Effect of neutron irradiation on single-crystal V_3Si: Heat capacity and resistivity

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We have measured the heat capacity and resistivity of a V_3Si single crystal as a function of neutron irradiation up to a fluence of 22.2×10^{18} n/cm^2. The structural transformation was found to be extremely sensitive to very-low-level irradiations ($\sim0.25\times10^{18}$ n/cm^2) whereas the superconducting transition temperature T_c was not affected up to $\sim3\times10^{18}$ n/cm^2. The residual resistivity showed minor variations up to $\sim3\times10^{18}$ n/cm^2 and started to change appreciably beyond that fluence. The electronic-heat-capacity coefficient and hence the density of states at the Fermi level was found to be considerably affected for *all* fluences. Further there is some evidence for the inhomogeneous nature of the irradiated state.

I. INTRODUCTION

The degradation of the superconducting transition temperature T_c of *neutron*-irradiated A-15 compounds is well documented.[1-4] However there is no consensus as to the nature of the defects and how they affect T_c in these irradiated superconductors.[4-6] To better understand this problem we set out to investigate for the first time the low-temperature heat capacity and resistivity of a V_3Si single crystal as a function of neutron irradiation up to a fluence of $\sim22.2\times10^{18}$ n/cm^2. On the same crystal, sound velocity, magnetic susceptibility, critical field, and neutron-scattering measurements were also performed and the results are reported in the accompanying papers.[7,8]

II. EXPERIMENT

A. Samples

The samples used in this experiment are from a single crystal of V_3Si grown by Greiner and Mason[9] using a floating-zone melting technique. The heat-capacity experiments were done on the same large crystal (~1.5 g) as the sound velocity, magnetic susceptibility, and neutron-scattering experiments. A resistivity sample was spark eroded to a uniform cross section of 0.54×1.32 mm from a piece adjacent to the large single crystal in the original boule.

B. Neutron irradiation

The neutron irradiations were all carried out in the High Flux Beam Reactor (HFBR) at Brookhaven National Laboratory. The HFBR used U^{235} as a fuel and is moderated and cooled with D_2O. The flux in the irradiation chamber is composed of a broad spectrum of energies and can be broken down as follows: 1.3×10^{14} n/cm^2 sec for $E>1$-MeV neutrons; 5.3×10^{14} n/cm^2 sec for $E>0.1$-MeV neutrons; 1.9×10^{14} n/cm^2 sec for $E<0.63$-eV neutrons (thermal neutrons); and a total flux of 12.1×10^{14} n/cm^2 sec. We have calculated our fluences on the basis of neutrons of $E>1$ MeV (i.e., using a flux of $\sim1.3\times10^{14}$ n/cm^2 sec). The determination of the flux was done by Argonne National Laboratory from a self-consistent computer fit to activation analysis data on 15 different pure metal foils irradiated in the HFBR.[10]

The samples for irradiation were packed in a water-cooled aluminum capsule using two different techniques. (i) The samples were wrapped in aluminum foil and tightly fitted into a sealed quartz tube, filled with $\sim\frac{1}{2}$ atm of helium gas, and fit tightly into the aluminum capsule. (ii) The samples were wrapped in aluminum foil and tightly packed in fine mesh aluminum powder in the aluminum capsule. The first three irradiations used technique (i)[11] and successive irradiations used technique (ii). We estimate that the temperature of the crystal was $\lesssim300-400$ °C for the first irradiation and $\lesssim200$ °C thereafter, and the temperature of the resistivity sample was $\lesssim200$ °C for all irradiations.[12]

C. Heat capacity

The heat capacity was measured by a conventional heat pulse technique. The sample was sandwiched between a Au(0.07-at.% Fe)-chromel thermocouple and an evaporated heater, each on a 6.25-mm-thick sapphire substrate. The heater resistance was measured by a four-probe technique for each data point. The thermocouple was referenced to a pumped ^{4}He pot with calibrated Ge and Pt thermometers through a reference copper block of ~10 g. The measured heat capacity had to be normalized by a factor of 1/2.5 to take into account the incomplete thermal isolation of the sample. This factor was found to be the same for different sample mountings and for all temperatures. The experiment was semiautomated using a Digitrend

220μp scanner and a HP9830A programmable calculator. The details of the experimental setup, which is similar in nature but quite different in detail to an earlier ac calorimetry arrangement,[13] will be published elsewhere.[14] The precision of the heat-capacity data is to within 0.5% and the absolute accuracy is ~ 2 to 3%.

D. Resistivity

The resistivity ρ was measured as a function of temperature from T_c to room temperature. A standard four-probe technique was employed using an ac excitation current in the range of 10 mA at a frequency of ~15 Hz.[15] The absolute values of ρ at room temperature were obtained using knife edge voltage contacts with separations on the order of 1–2 mm. The overall reproducibility of this absolute measurement was ~ 5%. The temperature dependence was measured either with the knife edge contacts (first three irradiations) or indium contacts (from the third irradiation on) which necessitated normalization of the data to the room-temperature values. The two techniques gave temperature dependences reproducible to better than 1%. The temperature scale was a calibrated germanium thermometer from 4.2 to 40 K and a calibrated platinum thermometer from 40 to 350 K. Thermal equilibrium was assured by thermally anchoring the sample and thermometers to a large copper block and making the measurement in helium exchange gas. Although the precision of the measuring instruments was ~ 0.1 to 0.2% the overall reproducibility of the temperature dependences was on the order of 0.5%

III. RESULTS

A. Heat capacity

The heat-capacity data for the unirradiated V_3Si and after the heavier irradiations (3rd and 4th) are plotted in Fig. 1 as C/T vs T^2. The data for the first and second irradiations not included in Fig. 1 are qualitatively similar to the data prior to irradiation. The various parameters which can be extracted from an analysis of this data are summarized in Tables I and II for all the irradiations. The superconducting transition temperature T_c is reported at the onset, midpoint, and peak of the transition. It should be noted that the T_c's and corresponding widths from heat capacity are truly representative of the bulk.

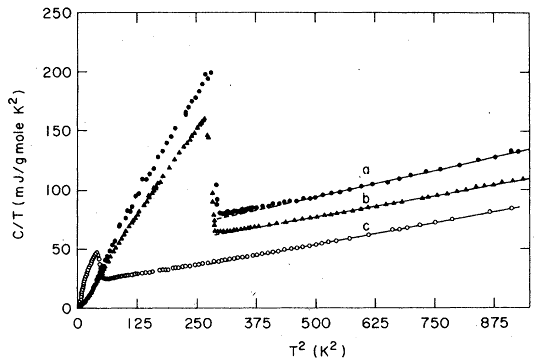

FIG. 1. C/T vs T^2 as a function of neutron fluence, ϕt, where ϕ is the flux (a) unirradiated, $\phi t = 0$, (b) $\phi t = 3.5 \times 10^{18}\, n/\text{cm}^2$, and (c) $\phi t = 22.2 \times 10^{18}\, n/\text{cm}^2$.

The deviation in the data between T_c and ~ 22 K prior to irradiation (Fig. 1) reflects the structural transformation from a cubic to tetragonal structure. The transformation, and thus the resulting deviation, is quite small. This makes it difficult to determine the structural transformation temperature T_m reliably. Rather than take T_m directly from Fig. 1 we plotted the slope of the C/T-vs-T^2 curve as a function of temperature in the region of interest which will show the T_m much more clearly.[16] This plot appears in Fig. 2. The entropy associated with the transformation $(\Delta S)_{T_m}$ which is the difference in entropy between the cubic and tetragonal states, was determined graphically. The cubic state was extrapolated from above T_m to T_c and it was assumed that the transformation was arrested at T_c. T_m and $(\Delta S)_{T_m}$ appear in Table I.

TABLE I. Sample parameters obtained from the heat capacity as a function of neutron fluence. T_m and $(\Delta S)_{T_m}$ are obtained as described in the text. T_c is correct to ~±0.05 K, T_m to ~±0.5 K, and $(\Delta S)_{T_m}$ correct to ~±1mJ/g mole K^2.

ϕt ($10^{18}\, n/\text{cm}^2$)	T_c(onset) (K)	T_c(midpoint) (K)	T_c(peak) (K)	T_m(onset) (K)	$(\Delta S)_{T_m}$ (mJ/g mole K^2)
0	17.3	17.0	16.8	21.5	8
0.25	17.25	16.85	16.65	21.5	2
1.65	17.2	16.95	16.7	21.5	1
3.53	17.0	16.75	16.4	21.5	2
22.2	7.5	6.8	6.2	19	1.5 (?)

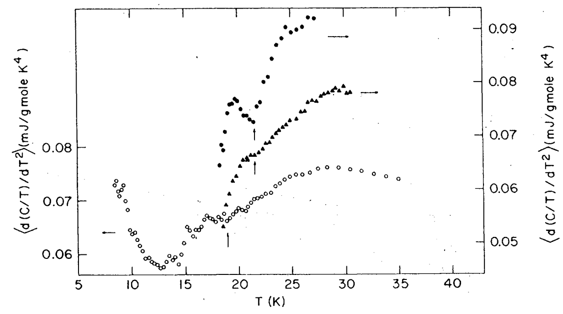

FIG. 2. Slope of C/T vs T^2 for a 13-point parabolic fit of the data, as a function of T; (●) unirradiated, $\phi t = 0$, (▲) $\phi t = 3.5 \times 10^{18}\ n/cm^2$, and (■) $\phi t = 22.2 \pm 10^{18}\ n/cm^2$. Arrows mark the onset temperature of the structural transformation T_m.

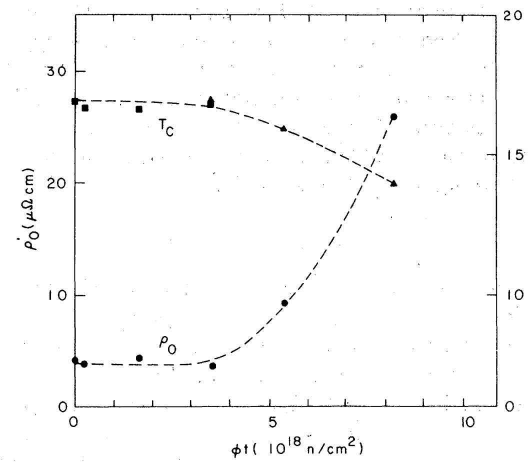

FIG. 3. Residual resistivity, ρ (19 K) and superconducting transition temperature T_c as a function of fluence.

Assuming $C_n = \gamma T + \beta T^3$, the linear term γ [$\propto N(E_F)$] and the cubic term β ($\propto 1/\Theta_D^3$) were determined by (i) linear extrapolation from above T_m and by (ii) equating the entropy in the superconducting state to that in the normal state and choosing $C_n(T_c)$ as the measured value at the onset of the transition. The values from (i) will be referred to as γ_c, β_c, and Θ_c, and those from (ii) as γ_T, β_T, and Θ_T. These values appear in Table II. It should be noted, however, that the simple Debye law is not expected to be valid for such high temperatures and in such systems (i.e., A-15 compounds). Therefore the γ and β values obtained have to be used with caution and they are most suitable for comparison purposes.

B. Resistivity

A plot of the residual resistivity, ρ_0 as a function of fluence appears in Fig. 3. ρ_0 was arbitrarily taken as ρ (19 K) for all irradiations since ρ vs T is very flat in this region. T_c at the midpoint of the transition, determined from the resistivity data, is also plotted as a function of fluence in Fig. 3. It should be noted that only the first three irradiations were carried out simultaneously for the resistivity and heat-capacity samples. Subsequently the irradiations for the resistivity sample were done separately in finer intervals of fluence.

IV. DISCUSSION

A. Unirradiated state

The values that we extract for γ and β for unirradiated V_3Si are comparable to previous results obtained by similar analyses.[17-20] To estimate the lattice contribution, C_L to the total heat capacity for unirradiated V_3Si in the harmonic approximation we have calculated C_L using the most plausible phonon density of states.[21] In this approximation $C_L = 3R \int (X/\sinh X)^2 F(\omega)\, d\omega$, where $X = \hbar\omega/2k_BT$ and $F(\omega)$ is the phonon density of states. The result is plotted in Fig. 4. The calculation based on the model density of states from acoustic data underestimates C_L, due to omission

TABLE II. Parameters obtained from the heat-capacity analysis as a function of neutron fluence. γ_T and β_T were obtained by the entropy constraint $[S_S(T_c) = S_N(T_c)]$ to a precision of $\lesssim 0.5\%$. γ_c and β_c were extrapolated from the high-temperature data (>22 K) using $C/T = \gamma_c + \beta_c T^2$ with precisions of $\lesssim 1\%$ and $\gtrsim 0.5\%$, respectively. C_∞ and β_∞ were obtained by fitting to $C = C_\infty + \beta_\infty T^3$ above 22 K with precisions of $\lesssim 1\%$ and $\lesssim 1\%$, respectively. Θ_T, Θ_c, and Θ_∞ are the Debye temperatures from β_T, β_c, and β_∞ with a precision of $\sim\pm 4$ K. The reduced jump $\Delta C/\gamma_T T_c$ is calculated to a precision of $\lesssim 1\%$.

ϕt ($10^{18}\ n/cm^2$)	γ_T (mJ/g mole K^2)	γ_c	β_T	β_c (mJ/g mole K^4)	β_∞	C_∞ (mJ/g mole K)	Θ_T	Θ_c (K)	Θ_∞	$\Delta C/\gamma_T T_c$
0	64.7	48.4	0.0511	0.0906	0.115	819	534	441	407	1.95
0.25	65.3	51.7	0.0512	0.0953	0.124	821	533	434	397	2.08
1.65	58.7	47.7	0.0484	0.0841	0.112	738	544	452	411	2.03
3.53	53.0	41.8	0.0414	0.0695	0.094	663	573	482	436	1.94
22.2	21.2	16.6	0.0604	0.0740	0.083	270	505	472	454	1.40

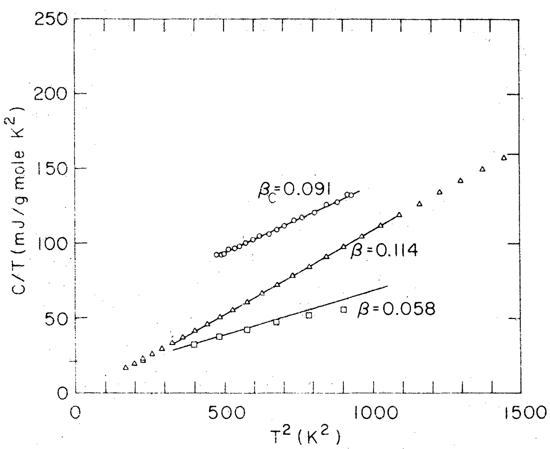

FIG. 4. C/T vs T^2 of the unirradiated sample from calculations and from the experimental data; ○ the experimental data with $C = C_L + C_{el}$, △ calculated from most plausible phonon spectrum (see text and Ref. 21) with $C = C_L$, and □ calculated from acoustic data (see text and Ref. 21) with $C = C_L$.

of high-frequency phonons, whereas that based on the neutron data slightly overestimates C_L, due to weighting factors.[21] We have included our experimental data for the total heat capacity on Fig. 4 and it can be seen that the slope of C/T vs T^2 which represents the lattice contribution lies between these two estimates, but closer to the estimate from the neutron data, as would be expected.

If we assume that C_L from the combined neutron and acoustic data is nearly correct, then the maximum contribution to the temperature dependence of C/T from terms other than T^3 is of the order of ~20%. These terms could result from anharmonic[22] or temperature-dependent electronic[23] contributions. If they are positive then they would have to increase as the temperature is reduced. Such a temperature-dependent contribution from the electronic term has been calculated[23] using the Labbé-Friedel model. It was shown[23] that in V_3Si above T_c, $C = C_\infty + \beta T^3$ where C_∞ is the saturated electronic term. For our data $\beta = 0.115$ from this type of fit (Table II) which is in good agreement with $\beta = 0.114$ from the C_L calculated from the most plausible phonon spectrum. Further, a similar dependence of the electronic term was reported[20] in V_3Ga by self-consistently analyzing heat capacity, magnetic susceptibility, and ^{51}V nuclear spin-lattice relaxation-time data.

The fact that γ_T and γ_c are not equal also indicates the presence of other temperature-dependent terms in the expression $C_N = \gamma T + \beta T^3$. We have attempted to fit C_N by including higher-order terms (odd powers up to T^{15}) and requiring that $S_N(T_c) = S_s(T_c)$. This did not yield values of γ that were significantly different from γ_T. We put an additional constraint by fixing β from the elastic constant data or from the calculated phonon density of states. Both yielded values of γ between γ_c and γ_T with a hump in the heat capacity below T_c. Similar results were obtained earlier by fixing β from the low-temperature ($T < 2$ K) heat-capacity data.[19] The discussion above indicates that the actual value for γ most probably lies somewhere between γ_c and γ_T.

B. Irradiated states

Examination of the values of γ in Table II shows a consistent trend as a function of irradiation in both γ_c and γ_T. Thus, it is reasonable to pick either γ for a comparison of the data as a function of irradiation. $N_d(E_F)$, the electronic density of states for the d band at the Fermi level, is related to γ by $\gamma = \frac{2}{3}\pi^2 k_B^2 N_d(E_F)(1+\lambda)$ where λ is the electron-phonon enhancement. λ was determined from McMillan's formulation[24] using T_c, Θ_D, and μ^*. μ^* was chosen to be 0.13 and the Debye temperature Θ_D to be Θ_∞ (Table II) for all fluences.[25,26] The values of $N_d(E_F)$ and λ for the various radiations assuming γ_T to be the correct γ, appear in Table III. The ratio of $N_d(E_f)$ in the unirradiated to the heavily irradiated state is 2.6, which is in excellent agreement with that calculated from the magnetic susceptibility data.[7] In addition, for the lowest dose where the change in γ is small, the magnetic-susceptibility data was the same to within the experimental error of 1%.

We observed a strong dependence of γ (Table II), and thus $N_d(E_F)$ on fluence for low levels of irradiation where T_c changes only slightly (Table I). Recently γ values have been determined indirectly from $(dH_{c2}/dT)_{T_c}$ and ρ_0 measurements on α-irradiated films of Nb_3Sn, and they also show a strong decrease for low doses.[27] The similarity of the results from our heat-capacity analysis to the critical-field data[27] (where anharmonicity should not be a factor) and the absence of change

TABLE III. Electronic density of states, $N_d(E_F) = 3\gamma_T / 2\pi^2 k_B^2(1+\lambda)$ as a function of neutron fluence. λ is calculated from McMillan's expression as described in the text.

ϕt (10^{18} n/cm^2)	λ	$N_d(E_F)$ (states/eV spin V-atom)
0	0.91	2.40
0.25	0.92	2.41
1.65	0.91	2.18
3.53	0.88	2.00
22.2	0.62	0.93

in the softening[7] (as observed in the sound velocity data) up to a fluence of 3.5×10^{18} n/cm^2 both indicate that the change in γ we observe at the low fluence levels is not related to a change in the anharmonic contributions, but is related to change in $N_d(E_F)$. These changes in γ may be understood by the suggestion, originally put forth by Labbé-Friedel[28] and more recently elaborated by Nettel and Thomas,[29] that T_c is not dependent on $N_d(E_F)$ in V_3Si, where there is a sharp density of states at the Fermi level, but rather on an average density of states $\bar{N}$. It is possible that for uniform damage, although $N_d(E_F)$ drops rapidly as the density of states is smeared by low-level radiation damage, $\bar{N}$ and hence T_c does not change.

One very striking feature of the dependence of γ on fluence is that γ_T and γ_c differ by approximately the same percentage for all fluences. This difference cannot be accounted for by anharmonic contributions to C_N due to lattice softening since the difference does not decrease even for the highest fluence, where the elastic constant data[7] indicate considerably less softening of the phonons. Similarly the temperature-dependent electronic density of states which results from the sharp peak may not account for this difference, since the susceptibility data[7] indicate considerable broadening of the sharp peak in the heavily damaged state. In addition, a recent calculation[30] based on disorder of the linear chains in the Labbé-Friedel model[31] shows that C_∞, in the expression $C = C_\infty + \beta T^3$, should increase with disorder. However, we observed a decrease (see Table II). The persistence of the percentage difference between γ_T and γ_c at all fluences suggests that some characteristics of the unirradiated sample are probably retained in the heavily damaged state. Certain observations in other properties studied also indicate the persistence (or retention) of the unirradiated state even in the heavily damaged V_3Si. The sudden loss of temperature dependence of susceptibility between ~17–20 K and T_c, the unexpectedly large negative shear strain dependence of T_c and the considerable peak broadening of the soft shear [110] mode at $T < 80$ K in inelastic neutron data are typical characteristics of unirradiated V_3Si.[7,8] Similar features are observed even for the heavily damaged state.[7,8]

Another interesting result is the variation of the reduced jump height $\Delta C/\gamma_T T_c$, where ΔC is the jump height at T_c as a function of irradiation, as given in Table II. $\Delta C/\gamma_T T_c$ for the unirradiated V_3Si indicates that it is a strongly coupled superconductor. The reduction of the reduced jump after the heavy irradiation can be interpreted in two ways. One could say that the sample becomes a weakly coupled BCS superconductor in this heavily damaged state. If correct, this would be the first such observation. All weakly coupled superconductors studied to date become strongly coupled as they are disordered.[32,33] In addition, Nb which is a strongly coupled superconductor, has recently been shown to be more strongly coupled when mechanically damaged.[34] An alternate explanation is to assume an inhomogeneous irradiated state, with regions of low T_c (perhaps even normal material) and regions of higher T_c, which would result in a decreased jump. It was recently shown by transmission electron microscopy that the neutron-irradiated Nb_3Sn is in fact quite inhomogeneous with disordered regions of diameter ~35 Å randomly distributed in the less disordered matrix.[6]

The heat-capacity data for the heaviest irradiation show an anomaly at ~13 K. This is most apparent in Fig. 2 where the slope of C/T vs T^2 is plotted. It is possible that the anomaly arises from anharmonic contributions, but such contributions are expected to be small in this case due to the greatly reduced softening. If it were due to temperature dependence of the electronic contribution, we would expect the anomaly to shift up in temperature as the peak broadens. However the anomaly occurs at nearly the same temperature as the anomaly observed[19] in the unirradiated state. Interestingly, a spread out superconducting transition in an inhomogeneous material could explain this observation. However, the magnetic susceptibility measurements[7] show no sign of diamagnetism above 7.5 K. Caution should be exercised in interpreting this as lack of superconductivity above 7.5 K, since if the higher T_c material were distributed on a fine scale ($\lesssim 100$ Å) there could be considerable penetration of the magnetic field[35] even if its volume fraction is not insignificant.

The structural transformation was quite small to start with, as evidenced by $(\Delta S)_{T_m}$ (Table I), which is less than 10% of the maximum value observed previously.[18,36] This could be due to either a small portion of sample transforming or a small distortion throughout the sample. The important observation with regard to the structural transformation is its extreme sensitivity for very low damage levels. The entropy of transformation decreases considerably for as low a fluence as 2.5×10^{17} n/cm^2 (Table I). This is in qualitative agreement with the disappearance of the transformation as seen in the inelastic neutron scattering[8] and sound velocity measurements.[7] Since the transformation is very small to begin with, the exact behavior of T_m and $(\Delta S)_{T_m}$ as a function of fluence requires further study.

The residual resistivity at low fluences is essentially independent of fluence except for minor fluctuations which are, however, outside experimental error (the same fluctuations are seen in the residual resistivity ratio (RRR) which is much more precisely determined). Previous observations of neutron-damaged high-purity metals[37] and α-particle damaged[38,39] A-15 materials always show an increase in ρ_0 with fluence in the initial region. However none of the A-15 samples previously investigated had a ρ_0 as low as that in this work (4 $\mu\Omega$ cm). When plotted as T_c vs RRR all of our data fall on the curve for V_3Si obtained for α-particle irradiation of thin films.[38]

The insensitivity of T_c to fluence up to $\sim 3.5\times 10^{18}$ n/cm^2 is remarkable since previous data[40] on polycrystalline V_3Si powder showed a strong degradation in this region. It is important to note that both heat-capacity measurements of T_c on the large and smaller single-crystal and resistivity measurements of T_c on the smaller single crystal showed the same insensitivity to low fluence, suggesting the existence of a "threshold fluence." Detailed discussions of the behavior of T_c at low fluences and the effect of possible annealing during irradiation have already been published.[12] We just make the additional comment here that it is very difficult to understand the consistent changes in γ for low fluences if the sample were annealing during irradiation. It is interesting to note that such an insensitivity of T_c to neutron fluences was observed for the first time in the well characterized Nb_3Sn specimens long ago.[41] More recently the existence of "threshold fluence" has been established explicitly for n-damaged polycrystalline Nb_3Sn.[42]

V. CONCLUSION

The following conclusions can be drawn from our data and analysis (i) We expect the electronic-heat-capacity coefficient γ to lie between γ_T and γ_c. γ and hence $N_d(E_F)$, the d-band density of states at the Fermi level decreases appreciably as a function of neutron damage. Similar observations can be made from the susceptibility data. (ii) We believe the most reasonable Debye temperature Θ_D prior to irradiation is ~ 410 K, which was calculated from the available phonon spectra. Similar values were obtained by analysis of heat-capacity data assuming a saturated electronic state; for the heaviest irradiation: $\Theta_D \sim 455$ K. And this would indicate the lattice is stiffened in the defected state. This is qualitatively similar to the elastic constant and neutron data. (iii) The structural transformation is found to be extremely sensitive even for low-level damages. (iv) There is evidence in our work to support the assumption that neutron-irradiated V_3Si is inhomogeneous.

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[15] The data for the V_3Si sample before irradiation were taken at ~ 200 Hz. Subsequent checks showed the data to be frequency independent from ~ 5 to 200 Hz.
[16] The slope was determined by fitting an odd number N of successive points to a parabola and choosing the slope at the midpoint. We chose $N=13$ to reduce the scatter by averaging more points. However the qualitative nature of the results did not change by choosing $N=5$ or fitting to a straight line instead of a parabola.

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