

**SUPERCONDUCTIVITY, INCLUDING HIGH-TEMPERATURE SUPERCONDUCTIVITY****Influence of internal stresses on the superconductivity of nanocrystalline vanadium films**

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Nanocrystalline vanadium films 7–20 nm thick are obtained by crystallization of amorphous condensates of this metal by heating to a temperature  $T < 60$  K. Immediately after completion of the crystallization the critical temperature of the superconducting transition  $T_c$  of these films is 3.1–4.3 K. When the films are heated to room temperature in an ultrahigh vacuum the values of  $T_c$  decrease by  $\approx 0.4$  K. It is shown that this decrease is due, in particular, to relief of the tensile stresses that arise in the films during crystallization. © 2005 American Institute of Physics. [DOI: 10.1063/1.1820544]

**INTRODUCTION**

It is known that when vanadium vapor is condensed on a substrate cooled by liquid helium the films formed up to thicknesses  $d \approx 20$ –40 nm have an amorphous structure.<sup>1–3</sup> When this critical thickness  $d_c$  is exceeded during condensation the whole volume of the film undergoes spontaneous explosive (avalanche) crystallization with the formation of the usual bcc phase. If the layer thickness is less than  $d_c$ , its amorphous structure is stable up to a temperature  $T_{a \rightarrow c} \approx 40$ –60 K, and the value of  $T_{a \rightarrow c}$  increases with decreasing  $d$  by an approximately hyperbolic law.<sup>2,4</sup> We have previously investigated the superconducting properties of the amorphous phase of vanadium.<sup>2</sup> The critical temperature of the superconducting transition (CTST) of the crystallized film is approximately 1.5 K higher than the value of  $T_c$  in the amorphous state. Further heating of the samples from  $T_{a \rightarrow c}$  to room temperature is usually accompanied by a lowering of the CTST.<sup>3</sup> This behavior has not yet been explained correctly in the published literature. The goal of the present study is to attempt an explanation.

**TECHNIQUE**

The techniques used to grow the films and to study their electronic properties are described in detail elsewhere.<sup>5,6</sup> We used welded-up glass ampoules with a flat polished substrate and welded-in leads of platinum wire for making electrical measurements of the metallic film condensed on the substrate. After being pumped down to a pressure  $p \sim 10^{-4}$  Pa the ampoules were hermetically sealed with a gas torch and removed from the vacuum apparatus. The ampoules were mounted in a helium cryostat which was then flooded with liquid helium, creating an ultrahigh vacuum ( $\sim 10^{-10}$  Pa) with respect to all components of air. The bulk vanadium used had a resistivity ratio of  $\approx 550$  between temperatures of 300 and 6 K.

The vanadium was evaporated by evaporators made of cleaned tungsten wire. To degas the mounting of vanadium and tungsten a significant part of the mounting was evaporated off while the substrate was shielded; the substrate remained in continuous contact with the liquid helium, and the average temperature of the vanadium layer during condensation was not over 18 K.

The electrical conductivity of the films was measured by a compensation method using a four-probe scheme with an accuracy of 0.01% or better. The critical magnetic field perpendicular to the plane of the film was produced by a superconducting solenoid. The thickness of the films during condensation was monitored by its conductance, and after the experiment was completed and the ampoule opened it was measured by an optical density method.<sup>7</sup>

The microstructure of the crystallized vanadium films and their surface topography were investigated at room temperature on a JEM-100CX analytical electron microscope.<sup>8</sup> The microstructure was investigated by photography in transmitted light. Both bright-field and dark-field images were obtained. The vanadium films were removed from the substrate by dissolving in water a thin ( $\sim 20$ –30 nm) NaCl layer that had been condensed on the glass substrate prior to deposition of the metal.

**EXPERIMENTAL RESULTS**

Figure 1 shows the change of the resistivity of a newly condensed vanadium film  $\approx 16$  nm thick upon heating to room temperature. The practically vertical segment of the curve corresponds to the transition of the film from the amorphous to the crystalline state ( $a \rightarrow c$  transition). Interesting, immediately after the  $a \rightarrow c$  transition ( $T_{a \rightarrow c} \approx 40$  K) and on up to room temperature the  $\rho(T)$  curve becomes practically reversible, i.e., the residual resistivity  $\rho_0$  at  $T = 6$  K is practically equal to its value  $\rho'_0$  for the same film after heating to room temperature. If the critical temperature was exceeded during the condensation process and explosive crystallization

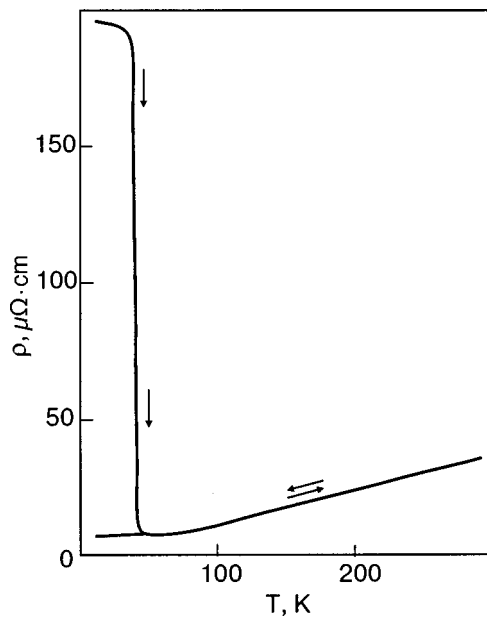


FIG. 1. Temperature dependence of the resistivity of a vanadium film of thickness  $\approx 16$  nm.

occurred, the  $\rho(T)$  curve is reversible from the start and is described by the lower part of the curve in Fig. 1 (without the vertical segment). Such behavior of  $\rho(T)$  is observed only for vanadium films. For all the other low-temperature condensates of pure metals (In, Sn, Al, Tl, etc.) known to us, the residual resistivity continually decreases as the film is annealed up to room temperature, owing to the annealing of defects and the growth of grains. For example, when an aluminum film ( $d = 70$  nm) is heated from helium to room temperature,  $T_c$  decreases from 3.25 to 1.45 K and the residual resistivity decreases from 16 to  $1.25 \mu\Omega \cdot \text{cm}$ .<sup>4</sup> Apparently in vanadium films the lattice defects that have a substantial influence on the resistivity are not annealed in the temperature interval  $T = 40\text{--}300$  K.

The critical temperature of the superconducting transition for the crystallized vanadium films directly after their heating to  $T = T_{a \rightarrow c}$ , denoted  $T_c$ , is always substantially higher than the value  $T'_c$  for the same films after annealing to room temperature. The value of the temperature  $T_c$  remains practically constant after the films have been annealed to  $T = 190\text{--}200$  K. Only after further increase in the annealing temperature does the CTST decrease from  $T_c$  to  $T'_c$ . If the film has crystallized as a result of explosive crystallization (at  $d = d_c$ ) the values of  $T_c$  and  $T'_c$  are equal.

Table I gives the values of  $T_c$  and  $T'_c$  for typical low-temperature condensates of vanadium. These values are lower than the CTST for for pure bulk vanadium. The mechanisms causing the decrease of the CTST with decreasing thickness of metallic films are discussed in detail in Ref. 4.

Some films show signs of cracking (a slight growth of the residual resistivity) already at room temperature (see Table I).

Sometimes a rather long hold at room temperature leads to a slight decrease in the residual resistivity (see the result for film  $V_5$ ). After heating to  $T = 330\text{--}340$  K all of the films

TABLE I. Some characteristics of the crystalline vanadium films investigated.

Sample	$d$ , nm	$T_c$ , K	$T'_c$ , K	$\frac{\rho'_0 - \rho_0}{\rho_0}$ , %	$\frac{\rho_{300}}{\rho'_0}$
$V_1$	7	3.10	2.7	0	2.2
$V_2$	8	3.35	2.98	5	3.5
$V_3$	15	3.90	3.52	2.5	3.7
$V_4$	16	4.20	3.78	0	4.76
$V_5$	22 ( $d_c$ )	4.30	4.30	-5.4	3.15

Note: The temperature smearing of the superconducting transitions for films  $V_1\text{--}V_5$  is 0.10–0.12 K [the interval  $(0.1\text{--}0.9)\rho_0$ ], and  $\rho_{300}$  is the value of  $\rho$  at 300 K.

exhibited a noticeable increase in residual resistivity which is due, as we shall show below, to an intensification of the cracking process. It should be emphasized that the films  $V_1\text{--}V_5$  were annealed to room temperature in ultrahigh vacuum ( $\sim 10^{-10}$  Pa). For this the upper part of the ampoule containing the substrate was heated by a furnace under a hood in the form an inverted Dewar while the lower part of the ampoule was immersed in liquid helium. This precludes the possibility of contamination of the annealed film by residual gases, the pressure of which without these precautionary measures would reach  $\sim 10^{-4}$  Pa at the higher temperatures (see Technique). In this case the decrease of the CTST on heating could be ascribed to impurities falling into the film from the surrounding medium, as it is known that vanadium is not attacked by air at ordinary temperatures.

Figure 2 shows the temperature dependence of the critical magnetic induction ( $B_{c2} \equiv B_{c\perp}$ ; Ref. 9) for the film  $V_4$ . Curve 1 was taken after heating to  $T = T_{a \rightarrow c}$  and corresponds to a value  $T_c = 4.2$  K; curve 2 was taken after heating to room temperature and corresponds to a value  $T'_c = 3.78$  K. Near the CTST the value of  $B_{c2}$  varies linearly with temperature, while at lower temperatures the dependence is approximately quadratic:

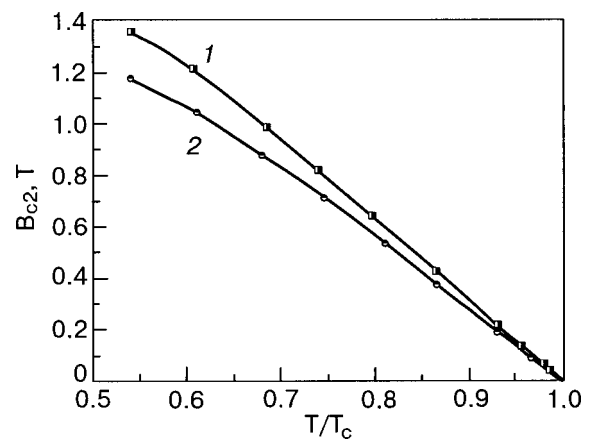


FIG. 2. Temperature dependence of the critical magnetic induction for a crystalline vanadium film  $\approx 16$  nm in diameter after annealing to  $T$  [K]: 50 (1), 300 (2).

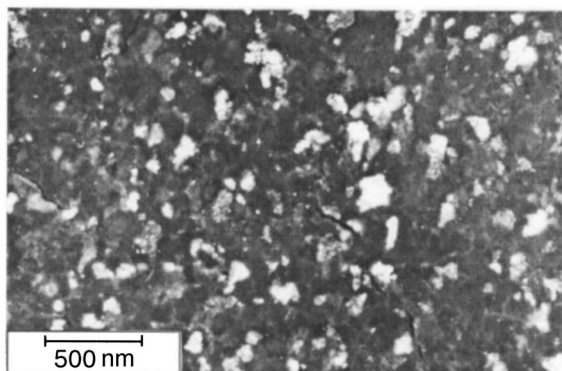


FIG. 3. Dark-field electron micrograph of the nanostructure of a vanadium film  $\approx 37$  nm thick.

$$B_{c2} = B_{c0} \left[ 1 - \left( \frac{T}{T_c} \right)^2 \right], \quad (1)$$

where  $B_{c0}$  is the value of  $B_{c2}$  at  $T=0$  K.

Electron diffraction patterns of the films show only the lines of the bcc phase of vanadium. The grain size in the films that have undergone explosive crystallization is practically the same as in films of the same thickness crystallized by heating. The mean grain size in both cases is 54–55 nm (Fig. 3). The grains have a flat shape and, judging from the Moiré patterns on some electron micrographs, they are often stacked on one another. Thus the mean grain size in the direction perpendicular to the plane of the film is less than its thickness. The electron mean free path  $l$  in the vanadium films studied here, calculated from the relation  $\rho l = 3.5 \times 10^{-12} \Omega \cdot \text{cm}^2$  (Ref. 10), amounts to 2–3 nm, a value attesting to a significant degree of intragrain disorder.

It is important to note that films condensed on a sublayer of NaCl had numerous cracks up to 700 nm long and 30 nm wide (Fig. 4). The cracks form at the time when the ampoule is opened to atmospheric pressure, as is manifested by the sharp increase in the resistivity of the films at the time of opening. This indicates that the films prior to the opening of the ampoule were in a maximally stressed state. Prior to the opening of the ampoule the films condensed on a NaCl sublayer, like films condensed on glass, displayed a reversible trend of  $\rho(T)$  from the point of the  $a \rightarrow c$  transition on up to room temperature. For the the films condensed on glass the resistivity changed insignificantly when the ampoule was

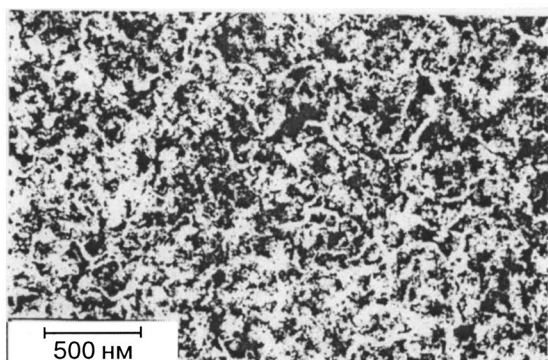


FIG. 4. Bright-field electron micrograph of the nanostructure of a vanadium film  $\approx 37$  nm thick.

TABLE II. Parameters for the calculation and the calculated values of  $N(0)$  of the film  $V_4$  after annealing to  $T=50$  and 300 K.

Heating temperature, K	$ dB_{c2}/dT _{T_c}$ , T/K	$\rho_0$ , $\mu\Omega \cdot \text{cm}$	$N(0)$ , $10^{47} \text{ J}^{-1} \cdot \text{m}^{-3}$
50	0.754	11.5	23.28
300	0.741	11.5	22.88

opened; from that we infer that crack formation is practically absent. The absence of cracks in those films is confirmed by the studies of their surface topography.<sup>8</sup> It was found that the surface of the vanadium films on glass did not exhibit noticeable relief after heating to room temperature, i.e., the micro-irregularities of the surface did not exceed 3–5 nm and there were no noticeable cracks. Cracks appeared only after the films were heated to  $\approx 340$  K.

### DISCUSSION OF THE RESULTS

It was shown in Refs. 10 and 11 that at the lowest temperatures (below 0.5 K) the heat capacity of vanadium is determined by the normal BCS energy gap ( $2\Delta_0 = 3.5k_B T_c$ ). Approximately the same value of  $2\Delta_0$  has been obtained in experiments in which the energy gap is measured by the methods of absorption of electromagnetic radiation, absorption of ultrasound, and in tunneling experiments (see Ref. 12 and the references cited in Refs. 10 and 12). For superconductors with strong coupling one has  $2\Delta_0 = (4.3-4.5)k_B T_c$ .<sup>11,13,14</sup> Sophisticated tunneling experiments<sup>12</sup> have given a value  $\lambda = 0.82$  for the electron-phonon coupling constant of vanadium; this value is considered too high by the authors. For superconductors with strong coupling the values of  $\lambda$  found experimentally equal 1.7–2.8.<sup>14</sup> Thus vanadium must be treated as a weakly coupled superconductor.<sup>10</sup> This conclusion is somewhat at odds with a theoretical study<sup>11</sup> that gave a value  $\lambda = 1.19$  for vanadium, but even in that case the coupling would be more aptly described as intermediate rather than strong.

For the films studied  $l \ll \xi_0$ , where  $\xi_0$  is the coherence length for pure “bulk” vanadium ( $\xi_0 \approx 45$  nm)<sup>10</sup> In that case it follows from the microscopic theory developed by Gor'kov for weakly coupled superconductors in magnetic field<sup>15</sup> that the electron density of states at the Fermi level (with allowance for the two spin directions) is given by the expression<sup>16</sup>

$$N(0) = \frac{\pi |dB_{c2}/dT|_{T_c}}{4k_B e \rho}, \quad (2)$$

where  $e$  is the charge of the electron and  $k_B$  is Boltzmann's constant.

For superconductors with intermediate and strong coupling a coefficient of the order of unity should be introduced in the denominator on the right-hand side of formula (2).<sup>17,18</sup>

Let us estimate the density of states for film  $V_4$  after heating to  $T=50$  and 300 K (see Table II) on the basis of formula (2) with the values of  $|dB_{c2}/dT|_{T_c}$  (Fig. 2) and  $\rho_0$ .

Qualitatively similar behavior of  $|dB_{c2}/dT|_{T_c}$  and  $N(0)$  as functions of the heating temperature are observed for films  $V_1$ – $V_3$  as well. In the case of the film  $V_5$  the values of  $|dB_{c2}/dT|_{T_c}$  and  $N(0)$  immediately after explosive crystallization and after heating to room temperature were the same within the experimental error.

The BCS theory for weakly coupled superconductors gives the value

$$T_c = 1.14\theta_D \exp\left(-\frac{1}{N(0)V^*}\right), \quad (3)$$

where  $V^*$  is the electron interaction parameter and  $\theta_D$  is the Debye temperature.

According to Eq. (3), in order for  $T_c$  to change from 4.2 to 3.78 (as in the case of film  $V_4$ ), the value of  $N(0)V^*$  should decrease by a factor of 1.023. A formula for  $T_c$  in the case of superconductors with strong or intermediate coupling was derived by McMillan<sup>19</sup> and later modified to<sup>20</sup>

$$T_c = \frac{\omega_{\log}}{1.2} \exp\left[-\frac{1.04(1+\lambda)}{\lambda - \mu^*(1+0.62\lambda)}\right]. \quad (4)$$

Here  $\omega_{\log}$  is the logarithmic mean phonon frequency,  $\mu^*$  is the Coulomb pseudopotential of Morel and Anderson, characterizing the electron-electron interaction, and  $\lambda$  is the electron-phonon coupling constant:

$$\lambda = \frac{N(0)\langle I^2 \rangle}{M\langle \omega^2 \rangle}, \quad (5)$$

where  $\langle I^2 \rangle$  is the square matrix element of the electron-phonon interaction, averaged over the Fermi surface,  $\langle \omega^2 \rangle$  is the mean square phonon frequency, and  $M$  is the mass of an atom.

According to Eq. (4), in order for  $T_c$  for film  $V_4$  to change from 4.2 to 3.78 K the value of  $\lambda$  [at  $\mu^*=0.15$  (Ref. 12) and  $\omega_{\log}=245$  K (Ref. 11)] should decrease from 0.654 to 0.635.<sup>1</sup> This decrease of  $\lambda$  by a factor of 1.030 agrees with the decrease of  $N(0)V^*$  estimated with the use of the BCS formula (3).

As we see from Table II, the value of  $N(0)$  decreases by a factor of 1.018 when the film is heated from 50 to 300 K. Thus the change of  $T_c$  for film  $V_4$  from 4.2 to 3.78 K can be attributed predominantly to the change in the density of states upon a slight change of the parameter  $V^*$  in formula (3) or of the parameters  $\langle I^2 \rangle$  and  $\langle \omega^2 \rangle$  appearing in Eq. (5). This conclusion pertains to all low-temperature crystalline vanadium films with thicknesses less than  $d_c$ . As is shown below, the most obvious reason for the behavior described is internal stresses in nanocrystalline vanadium films.

In concluding this analysis we should mention the domain of applicability of formula (4) without corrections for spin fluctuations. Contrary to some previous papers, in Ref. 12 a weak influence of spin fluctuations on the superconductivity of vanadium was mentioned, although it is not ruled out at the level  $\lambda_s \sim 0.1$ . The theoretical paper of Ref. 11 denies any dependence of the parameter  $\lambda$  on the spin fluctuations in the case of vanadium and many other superconductors except palladium. Therefore, for qualitative analysis

of our results above we have used the modified McMillan formula without renormalization of the parameters  $\lambda$  and  $\mu^*$ .

The reversibility of  $\rho(T)$  in the temperature interval from 6 to 300 K in vanadium films immediately after the  $a \rightarrow c$  transition attests to the fact that the number and size of the grains (and, hence, the area of the grain boundaries, which gives the main contribution to the resistivity of nanocrystalline films) remains practically unchanged during annealing of the films after the  $a \rightarrow c$  transition. Meanwhile, the internal stresses, which have little effect on the resistivity, can change substantially.

Indeed, internal stresses are absent in amorphous metallic films.<sup>21</sup> The density of vanadium films increases upon crystallization, and because of the coupling of the film with the substrate, that gives rise to tensile stresses. The tensile stresses should increase when the film is heated, since the mean values of the thermal expansion coefficients  $\bar{\alpha}$  of vanadium are much smaller than those of the soft chemical-laboratory glass S89-2 (No. 23) used for the body of the ampoule and the substrate.

At room temperature one has  $\bar{\alpha} = (8.8-9) \times 10^{-6} \text{ K}^{-1}$  for glass No. 23 (Ref. 22) and  $\bar{\alpha} = 7.75 \times 10^{-6} \text{ K}^{-1}$  for vanadium.<sup>23</sup> For NaCl the  $\bar{\alpha}(T)$  curve lies considerably higher than for glass or vanadium and at 300 K it has a value of around  $39 \times 10^{-6} \text{ K}^{-1}$  (Ref. 23). Therefore, the vanadium films condensed on a NaCl sublayer are subjected to much higher tensile stresses on heating than are the films condensed on glass.

It should be kept in mind that from 73 to 273 K the ultimate tensile strength of vanadium decreases from  $\approx 980$  MPa to  $\approx 490$  MPa, while the relative contraction increases from  $\approx 64\%$  to  $\approx 80\%$  (Ref. 24) owing to the cold-shortness effect. Vanadium films could be expected to have qualitatively similar behavior. It can be assumed that the tensile stresses arising as a result of the  $a \rightarrow c$  transition cause elastic straining of the film because of the high values of the ultimate strength and yield stress at  $T \approx T_{a \rightarrow c}$ . It appears that it is these stresses and the elastic strains they produce that are responsible for the increased values of the density of states and CTST of low-temperature condensates of vanadium immediately after their crystallization (see the  $T_c$  values in Table I). As the heating temperature is increased to  $\approx 200$  K this behavior continues in spite of the growth of the tensile stresses in the film due to the higher thermal expansion of the glass as compared to vanadium. Therefore on the return to helium temperature the elastic strains in the film are approximately the same as immediately after the  $a \rightarrow c$  transition, and so is the CTST.

When the temperature is increased from  $\approx 200$  to  $\approx 300$  K the tensile stresses apparently exceed the lowered yield point of the vanadium film, and plastic deformation of the latter occurs, which is often manifested in fractionation of the blocks inside the grains.<sup>24</sup> During plastic deformation the internal stresses in the film are relieved, lowering the density of states and the CTST (see the values of  $T'_c$  in Table I). Heating in the temperature interval 200–300 K can also lead to annealing of some lattice defects that have an appreciable effect on the CTST but not much effect on the resistivity of the film. The values of  $T'_c$  in Table I agree with the

values of the CTST for vanadium films of the same thickness obtained in ultrahigh vacuum ( $p \sim 10^{-6}$  Pa) on substrates above room temperature.<sup>25,26</sup> A situation analogous to that described above for vanadium films has been observed for "bulk" vanadium.<sup>27</sup> Specifically, the elastic internal tensile strains arising as a result of the plastic deformation of "bulk" vanadium at  $T \approx 4.2$  K were considered by the authors to be responsible for the increase in the CTST of the samples by as much as 0.5 K. The relief of the internal stresses on heating the samples to room temperature lowered the value of  $T_c$  to nearly the initial value. A qualitatively similar result on "bulk" vanadium was also obtained in Ref. 28. The lattice defects that determine the resistivity do not depend much on the internal stresses; this accounts for the lack of a clear connection between  $T_c$  and  $\rho$  in the films studied here and in "bulk" vanadium samples.<sup>27</sup>

Cracking of the vanadium films on a glass substrate, as we have said, starts at temperatures close to room temperature, while for films on a rock salt sublayer the films crack when the ampoule is opened at room temperature. The cracking process is evidence that the internal tensile stresses in the films have exceeded the ultimate strength. Consequently, we were correct in assuming that at lower temperatures they exceeded the yield stress and plastic deformation of the films occurred.

At first glance it seems surprising that for vanadium films that have undergone explosive crystallization during condensation the values of the CTST immediately after the  $a \rightarrow c$  transition and after the films had been annealed up to room temperature are the same (see  $V_5$  in Table I). Here one should remember the nature and the features of the explosive crystallization mechanism.<sup>5,6</sup> In amorphous vanadium films explosive crystallization occurs spontaneously when the film reaches a critical thickness. Under conditions of intensive evolution of the latent heat of the transition (and poor heat removal from the sample) a self-acceleration of the crystallization occurs, and the rate of the transformation front reaches tens of meters per second. This process is close to adiabatic, and the temperature of the explosive crystallization front in the vanadium film is estimated as 340 K.<sup>5,6</sup> The massive glass substrate remains cold during this process owing to the small mass of the film and the high velocity of the transformation front ( $\approx 50$  m/s for vanadium). It appears that in this case the internal stresses due to the difference of the densities of the amorphous and crystalline phases of vanadium are relieved during the explosive crystallization process. This leads to stabilization of the CTST of the film during its subsequent heating to room temperature.

## CONCLUSION

Tensile stresses appear during crystallization of amorphous thin films of vanadium because of the higher density of the crystalline phase and the adhesion of the film to the substrate, and these lead to elastic straining of the films. This changes the distance between atoms in the crystal lattice of the metal. Apparently in the case of bcc films of vanadium this causes a certain change of the band structure, manifested in a slight increase in the density of states. This increase is the main cause of the increase of  $T_c$  in the elastically strained vanadium films in comparison with the annealed state of the

same films. The internal stresses, which have an appreciable influence on  $B_{c2}$  and  $T_c$ , clearly have little effect on the resistivity. It may be that the change of  $T_c$  is influenced by some lattice defects that are annealed when the films are brought to room temperature but which affect the resistivity only weakly.

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<sup>1</sup>For bulk vanadium with  $T_c = 5.4$  K the analogous estimate gives  $\lambda = 0.702$ .

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