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Influence of radiation-induced disordering on the superconducting transition temperature of Nb_3Ir films

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Evaporated single-phase $A15$ Nb_3Ir films were irradiated at room temperature with protons and helium ions to study the influence of the induced defect structures determined by x-ray diffraction measurements on the superconducting transition temperature T_c. A reduction in T_c from 2.1 to 1.8 K was observed for energies deposited into nuclear collisions below 1 eV/atom. Beyond this threshold T_c increased and revealed values above the initial transition temperature of the unirradiated films depending on the irradiation conditions: 2.8 K for proton and 3.7 K for helium-ion irradiation. The changes in T_c were accompanied by a decrease of the Bragg-Williams long-range order parameter, a defect structure consisting of static displacements of the lattice atoms with average rms amplitudes in the range of 0.005 to 0.009 nm, and by partial amorphization. The maximum T_c value of 5.7 K was determined in totally amorphized films. Both the depressions and enhancements in T_c can be explained by changes of the electronic density of states at the Fermi energy due to smearing effects.

I. INTRODUCTION

High-T_c superconductors with the $A15$ structure are very sensitive to disorder. In recent years therefore many investigations have been performed to study the influence of particle irradiation on T_c. As a common result all these experiments revealed a strong degradation of the superconducting transition temperature which was correlated to structural defects and changes of the electronic properties of the materials. Less attention has been paid to the radiation-induced properties of low-T_c $A15$-type superconductors. Exceptions are the irradiation studies of Mo_3Ge with α particles by Gurvitch *et al.*[1] and of Mo_3Si with high-energy sulphur ions by Lehmann and Saemann-Ischenko.[2] In contrast to the behavior of the high-T_c $A15$ superconductors, in these compounds T_c enhancements have been observed and related to disorder. The disorder structure, however, has not been considered in greater detail.

An increase of T_c has also been observed in Nb_3Ir single crystals irradiated either with He or with heavy Kr ions by Meyer *et al.*[3] Here the defect structure was studied by channeling-Rutherford backscattering experiments. It was found that heavy-ion irradiation leads to amorphization of the $A15$ structure with a T_c value of 5.7 K. By He-ion irradiation T_c increases to about 3 K were accompanied by the formation of disorder consisting of small static displacements of the target atoms with average displacement amplitudes increasing with irradiation fluence.

It is known, however, that in high-T_c $A15$ superconductors, T_c changes are also strongly correlated with variations of the Bragg-Williams long-range order parameter S, i.e., with the formation of antisite defects.[4] In addition, it was found previously that changes of S are accompanied by the occurrence of static displacement values, and it was assumed that these defects might be inherently interconnected.[5] It was therefore of interest to include the long-range order parameter into the discussion of defect structures accompanying the T_c changes in disordered low-T_c $A15$ superconductors. To this end the channeling studies of irradiated Nb_3Ir single crystals have been extended to thin-film experiments with an emphasis on the determination of S by x-ray diffraction measurements. First results of these experiments have been reported already.[6]

II. FILM PREPARATION, IRRADIATION, AND ANALYSIS

Single-phase polycrystalline $A15$ Nb_3Ir films have been prepared by simultaneous electron-beam evaporation of niobium and iridium from two separate sources onto sapphire substrates at temperatures of 850 °C. The pressure in the evaporation chamber was typically 7×10^{-7} Pa before and 8×10^{-6} Pa during evaporation. The film thicknesses and composition were determined by Rutherford backscattering analysis.[7] The values ranged from 170 to 300 nm, and from 21 to 27 at % iridium for the thickness and composition, respectively.

The superconducting transition temperature T_c was measured resistively using a standard four-point probe arrangement and inductively to discriminate between single superconducting filaments and networks of such filaments.

For the irradiation experiments, films have been selected with compositions close to stoichiometry (23.5—25 at. % Ir). These films had residual resistivity ratios $r[=R(300\text{ K})/R(4.2\text{ K})]$ around 2 and the lattice constant and T_c values were in the range of 0.5138—0.5131 nm and 1.6—2.1 K, respectively.

The irradiations were performed with 300-keV protons and He^+ ions at room temperature. The energy of the particles was high enough to provide a homogeneous energy deposition into nuclear collisions throughout the depth of a film with the particles coming to rest in the substrate. Some important calculated parameters concerning irradiation, energy deposition, and defect production are summarized in Table I. The data have been obtained either by using the TRIM code of Biersack and Haggmark,[8] or, in an approximate fashion with analytical expressions based on the Nielsen and Coulomb potentials.[9] The values for Kr-ion irradiation have been included for comparison.

The crystallographic structure, lattice constants, and the order parameter S have been determined by x-ray diffraction experiments employing a Guinier thin-film diffractometer.[10] The integrated line intensities have been measured before and after each irradiation step and analyzed by modified Wilson plots.[11] In this procedure the intensities of each line after irradiation are referred to the corresponding lines before irradiation to overcome possible influences of preferred grain orientation on the x-ray intensities. Assuming that the dynamic behavior of a crystal does not change under irradiation and neglecting small changes of the diffraction angles θ by the irradiation, the following equation is obtained by taking the ratio of the intensities after irradiation, I^D_{hkl}, to those before irradiation, I^0_{hkl}, for an $A\,15$ structure:

$$\ln(I^D_{hkl}/I^0_{hkl}) = -16\pi \overline{u^2_{st}} \sin^2\theta/\lambda^2 + \ln\left[C\frac{|F^D_{hkl}|^2}{|F^0_{hkl}|^2}\right]. \quad (1)$$

In this equation $\overline{u^2_{st}}$ is the mean-square-displacement amplitude of the lattice atoms perpendicular to the reflecting planes and C is the ratio of the material volumes V_0 and V_D contributing to diffraction before and after irradiation, respectively. F^0_{hkl} and F^D_{hkl} are the structure factors before and after irradiation, and λ is the x-ray wavelength. Equation (1) has the form of a straight line if $\ln(I^D/I^0)$ is plotted versus $\sin^2\theta/\lambda^2$. The static displacement values can be deduced from the slopes of such lines while the intercepts on the ordinate yield $|CF^D/F^0|^2$.

For the analysis of defect structures using Eq. (1), the 22 observed x-ray diffraction lines have been divided into four groups shown in Table II. Within one group the concentration-dependent structure factors $|F_{hkl}|$ of the reflections hkl have the same functional dependence on the Ir concentration β, the atomic form factors f_{Nb} and f_{Ir} of the Nb and Ir atoms, respectively, and the Bragg-Williams long-range order parameter S defined by the equation:

$$S = [r - (1-\beta)]/[1-(1-\beta)]$$

[r is the fraction of $6(c)$ chain sites of the $A\,15$ structure occupied by Nb atoms]. The common characteristics of the structure factors F_{hkl} in the different groups are the following: The reflections of the second group are independent of the order parameter S. Reflections of the first and third group are the same with the exception of the opposite sign of S. In the third group only the atoms on the $6(c)$ chain sites contribute to the structure factor. The reflections of the fourth group are the so-called superstructure lines. Their structure factor is proportional to S. This means that the second term in Eq. (1) reduces to $\ln[C(S^D/S^0)^2]$ and appears suitable for the determination of the ratio of order parameters S^D after and S^0 before irradiation. It should be noted that the $A\,15$ compound Nb_3Ir is well suited for the determination of the order parameter, since the intensity of the reflections of the fourth group is large due to the large difference between the atomic form factors of niobium and iridium.

With the separation of the reflections into different groups, the defect analysis was performed in the following sequence.

(1) One obtains the mean-square-displacement amplitudes $\overline{u^2_{st}}$ of the atoms averaged over all sites from the slope of the straight line of the group-1 reflections.

(2) With the result of (1), one gets the constant C from the intensities of the group-2 reflections. It should be noted that C may change during irradiation either due to material removed by sputtering or to partial amorphization of a sample.

(3) $\overline{u^2_{st}}$ averaged only over the chain atoms is obtained from the slope of the modified Wilson plot of the group-3 reflections.

(4) With the known value of C we can determine the ra-

TABLE I. Calculated parameters for the ion target interactions in Nb_3Ir. The parameters are defined as follows: E, incident energy of the particles; R_p, and ΔR_p, projected range and range straggling of the incident particles, respectively; T_m, maximum energy transfer to the target atoms; $\overline{T}$, mean energy transfer to the target atoms; σ_d, total displacement cross section; n_{PRA}, number of primary recoil atoms per incident ion and distance dx; λ, mean free path of the incident particles between two collisions; S_n, total energy loss of a particle deposited into nuclear collisions; C_d displacements per atom (DPA). Values with an asterisk have been obtained by using TRIM code (Ref. 8). The other values have been determined with analytical formulas based on the Nielsen potential (Ref. 9) with mass and Z values averaged for Nb_3Ir. A displacement energy E_d of 25 eV has been assumed.

Projectile	E (keV)	R_p^*; ΔR_p^* (nm)	T_m (keV)	$\overline{T}$ (eV)	σ_d (cm^2)	$\frac{n_{\mathrm{PRA}}}{dx}$ $\left(\frac{\text{atoms}}{\text{ion nm}}\right)$	λ (nm)	S_n $\left(\frac{\text{eV cm}^2}{\text{ion atom}}\right)$	$\frac{c_d}{\phi t}$ (cm^2)
H^+	300	1583; 280	10.1	150	1.9×10^{-19}	1.1×10^{-3}	910	2.8×10^{-17} $2.7\times10^{-17*}$	5.9×10^{-19} $5.0\times10^{-19*}$
He^+	300	818; 243	38.2	183	3.0×10^{-18}	1.7×10^{-2}	57	5.4×10^{-16} $6.4\times10^{-16*}$	1.1×10^{-17} $1.1\times10^{-17*}$
Kr^{2+}	700	143; 58	680.2	4124	7.03×10^{-17}	4.1×10^{-1}	2.4	2.9×10^{-13} $3.8\times10^{-13*}$	3.8×10^{-15} $6.1\times10^{-15*}$

TABLE II. Groups of reflections having the same structure factor F_{hkl} in the A 15-type structure. hkl are the Miller indices, S is the Bragg-Williams long-range order parameter, β the iridium concentration, and f_A and f_B are the atomic scattering factors for the Nb and Ir atoms, respectively.

Group	h	k	l	Structure factor
1	2	0	0	$\lvert F_{hkl} \rvert = 4\{[1-\beta(1+S)]f_A + \beta(1+S)f_B\}$
	2	1	1	
	3	2	1	
	4	2	0	
	3	3	2	
	5	2	1	
	600	+	422	
	611	+	532	
2	4	0	0	$\lvert F_{hkl} \rvert = 8[(1-\beta)f_A + \beta f_B]$
	4	4	0	
3	2	1	0	$\lvert F_{hkl} \rvert = 4\{[1-\beta(1-S)]f_A + \beta(1-S)f_B\}$
	3	2	0	
	4	2	1	
	520	+	432	
	6	1	0	
4	2	2	0	$\lvert F_{hkl} \rvert = 8\beta S(f_B - f_A)$
	3	1	0	
	411	+	330	
	4	2	2	
	510	+	431	
	530	+	433	
	6	2	0	

tio S^D/S^0 of the order parameters before and after irradiation using the axial section value on the ordinate of the straight line through the data of the group-4 reflections.

III. RESULTS

The *superconducting transition temperature* T_c of proton-and helium-ion irradiated films is shown in Fig. 1 as a function of the mean energy density Q deposited into nuclear collisions by the bombarding particles, which is an integral measure of disorder. Q (1 eV/atom corresponds to approximately 0.02 displacements per atom) is independent of the ion species and is obtained as the product of the nuclear energy loss S_n as tabulated in Table I with the applied ion fluence. The maximum fluences which could be applied in the irradiation experiments without mechanical disruption of the films due to bubble formation in the substrate were of the order of 10^{17} ions/cm^2. Therefore the maximum Q values attainable were limited to 15 eV/atom for proton irradiation and 200 eV/atom for helium-ion bombardment. The T_c behavior was dependent on the Q ranges. For small Q values ($\lesssim 1$ eV/atom) and proton irradiation T_c decreased by about 14%[Fig. 1(a)]. The same observation, though less pronounced, was made for helium-ion irradiation [Fig. 1(b)]. This is a new effect not detected in the previous investigations, which agrees with the "normal" behavior of high-T_c A 15-type superconductors. For higher Q values T_c increased above its initial value and reached saturation values of 2.8 and 3.7 K for proton and helium irradiation, respectively. A further T_c enhancement up to 5.7 K was achieved by helium irradiation at liquid-nitrogen temperature or by Kr-ion bombardment at room temperature.

The *normal conducting properties,* i.e., the residual resistivity ratio r and the residual resistivity ρ_0 (4.2 K) are shown in Figs. 2(a) and 2(b) for proton- and helium-irradiated samples, respectively. r decreases and ρ_0 increases very steeply in the damage range $Q \leqq 1$ eV/atom, where the T_c depressions were observed. In the Q range where a T_c enhancement occurred, r and ρ_0 change at a lower rate and saturate in the T_c saturation region.

It was the main intention of this paper to determine the different types of disorder produced by ion irradiation which lead to the observed T_c changes. As an example of the analysis of defect structures, modified Wilson plots, i.e., plots of $\ln(I^D/I^0)$ versus $(\sin^2\theta)/\lambda^2$ according to Eq. (1), are displayed in Fig. 3 for a sample irradiated with 1.5×10^{16} He$^+$/cm^2. Straight lines have been fitted through the data points of group-1, -3, and -4 reflections, and the values for the lines 400 and 440 of group 2 have been included in the figure. The intensity decrease of the superstructure lines of group 4 is most pronounced. It should be mentioned, that once the ratio of the order parameters S^D/S^0 has been determined from the data of this group, the absolute value S^0 can be deduced from the axial section on the ordinate of group-1 reflections.[12] This procedure leads to a rather large error in the determination of S^0. The estimated value of 0.96±0.04, however, is in good agreement with data of Flükiger[4] reported in the range of 0.93 to 0.97 for bulk Nb_3Ir samples.

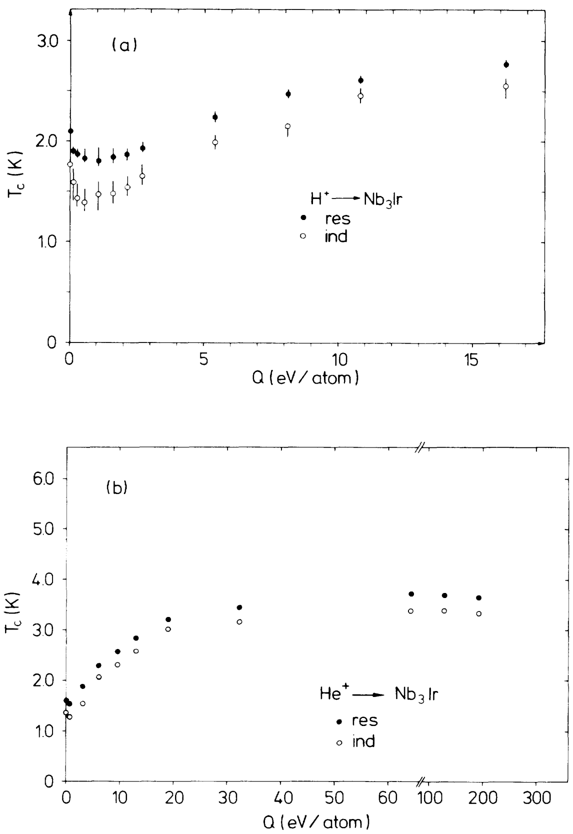

FIG. 1. Resistively and inductively determined transition temperatures T_c as a function of the energy density Q deposited into nuclear collisions for (a) proton- and (b) He-ion irradiated Nb_3Ir films.

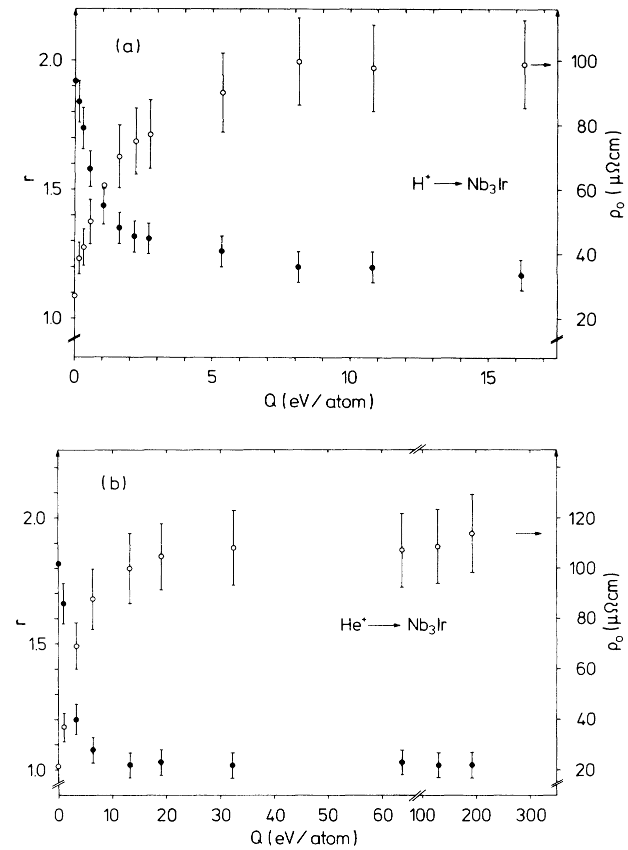

FIG. 2. Residual resistivity ρ_0 and residual resistivity ratio r vs Q for (a) proton- and (b) He-ion irradiated Nb_3Ir films.

The *order parameter* in the irradiated samples decreases with progressive irradiation. This behavior is illustrated in Figs. 4(a) and 4(b) for proton and helium irradiations, respectively, where the ratio S^D/S^0 is plotted versus Q. The minimum order parameter observed for proton irradiation is 0.4. For helium irradiation S saturates for Q values above 30 eV/atom at a value of 0.3. This saturation is thought to be due to a temperature-dependent equilibrium between radiation-induced disordering and radiation-assisted thermal ordering. Therefore a total disordering so far not observed in *A* 15 compounds could also not be achieved by ion irradiation performed in our experiments at room temperature (RT).

A second disorder component deduced from the x-ray analysis is a defect structure consisting of small *static displacements* of the lattice atoms, already detected previously in other irradiated *A* 15 compound thin films.[5,13,14] The quantitative data of the displacement values $(\overline{u^2})^{1/2}$ are shown as a function of Q in Figs. 5(a) and 5(b). The observed increase of u depends on the ion species used in the irradiation experiments. For proton irradiation [Fig. 5(a)] the static displacements are small in the Q range smaller than 1 eV/atom. A distinct increase then is observed for Q values up to 3 eV/atom and finally saturation occurs at a value of about 0.007 nm for higher irradiation fluences. For helium irradiation [Fig. 5(b)] the Q range smaller than 1 eV/atom was not accessible. For higher-Q values a behavior similar to that for proton irradiation is observed, although quantitatively the u values increase in a larger Q range up to 30 eV/atom and saturate at a value of 0.0085 nm which is larger than for proton irradiation. This value is only slightly lower than that of 0.01 nm determined in the channeling experiments.[3]

A third defect type are *amorphous regions* detected in

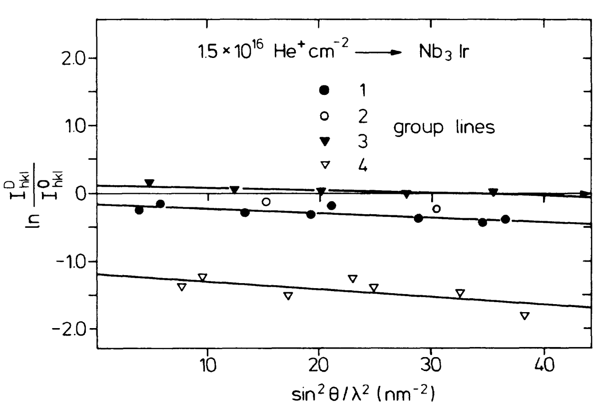

FIG. 3. Modified Wilson plots for a Nb_3Ir film irradiated with 1.5×10^{16} He^+/cm^2.

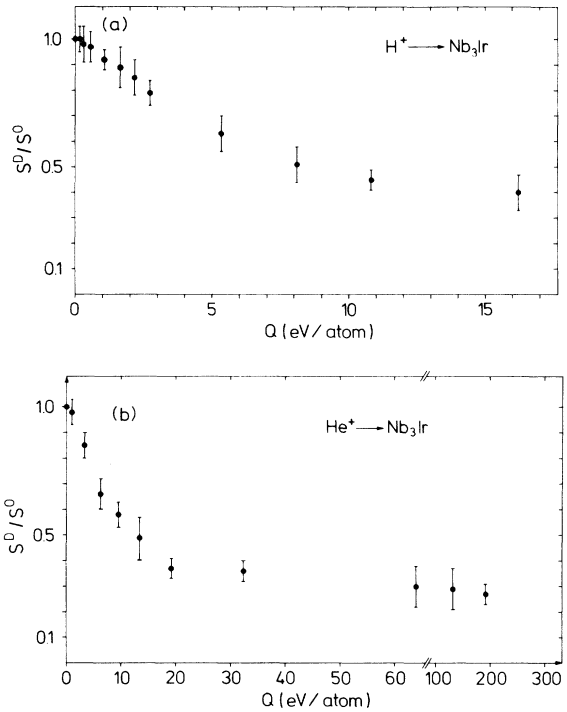

FIG. 4. Ratio of the long-range order parameters S^D after, and S^0 before, irradiation versus the energy density Q deposited into nuclear collisions. (a) Proton irradiation; (b) He-ion irradiation.

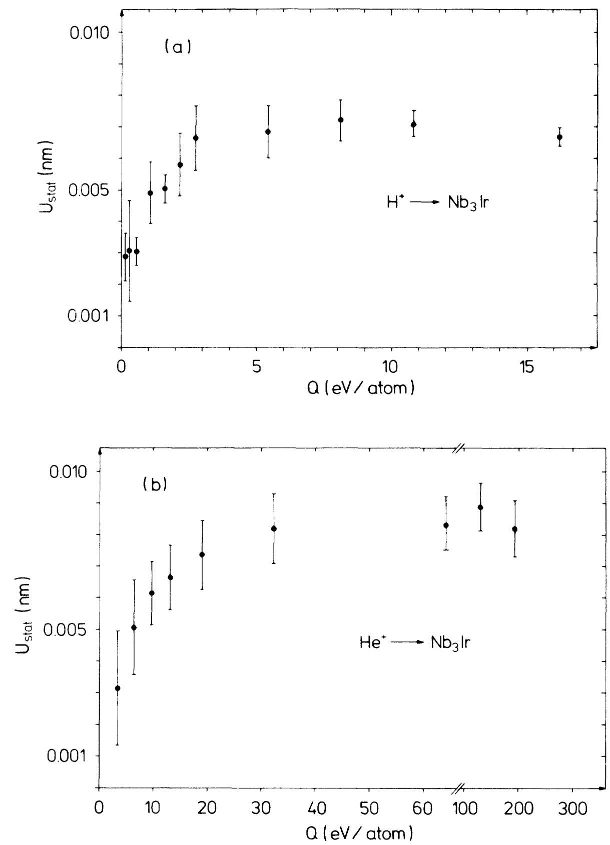

FIG. 5. Mean static displacement values $(\overline{u^2})^{1/2}$ averaged over all lattice atoms in the A 15 structure plotted versus Q (a) p irradiation; (b) He-ion irradiation.

samples irradiated with high fluences of He ions at RT. We have found that about 15% of the x-ray intensity decrease in the Q range above 30 eV/atom could be ascribed to partial amorphization since no material loss was observed by sputtering. No amorphization was detected in the proton irradiated samples within the detection sensitivity of about 4%. Total amorphization was observed in Nb_3Ir films irradiated with He ions at liquid-nitrogen temperature.

The *lattice constant* changes in the irradiated samples as a function of growing disorder are consistent with the defect types already discussed. The data displayed in Figs. 6(a) and 6(b) for He and p irradiation, respectively, depend on the ion species and reveal in plots versus Q, a relationship similar to the static displacement values. As long as the displacement values are small as for p irradiations, the variations of the lattice constants also are small, except for a jump of a in the first irradiation step. This step could be due to strain relaxation depending on the condition of the as-evaporated sample. With increasing values of the static displacement, then also the lattice constants increase and saturate in a Q range where the static displacements saturate. The higher saturation value of the lattice constant measured in the He-irradiated films coincides with the larger displacement amplitudes produced by He irradiation.

The experimentally observed correlation between the changes of the lattice constant and the static displacements are in accordance with discussions of Flükiger.[15] Flükiger argues that the variations of the lattice constant and the distortions described by static displacements have a common cause, namely a vacancy-type defect on the Nb sublattice which occurs in a split configuration. Such a defect type was detected in computer simulations of defects in Nb_3Sn by Welch *et al.*[16] The occurrence of this defect also may explain the observation in the proton-irradiated films that the order parameter saturates at higher Q values than the lattice constant and the static displacements. In the helium-irradiated samples the saturation range of the order parameter coincides with that of the lattice parameter and the static displacements. Here the occurrence of amorphous zones may act as a stabilizing factor for a higher concentration of antisite and other point defects.

IV. DISCUSSION

Different types of disorder like a decrease of the Bragg-Williams long-range order parameter, chain buckling described by small static displacements of the lattice atoms, and partial or total amorphization have been observed in Nb_3Ir thin films upon ion irradiation. The superconducting transition temperature T_c revealed an unusual behavior so far not observed in A 15 superconductors, namely first a small depression for low irradiation fluences followed by an enhancement above the initial value for higher damage levels. Irrespective of the dif-

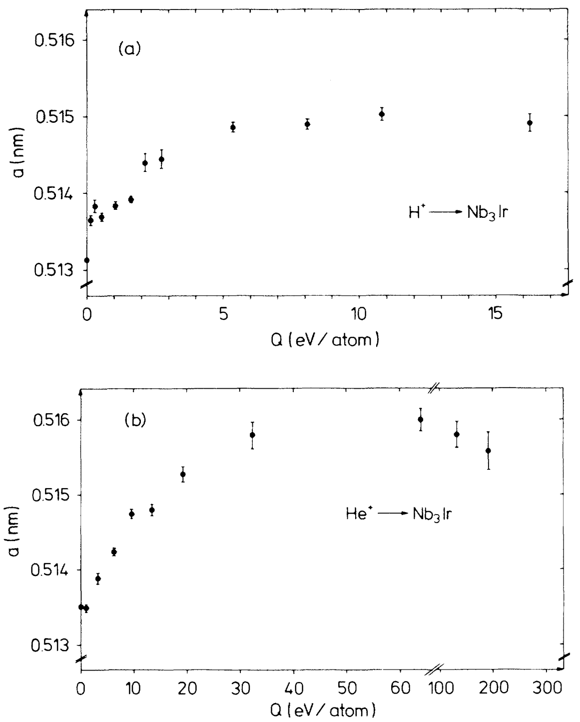

FIG. 6. Lattice constant values a versus energy density Q deposited into nuclear collisions in irradiated Nb_3Ir films. (a) Proton irradiation; (b) He-ion irradiation.

ferent types of disorder this behavior at least qualitatively can be understood within the parameters determining T_c in the theory of superconductivity. The T_c depressions in high-T_c $A15$ compounds often have been explained by a decrease of the electronic density of states (DOS) at the Fermi energy, $N(0)$, due to smearing effects by lifetime broadening with increasing disorder.[17] This picture has been generalized by Gurvitch *et al.*[1] for low-T_c $A15$ compounds with the example of Mo_3 Ge where a T_c enhancement has been observed by disordering. In this compound the Fermi level in contrast to the high-T_c $A15$ superconductors is situated in the region of a low density of states between two peaks arising from the bonding bands at low energies and the antibonding bands at higher energies. In this situation, due to smearing effects, $N(0)$ and thus T_c are increased upon disordering. These arguments are similar to those discussed previously to explain T_c changes in disordered bcc transition metals.[18] The T_c changes in our experiments, showing a decrease followed by an enhancement over the initial value at first glance, seem to be an exception which is in contradiction to the generalized picture. Energy-band calculations of Jarlborg, Junod, and Peter,[19] however, show that for Nb_3Ir the Fermi level is located at a small sharp peak adjacent to a broader and higher maximum of the DOS. A continuous smearing of such structures would thus first cause a depression of $N(0)$ due to a broadening of the small peak before the contributions of the smeared adjacent large structures lead to an increase of $N(0)$. Presuming that $N(0)$ is the decisive parameter governing T_c in $A15$ compounds[20] smearing effects at least qualitatively explain the observed T_c changes also in Nb_3Ir. A quantitative parameter coinciding with the T_c variations is the residual resistivity ρ_0 shown in Fig. 2. This parameter according to Testardi and Mattheiss[17] via the plasma energy Ω_p determines the mean electron lifetime $\bar{\tau}$ ($\rho^{-1}=\langle\Omega_p\rangle^2\bar{\tau}/4\pi h^2$) which directly enters the broadening function which determines the quantity of smearing of the DOS and thus the T_c changes. A comparison of the T_c variations with the behavior of ρ_0 shows that the largest T_c changes are correlated with the largest changes of ρ_0, and saturation is achieved in approximately the same irradiation ranges for both, p and He irradiations. With these results the validity of the generalized model discussed by Gurvitch *et al.*[1] could be extended to low-T_c $A15$ structures composed of two transition metals.

It is of course tempting to correlate the T_c changes with the different types of disorder determined from the x-ray data, though such a correlation can only be indirect via the influence of the defects on the electronic and dynamic properties of a superconductor. Only the highest T_c value of 5.7 K achieved at low-temperature irradiations unambiguously can be ascribed to total amorphization of the films in accordance with previous results of the channeling experiments on bulk Nb_3Ir crystals. At lower irradiation fluences at RT two types of defects appear simultaneously; a continuous decrease of the order parameter is accompanied by an increase of the average static displacement values. Therefore the T_c enhancements cannot be uniquely assigned to a certain type of defects. However, here a quantitative higher damage level observed for He irradiations as compared to the p irradiations leads to a larger increase of the residual resistivity which may be considered as an integral measure of disorder and thus to a higher T_c value.

It is a question of whether in the low damage region ($Q<1$) the observed decrease is solely accompanied by a decrease of the order parameter. This is an important point since chain integrity often has been discussed as an important parameter for T_c in high-T_c $A15$ superconductors, and it is a question of whether this argument also applies to the low-T_c compounds. Although the static displacement values for $Q<1$ appear small and show little variation a definitive conclusion cannot be drawn due to the large uncertainty in the experimental data. It should be noted that irrespective of the defects the resistivity increases sharply in this damage range leading to the T_c decrease within the smearing model discussed above.

V. SUMMARY

Different types of defect structures induced by particle irradiation (p,He) have been determined by x-ray diffraction in Nb_3Ir thin films. The disorder leads to an increase of the resistivity and to T_c variations revealing a T_c decrease for very low damage levels followed by a T_c enhancement above the initial value for higher irradiation fluences. A unique assignment of the T_c behavior to different types of defects could not be performed since several defect components appeared simultaneously and probably were interrelated. The highest T_c value of 3.7 K in a sample preserving the $A\,15$ structure was observed for high fluence He irradiation at RT. The defect types determined in this region were a low order parameter, static displacements of the lattice atoms, and probably amorphous zones. The maximum T_c value of 5.7 K was observed in a totally amorphized film. The T_c variations microscopically could be explained by a model describing changes of $N(0)$ by smearing of sharp structures in the electronic density of states. The residual resistivity which enters the smearing model as a parameter is in accordance with the observed T_c changes.

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