PHYSICAL REVIEW B VOLUME 18, NUMBER 1 1 JULY 1978

Effect of neutron irradiation on single-crystal V_3Si: Sound velocity, magnetic susceptibility, and upper critical field

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(Received 9 January 1978)

We have measured the sound velocity, magnetic susceptibility, and upper superconducting critical field H_{c2} of a single crystal of V_3Si as a function of neutron irradiation up to a fluence of 22.2×10^{18} n/cm^2. Very small doses affect the structural transformation, while leaving the superconducting transition temperature T_c, the soft acoustic shear mode, the susceptibility χ, and H_{c2} essentially unaltered. At higher defect concentrations, the strong anomalous temperature dependences of χ and the elastic modulus c_s are considerably reduced, although significant anomalies remain for the highest fluence (which lowers T_c from 17.2 to 7.4 K). Within models which assume unusual fine structure in the electronic density of states near the Fermi level, our results imply that defects produce broadening and smearing of the density-of-states function.

INTRODUCTION

Compounds with A-15 structure which exhibit high superconducting transition temperatures T_c and structural instabilities are also unusually susceptible to defect formation. Neutron, ^{4}He, and heavy-ion radiation-damage studies[1-3] have shown a rapid reduction in T_c and an increase in electrical resistivity with defect concentration. While the structural details of the defects are not yet fully known, it is important to learn how defects influence the behavior of these materials.

The aim of the experiments reported in this and in the accompanying articles[4,5] was to investigate the effect of defects on the anomalous physical behavior of A-15 materials and, by measuring a range of different properties, to obtain the most extensive characterization to date of a single sample of V_3Si.

Measurements were made on a single crystal of V_3Si in its initial state, and after successive neutron irradiations to a maximum fluence of 22.2×10^{18} n/cm^2. The following properties were measured: sound velocities, susceptibility, upper critical field (this paper), neutron scattering, specific heat, and resistivity (Refs. 4 and 5).

We report below the first measurements which show how defects modify the strongly temperature-dependent magnetic susceptibility χ, the soft acoustic shear mode, and the structural transformation. Measurements of dH_{c2}/dT, and the Ginzburg-Landau parameter κ derived from these, are also given for different defect levels.

EXPERIMENTAL PROCEDURE AND RESULTS

The susceptibility and sound velocity of a single crystal of V_3Si grown by Greiner and Mason[6] were measured in the unirradiated state (resistance ratio approximately 20 to 22), and after successive neutron irradiation[7] to fluences of 0.25, 1.66, 3.54, and 22.2×10^{18} n/cm^2. The superconducting transition temperature $T_c=17.2$ K of the unirradiated sample was reduced to $T_c=7.4$ K after the heaviest neutron dose.

Measurements[8] of sound velocities using the pulse superposition method at 20 MHz were made between 300 and 4.2 K. Longitudinal waves propagating along [001] and [110], and shear waves propagating along [001] were measured before irradiation and after each dose (except the third), from which the complete elastic moduli c_{11}, c_{12}, and c_{44} of this cubic crystal were obtained. The soft shear mode corresponding to $c_s=\frac{1}{2}(c_{11}-c_{12})$ was not directly determined in the unirradiated state or following the first two doses because of high attenuation. After the final irradiation (which gave $T_c=7.4$ K), this mode exhibited considerably less attenuation and direct measurements of its velocity showed agreement with the indirect measurement to generally within about 1%.

Micrometer measurements of the crystal size along the [001] and [110] directions showed fractional increases in length of 0.3 and 0.4 (±0.3) $\times10^{-3}$ after a dose of 0.25×10^{18} n/cm^2 and increases of 1.9 and 1.8 (±0.3)$\times10^{-3}$ after a dose of 22.2×10^{18} n/cm^2 in the two respective directions. The length increases for the latter dose, clearly outside experimental error, are comparable to those determined from lattice parameter measurements[9] and show that the defect induced expansions are nearly isotropic.

In the unirradiated state the elastic moduli (see Figs. 1 and 2) were found to be similar to those previously reported[8] for V_3Si. Small anomalies in several of the elastic moduli at $T = 20.5$ K marked the onset T_m of the Batterman-Barrett transformation. These anomalies, however, were smaller than previously observed[8] and suggest that only part of the sample was transforming. More convincing evidence of this has come from the specific-heat studies of Viswanathan *et al.*[4]

Following the first dose, T_c was found to be unchanged to within 0.2 K, and all measured sound velocities between 300 and 4.2 K were reproduced to ~0.1%. However, the small moduli anomalies which mark T_m at 20.5 K in the unirradiated sample were no longer observed, and new small anomalies occurred at ~18 K. These may be due to a structural transformation at this lower temperature. However, the character of this new anomaly was different from that which marked T_m in the unirradiated state; the sound pattern in the pulse superposition mode indicated an increase in sample inhomogeneity, resulting in spatial variations in sound velocity of ~0.1%.

Following the second dose, no changes in T_c or sound velocities were observed, again to within the above stated uncertainties. However, it was no longer possible to identify any clear anomaly as marking T_m. Thus, defects in very small doses affect the structural transformations, while the high T_c, soft mode, magnetic susceptibility, and H_{c2} remain essentially unaltered.

Figure 1 shows the temperature dependence of $\frac{1}{2}(c_{11} - c_{12}) = c_s$ in the unirradiated state ($T_c = 17.2$ K) and after the final dose of 22.2×10^{18} n/cm^2 which lowers T_c to 7.4 K. The shear mode softening of the high-T_c state is considerably reduced as a result of the radiation, and the unusually large defect-induced mode stiffening is greatest at low temperature.

Note that c_s for the irradiated sample, shown at low temperatures in the insert of Fig. 1, continues to change with temperature between 17 K and its new superconducting transition temperature of 7.4 K.

The magnetic susceptibility $\chi(T)$ was measured between 300 K and the superconducting transition temperature T_c in a field of 5 kG by the Faraday method using a Cahn RH electrobalance with a sensitivity of 2 μg. The magnetization was found to be proportional to field up to at least 10 kG

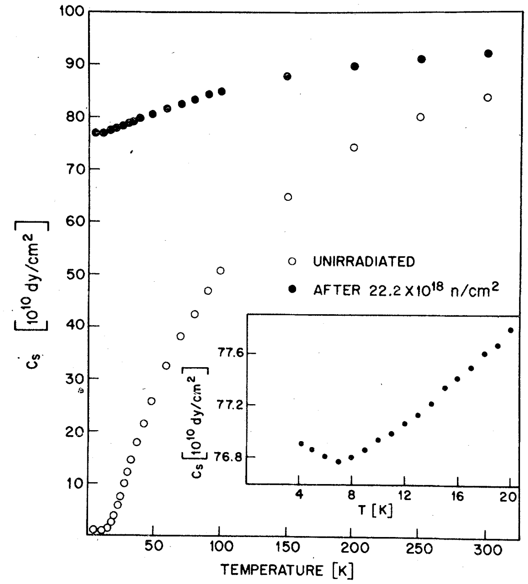

FIG. 1. $c_s = (c_{11} - c_{12})/2$ vs temperature T, initially and after irradiation to a fluence of 22.2×10^{18} n/cm^2. Insert shows low-temperature details for the irradiated sample.

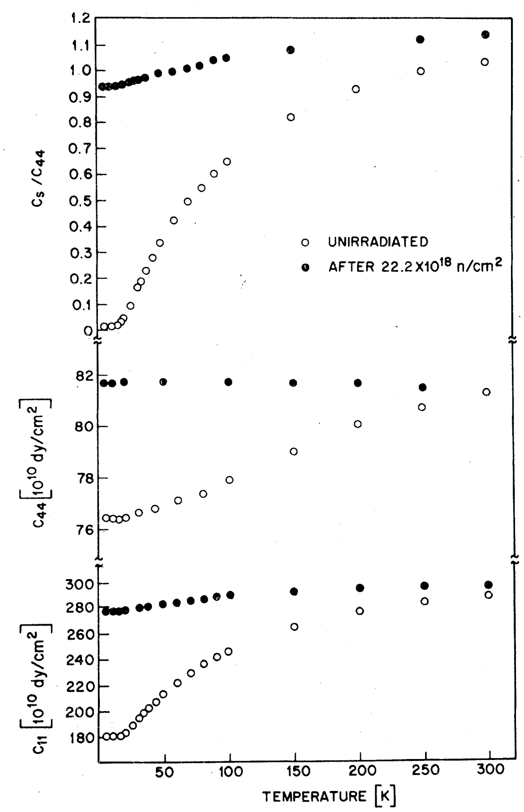

FIG. 2. c_{44}, c_{11}, and c_s/c_{44} vs temperature T, initially and after irradiation to a fluence of 22.2×10^{18} n/cm^2.

in the normal state. The absolute accuracy for χ was about 1% from run to run, while the precision within a given run was about 0.05%. The upper critical field H_{c2} was measured below T_c by plotting the output of the electrobalance as a function of external field on an X-Y recorder, and observing when the sample returned to the normal state as a function of applied field.

The susceptibility χ is shown in Fig. 3 as a function of temperature up to 300 K for unirradiated V_3Si, and for the same single-crystal sample after irradiation to the maximum fluence of 22.2×10^{18} n/cm^2. For a smaller fluence of 0.25×10^{18} n/cm^2, the value of the susceptibility was the same as for the unirradiated sample within the absolute experimental error of 1%, and the temperature dependence was unchanged to within the 0.05% relative experimental error. The inset of Fig. 3 shows the susceptibility of V_3Si after the largest dose at low temperatures on an expanded scale.

The susceptibility of the V_3Si sample before irradiation agrees very well numerically with measurements of Sherwood, *et al.*[10] No peak was observed at T_m such as that found by Maita and Bucher.[11] One should note, however, that the specific-heat data[4] indicate that less than 10% of the sample transforms to the low-temperature tetragonal phase.

Irradiation to a fluence of 22.2×10^{18} n/cm^2 lowered the susceptibility of V_3Si at 20 K by almost a factor of 2 and decreased the temperature dependence markedly. The susceptibility is still temperature dependent however, and comparable with that of other A-15 superconductors, such as V_3Ge, which have similar T_c's of ~7 K. We note that the inflection point in $\chi(T)$ increased from 35 ± 15 K for the unirradiated sample to 80 ± 40 K after the final irradiation.

A striking feature of the data is presented in the inset of Fig. 3, which shows that the susceptibility of the irradiated sample of V_3Si flattens rather suddenly at about 17 K (the T_c of the original, unirradiated sample), and remains independent of temperature to within experimental error down to the new T_c of 7.4 K. (Note that the sound velocity continues to vary in this temperature range.) Superconductivity, which would give measurable diamagnetic contributions,[12] seems to have been suppressed everywhere within the sample at temperatures down to 7.4 K.

Upper-critical-field measurements yielded $-(dH_{c2}/dT)_{T_c}=20.5\pm 1$ kG/K for unirradiated V_3Si, a value which was unchanged to within experimental error by an irradiation of 0.25×10^{18} n/cm^2. Following irradiation to 22.2×10^{18} n/cm^2, $-(dH_{c2}/dT)_{T_c}$ increased to 27.6 ± 1.3 kG/K. H_{c2}

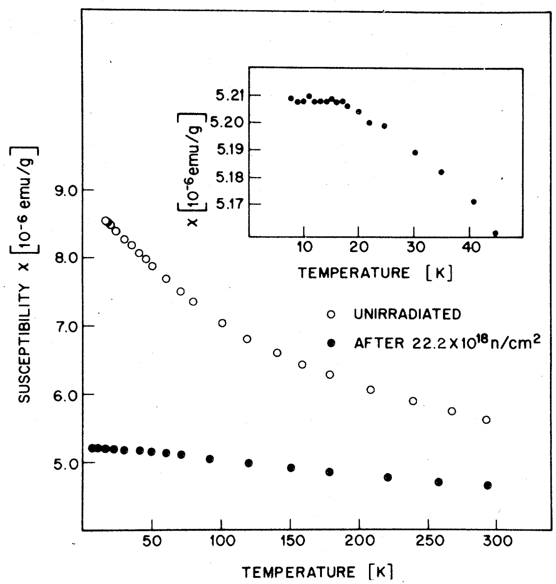

FIG. 3. Susceptibility χ vs temperature T, initially and after irradiation to a fluence of 22.2×10^{18} n/cm^2. Insert shows low-temperature details for the irradiated sample.

was linear in $(T-T_c)$ up to at least 30 kG for all samples measured, and extrapolations to $H_{c2}=0$ yielded $T_c=17.15\pm 0.05$ K for the unirradiated sample (a value unchanged by an irradiation of 0.25×10^{18} n/cm^2), and $T_c=7.4\pm 0.05$ K following an irradiation of 22.2×10^{18} n/cm^2. These values for T_c are in good agreement with those obtained from heat-capacity measurements on the same sample.[4]

DISCUSSION

We have found that the following major changes are produced in V_3Si by irradiation to a fluence of 22.2×10^{18} n/cm^2: (a) the superconducting transition temperature T_c is reduced from 17.2 K to 7.4 K; (b) there is a large defect-induced stiffening of the soft mode corresponding to $c_s=\frac{1}{2}(c_{11}-c_{12})$, and a much reduced temperature dependence for all elastic moduli; and (c) the susceptibility χ is lower by almost a factor 2, and there is a marked reduction in its temperature dependence.

The large reductions in the temperature dependences of c_s and χ indicate that both anomalies are being diminished with defects at comparable rates. In the simplest comparison, we may write

$$c_s(300\text{ K})-c_s(20\text{ K})=A[\chi(300\text{ K})-(20\text{ K})] \qquad (1)$$

and note that the proportionality factor A in the high-T_c unirradiated state is, to within several percent, the same as that in the heavily defected low-T_c state, although the quantities on either side of Eq. (1) are changing by somewhat more than a factor of 5. Correlations of quantities which may depend on the electronic density of states, such as that of Eq. (1), long ago provided the framework for the Labbé-Friedel[13] model in which unusual fine structure in the density of states near the Fermi level can contribute to the temperature dependences of c_s and χ, and lead to structural transformations as well. For such models, our results would imply that the disorder induced by irradiation causes considerable broadening and smearing of the density-of-states function.

If we attribute the decrease in the measured susceptibility to a decrease mainly in $N_d(0)$, the bare density of d-electron states at the Fermi energy, we can obtain an estimate for the change in $N_d(0)$ from the observed change in χ. In order to do this, it is necessary to attempt a separation of the various contributions to the measured χ. The two dominant contributions are the temperature-independent, orbital susceptibility χ_{orb} and the d-spin susceptibility $\chi_d(T)$ given at $T=0$ by

$$\chi_d = 2\,\mu_B^2 N_d(0)/[1 - N_d(0)\,V],$$

where V is an electron-electron exchange interaction. We adopt Clogston's[14] estimate for $\chi_{\text{orb}} = 7\times 10^{-4}$ emu/mole $= 3.87\times 10^{-6}$ emu/g, and use his analysis to obtain $N_d(0) = 2.5$ states/(spin eV V-atom), and $V = 0.173$ eV for unirradiated V_3Si. We note that this estimate for $N_d(0)$ is in good agreement with a value proposed for V_3Si by Fradin and Williamson[15] from an analysis of NMR measurements.

Using the preceding expression for χ_d and assuming that the interaction potential V remains constant with irradiation,[16] we deduce that the bare density of states for V_3Si after irradiation to a fluence of 22.2×10^{18} n/cm^2 is reduced by a factor of approximately 2.5 as compared to the unirradiated state.

Using these estimates for $N_d(0)$ and for its decrease due to irradiation, along with the measured value[4] for the coefficient of the linear term γ in the specific heat, we can obtain estimates for the electron-phonon enhancement factor $(1+\lambda)$. We note that $\gamma = \frac{2}{3}\pi^2 N_d(0) k_B^2 (1+\lambda)$, and use $\gamma = 64.7$ mJ/g mole K^2, and $N_d(0) = 2.5$ states/(spin eV V-atom) to obtain $1+\lambda = 1.82$ for unirradiated V_3Si. By comparison, McMillan's expression for T_c [see Eq. (18) of Ref. 17] yields an approximate value $1+\lambda = 1.89$ if $\mu^* = 0.10$ and $\hbar\langle\omega\rangle = 25.7$ meV[18] are used for the Coulomb pseudopotential and average phonon energy.

After irradiation to a fluence of 22.2×10^{18} n/cm^2, using[4] $\gamma = 21.2$ mJ/g mole K^2 and $N_d(0) = 1.0$ states/(spin eV V-atom), we obtain $(1+\lambda) = 1.49$. For the same values[16] of μ^* and $\hbar\langle\omega\rangle$, but with $T_c = 7.4$ K, McMillan's expression yields $1+\lambda = 1.62$.

From our results for $(dH_{c2}/dT)_{T_c}$ and the results of the companion paper[4] for $\Delta C(T_c)/T_c$, we can determine how the Ginzburg-Landau parameter $\kappa(T_c)$ varies with irradiation for the V_3Si sample studied. We use $dH_{c2}/dT = \sqrt{2}\,\kappa\, dH_c/dT$ as the definition of κ, and the thermodynamic relation $\Delta C(T_c)/T_c = (4\pi)^{-1}(dH_c/dT)^2_{T_c}$, in order to obtain the slope of the thermodynamic critical field at T_c. From[4] $\Delta C(T_c)/T_c = 126$ mJ/g mole K^2, we obtain $-(dH_c/dT)_{T_c} = 0.708$ kG/K which, along with $-(dH_{c2}/dT)_{T_c} = 20.5$ kG/K yields $\kappa(T_c) = 20.5$ for the unirradiated sample. For the sample following an irradiation of 22.2×10^{18} n/cm^2, a similar analysis yields $\kappa(T_c) = 56.9$.

We compare our results with the Gorkov-Goodman[19] expression $\kappa(T_c) = \kappa_0 + 7.5\times 10^3\,\gamma^{1/2}\rho_0$, where γ is the coefficient of the linear term in the specific heat (in units of erg/cm^{3}K^2) and ρ_0 is the residual resistivity (in units of Ω cm). Using $\gamma = 64.7$ mJ/g mole K^2 ($= 2.05\times 10^4$ erg/cm^{3}K^2) and $\rho_0 = 4$ $\mu\Omega$ cm (at 19 K) for the unirradiated sample,[4] we find $\kappa_0 = 20.5 - 4.3 = 16.2$ from the Gorkov-Goodman expression. If one now uses the Gorkov-Goodman expression, the above value of κ_0 can be used to predict $\kappa(T_c)$ for the sample following the irradiation of 22.2×10^{18} n/cm^2. Using $\gamma = 21.2$ mJ/g mole K^2 ($= 6.70\times 10^3$ erg/cm^{3}K^2) and[4] $\rho_0 = 92$ $\mu\Omega$-cm (at 19 K), one obtains $\kappa(T_c) = 16.2 + 56.4 = 72.6$, which is higher than the measured value of 56.9. It should be noted, however, that the Gorkov-Goodman expression is valid with a constant κ_0 only if the band-structure parameters do not change with radiation. There is, however, a large reduction in the density of states with irradiation, indicated by the reduction in γ and χ. An approximate calculation[20] indicates that this reduces κ_0 by an order of magnitude for the most heavily defected sample. The resulting value of $\kappa(T_c) \sim 60$, is in much better agreement with the measured value of 56.9

The anomalies in χ and c_s are diminished with defects at comparable rates over most of the measured temperature range, as pointed out at the beginning of this section. However, this "coupled" behavior breaks down below 17 K in the heavily defected state. Here $\chi(T)$ exhibits appreciable flattening while $c_s(T)$ continues softening to the new T_c of 7.4 K, and shows no major change in absolute value or slope at 17 K. This behavior, and the abruptness of the change in $d\chi/dT$ below

17 K, would appear to preclude an explanation based only on broadened fine structure in the density of states. At present we have no explanation for why χ of the defected material should have some memory of the high T_c state.

At the superconducting T_c, the temperature derivative of c_s undergoes a negative discontinuity for both the high- and low-T_c states. The magnitude of this discontinuity[21] is proportional to $d^2T_c/d\epsilon_s^2$ where ϵ_s is the shear (or tetragonal) strain associated with $\frac{1}{2}(c_{11}-c_{12})$. In the high-$T_c$ (cubic) state, previous work[21] has yielded the unusually large value $d^2T_c/d\epsilon_s^2=-9.5\times 10^4$ K. In the defected state with $T_c=7.4$ K, we now find $d^2T_c/d\epsilon_s^2=-17\times 10^4$ K. Thus, defects have reduced T_c, the mode softening and the temperature dependence of χ, but have unexpectedly increased the large negative shear strain dependence of T_c. In existing models of the A-15 compounds, the large magnitude of $d^2T_c/d\epsilon_s^2$ is expected from the large strain-dependent peak in the density of states. Since defects appear to reduce this peak, we have no explanation for the increase in $|d^2T_c/d\epsilon_s^2|$.

Figure 2 shows the elastic moduli c_{11}, c_{44}, and the anisotropy factor $(c_{11}-c_{12})/2c_{44}$. Like c_s, the moduli c_{11} and c_{44} stiffen with defects at all temperatures. For c_{44} the temperature coefficient with defects has reverted to the "normal" negative sign. Not shown is the bulk modulus $\frac{1}{3}(c_{11}+2c_{12})$, which increases by only a little more than 1% after the highest dose and increases by only ~$\frac{1}{2}$% between 300 and 4.2 K irrespective of dose.

The elastic anisotropy factor c_s/c_{44} (see Fig. 2), while largely reflecting the decrease in the temperature dependence of c_s, also shows that defects leave the material elastically isotropic to within 1% to 2% over the temperature range studied. It should be noted that elastic isotropy is rare even for cubic materials.[22] (Tungsten is the only element with comparable isotropy.) It does not generally result from chemical disorder alone, since random solid solution alloys show anisotropy. The result shows the loss of bonding or structural anisotropy on a large scale (the sound wavelength is ~150 microns) similar to an amorphous material. X-ray Bragg diffraction peaks, however, are generally found in V_3Si after comparable damage. The elastic isotropy provides a clue to the defect microstate which is not yet understood.

Calculations of the Debye temperature[23] from the low-temperature elastic moduli show $\Theta_D \sim 300$ K in the undamaged crystal and $\Theta_D \sim 520$ K for the heavily irradiated state with $T_c=7.4$ K. The former is of little use for comparison with specific-heat Debye temperatures Θ_c, because the large long-wavelength dispersion restricts its validity to temperatures considerably below T_c where a straightforward determination of Θ_c is not possible. Our Θ_D's can be compared with the values $\Theta_c=505$ K deduced from the specific-heat measurements of Viswanathan *et al.*[4] after irradiation by 22.2×10^{18} n/cm^2. With defects the agreement is better, as expected.

Our present results show a far greater stiffening of the low-frequency shear waves with defects, the disappearance of the anomalies at T_m, and possibly the disappearance of the structural transformation. Our understanding of the large changes in c_s and χ due to defects is limited by our knowledge of the accompanying microstate. ^{4}He channeling studies[24] and neutron studies[5] have indicated ~0.05 Å displacement of atoms from lattice sites in the damaged state. These and other defects (e.g., long-range disorder, small amorphous regions, etc.) are also accompanied by a stiffer lattice at high frequencies.

It was suggested by Testardi *et al.*[24] that both thermal and static (defect) displacements reduce the electron-phonon interaction and many of the anomalies in qualitatively similar ways. Huntington[25] has observed that the change in moduli with thermal disorder may predict the change in modulus with random displacements due to defects. Thus, increasing defect concentrations (static displacement magnitudes) may lead to behavior at low temperatures similar to what occurs at higher temperatures (larger thermal displacements) in the near perfect crystal. This is at least qualitatively consistent with observations for $c_s(T)$ and $\chi(T)$.

CONCLUSIONS

We have presented results of measurements of the sound velocity, susceptibility and upper-critical-field H_{c2} for a single crystal of V_3Si in its initial state and after successive neutron irradiations up to a fluence of 22.2×10^{18} n/cm^2. These measurements, together with the specific heat, electrical resistivity, and Bragg-Williams long-range-order parameter reported in the accompanying papers,[4,5] yield the most extensive characterization of a single specimen of V_3Si so far obtained.

Very low defect levels affect the structural transformation, while leaving the superconducting transition temperature T_c, the soft mode, the susceptibility, and H_{c2} essentially unaffected. Higher defect concentrations, which reduce T_c, also considerably reduce the anomalous temperature dependences of the susceptibility and elastic moduli. However, significant anomalies, comparable with those for V_3Ge, remain for the highest fluence which produces a moderate T_c of 7.4 K. The sound velocity measurements show that high neutron doses leave the material elastically isotropic to within 1 to 2% over the entire temperature range studied, indicating a loss of bonding or structural anisotropy on a large scale similar to an amorphous material. Unexpectedly, the highly defected material appears to show an increase of the large, negative shear strain dependence of T_c. Further, there is an abrupt change in $d\chi/dT$ at about 17 K (the T_c of the original, unirradiated sample), below which χ remains constant while the elastic modulus c_s continues to soften.

Almost all models for the A-15 compounds assume, or attempt to calculate, unusual fine structure in the electronic density of states near the Fermi level. Within such models, our data can be given a qualitative explanation by assuming that defects broaden and reduce this peak. We have presented qualitative calculations of this effect. Whether any model can explain all of the observed effects with a single broadened density of states remains an untested challenge of the proposed theories.

ACKNOWLEDGMENT

This work was supported in part by the City University of New York Faculty Research Award Program.

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