Thermal expansion of V_3Si with controlled martensite-phase morphology

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High-resolution thermal-expansion measurements along the tetragonal a and c axes have been made for a martensitically transforming V_3Si single crystal by using external stress fields to control the morphology of the low-temperature-phase domains. Thermal-expansion anomalies at low temperatures below T_c were found for both the a and c axes. The tetragonality continues increasing on cooling at low temperatures, which, predicted by theory, should have been inhibited by the onset of superconductivity. The discontinuities of the linear-expansion coefficients for the a and c axes at T_c are of opposite sign. The results are consistent with the picture of an electronically driven structural distortion that is destroyed by the thermally induced lattice distortion.

I. INTRODUCTION

The $A15$ compound superconductors V_3Si and Nb_3Sn have been studied extensively on account of the possible correlation between their structural properties and superconductivity.[1] It is well known that V_3Si undergoes on cooling a cubic-to-tetragonal, structural, phase transition with a transformation temperature $T_m \approx 21$ K, just a few degrees above its superconducting transition temperature ($T_c \approx 17$ K).[2] The transformation has been characterized as martensitic, namely, a diffusionless (weakly), first-order, displacive, structural, phase transition, with twin bands formed in the low-temperature phase. The tetragonal distortion has a c/a ratio of about 1.002,[2] and effectively, the tetragonal axes are parallel to $\langle 100 \rangle$ of the cubic phase. (All the indices hereafter referred to are for the cubic axes.) The twin bands are always parallel to $\langle 001 \rangle$ in $\{110\}$ planes.[3] The transformation is sample dependent and does not always occur. It has been widely accepted that the transformation is electronically driven and due to a band Jahn-Teller (BJT) effect.[4–9]

High-resolution dilatometry studies on polycrystalline and single-crystal samples of V_3Si have been performed by several authors.[10–16] Large peaks in linear thermal-expansion coefficient α were observed at T_m for transforming, single-crystal samples measured along $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$ and polycrystalline samples. Premartensite thermal-expansion anomalies were detected as high as 40–70 K above T_m.[12–16] The anomalous expansion behavior appeared to continue to temperatures below T_c.[10–12]

The single-crystal measurements also showed that the peaks could be positive or negative, depending on the crystallographic direction of the measurements. The crystallographic direction was dependent on the sample and the magnitude of the applied uniaxial compressive stress along the measurement direction.[13,16] These effects demonstrated that the formation of the low-temperature-phase microstructural domains in V_3Si is sensitive to applied and/or residual internal stress fields.

A positive peak was observed for α along $\langle 100 \rangle$ in these earlier studies, with the exception of one set of measurements[15] where the sign of α changed and became negative below T_m. Thus, it appears that, with one exception, the tetragonal a-axis expansion behavior was measured along $\langle 100 \rangle$ when the sample was stressed uniaxially along this direction. The negative peaks which have been observed in the $\langle 110 \rangle$ and the positive peak in the [111] directions represent mixed a- and c-axis behavior.

For some nontransforming samples, similar anomalous expansion behaviors were observed[16] above T_c but with much smaller magnitudes (usually one order), indicating that there is a tendency to transform at a lower T_m for these samples, which may be suppressed by the onset of superconductivity. Measurements[17] of the elastic properties as a function of temperature had previously revealed that for nontransforming V_3Si the onset of superconductivity arrests the lattice softening, which continues to lower temperature when the superconductivity is suppressed in a magnetic field.[18]

Theoretical models of the structural stability of the $A15$ compounds have centered on the raising of the degeneracy of the d bands by the tetragonal distortion. Two models have been discussed in detail:[7] a band Jahn-Teller effect and a Peierls instability. Both models result in the formation of an energy gap at part of the Fermi surface which lowers the electronic energy of the system sufficiently to compensate for the increase in lattice strain energy due to the lattice distortion. The onset of superconductivity and the formation of the single electron excitation energy gap inhibits further lowering of the electronic energy due to the electronic band splitting.

There have been several calculations of the effect of superconductivity on the martensitic transformation. That

by Kataoka[8] in the context of very narrow, twofold-degenerate d bands, predicts a marked reduction in the growth of the tetragonal distortion at T_c and a value of $1-(c/a)_0$ at $T=0$ approximately one-half that in the absence of superconductivity.

There has been no direct observation of the arrest in the growth in tetragonality at T_c. The only direct measurements of the tetragonal a- and c-axis expansions for V_3Si have been made by x-ray diffraction[2,19] and these lack the resolution to determine whether there is an arrest in the growth of c/a at T_c. The existence of low-temperature-phase domains has prevented direct measurements of the expansion behavior by capacitance dilatometry which has a resolution far surpassing diffraction methods. Thus, a complete picture of the expansion behaviors along the c and a axes for transforming V_3Si has not been given by the previous studies.

The observed discontinuity in α at T_c, $\triangle\alpha^{n-s}=\alpha^n-\alpha^s$ (n and s denote normal and superconducting), was observed to be positive when it occurred in the positive anomalous peak (i.e., a-like direction) and negative in the negative anomalous peak (i.e., c-dominated direction).[13,16] This implies that the sign of the strain dependence of T_c is dependent upon the crystallographic direction of the applied stress.

Ott *et al.*[15] concluded from their measurements on a single-crystal sample that there is a small volume change accompanying the transformation. This represents the only experimental evidence for the first-order nature of the transformation, which had long been theoretically predicted[20,7] and experimentally sought.

As part of our systematic investigation of first-order displacive phase transitions by high-resolution, capacitance dilatometry,[21,22] a V_3Si single crystal has been studied. Following the example of our recent thermal-expansion measurements for an In-26.5 at. % Tl single crystal[22] which shows a martensitic transformation comparable to that in V_3Si, we subjected our V_3Si crystal to different external stress fields to control the morphology of the low-temperature-phase domains. This has enabled us to measure the bulk tetragonal a- and c-axis expansion behaviors as a function of temperature through the transformation and down to 2 K. The results are reported here.

II. EXPERIMENTAL DETAILS

The sample was a $3.5\times4.6\times4.8$ mm^3 cube with {100} faces, spark cut from a disk 8 mm in diameter and 6 mm thick, which had been cut from a single-crystal rod grown by E. S. Greiner (referred to in Ref. 23). The excellent quality of the crystal was indicated by a small FWHM (full width at half maximum) value of 0.15° for the (200) x-ray Bragg reflection for this crystal.

The sample was aligned to the desired directions within 1° using the conventional x-ray Laue method. All the Laue photos taken from different {100} faces showed well-defined Laue patterns, which agree well with the corresponding computer-simulated patterns[24] for a β-W ($A15$) crystal structure with V and Si atoms occupying their appropriate lattice sites. The forbidden reflections additional to those from the simple lattice, which are generally observed for V_3Si and are due to the deviation of the V atom charge density from spherical symmetry caused by the anisotropy and anharmonicity of thermal vibration,[25] were also observed. For identification, we designate the three cube directions as A ([100]), B ([010]), and C ([001]).

Thermal-expansion measurements were made using a low-temperature, high-resolution, three-terminal-capacitance dilatometer identical to the design of White and Collins.[26] The experimental apparatus and procedure have been described previously.[27] The maximum resolution of this dilatometer for change in sample length was about 5 Å. This corresponds to, for example, a resolution of about 10^{-7} K^{-1} in α at 4.2 K for a 5 mm long sample and a 1 K temperature interval between two neighboring datum points. Data were collected during heating runs from 4.2 K to 60 K and cooling runs of 4.2–1.5 K.

The T_c was determined by the induction method, having a pair of coils around the sample, in which a small ac field (about 5 G at 36 kHz) was applied, while the expansion measurements were being simultaneously made. The T_c determined from a sharp change of the signal from the pickup coil with increasing temperature was 16.7 K (midpoint), with the width of the transition $\triangle T_c$ being 0.3 K.

Mounting the sample in the capacitance cell subjects it to an unavoidable uniaxial stress ($s\approx0.5$ bar) from the sample mounting springs along the measurement direction (l). The measured strain indicated that this forced the crystal to transform with the shorter axis (a axis) of the tetragonal unit cell along l (see the bottom inset to Fig. 1).

The magnitude of the stress was limited to that necessary to control the morphology of the crystal on cooling below T_m and was well below that to produce any measurable offset on the values for T_c and T_m. This will be further discussed below.

Biaxial stresses $\sigma_{\langle100\rangle}\gg s$ were applied along the two $\langle100\rangle$ axes perpendicular to l (the third $\langle100\rangle$ direction) (see the top inset to Fig. 1). It was found that a minimum stress of $\sigma^m_{\langle100\rangle}\simeq16$ bars was required to force the *whole* crystal to transform with the c axis along the l. This was only completely achieved for measurements along the C direction and details will be given in the next section. For all three directions of the sample, measurements were made with the uniaxial stress along l and with different levels ($\sigma_{\langle100\rangle}\leq16$ bars) of the biaxial stresses perpendicular to l.

III. RESULTS

Thermal strains $\epsilon=\Delta L/L_0$ (L is the length of the sample, L_0 the length at room temperature), as a function of temperature (T) from three runs with different applied stresses, measured along the C axis, are shown in Fig. 1. Curve (c) in Fig. 1 is the case of a biaxial stress

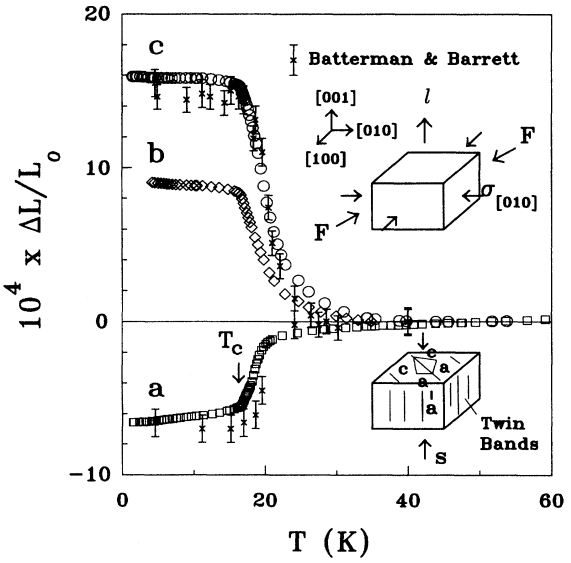

FIG. 1. Thermal strain vs temperature curves for V_3Si measured along [001]. Curve (a) is for an uniaxial stress ($s \simeq 0.03\sigma^m_{\langle 100\rangle}$) along [001]; (b) and (c) are for biaxial stress applied along [100] and [010] with $0.5\sigma^m_{\langle 100\rangle}$ and $\sigma^m_{\langle 100\rangle}$, respectively. The x-ray data of Batterman and Barrett (Ref. 2) are also plotted for comparison. The insets show the directions of applied stresses and [in the case of curve (a)] the martensite-phase domains. See text for the detailed explanation to the insets.

(see the top inset) with $\sigma_{\langle 100\rangle} = \sigma^m_{\langle 100\rangle} \approx 16$ bars perpendicular to l, curve (a) is the case of an uniaxial stress (see the bottom inset) s ($\simeq 0.03\sigma^m_{\langle 100\rangle}$) along l, and curve (b) is the case of a biaxial stress but with $\sigma_{\langle 100\rangle} \approx 0.5\sigma^m_{\langle 100\rangle}$.

The applied stresses (0.5–16 bars) are well below those required to shift T_m and T_c significantly. To suppress the transformation in V_3Si, for example, an uniaxial compressive stress of $s \geq 400$ bars is required along [100].[28] From the experimental values of $(dT_m/d\sigma_{001}) = 2.4 \times 10^{-3}$ K bar^{-1} (Ref. 29) and $(dT_c/d\sigma_{001}) = -5 \times 10^{-4}$ K bar^{-1} (Ref. 30), the possible shifts of T_m and T_c ($\triangle T_m \simeq 0.04$ K, $\triangle T_c \simeq -0.001$ K, for an uniaxial stress of 16 bars along [100]) are below the experimental resolution (0.05 K) in determining the temperature. For the case of biaxial stress, which is more isotropic than an uniaxial stress, the values of the shifts in T_c and T_m are expected to be even smaller because the transformation is less sensitive to a more isotropic stress field.[31] Therefore, it is believed that the stresses applied are only sufficient to manipulate the formation of the martensite domains in the crystal.

Thermal strains for the a and c axes derived from the x-ray measurements of Batterman and Barrett[2] are also included in Fig. 1 for comparison. It is clear that our dilatometry data agree well with the x-ray results. Therefore, it is concluded that a full c- or a-axis orientation was formed along l in the cases (c) and (a), respectively. For a biaxial stress with $\sigma_{\langle 100\rangle} < \sigma^m_{\langle 100\rangle}$, a smaller magnitude of the c-like transformation strain was observed, as illustrated for the typical case (b) in Fig. 1, indicating mixed c- (predominantly) and a-axis orientation along l.

Similar stress dependence of the martensite morphology has been observed for In-Tl.[22] In this case, the level of the biaxial stress required to produce full c-axis behavior is about one-fifth ($\sigma_{\langle 100\rangle} \leq 3$ bars) of that for V_3Si. This could simply be due to the different magnitudes of their elastic stiffnesses, e.g., $c_{11} = 2.87 \times 10^{11}$ N m^{-2} for V_3Si (Ref. 17) and $c_{11} = 0.397 \times 10^{11}$ N m^{-2} for In-27 at. % Tl (Ref. 32) at room temperature.

To check whether the stress-dependent results are reproducible, a series of measurements were made with alternating uniaxial and biaxial stresses being applied, with different levels of the latter. It was observed that the expansion behavior does not depend on the sequence of applying different stresses, and its reproducibility was only related to the type of applied stress.

The *same* a-axis behavior was always obtained when the uniaxial stress was applied. The c-axis behavior was reproducible in all the runs with biaxial stress applied, but with the magnitude of the c-like transformation strain roughly proportional to $\sigma_{\langle 100\rangle}$ up to $\sigma^m_{\langle 100\rangle}$. This demonstrates the reproducibility of the transformation behavior and the control of the martensite domains at these stress levels.

Similar stress-dependent measurements were also carried out for the A and B directions. The observed expansion behavior was the same as that described above, except that the magnitude of the c-like transformation strain at $\sigma^m_{\langle 100\rangle}$ only reached about 2/3 of the full value. This behavior is attributed to an internal stress field in the as-grown crystal which offsets the external stress field and thus a higher value is required to drive the crystal into a single domain. The A and B axes are symmetrical to the crystal growth direction, and therefore it is reasonable to assume that the internal stress field is symmetrical for these two directions. Thus, the stress-dependent expansion behavior for the A and B directions could be expected to be similar, and different from that for the C, as observed. It is expected that with a higher level of the biaxial stress a single-domain behavior would be observed for the A and B axes. Because of concern that excessive stress would introduce significant defects or even damage this brittle crystal, this was not pursued. Nevertheless, the measurements for the C direction have already provided the information on the c-axis expansion behavior that was the objective of this study.

It can be seen in Fig. 1 that the tetragonal distortion starts at a temperature well above T_m and changes rapidly around T_m. This rapid change ceases below T_c, but the tetragonality *continues to increase slowly* with decrease of temperature. This can be seen in Fig. 2 where $(1 - c/a)$, normalized by its $T = 0$ value $[1 - (c/a)_0]$, is plotted as a function of T/T_m. The c/a ratio here was derived using the relation $c/a = [a_0(1 + \epsilon_c)] / [a_0(1 + \epsilon_a)]$ (where a_0 is the lattice parameter of the cubic phase not far above the transformation) using the interpolated values of ϵ_c and ϵ_a from curves (c) and (a) in Fig. 1. The normalized $1 - c/a$ data for In-26.5 at. % Tl are also plotted as the dot-dashed line in Fig. 2 for comparison. X-ray measurements lack the resolution to detect this small change in tetragonality below T_c and only showed approximately a constant c/a below T_c. The derived value

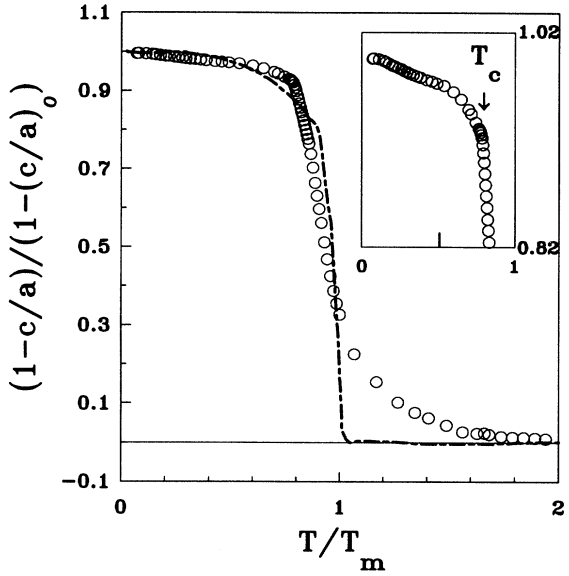

FIG. 2. The temperature dependence of the crystal distortion for V_3Si (○), derived from the data in Fig. 1 [curves (a) and (c)], as discussed in the text. The dot-dashed curve shows the same distortion parameter for In-26.5 at. % Tl. The inset shows in detail the data below T_c.

of $c/a = 1.0022$ for V_3Si at 4.2 K is in excellent agreement with that obtained from x-ray data.[2]

The thermal-expansion coefficients $\alpha(T) = \triangle\epsilon/\triangle T$ derived from curves (c) and (a) in Fig. 1 for the tetragonal c and a axes are shown in Fig. 3. It can be seen from the inset to Fig. 3 that the onset of pretransformation anisotropy starts at about 50 K, 30 K above T_m. On further cooling to T_m, positive and negative peaks in α_a and α_c, respectively, associated with the transformation are observed. There is no evidence of a volume change at T_m.[15]

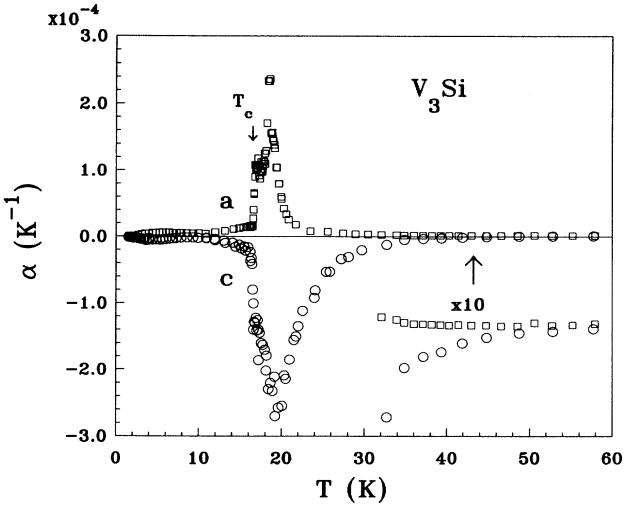

FIG. 3. Linear-thermal expansion coefficients of V_3Si for the a and c axes. The inset shows the onset of the premartensite anisotropy at about 50 K.

At T_c, the discontinuities in α_a and α_c are of opposite sign and have the values $\triangle\alpha_a^{s\text{-}n} = -(9 \pm 1) \times 10^{-5}$ K^{-1} and $\triangle\alpha_c^{s\text{-}n} = (14 \pm 2) \times 10^{-5}$ K^{-1}. The widths of the discontinuities are consistent with $\triangle T_c$ determined by the induction method.

An important new feature in the present data is the broad, anomalous peak centered at about 5 K for each axis, as shown in detail in Fig. 4. This peak is positive for the a and negative for the c axis. Thus when the sample is heated from the lowest temperature, the a axis expands and the c axis contracts, with a corresponding decrease of the tetragonality with *increasing* temperature. This anomalous expansion behavior is similar to that observed for dilute, In-rich, In-Tl alloys[33] and In.[34] The low-temperature thermal-expansion data for In-26.5 at. % Tl are also plotted in Fig. 4 for comparison.

IV. DISCUSSION

The present study provides high-resolution measurements of the bulk a- and c-axis thermal strain for V_3Si over a temperature range spanning the premartensite, anisotropic, thermal-expansion behavior and $T \to 0$ K. This allows a detailed evaluation of the effect of the superconducting transition on the martensitic transformation.

The normalized plot of $(1 - c/a)/[1 - (c/a)_0]$ vs T/T_m for V_3Si in Fig. 2 does show a marked reduction in the growth of the tetragonal distortion for temperatures below T_c ($T/T_m \sim 0.8$). However, when compared with the same plot of for In-26.5 at. % Tl, where the onset of superconductivity is at $T/T_m \sim 0.03$, there is no significant difference in the shapes of the two plots for $T/T_m < 1$.

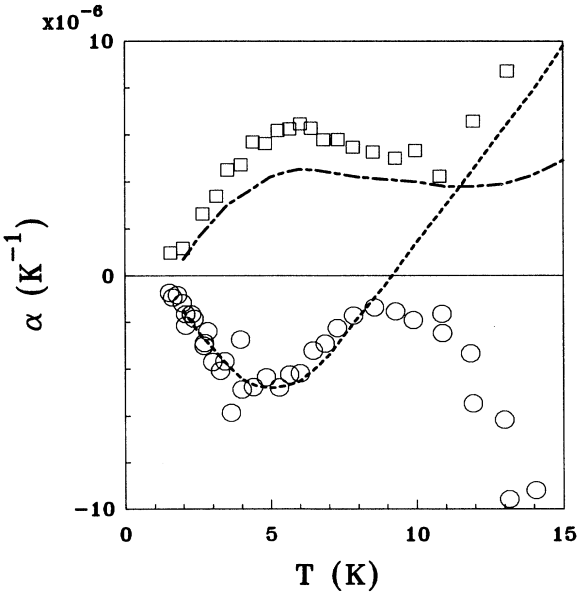

FIG. 4. Linear-thermal-expansion coefficients of V_3Si for the a (□) and c (○) axes in the low-temperature range. The dot-dashed and dashed curves are the a- and c-axis expansions for In-26.5 at. % Tl ($T_m \simeq 105$ K, $T_c \simeq 3$ K) (Ref. 22).

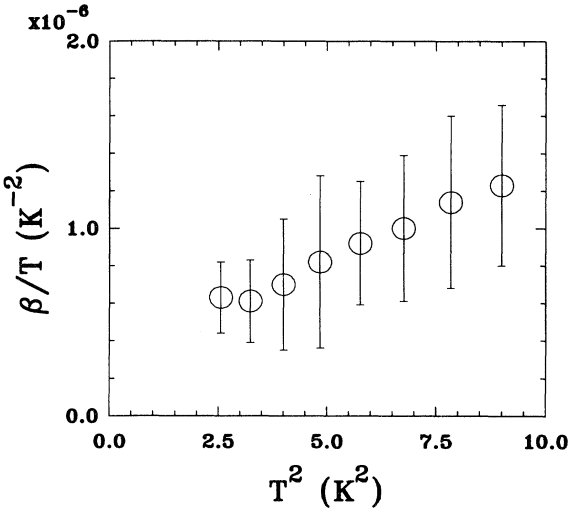

FIG. 5. Volume-expansion coefficient β below 3 K for V_3Si plotted in the form β/T vs T^2.

Thus, it is concluded that the reduction in the growth of tetragonality for $T/T_m < 0.8$ is *not* a consequence of the onset of superconductivity. However, to be absolutely certain it would be necessary to make the measurements with superconductivity suppressed by a magnetic field.

The most significant new feature that is observed is the low-temperature peaks in α_a and α_c at ~ 5 K. While there is evidence for anomalous behavior at these low temperatures in earlier measurements on polycrystalline material,[11] this was only a measure of the partial strain to be expected along the a or c axis. The previous single-crystal studies[10,12,13,15,16] also suffered from only being able to measure mixed c- and a-axis behavior.

The volume expansion coefficient $\beta = 2\alpha_a + \alpha_c$ below 3 K is plotted as β/T vs T^2 in Fig. 5. The data are consistent with a straight line intercept with the origin, i.e., the electronic component $\beta_e \approx 0$. This is in accordance with BCS theory, in which the electronic contribution to the thermal expansion in the superconducting state falls off rapidly with close to an exponential variation with T/T_c.[35] Thus, we associate the expansion behavior for $T < 3$ K with the lattice.

The similarity between the anomalous expansion behaviors of V_3Si and In-Tl below 10 K is striking (Fig. 4). In-Tl alloys in the composition range 15–31 at.% Tl undergo a fcc-fct martensitic transformation that has been attributed to Fermi-surface–Brillouin-zone overlap.[33] Thus the $T = 0$ crystal structure is determined by a balance between the electronic band energy (kinetic energy) and the electrostatic lattice energy (potential energy). On raising the temperature the lattice vibrations introduce thermal disorder energy that tips the delicate balance between the kinetic and potential energies. Thermodynamically the difference in free energy between the cubic and tetragonal states may be written as

$$\triangle F = \triangle U - T\triangle S_{\text{vib}} , \quad (1)$$

where $\triangle U$ is the difference between the $T = 0$ kinetic and potential energies, and $\triangle S_{\text{vib}}$ is the change in entropy associated with the lattice vibrations.[36] Recent measurements of $\triangle S_{\text{vib}}$ (Ref. 36) associated with order-disorder transitions indicate that it is large enough to have a significant effect on the relative thermodynamic stability of the ordered and disordered states in Ni_3Al and Fe_3Al.

It is proposed that this model also applies to V_3Si.

As the temperature is raised from $T = 0$ the presence of an energy gap at the Fermi surface due to superconductivity will have a strong influence on the electronic excitations at the Fermi surface and thus on the electronic contribution to the thermal expansion. However, such an energy gap will not prevent displacements in the atomic positions due to thermal excitation of the lattice. In the case of V_3Si, the soft-mode behavior is indicative of a highly anisotropic lattice potential,[1] resulting in a high degree of thermal disorder close to T_m.

V. CONCLUSION

The thermal strains for the tetragonal a and c axes of V_3Si have been measured by using the external stress field to control the morphology of the martensite domains. These measurements show no indication of a volume change at the transformation temperature T_m or evidence for the arrest of the transformation with the onset of superconductivity.

It is concluded that the tetragonal distortion from the cubic structure is electronically driven, but it is the thermal excitation of lattice that is responsible for the structural phase transition.

ACKNOWLEDGMENTS

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