher than the experimental result and as calculated from quadratic summation laws is two to three orders of magnitude smaller than measured.

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267 470

The research completed under this grant followed the outline of the original proposal.[1] The emphasis was on the first and primary aim of the proposal which was to study the flux pinning and critical current density in type II superconductors due to defects produced by high energy proton irradiation of Nb and Nb alloys.

The interim scientific report[2] covering the first year of the grant (Jan. 1975 to Jan. 1976) described the apparatus constructed for sample preparation before irradiation, the irradiation of samples, and the data collection system. Preliminary results obtained in the first year were also provided in that report.[2]

The interim scientific report[3] covering the second year of the grant (Jan. 1976 to Jan. 1977) provided a block diagram of the program which must be followed in order to understand the effect of defects on flux-pinning in terms of theoretical model calculations.

The primary system used in our study for comparison between measured flux pinning and model calculations was very-well-annealed niobium which was then irradiated with high energy protons. The second interim report[3] included various aspects of the results of measurements of flux-pinning due to defects produced by proton-irradiation of several annealed niobium samples. These results were reported at professional meetings[4,5] and published in the Journal of Applied Physics.[6]

The third and final year of the grant (Jan. 1977 to March 1978) provided for the identification of the nature of the damage in the proton-irradiated Nb samples using transmission electron microscopy (TEM). The TEM was done in collaboration with Dr. J. Bentley at Oak Ridge National Laboratory. The analysis of the TEM photographs of the damage allowed us to make a quantitative comparison of our measured critical currents and volume pinning force, F_v, with theoretical calculations. A report of this analysis was given very briefly during discussions[7] at the International Discussion Meeting on Radiation Effects on Superconductivity, Argonne, Illinois, June 13-16, 1977 (see Appendix A), and was reported at the March 1978 meeting of the American Physical Society[8] (See Appendix B). A detailed paper reporting more completely on this and several other aspects of our work has been submitted to the Journal of Applied Physics[9] and is included as Appendix C.

In addition the above results which have been or are being reported in publication form, several other measurements were done the results of which are still in preliminary form. Work is still continuing in many of these areas and any completed reports of results will be added as supplements to this report.

Work still underway includes the following:

(i) studies of the rate at which the defects and the resultant flux-pinning anneal out at various annealing temperatures. Preliminary measurements show a very slight reduction in pinning after a one hour anneal at 135 C followed by approximately 50% reduction in pinning after one hour at 365 C and another 50% reduction after one hour at 455 C. Finally a 75% reduction was observed after a one hour anneal at 580 C.

(ii) studies of the flux-pinning as a function of the total proton fluence. Preliminary measurements show a very rapid increase in total flux-pinning with increasing proton fluence. The total pinning appears to go as the total proton dose to the n^{th}

power where n is between 2 and 3. The observed rapid change in flux-pinning with total irradiation dose appears to be in conflict with model calculations. This is still under investigation.

(iii) we have observed the total flux-pinning for a given proton-irradiation can vary by as much as a factor of 20 in different samples. These samples were prepared by the same procedure but we expect the difference in pinning is the result of a different microstructure in the samples before irradiation. We have also observed a different magnetic field dependence for the flux-pinning in these two cases--a strong "peak effect"[10] is observed in one case and not in the other. This interesting behavior is under continuing study with the hope of gaining further understanding of those mechanisms which cause the "peak effect".

Any reports or publications on the above studies will be added as supplements to this report.

We also irradiated a $Nb_{0.42}Ta_{0.58}$ alloy sample with a total proton fluence of about 2×10^{17} p/cm^2. This represents about 3 to 4 times the fluence given the pure Nb samples. However, no change in the flux-pinning was observed at all. We conclude that irradiation damage is much more effective in pinning flux in pure type II superconductors than in alloys. No further work on the alloy samples is planned at this time.

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APPENDIX A

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[87] H.C. Freyhardt, I.D.M.F.P.S. (see ref. 16) p. 98.
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Discussion

H. Kerchner: Would you explain why you neglected the $\Delta\kappa$ interaction in calculating the elementary pinning force of dislocation loops?

E. Kramer: The problem of computing f_p due to the $\Delta\kappa$ change is that one must average changes in κ over a volume approximately equal to the electron mean free path l^3. (It makes no sense to talk about a value of κ more localized than l^3.) Since l in these samples is larger than the FLL parameter and approaches or exceeds the distance between defects, any $\Delta\kappa$ changes will be very smeared out and in my opinion negligible compared to the stress field interaction. [This discussion continues after Kerchner's paper.]

H. Weber: Concerning the master curve ($Q - f_p$) shown in your last slide I should draw your attention to our new results on the pinning forces exerted by normal conducting Nb_2N precipitates in NbTa-polycrystals (I. Adaktylos and H.W. Weber: Phil. Mag. 35 (1977) 83. and Nb single crystals (work in progress). In both cases, which refer to relatively low precipitate concentrations, the results follows more closely to the Labusch line than indicated in your figure. So I think, that in these systems the quadratic summation law is correct and the problems with the threshold criterium could be resolved on the basis of your argment, i.e. by taking the flux line lattice defects into account.

E. Kramer: 1) As I indicate in my text, but unfortunately had not time to cover in my lecture, the quadratic summation is really not correct theoretically. As was first shown by Campbell, when one uses a correct periodic potential rather than a single well potential one gets a Q vs. f_p curve which is asymptotic to a linear, rather than a quadratic dependence.

2) There are severe problems with trying to compute the core interaction f_p for a large disk-shaped particle. I am not convinced that your method of estimating f_p the particle on the FLL as the force exerted on an isolated flux line is anything better than an order of magnitude estimate for such particles and your data fall within an order of magnitude of the summation curve for voids.

R. Rollins: We have recently collaborated with Dr. J. Bentley at ORNL to obtain transmission electron micrographs of the irradiation damage structure in our proton-irradiated Nb samples. We observe the damage to be dislocation loops and, relevant to your paper, we have measured the volume flux pinning, F_p, and the average size and density of the dislocation loops in the same sample. Thus, we can add a point to your plot of F_p/ρ vs. f_p. Our numbers are:

$$F_p(b = 0.7) = 10^6 \text{ dynes/cm}^3 , \quad b \equiv B/B_{c_2} ,$$

$$D_{ave} \simeq 300 \text{ A} \rightarrow f_p(b = 0.7) \simeq 10^{-8} \text{ dynes} ,$$

and

$$\rho \simeq 8 \times 10^{14} \text{ loops/cm}^3 ,$$

or

$$F_p/\rho \sim 10^{-9} \text{ dynes} = 10^{-14} \text{ N} ,$$

$$f_p \sim 10^{-8} \text{ dynes} = 10^{-13} \text{ N} .$$

E. Kramer: That point falls right on the summation curve in very good agreement with both our dislocation loop data and the void data. Significantly your loop density of 8×10^{20} m^{-3} is an order of magnitude below our lowest loop density giving further proof that Q is really independent of defect density in this density range.

Discussion

The following comment was given by R. Labusch of the Institut fur angewandte Physik der TU Clausthal, Germany.

D.1. Introduction

Since its publication the statistical theory of flux pinning in type II superconductors [1] has been applied to many experimental results and has found acceptance as well as criticism. According to this theory the volume pinning force F_p of a random distribution of pinning centers is approximately

$$F_p = \frac{B}{2\phi_0} \rho d f_0^2 G'(0) , \tag{D.1}$$

where ρ is the volume density of pins, d their diameter (or range of interaction) perpendicular to the driving force, f_0 the maximum interaction force between a single center and a flux line and $G'(0)$ the Green's function of the flux line lattice (FLL), representing the response of the FLL to a localised force of unit strength. $G'(0)$ was calculated in terms of the magnetic induction B and the elastic constants c_{66}, c_{11}, c_{44} of the FLL, using a continuum approximation and linear

APPENDIX B

Abstract Submitted
for the Washington, D.C. Meeting of the
American Physical Society
March 27 - 30, 1978

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Experimental Superconductivity

Flux Pinning and Dislocation Loops Observed in Proton-Irradiated Nb.* Y. ANJANEYULU† and R.W. ROLLINS, Ohio University**--Very pure annealed Nb samples were irradiated with 3.2 and 5 MeV protons to fluences of about 6.8×10^{16} p/cm^2. Local magnetic induction profiles, when the samples were in the superconducting mixed state, were obtained using an ac technique. Electron micrographs of the same irradiated samples show dislocation loops in the damaged region of the samples. The critical current and the volume flux pinning force F_v, are determined from the slope of the measured field profiles. Assuming pinning due to the observed dislocation loops, F_v calculated from a direct summation law is an order of magnitude higher than the experimental result and F_v calculated from quadratic summation laws is two to three orders of magnitude smaller than measured.

*Submitted by R.W. Rollins

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Prefer Poster Session

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Y. Anjaneyulu and R. W. Rollins, J. Am. Phys. Soc. 23, 322 (1978).

APPENDIX C

Magnetic Flux Pinning in Proton-Irradiated Thick Nb Samples*

by

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Departement of Physics, Ohio University
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Abstract

Very pure annealed Nb Samples having resistivity ratio, $\Gamma \equiv \rho_{296}/\rho_{4.2}$, in the range of 1300 to 1700 were irradiated with 3-8 MeV protons to fluences of 6.8×10^{16} p/cm^2 and 3.4×10^{16} p/cm^2. Local magnetic induction profiles, when the samples were in the superconducting mixed state, were obtained using the ac technique of Rollins, Küpfer and Gey. Electron micrographs of the irradiated samples show that the damage caused by proton irradiation was in the form of dislocation loops. The volume pinning force F_v, calculated for the dislocation loops using flux pinning models and theories for b = 0.7 and T = 4.2 K, is compared with the experimentally obtained F_v. The F_v calculated from direct summation law is an order of magnitude higher than the experimental result and the F_v calculated from models, which use Labusch quadratic summation law, is two to three orders of magnitude smaller than the experimental value. Our experimental pinning force per dislocation loop is in agreement with a master curve obtained by Kramer as an experical solution to the summation problem. Experimentally

obtained F_v for temperatures lying between 2 K and 7 K obey a scaling law. If the temperature dependence is written as $F_v \propto H_c^n$, then we found n to be slightly dependent on depth x from the sample surface. At x = 20 μm, n = 3.2 ± 0.2; while at x = 84 μm, n = 2.7 ± 0.2. The experimental scaling law is compared with the scaling laws obtained from Labusch model for point pinning centers and Kramer's model for line pinning centers. Irradiated samples were observed to have a region at the surface with no apparent pinning. This observation is discussed in terms of two different effects; (i) reversible motion of flux lines and (ii) a threshold for flux pinning.

I. Introduction

We have made local magnetic induction profile measurements, when the proton-irradiated Nb samples were in the superconducting mixed state, using the ac technique of Rollins et al.[1] The position dependent volume pinning force F_v has been obtained from the flux profile measurements. The experimental set up to obtain the local magnetic induction profiles and annealing of Nb samples prior to irradiation have been described previously by the authors.[2] The damage caused by the proton irradiation of our Nb samples at room temperature was observed by transmision electron microscopy and found to be in the form of dislocation loops. Limited electron microscopic work indicated the size and concentration of the loops to be dependent on the depth in the region between the outer surface and the range of protons. The dislocation-loop/flux-line-lattice pinning models,[2] which we described previously to explain the position dependent flux pinning in proton-irradiated Nb samples, are discussed in terms of pinning by the observed dislocation loops. A plot of F_v, measured at a particular depth from the sample surface, versus reduced field, $b = B/B_{c2}$, shows a peak near H_{c_2} similar to the results in neutron irradiated Nb observed by Agrawal, et al.[3] The experimentally determined F_v for reduced fields below the peak region is discussed in terms of flux pinning theories and models. The peak in F_v near H_{c2} is discussed in terms of Pippard's phenomenological model[4] for "peak effect" together with Kramer's criterion for line forces[5] and the elementary pinning force f_p between a dislocation loop and the flux line lattice.[6] We have made magnetic induction profile measurements for temperatures ranging from 2 K to 7 K to obtain the temperature dependence of F_v. Experimentally obtained temperature scaling laws for F_v are compared with Kramer's model for scaling laws for flux pinning in hard superconductors and Labusch flux pinning model[7] for point like pinning centers. We have observed what appears to be a pinning free surface layer of 30 - 40 μm in the case of 5 MeV proton irradiated sample and 5 - 10 μm in 3.2 MeV proton irradiated sample. We discuss this region in terms of two different effects: (i) reversible motion of flux lines proposed by Campbell[8,9] and (ii) a threshold for flux pinning.

II. Characterization of Annealed Nb Samples

Preirradiation sample preparation was as previously described.[2] Four probe resistivity ratio, $\Gamma \equiv \rho_{296}/\rho_{4.2}$, of the annealed Nb samples ranged from 1300 to 1700. Koch et al.[10] have studied the effects of interstitial oxygen on the superconducting properties of Nb. Using their linear relationship between electrical resistivity at 4.2 K, $\rho_{4.2}$, and atomic percent of oxygen, we estimate that our annealed Nb samples may contain less than or equal to 20 to 26 ppm of oxygen. Magnetization curves obtained on a similarly annealed Nb cylinder show practically no hysteresis. We have calculated the elastic constants C_{11}, C_{44} and C_{66} of the flux line lattice from the magnetization curves using the relationships obtained by Labusch.[11,12] The temperature dependence of the elastic constants has been determined from the magnetization curves, which were obtained from 1.5 K to near T_c. Fig. 1 shows a plot of C_{44}, C_{66} and $(C_{44}\ C_{66})^{\frac{1}{2}}$ as a function of H_{c2} for a reduced field b = 0.7. Here we have used the Labusch[12] relation near H_{c2} to calculate C_{66}. Our estimate of error in determining C_{44} is about 2% and C_{66} is about 5%. Recently Schmucker and Brandt[13] have obtained elastic constants of the flux line lattice with non-local effects taken into consideration. Using this theory, we find the displacement of flux line lattice per unit pinning force is larger by a factor of three than that obtained from Labusch relationships for b = 0.7.

We made pre-irradiation induction profile measurements on the annealed Nb samples to see that the samples have low pinning before proton bombardment. The annealed samples, which had practically negligible pinning, were selected for proton bombardment. Our criterion for practically negligible pinning was that an ac field of amplitude 10 Oe should penetrate from the outer surface to the center of sample for dc fields lying between H_{c1} and H_{c2} at 4.2 K.

III. Proton Irradiation of Nb Samples at Room Temperature

Protons in the energy range of 3 to 8 MeV were used to irradiate the Nb samples. The required energy proton beam was supplied by the 11 MeV Tandem Van de Graaff accelerator of the Ohio University Accelerator Laboratory. The beam current was adjusted so that all the samples were exposed to the same amount of incident beam power, which we maintained at 9 Watts. A complete description of the radiation damage station placed in the beam line at the Accelerator Laboratory to irradiate the samples at room temperature is given by Anjaneyulu.[14] The radiation chamber was connected to an evacuated 2 inch beam line. An aperture 12.3 mm in diameter, and a rectangular slit (1 mm X 3 mm) were used to collimate the beam on to the sample. The sample was epoxied to a brass tube which was soldered to a 0.25 mm thick and 6.3 mm diameter stainless steel tube. A stainless steel capillary was positioned coaxially inside the stainless steel tube to circulate water through the sample during proton bombardment. Deionized distilled water, which was circulated from a tank to the sample, slit and aperture using a pump, was chilled just before it entered the sample through the capillary.

With the sample pulled out of the way, the proton beam was collimated and tuned to obtain the required beam current on the beam stop with minimum power dissipation on the slit and aperture. The sample with a dynamic vacuum seal on the stainless stell tube was lowered into the radiation chamber and positioned so that the proton beam struck the cylindrical surface of the sample perpendicularly. The stainless steel tube was coupled to a motor with a toothed belt to rotate the sample at a rate of about 1 rev/sec and at the same time the frame with the stainless steel tube and sample slewed on linear bearings back and forth at a rate of 0.1 cm per minute. The radiation chamber along with the beam line was electrically grounded and the electrical impedance between the sample and the ground was > 10 MΩ. The sample was uniformly irradiated with protons except for a length of about 4 mm at both ends. The vacuum in the radiation chamber and the beam line was about 2×10^{-6} torr during the proton bombardment.

The total fluence of protons incident on the sample was obtained by integrating the sample current. The measured sample current was found to be 10 - 15% larger than that measured on the beam stop. This increase in sample current was due to the electron emission from the sample surface. A correction of 10 -15% was made in the total charge to calculate the fluence of protons. Typically it took about 5 hours to irradiate about 2.5 cm of the central portion of a 4.2 mm diameter sample to a fluence of 6.8×10^{16} p/cm^2 using 5 MeV incident protons at a beam current of about 1.8 μamp. The beam current was changed when exposing the sample to protons of other energies such that the power dissipated in the sample was constant.

IV. Local Magnetic Induction Profiles of Proton-Irradiated Nb Samples

Local magnetic induction profile measurements on the proton irradiated Nb samples show a drastic increase in flux pinning at a depth approximately equal to the range of protons. Protons of 3 - 8 MeV energy have a range of about 47 - 221 μm in niobium.[15] Transmission electron microscopic study of the irradiated samples has shown that the damage caused by proton irradiation was in the form of dislocation loops. We believe that the dislocation loops act as pinning centers for the magnetic flux lines when the sample was in the superconducting mixed state.

Fig. 2 shows the deviation in local magnetic induction as a function of depth for some dc fields lying between H_{c1} and H_{c2}. These profiles were obtained at 4.2 K. 3.2 MeV protons have a range of about 51 μm in Nb. The sharp bend in the profiles at a depth of about 50 μm is an indication of the range of protons. The portion of the profiles close to the surface up to a depth of about 10 μm indicates practically no pinning and the portion of the profiles beyond the range of protons was found to be similar to the annealed sample profiles.

Local magnetic induction profile measurements for temperatures ranging from 2 K to 7 K were made to obtain the temperature dependence of volume pinning force F_v. The profiles obtained at 4.2 K when the sample was wet immersed in liquid helium and when it was dry in vacuum with the temperature controlled to 4.2 K agree with each other, and this agreement confirms that there was no heating of the sample during the measurements. The profiles obtained at three different temperatures at approximately same reduced field b are shown in Fig. 3.

Samples irradiated with different energy protons showed approximately similar results except for the obvious change in the range of the protons. Fig. 4 shows the profiles obtained on three samples irradiated with 3.2 MeV, 5 MeV and 8 MeV protons having approximate ranges of 47 μm, 100 μm and 220 μm respectively.[15] The difference in the shape of the profiles is possibly due to the difference in the nature of annealed samples before irradiation.

We have checked the frequency dependence of magnetic induction profiles. The profiles obtained at 0.7 Hz and 2.4 Hz agree with each other, where as those obtained at 13 Hz were quite different. The penetration of ac field in the proton irradiated samples was usually less than 0.15 mm, which is a factor of 10 smaller than the flux flow skin depth at 13 Hz. The change in the shape of induction profiles at 13 Hz is probably attributable to small phase shifts caused by the normal skin effect in these samples which have such high resistivity ratios. No detailed study was done and we mention it only as an experimental indication that care must be taken to insure the frequency is low enough when using this technique.

A surface layer of up to 40 μm, in the case of 5 MeV proton irradiated samples, showed apparently no pinning. We used ac field amplitudes of ~ 5 Oe to see the shape of the profiles in this region and Fig. 5 shows some of the profiles. The profiles intersect the x-axis instead of passing through the origin. We observed the intersection of the induction profile with B = Bav at a depth of 30 - 40 μm in 5 MeV proton irradiated samples and 5 - 10 μm in the 3.2 MeV proton irradiated sample. This apparent zero pinning region will be discussed further in section IV.

V. Transmission Electron Microscopy

We used the JEOL JEM 100C transmission electron microscope at Oak Ridge National Laboratory to study the defect structure in the proton irradiated Nb samples. The microscope was equipped with ± 45 degree double tilt side entry stage and operated at 120 KV. We collaborated with Dr. J. Bentley at ORNL to do themicroscopic work on our samples.

The thin specimens for electron microscopy were prepared by first chemically polishing the outer surface of the sample to the required depth and then back thinning until a performation was observed. The sequence of steps in preparing the specimens for the microscopy is shown in Fig. 6. Initially we cut the sample into two halfs. The outer surface of one half was chemically polished to the required depth in a solution of 70% HNO and 30% HF by volume at 0 C. We were chemically polishing the surface at a removal rate of about 10 μm per minute as determined by weight loss measurements. A 3 mm long cylinder was sliced out of the polished half of the sample and then the 3 mm cylinder was sectioned to obtain a 0.5 - 0.75 mm thick plano-cylindrical shaped specimen. The plane surface of the specimen, which was a square approximately 3 mm on a side, was dimpled to a depth of 0.4 - 0.6 mm using jet electropolishing technique.[16] We used an electrolyte consisting of 300 cc methyl alcohol, 36 cc H_2SO_4 and 4cc HF for the jet.

The specimen was maintained at +255 Volts with respect to the jet and a current of 150 MA was measured during dimpling. Finally, the dimpled area was chemically polished with the jet until a performation was observed using the standard light source and microscope method.[16] In preparing a specimen, the main error in depth was due to the assumption that the outer surface of the sample was chemically polished uniformly. We estimate an error of about ± 15% depth for which the specimen was prepared.

Figs. 7a and 7b show the bright field electron micrographs of the proton irradiated Nb samples. The dark paranthesis like structure is characteristic of inclined dislocation loops.[17] Oen et al.[18] have observed a similar defect structure in copper irradiated with protons at room temperature.

The electron micrograph shown in Fig. 7a was obtained at a depth of 4 ± 0.5 μm from the surface of the 5 MeV proton irradiated sample. In this case we found $\langle D \rangle$ = 239.5 Å, $\sqrt{\langle D^2 \rangle}$ = 239.5 Å and ρ_L = 4.53 X 10^{14} loops/cm^3. The electron micrograph shown in Fig. 7b was obtained at a depth of 30 ± 5 μm from the 3.2 MeV proton irradiated sample. The density of dislocation loops has approximately a gaussian distribution when plotted as a function of diameter of the loops. The average diameter $\langle D \rangle$ and the root mean square diameter $\sqrt{\langle D^2 \rangle}$ of the loops was found to be 301.5 Å and 311.0 Å respectively and the concentration of the loops ρ_L = 7.63 X 10^{14} loops/cm^3. The percentage of point defects residing in the loops ranged from 1 to 3% of the calculated total point defects produced. Similar low retention of point defects was observed by Oen et al.[18] in copper irradiated with protons at room temperature. We did not see any dislocation loop structure in the case of a specimen prepared for a depth greater than the range of protons. Nearly all the micrographs that we had taken corresponded to a depth of 60 to 80% of the range of the protons. From the analysis completed on these micrographs, we are not able to draw definite conclusions about the size and density of loops as a function of depth. It appears that both size and density of loops increase as we approached the range of the protons.

VI. Analysis and Discussion

The depth dependent volume pinning force F_v (dynes/cm^3) = {1/10 μ_{eq}(B)} {B(G) j_c(amp/cm^2)} was calculated by numerically differentiating a smooth curve drawn through the data obtained at 4.2 K. Fig. 8 shows F_v plotted as a function of reduced field b for three different depths, x = 65, 80 and 90 μm. We will try to compare our experimental result for F_v obtained for a field away from the peak at b = 0.7 corresponding to x = 80 μm with the calculated values for F_v using different flux pinning theories and models. We will use the size and density of dislocation loops shown in micrograph (Fig. 7b), which was obtained from 3.2 MeV proton irradiated sample at x = 30 μm, to calculate F_v. The dislocation loops have a root mean square diameter $\sqrt{\langle D^2 \rangle}$ = 311 Å and density ρ_L = 7.63 X 10^{14} loops/cm^3. It is reasonable to expect similar dislocation loop structure at x = 80 μm in 5 MeV proton irradiated sample for a given fluence of protons, since 3.2 and 5 MeV protons have ranges of about 50 and 100 μm respectively in Nb. Experimentally we find F_v = 0.9 ± 0.05 X 10^{16} dynes/cm^3 at x = 80 μm in 5 MeV proton irradiated sample and F_v = 1.4 ± 0.1 X 10^6 dynes/cm^3 at x = 30 μm in 3.2 MeV proton irradiated sample at 4.2 K. To calculate F_v, first we need to know the elementary pinning force f_p between a dislocation loop and the flux line lattice. Kramer[6] calculated numerically the so called first-order and second-order interactions of a dislocation loop with the flux line lattice. Pande[19] on the other hand, found an analytical solution for the first order interaction. Kramer's results agree with Pande's results for dislocation loops having diameter D smaller than the flux line lattice spacing a_o. The first order interaction seems to be somewhat larger than the second order interaction.[20] We use only first order interaction to estimate f_p between a dislocation loop and the flux line lattice. From Kramer's numerical results for first order interaction[6], we find $f_p \simeq 1.0 \times 10^{-8}$ dynes at b = 0.7 and T = 4.2 K for dislocation loops of diameter 311 Å.

Next problem to solve after obtaining f_p is, how to sum these elementary forces f_p to get the volume pinning force F_v. A simple solution to the summation problem is the direct summation model proposed by Dew-Hughes.[21] Assuming every pinning center exerts a force of f_p on a flux line, $F_v = \rho_v f_p$, where ρ_v is the volume density of pinning centers. Using $\rho_v = \rho_L = 7.63 \times 10^{14}$ loops/cm^3 and $f_p = 1 \times 10^{-8}$ dynes, $F_v = 7.63 \times 10^6$ dynes/cm^3, which is about an order of magnitude larger than the experimental result. The other summation model is the quadratic summation model developed by Labusch.[7] Labusch calculated F_v for point like pinning centers, in which he takes into account the finite rigidity of flux line lattice. According to this theory, F_v is given by (Campbell and Evetts[22] equation 6-12)

$$F_v = \frac{2d\ \rho_v\ f_p^2}{8\pi\ \mu_{ep}\ \beta} \left(\frac{B}{\phi_o}\right)^{3/2}, \tag{1}$$

where 2d is the range of interaction, $\mu_{ep} \simeq (C_{44}C_{66})^{\frac{1}{2}}/\beta\sqrt{\pi}$, $\beta = (2/\sqrt{3})^{\frac{1}{2}} = 1.07$ and $\phi_o = 2 \times 10^{-7}$ gauss - cm^2. Using the range of interaction 2d to be equal to the diameter of the loops D and also using the elastic constants shown in Fig. 1 at 4.2 K, we find $F_v = 7.9 \times 10^2$ dynes/cm^3 at b = 0.7. The F_v calculated from Labusch theory is about three order's of magnitude smaller than experimental result. Schmucher and Brandt[13] calculated F_v for point pinning centers using a non-local theory. According to them, it is necessary to consider non-local effects on elastic constants to calculate the displacement of flux line lattice and the non-local effects become important for $bk^2 > (1-b)$, where $b = B/B_{c2}$ and k is the Ginsburg-Landau parameter. Using Schmucker and Brandt model, we find $F_v = 2.7 \times 10^3$ dynes/cm^3, which is about a factor of three larger than F_v calculated from Labusch model.

We have also checked the possibility that the dislocation loops may be dense enough so that the loops in a row may act as line force. If the point pinning centers are closer than certain critical distance $\ell^* = \sqrt{C_{44}\phi_o/C_{66}B}$, then the effect of point pinning centers can be considered as line forces.[5] The spacing between the dislocation loops ℓ is approximately given as $\ell = \rho_L^{-1/3} = (7.63 \times 10^{14})^{-1/3} = 1.1 \times 10^{-5}$ cm; where as the $\ell^* = 1.5 \times 10^{-4}$ cm for b = 0.7 and at T = 4.2 K. Following Kramer, we find the dislocation loops do satisfy the criterion for line forces. The elementary pinning force per unit length $f_p^\ell \equiv f_p^\ell/\ell \simeq 10^{-3}$ dynes/cm. The displacement of flux line lattice due to f_p^ℓ is given as[5] $u^\ell = (f_p^\ell/4\sqrt{\pi}\, C_{66}) = 10$ Å and the ratio between the displacement and flux line lattice spacing $u^\ell/a_o = 0.009$. An expression similar to the equation (1) can be used to find F_v due to line pinning, which is given by

$$F_v = d\, \rho_a\, f_p^\ell\, u^\ell \left(\frac{B}{\phi_o}\right), \qquad (2)$$

where d is half the range of interaction, ρ_a is the area density of pinning centers and u^ℓ is displacement of flux line lattice due to f_p^ℓ. A similar expression for point pinning centers was used by Schmucker and Brandt.[13]

Using (2), we find $F_v = 1.14 \times 10^4$ dynes/cm^3, which is about two orders of magnitude smaller than the experimental result. The F_v, calculated from models which use Labusch quadratic summation law, is two to three orders of magnitude smaller than the experimentally determined value, whereas the direct summation law gives an upper limit to F_v.

Since neither summation model gives the right value of F_v, Kramer[20] has shown an empirical solution to the summation problem. He plotted experimentally obtained specific pinning force per defect $Q \equiv F_v/\rho_v$ versus the theoretically determined f_p for pinning centers introduced by particle irradiation. The points fall on a master curve, which lies in between the curves obtained by direct summation and quadratic summation models. For the dislocation loops that we have been discussing, the points ($Q = 1.2 \times 10^{-9}$ dynes for 5 MeV sample and $Q = 1.8 \times 10^{-9}$ dynes for 3.2 MeV sample; $f_p = 10^{-8}$ dynes) fall on the Kramer's master curve.

Magnetic flux pinning in proton irradiated Nb samples was found to be a strong function of depth. The electron micrographs of the irradiated samples show that the damage was in the form of dislocation loops. The point defects produced by proton bombardment are mobile at room temperature and form into dislocation loops. The size and density of dislocation loops depend very much on the nucleation process. We explained previously[2] the local magnetic induction profiles in terms of pinning models based on the interaction between the dislocation loops and the flux line lattice. We considered three models to obtain estimates for the density of dislocation loops $\rho_L(x)$. The three models differ in the nucleation process responsible for dislocation loop formation; (i) cluster nucleation model, (ii) induced defect nucleation model and (iii) precursory defect nucleation model.

In the case of cluster nucleation model, it was assumed that the vacancies and interstitials of a defect cluster, produced by a primary knocked off Nb atom, do not diffuse very much from the location where they were created and the point defects of the cluster form into a dislocation loop. It turns out that it is not possible to obtain the size of dislocation loops, which we see in the electron micrographs, from the defect clusters. For example, the electron micrograph shown in Fig. 7b has dislocation loops of $\sqrt{<D^2>}$ = 311 Å, where as a dislocation loop that could form from the largest defect cluster corresponding to this micrograph, calculated on the basis of Rutherford scattering theory and Kinckin-Pease model , was found to have a diameter of 130 Å. So the cluster nucleation model does not lead to a reasonable description of either the induction profiles or the TEM micrographs for our proton irradiated Nb samples. The induced defect nucleation model assumes $\rho_L(x)$ to be proportional to the density of point defects produced by the proton bombardment, whereas the size of loops was assumed to be a constant. The precursory defect nucleation model assumes that the dislocation loops are nucleated from the impurity atoms and/or such inhomogeneities, which were present in the sample before irradiation, and hence ρ_L was assumed to be independent of depth x. In this model the diameter of loops D is proportional to the local density point defects calculated from Rutherford Theory and Kinchin-Pease model.[23]

We believe, from the limited electron microscopic work, both the size and density of dislocation loops increase as we approached the range of protons from the outer surface. Both the induced defects produced by proton irradiation and the precursory defects existing in the sample before irradiation may act as nucleation sites for dislocation loops. The relationship connecting both the size and density of loops is $\rho_L \langle D^2 \rangle \propto \rho_\nu(E_1)$, where ρ_L is the density of loops, $\langle D^2 \rangle$ is the mean square diameter and $\rho_\nu(E_1)$ is the calculated density of point defects. The ρ_L is a sum of two densities, one nucleated from the precursory defects and the other nucleated from induced defects; i.e. $\rho_L = \rho_o + A\,\rho_\nu(E_1)$, where ρ_o and A are constant parameters. Using this approach, it would be possible to build a model that includes both the size and density of loops.

Our experimental results of F_v versus b show the so called "peak effect" near H_{c2} (See Fig. 8). Similar peaks in F_v have also been observed in neutron irradiated Nb.[3,24,25] Fig. 8 shows that the peak in F_v increases in height, broadens and shifts to lower b as the depth approaches the range of protons. Pippard[4] proposed an explanation for the peak effect based on the idea that the resistance of flux lines to shear decreases as the field approaches H_{c2}. The peak effect may be expected to occur when the pinning is due to line forces.[26] Using the Kramer's criterion for line pinning, we find $f_p^\ell = 2.6 \times 10^{-4}$ dynes/cm for b_p = 0.92 at which the peak occurs corresponding to the depth x = 80 μm (see Fig. 8) and the ratio of displacement of flux line lattice to its spacing $K \equiv u^\ell/a_o = 0.04$, where K is the Pippard's phenomenological factor.[22] At the peak the flux lines shear to reach the pinning centers. The volume pinning force at the peak is given by[22]

$$(F_v)_{peak} = \left(\frac{B_p}{\phi o}\right) f_p^\ell = 3.3 \times 10^6 \text{ dynes/cm}^3 \tag{3}$$

where B_p is the magnetic induction at the peak. The value given by (3) is comparable to the experimental result. We found $(F_v)_{peak} = 2.4 \pm 0.2 \times 10^6$ dynes/cm^3 in 3.2 MeV proton irradiated sample at x = 30 μm and $(F_v)_{peak}$ = $2.2 \pm 0.2 \times 10^6$ dynes/cm^3 in 5 MeV proton irradiated sample at x = 80 μm.

Pippard's model with Kramer's criterion for line force does give a reasonable value at the peak for F_v.

The temperature dependence of F_v was obtained from the induction profile measurements made at temperatures between 2 K and 7 K on 8 MeV proton irradiated sample. Fig. 9 shows reduced volume pinning force F_v/F_{vR} plotted as a function of b for four different temperatures. In this case the slopes on the profiles were taken at a depth x = 20 μm. F_{vR} is the volume pinning force corresponding to the flat portion of F_v vs b curve for each temperature. The F_v obtained at different temperatures seem to obey a scaling law. If the temperature dependence of F_v is written as $F_v \propto H_{c2}^n$, then we find n to be slightly dependent on depth. At a depth of x = 20 μm, n is equal to 3.2 ± 0.2; while at x = 84 μm, n has a value of 2.7 ± 0.2. In Kramer's model for scaling laws for flux pinning in hard superconductors, the pin breaking volume pinning force F_p has a temperature dependence which is given by $F_p \propto H_c^{8.5}/\kappa_1^8 C_{66}^3$ (Ref. 5; using equations (13 and (15)). Labusch[7] expression for F_v for point pins has a temperature dependence which is given by $F_v \propto (f_p^2/\mu_{ep})(B/\phi_o)^{3/2} \propto H_{c2}^{5.5}/\{\kappa_1^4(C_{44}C_{66})^{\frac{1}{2}}\}$, where we have used[27] $f_p = \Omega H_c^2(T)(1-b)$ and Ω is relatively temperature and field independent. The experimentally obtained F_v in arbitrary units plotted as a function of H_{c2} is shown in Fig. 10 along with the temperature scaling laws obtained from Kramer[5] and Labusch[7] models. As it turns out, the experimental results are in general agreement with both models.

Magnetic induction profile measurements using very small ac field amplitudes of 5 Oe show that the surface layer of 5 - 10 μm in 3.2 MeV proton irradiated sample and 30 - 40 μm in 5 MeV proton irradiated sample behave as if they were normal metal layers. The profiles, instead of passing through zero at the surface, intersect the x-axis at a finite depth. A somewhat similar phenomenon was observed by several authors (see references in Ref. 8) using different experimental techniques. The signal at small ac field amplitudes from a low pinning material has been observed[8,9] to be a linear function of amplitude, whereas the critical state model would predict the signal to be proportional to the square of the amplitude. Campbell[8,9] has described the linear response in terms of the reversible motion of the flux lines in potential wells representing the effect of pinning centers. As a result there is a linear and reversible penetration of the ac field up to a depth λ', known as the pinning penetration depth, which is similar to the penetration depth in type I superconductors. For the case of an infinite slab $\lambda'(\text{cm}) = (10\ B_o\ d/4\pi j_c)^{1/2}$ $(\text{G cm}^3/\text{Amp})^{1/2}$, where B_o is the magnetic induction obtained from reversible magnetization curve, d is the distance that the flux lines must be moved before most of them become free from the pinning potential wells and j_c is the critical current density.

Applying the Campbell model[8] to our technique for obtaining the induction profiles leads to a profile which should suddenly drop to zero at the depth λ'. Our estimation of λ' in the case of 5 MeV proton irradiated sample is about 15 μm for B_o = 2250 G. Campbell model assumes j_c and d to be independent of x and so does not really apply to our case. On the basis of Campbell model, it is reasonable to expect that the actual induction profiles approach zero with j_c values much less than those obtained at the intersection of profiles with the x axis. The reversible motion of flux lines could lead to large errors at the surface, but this error decreases rather drastically as j_c increases.

Perhaps we should mention that this apparent no pinning region may also be explained in terms of the Labusch[7] threshold criterion for pinning. Fig. 7a shows the dislocation loop structure observed at a depth of 4 μm in the 5 MeV proton irradiated sample. These dislocation loops, or for that matter any other dislocation loops that we have observed so far in proton irradiated Nb samples, do not satisfy the Labusch threshold criterion for pinning. This situation has been discussed recently by Kramer.[20] If the region near the surface has such weak pinning that the threshold criterion of Labusch actually applies, then we would expect the apparent no pinning region as observed.

VII. Summary and Conclusions

Very pure annealed Nb samples having practically negligible pinning were irradiated at room temperature with protons in the energy range of 3-8 MeV. The magnetic flux pinning in the proton irradiated Nb samples, when they were in the superconducting mixed state, was determined using the technique of Rollins, Küpfer and Gey. Transmission electron micrographs or regions in the irradiated samples confirmed that the damage caused by proton irradiation was in the form of dislocation loops. From the limited electron microscopic work, we are not able to conclude definitely about the size and density of loops as a function of depth. It appears though both the loop size and density of the loops increase as we approached the range of protons from the outer surface of the sample.

The depth dependent volume pinning force F_v was determined from the experimental induction profile data. A plot of F_v versus b shows the peak effect near H_{c2}. Experimentally determined F_v for a field away from the peak at b = 0.7 and T = 4.2 K was compared with the calculated values for F_v using different flux pinning theories and models. Experimentally F_v was determined to be $0.9 \pm 0.05 \times 10^6$ dynes/cm^3 at a depth x = 80 μm in 5 MeV proton irradiated sample and $1.4 \pm 0.1 \times 10^6$ dynes/cm^3 at x = 30 μm in 3.2 MeV proton irradiated sample. Both the samples were irradiated to a fluence of 6.8×10^{16} p/cm^2. The electron micrograph for x = 30 μm of 3.2 MeV sample show that the dislocation loops have a root mean square diameter of 310 Å and density $\rho_L = 7.63 \times 10^{14}$ loop/cm^3. The f_p for the loops is about 1×10^{-8} dynes. The calculated values for F_v are 7.63×10^6 dynes/cm^3 (direct summation model), 7.9×10^2 dynes/cm^3 (Labusch model for point pins), 2.7×10^3 dynes/cm^3 (Sckmucker and Brandt non local theory for point pins) and 1.4×10^4 dynes/cm^3 using line pinning criterion of Cramer and the Labusch summation model. The experimental value of the pinning force per loop, F_v/ρ_L, vs f_p falls on the master curve obtained by Kramer as an empirical solution to the summation problem. Pippard's phenomenological model combined with Kramer's criterion for line pinning seem to explain the peak in F_v near H_{c2} to within a factor of two. Experimentally determined F_v for temperatures between 2 K and 7 K obey a scaling law. If the temperature dependence is written as $F_v \propto H_{c2}^n$ then we found n to be slightly dependent on depth. At x = 20 μm, n = 3.2 ± 0.2; while at x = 84 μm, n = 2.7 ± 0.2. The experimental scaling laws are found to be approximately in agreement with the scaling laws obtained from Labusch model for point pins and Kramer's model for scaling laws in hard superconductors.

We have discussed the apparently no pinning region which is observed near the surface in terms of the Campbell model for reversible motion of a flux line in the potential wells representing the effect of pinning centers along a flux line and in terms of a region where the pinning is so weak that the Labusch threshold criterion applies and is not met. Further work is necessary if we are to further understand this region.

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FIGURE CAPTIONS

Fig. 1. Temperature Dependence of the flux-line lattice elastic constants.

Fig. 2. Magnetic induction profiles after proton irradiation. Essentially the same profile was obtained from the increasing and decreasing field portions of the cycle in all cases except when B_{ave} is in the neighborhood of the peak in $F_v(B)$ where F_v is changing rapidly with B. This asymmetry is evident for B_{ave} = 2450 G where ● are from db/dt > 0 and ○ are from db/dt < 0 portions of the cycle.

Fig. 3. The temperature dependence of the magnetic induction profiles.

Fig. 4. Magnetic induction profiles for samples irradiated with protons of different energies.

Fig. 5. Magnetic induction profiles obtained from very small amplitude ac field measurements shows an apparent zero pinning region near the surface.

Fig. 6. The sequence of steps used to prepare the sample for electron microscopy.

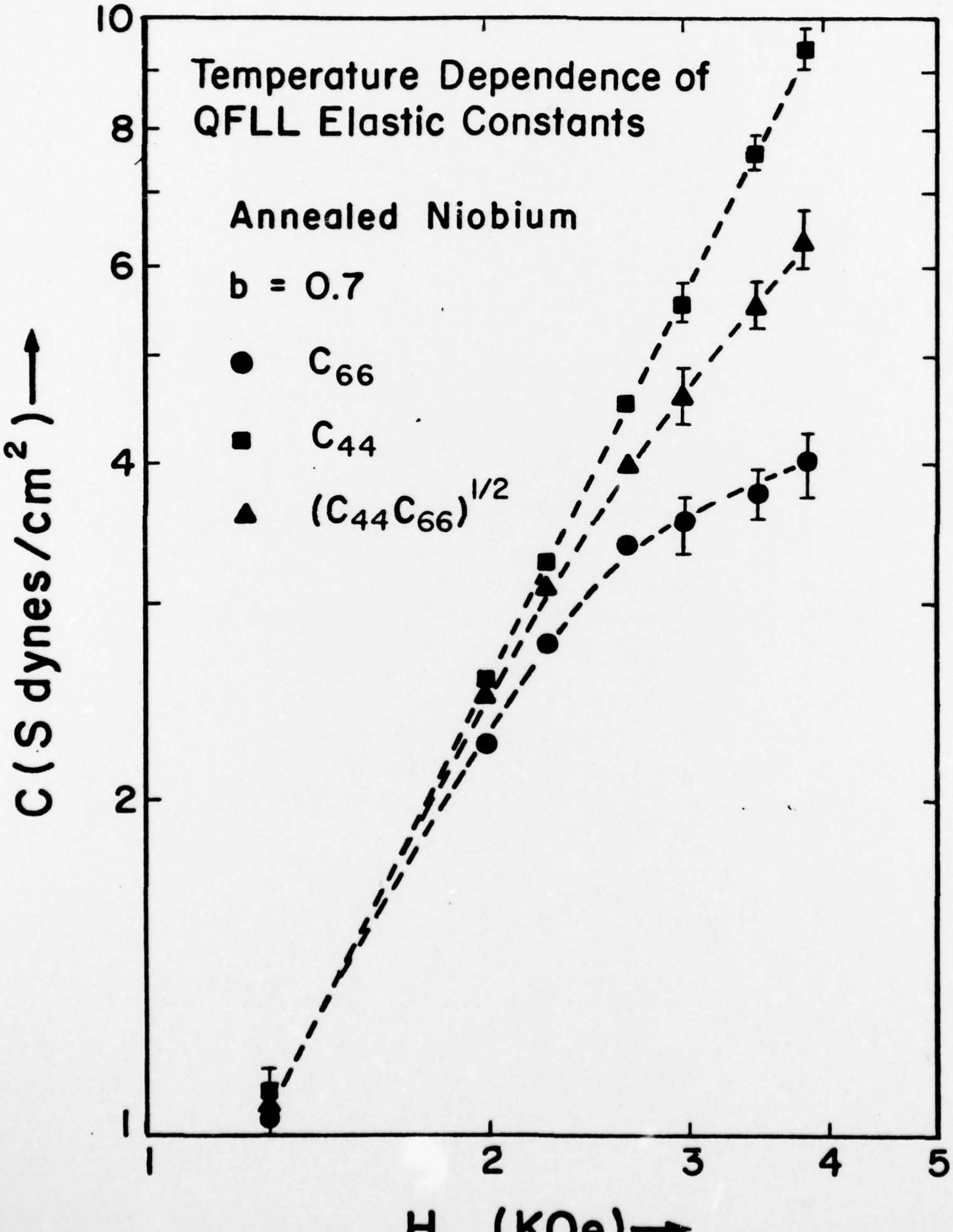
Fig. 7. Electron micrographs showing the defect structure. 7a shows an area taken from a depth of 4 μm. 7b shows an area taken from a depth of 30 μm.

Fig. 8. Volume pinning force, F_v, as a function of reduced field.. All with the temperature at 4.2 K.

Fig. 9. Reduced volume pinning force as a function of reduced field showing an approximate scaling law is obeyed.

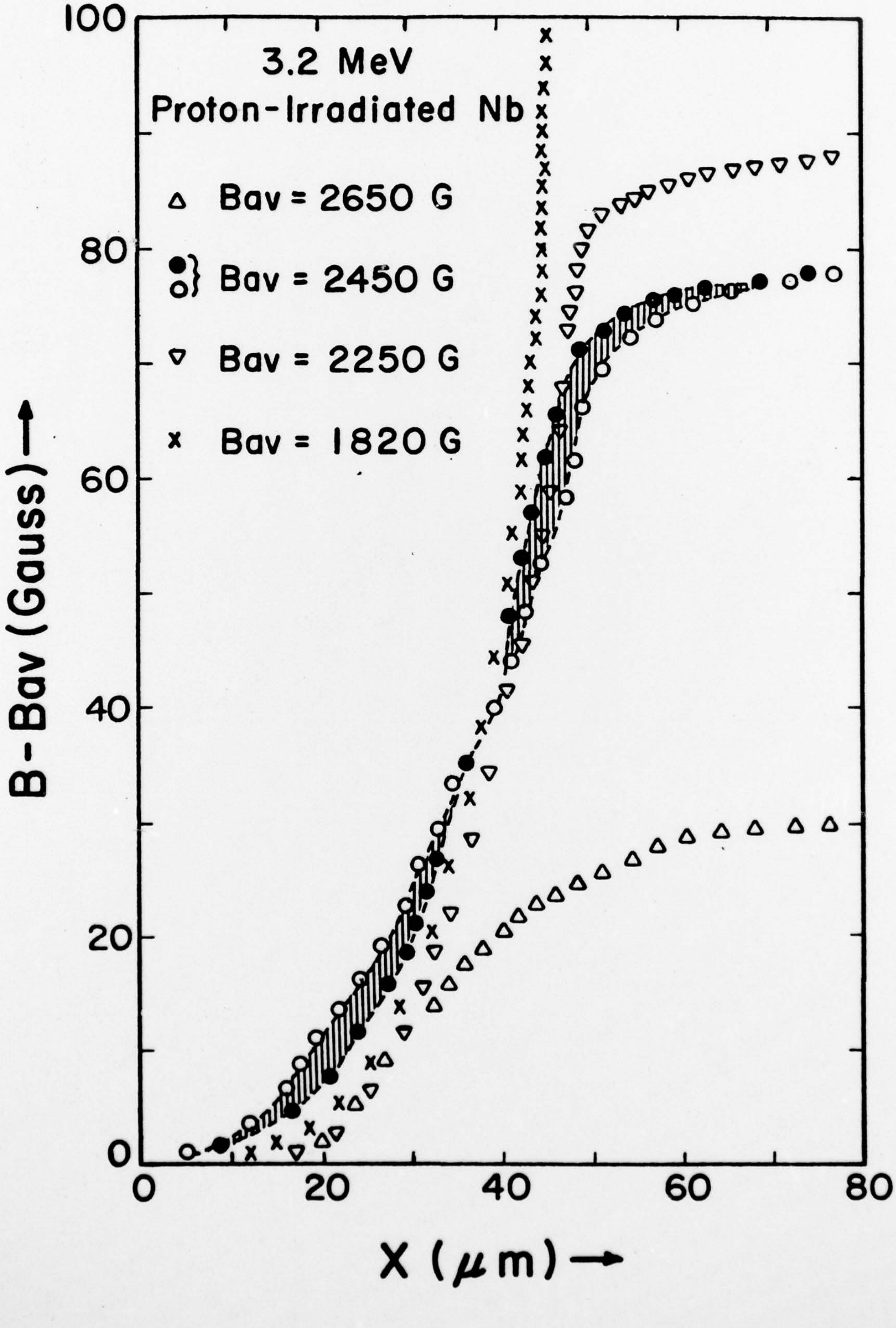
Fig. 10. Volume pinning force data compared with scaling laws from different models discussed in the text.

Fig. 1



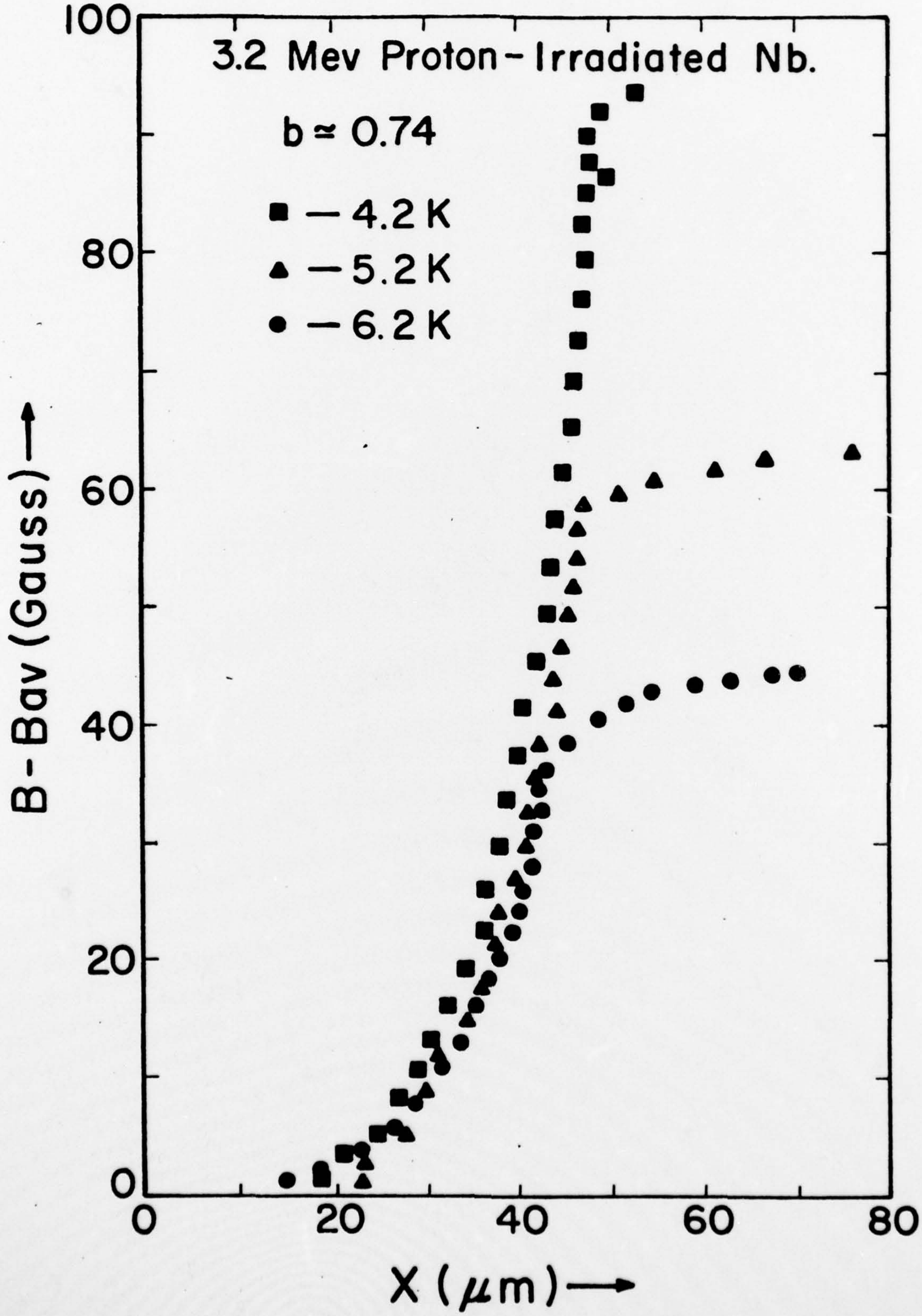
Temperature Dependence of
QFLL Elastic Constants
Annealed Niobium
b = 0.7
C66
C44
(C44C66)^1/2
C (S dynes/cm²) →
H_c2 (KOe) →
10
8
6
4
2
1
1
2
3
4
5

Fig. 2



3.2 MeV
Proton-Irradiated Nb
Bav = 2650 G
Bav = 2450 G
Bav = 2250 G
Bav = 1820 G
B - Bav (Gauss) ⟶
X (μm) ⟶
0
20
40
60
80
100

Fig. 3



3.2 Mev Proton-Irradiated Nb.
b ≃ 0.74
— 4.2 K
— 5.2 K
— 6.2 K
B - Bav (Gauss) ⟶
X (μm) ⟶
0
20
40
60
80
100

Fig. 4

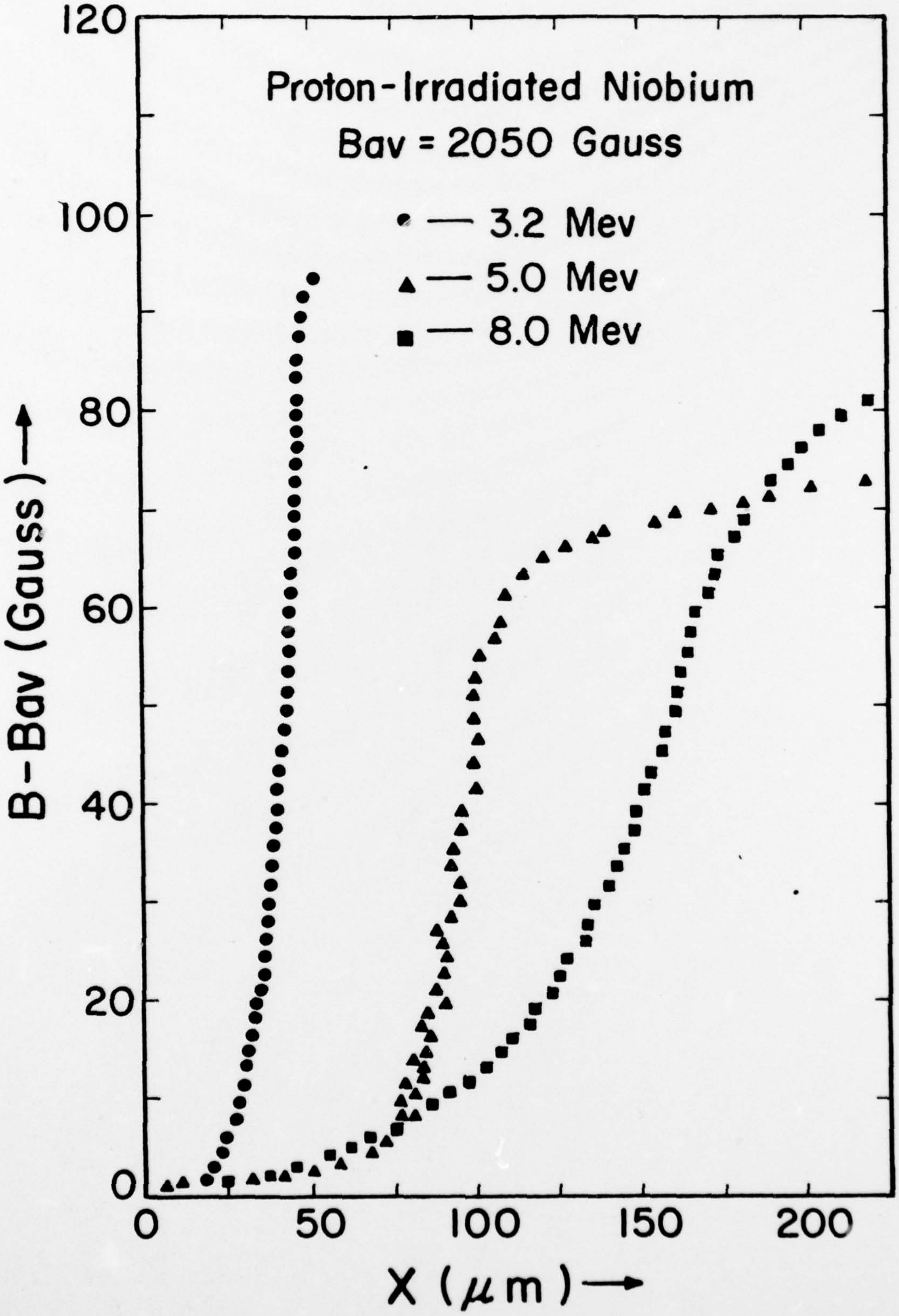
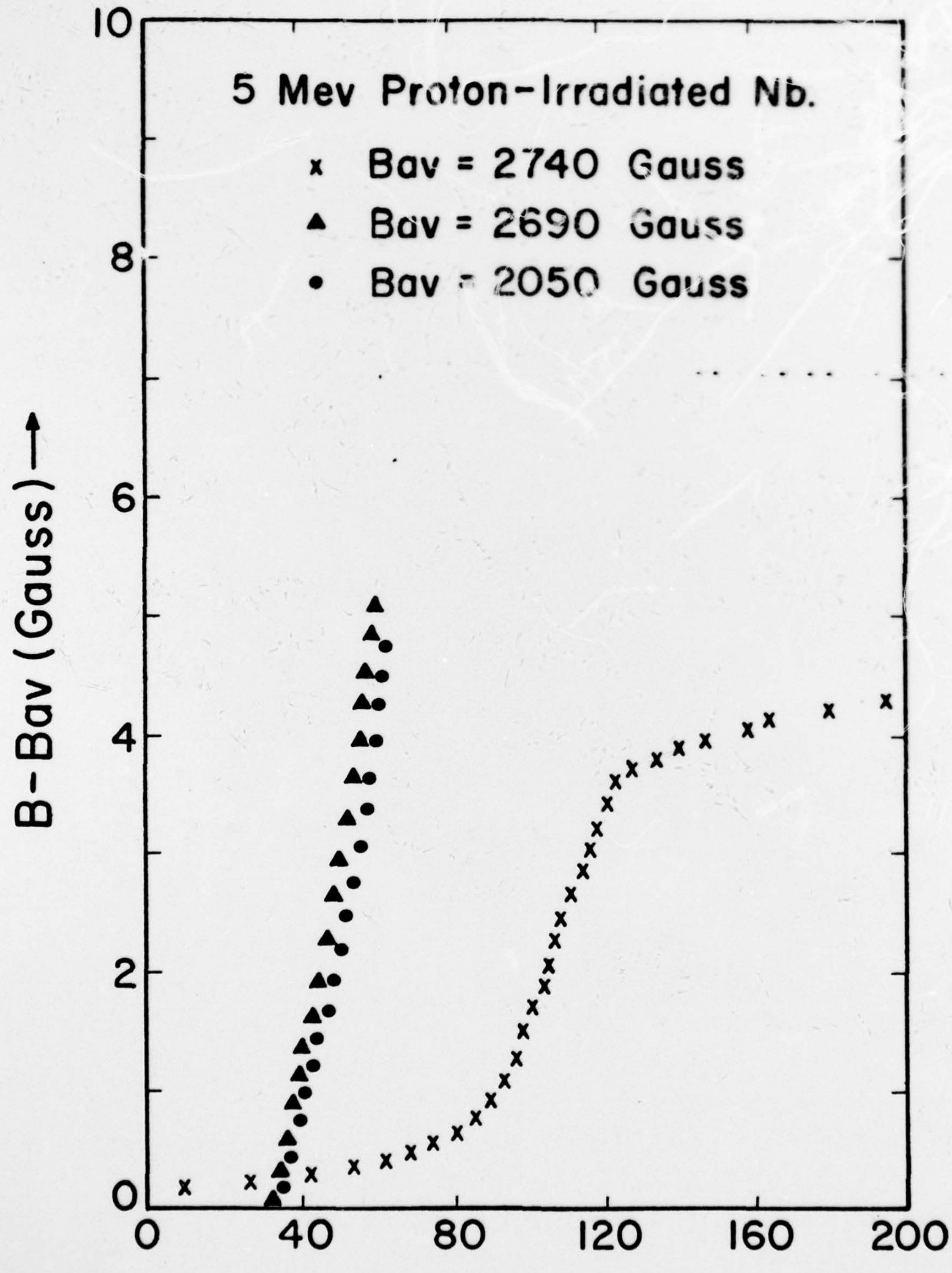

Proton-Irradiated Niobium
Bav = 2050 Gauss
— 3.2 Mev
— 5.0 Mev
— 8.0 Mev
B-Bav (Gauss) →
X (μm) →
0
20
40
60
80
100
120
0
50
100
150
200

Fig. 5



5 Mev Proton-Irradiated Nb.
x Bav = 2740 Gauss
▲ Bav = 2690 Gauss
● Bav = 2050 Gauss
B-Bav (Gauss) ⟶
X (μm) ⟶
0
2
4
6
8
10
0
40
80
120
160
200

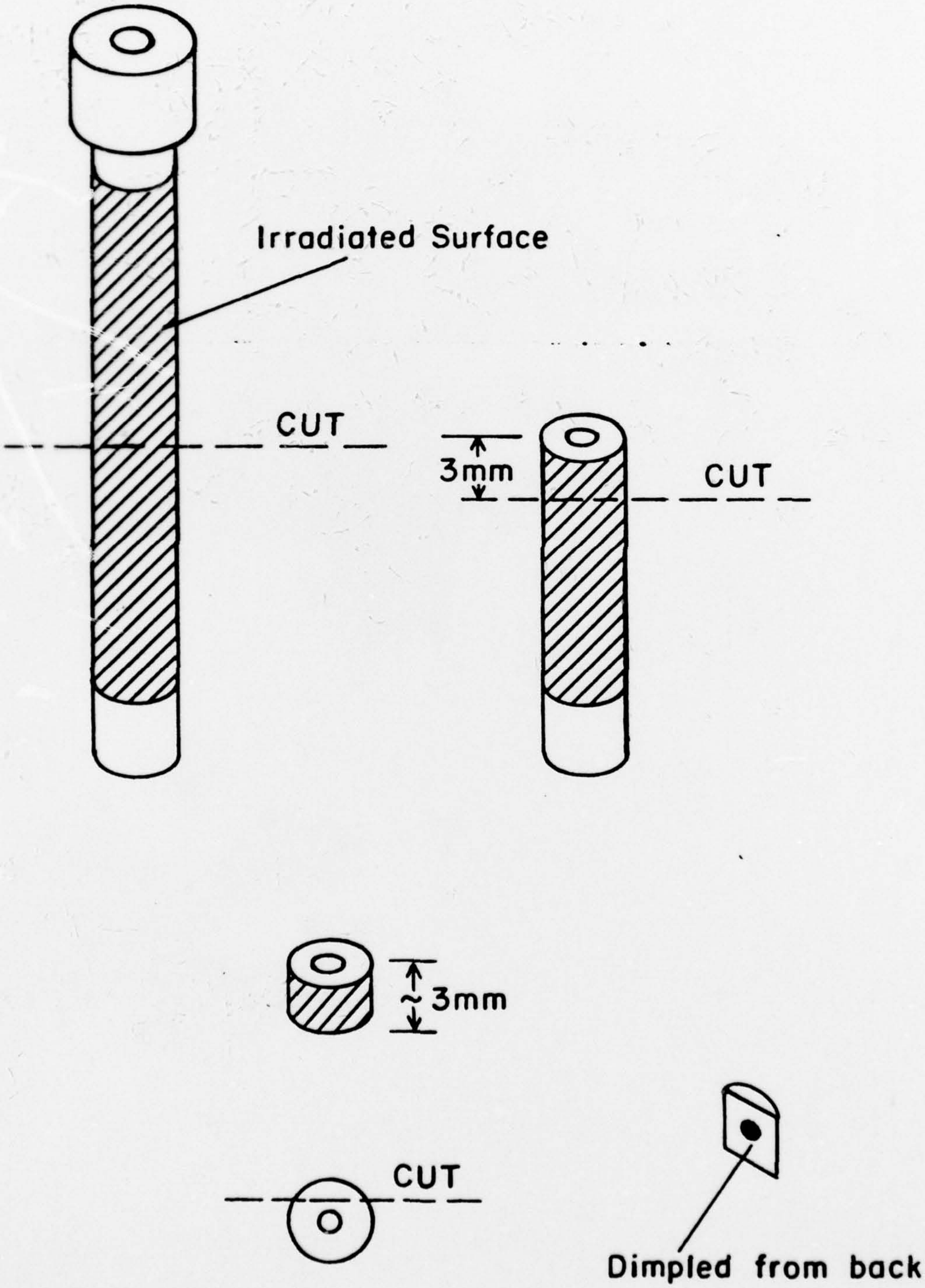


Fig. 6

Fig. 7a

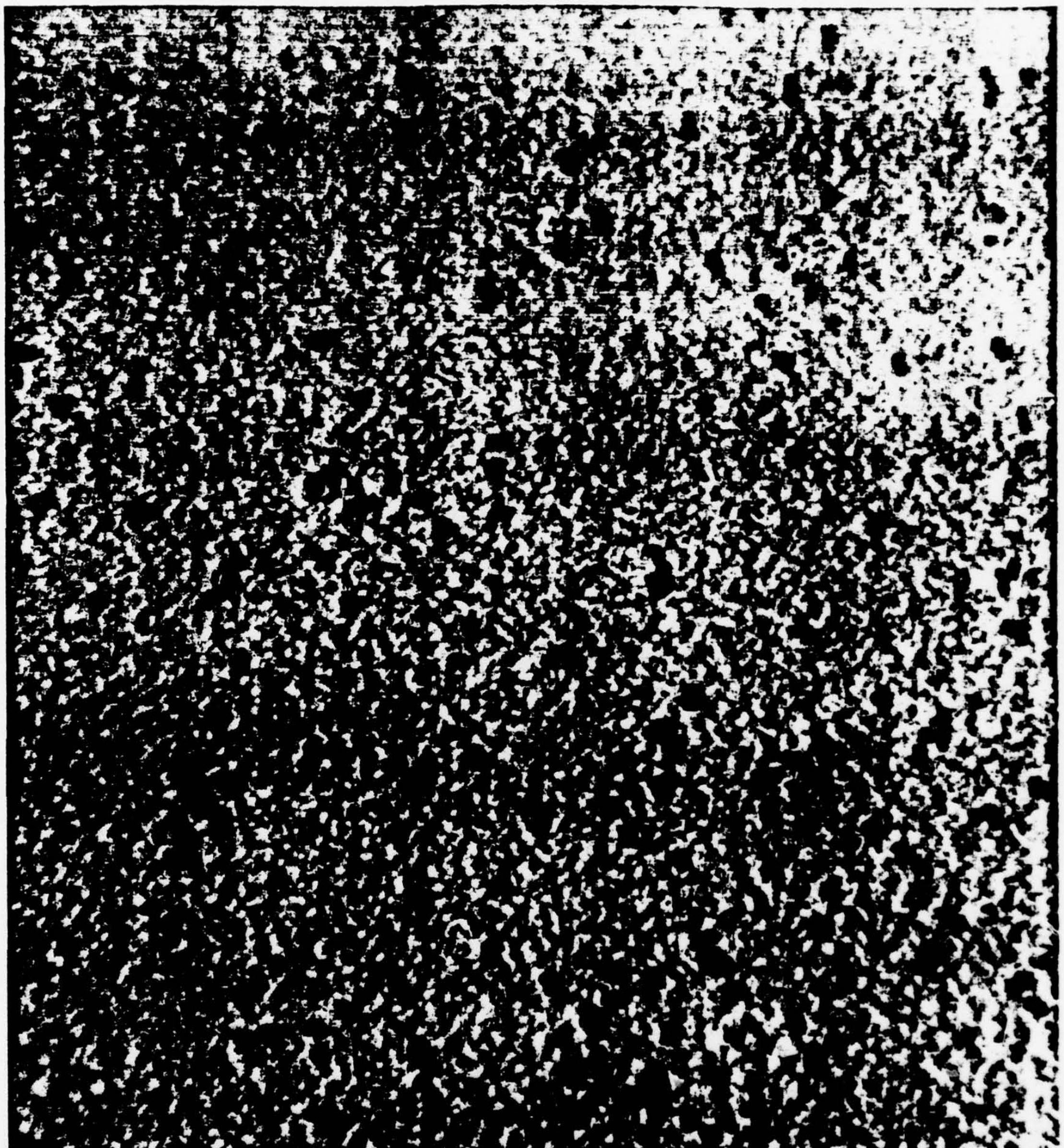


Fig. 7b

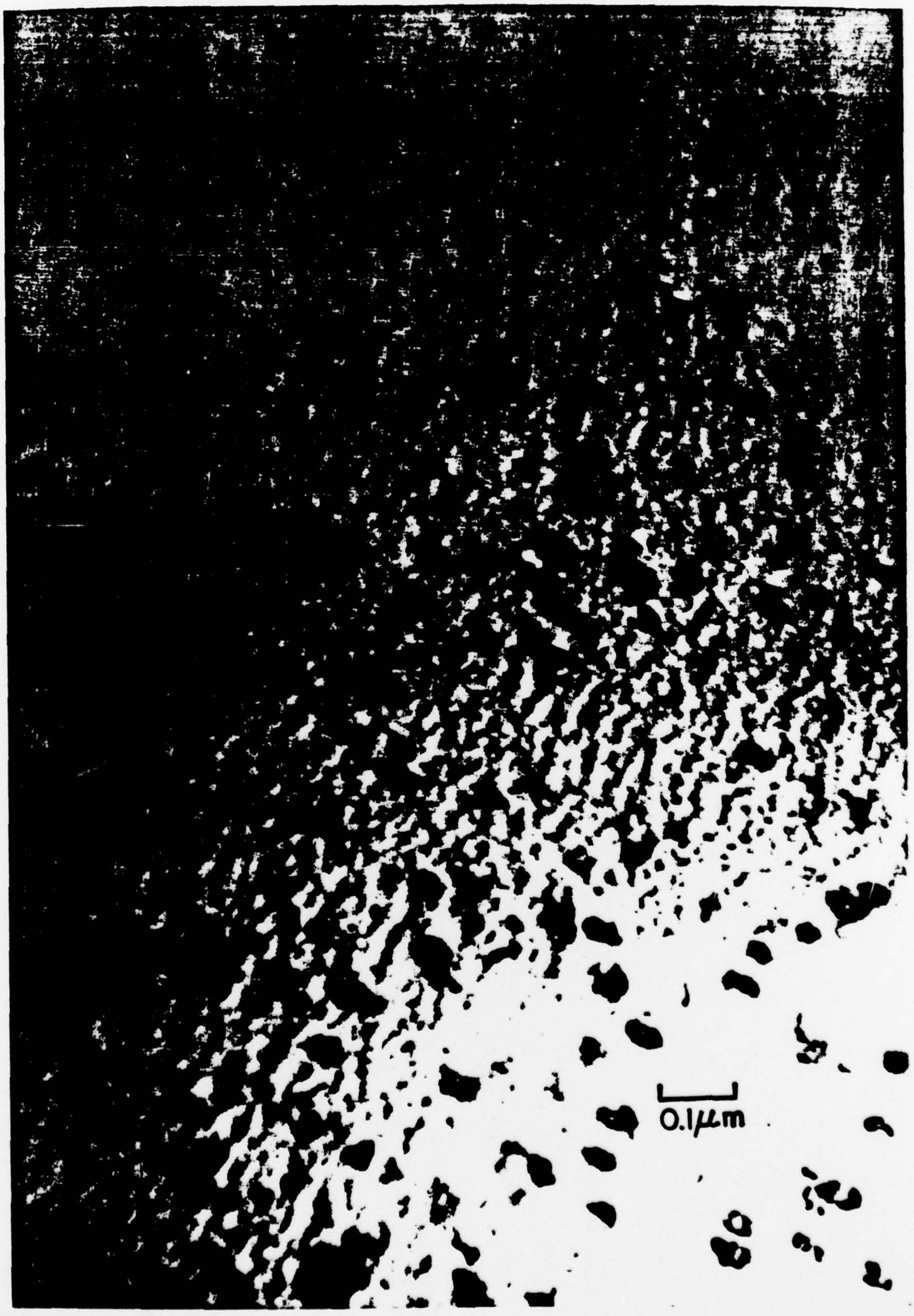
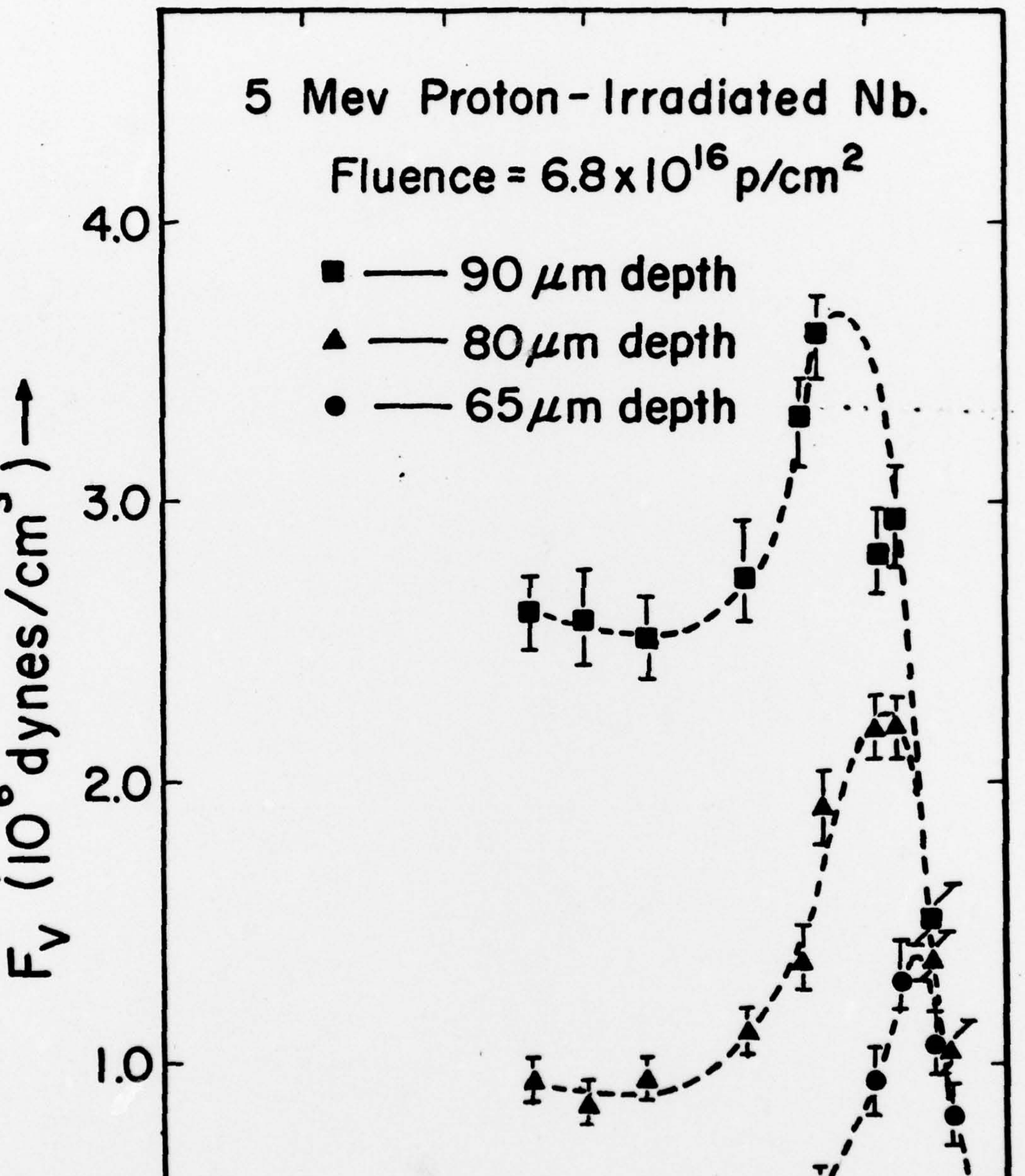


Fig. 8.



5 Mev Proton-Irradiated Nb.
Fluence = 6.8x10¹⁶ p/cm²
90 μm depth
80 μm depth
65 μm depth
Fv (10⁶ dynes/cm³) →
4.0
3.0
2.0
1.0
0
0.4
0.6
0.8
1.0
b →

Fig. 9

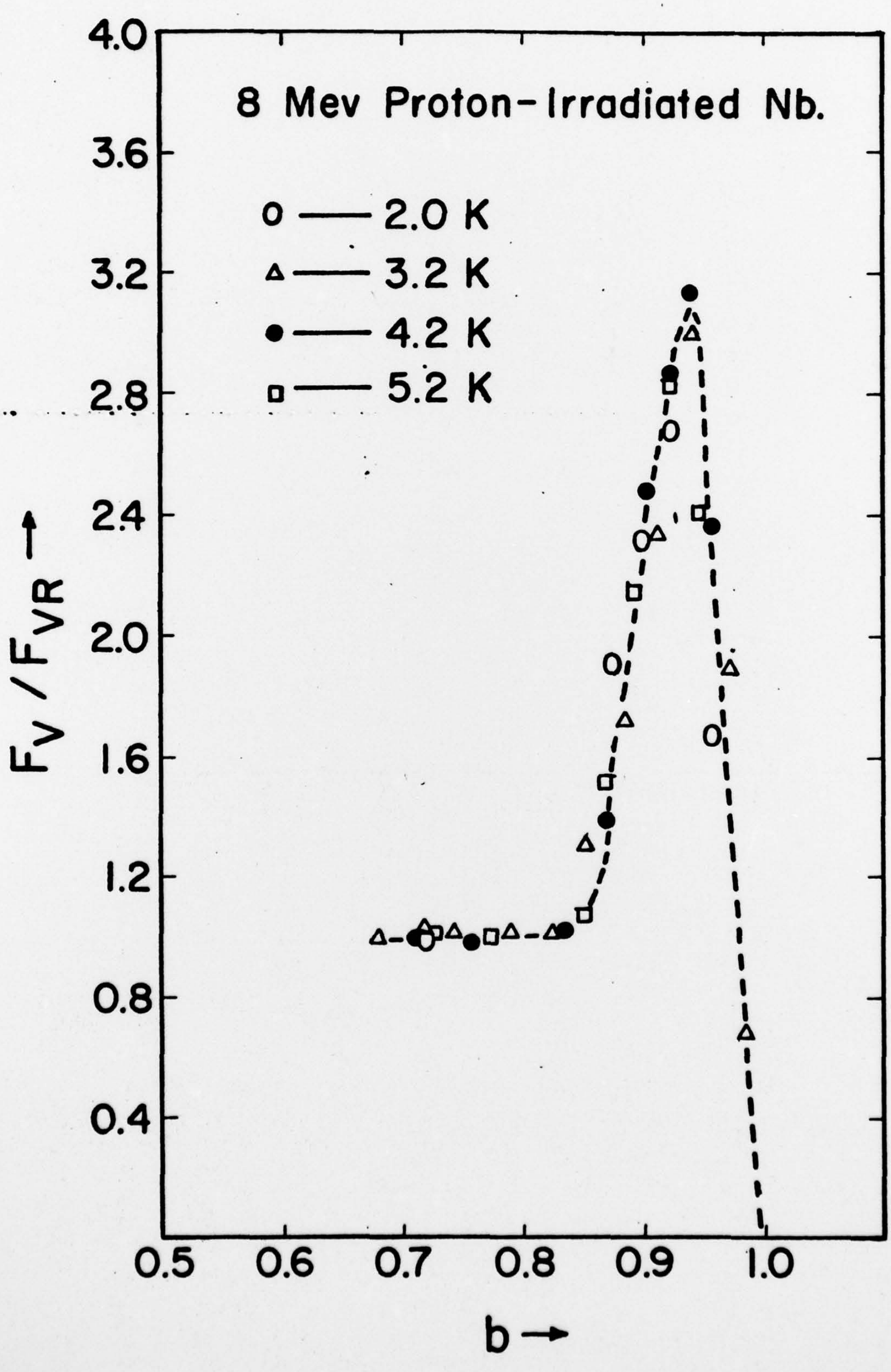
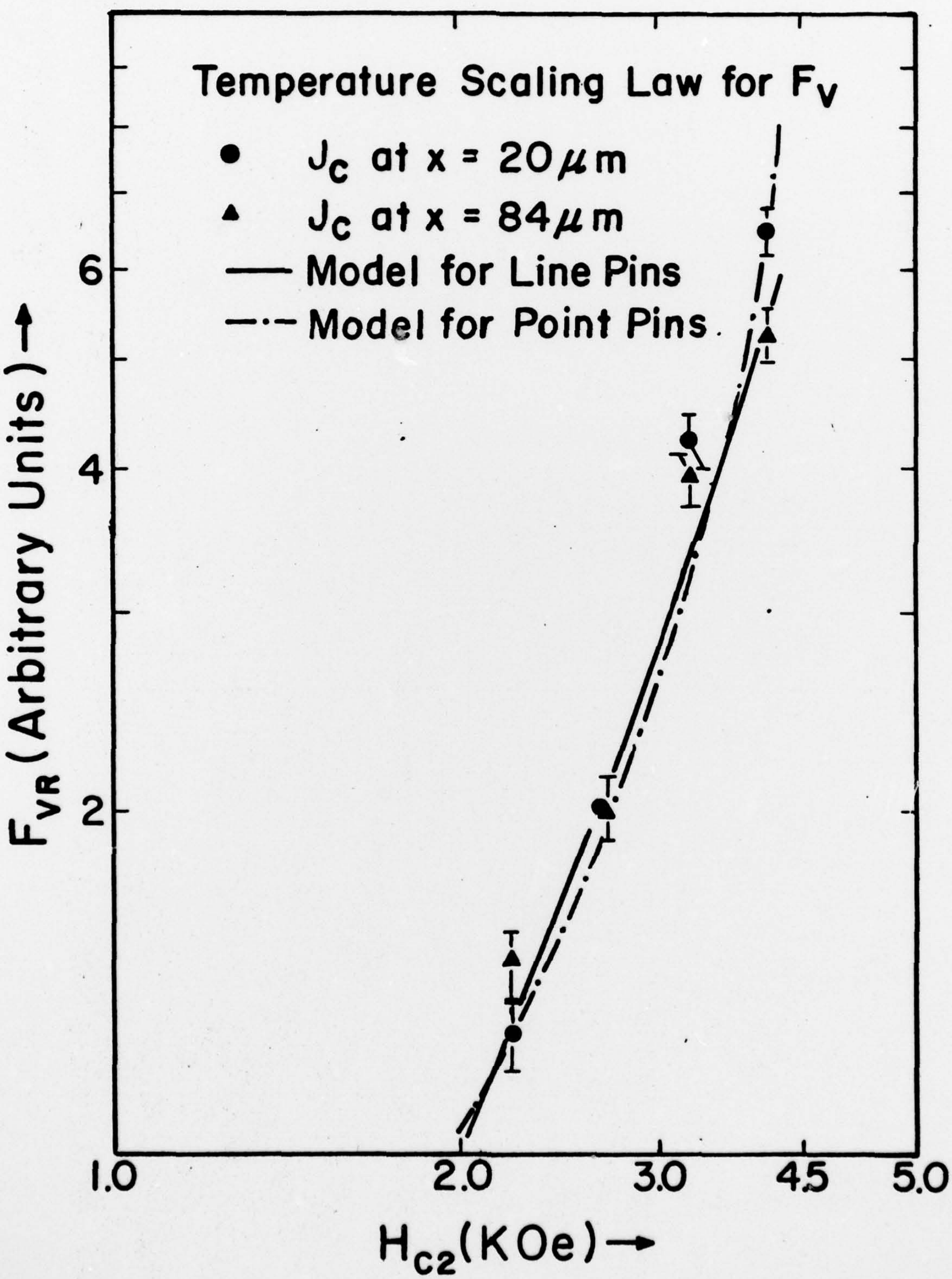

8 Mev Proton-Irradiated Nb.
o —— 2.0 K
△ —— 3.2 K
● —— 4.2 K
□ —— 5.2 K
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
0.8
0.4
0.5
0.6
0.7
0.8
0.9
1.0
F_V / F_{VR} →
b →

Fig. 10



Temperature Scaling Law for F_V
● J_c at x = 20 μm
▲ J_c at x = 84 μm
—— Model for Line Pins
—·— Model for Point Pins
6
4
2
F_{VR} (Arbitrary Units) →
1.0
2.0
3.0
4.5
5.0
H_{c2}(KOe) →