

Critical magnetic fields, electric resistance, and density of electronic states in superconducting crystalline and amorphous technetium-carbon films

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The superconducting transition temperatures T_c , perpendicular critical magnetic fields H_{cl} , and resistivities are measured in technetium-carbon films for three metastable states: for a supersaturated hcp solid solution of carbon in technetium, for the amorphous phase, and for the cubic phase. The density of the electronic states is determined on the basis of data on $|dH_{cl}/dT|$ and the residual resistivity ρ_0 . The properties of the films in the crystalline and amorphous phases are compared. The correlation between the variation of T_c and N on going from one phase to another, or when the composition is varied within a given phase, is investigated. It is concluded that the amorphous and cubic phases are characterized by a stronger electron-phonon interaction than the hcp phase.

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There is at present no complete understanding of the relative role of the changes of the electron and phonon spectra and of the electron-phonon interaction when the superconducting properties of transition metals and alloys on their base are altered by various interactions (e.g., introduction of impurities, structural transformations, and others). By analyzing data on transition metals and their alloys, McMillan¹ reached the conclusion that the decisive influence on the temperature T_c of the superconducting transition is exerted by changes of the phonon spectrum. Later Varma and Dynes² and Gomersall and Gyorffy³ advanced arguments that the crucial role is assumed by changes of the electronic states.

We investigate in the present paper the superconducting-transition temperature, the critical magnetic fields, and the resistivity of superconducting films of a two-component system based on a transition metal, viz., the technetium-carbon system. Principal attention is paid to determination of the correlation between the superconducting transition temperature T_c and the density of the electronic states N when the carbon concentration is increased, as well as when phase transitions occur in this system, particularly the transition from the crystalline to the amorphous state. It should be noted that traditional methods of measuring N cannot be used for thin films because of the small mass of the matter; N was therefore determined from data on the critical magnetic fields and on the resistivity.^{4–6}

The use of films as the object of such investigations offers certain advantages connected with the possibility of obtaining and stabilizing phases not observed in the bulk. In addition, in transition-metal films it is possible to extend considerably the solubility limit of the nonmetallic component, so that the variation of T_c and N can be tracked over a wide range when the concentration of the dissolved component is increased until the sample becomes amorphous.

The Tc-C films were prepared by ion sputtering in ultrahigh vacuum apparatus^{5–7} with layer-by-layer deposition of alternating monatomic layers of technetium and of the fraction of monatomic carbon layer corresponding to the

specified concentration. Investigations of the superconducting-transition temperatures together with electron diffraction and electron-microscopy studies of these films have shown that several phases not observed in bulky samples can be observed in this system, depending on the film thickness, carbon concentration, and heat treatment conditions. Thus, non-annealed films ~ 500 Å thick with concentrations $x = 6.5$ and 12 at. % carbon are deposited as a supersaturated hcp phase of a solid solution of carbon in technetium (the solubility limit in the bulk is much lower than these concentrations, merely 1 at. % C at 900 °C, Ref. 9). At higher carbon concentrations (22, 25, 29, 32, 42, and 55 at. %) the films are deposited as an amorphous (or quasi-amorphous) phase with crystallite size ~ 10 Å. Whereas annealing at $x < 12$ at. % C does not change the structure, annealing of samples with $22 < x < 32$ at. % C at 400–500 °C produces a phase with cubic structure and with a lattice parameter $a = 6.66$ Å, while at a higher annealing temperature the samples go over into the hcp phase of the supersaturated solid solution. It must be noted that a feature of the cubic Tc-C phase is high T_c (up to ≈ 14 K).

To investigate the correlations between T_c and N in the Tc-C system we chose several film samples representing the following phases: 1) hcp phase of supersaturated solid solution of carbon in technetium, 2) cubic phase, and 3) amorphous phase.

The film thickness ranged from 400 to 550 Å. The thickness was calculated from the rates of deposition of the technetium and carbon and from the time of exposure of each layer. The thickness of certain non-annealed films (with concentrations 12.5, 32, 42, and 55 at. % C) were measured directly with a profile recorder. The values obtained in this manner agreed with the calculated ones within 5%.

The perpendicular critical magnetic fields were measured by a resistive method using clamp contacts. The sample was so mounted that the film plane was perpendicular to the axis of the superconducting solenoid. The maximum solenoid field was 80 kOe.

The critical field were determined from the supercon-

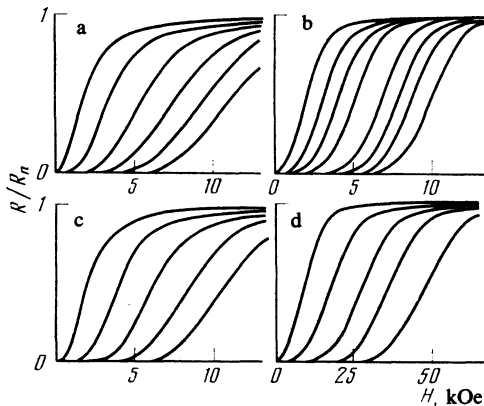


FIG. 1. Superconducting transition $R(H)$ curves: a—nonannealed Tc + 29 at. % C (amorphous) film at temperatures (in order of increasing magnetic field) 7.61, 7.52, 7.39, 7.27, 7.18, and 7.08 K; b—for film of the same composition annealed at 600°C (hcp solid solution) at temperatures 2.90, 2.79, 2.69, 2.58, 2.40, 2.30, 2.21, and 2.10 K; c—for film of the same composition annealed at 500°C (cubic phase at temperatures 13.44, 13.33, 13.25, 13.14, and 13.04 K; d—for the same sample at temperatures 13.14, 12.72, 12.26, 11.94, and 11.38 K.

ducting-transition $R(H)$ curves at constant temperature. The transition curves for the investigated samples were smooth and had no "steps." This agrees with the electron-diffraction-based conclusion that the chosen samples were single-phase. Typical transition curves for films in the hcp and cubic phases as well as for amorphous films are shown in Fig. 1. The widths of the transition curve increase somewhat with rising temperature.

H_{c1} was taken to be the field at which, at a given temperature, the sample resistance R amounted to half the normal-state resistance R_n . Plots of $H_{c1}(T)$ for the investigated samples are shown in Fig. 2. The values of H_{c1} near T_c vary linearly with temperature, at least within one degree Kelvin, as functions of T_c . From the slope of the straight line $H_{c1}(T)$ near T_c we determined the derivative $|dH_{c1}/dT|_{T_c}$.

The density N of the electronic states was determined in the following manner. In the case of a small electron mean free path l ($l < \xi_0$, where $\xi_0 = 0.18\hbar v_F/kT_c$ is the coherence length and v_F is the electron Fermi velocity) it follows from Gor'kov's theory⁹ that

$$|dH_{c2}/dT|_{T_c} = 4kc/\pi eD, \quad (1)$$

where

$$D = lv_F/3 \quad (2)$$

is the electron diffusion coefficient. According to Einstein's equation for the electric conductivity

$$1/\rho_0 = 2e^2DN. \quad (3)$$

The residual resistivity ρ_0 of our samples can be replaced with high accuracy by the resistivity ρ_n measured directly after the transition from the superconducting to the normal state. For type-II superconductor films,¹¹ $H_{c2} = H_{c1}$ (to the perpendicular critical magnetic field). With allowance for these remarks, it follows from (1) and (3) that

$$N = |dH_{c1}/dT|_{T_c} / 2.55kcp_0. \quad (4)$$

That this method of determining N can be used for our sample is indicated by the fact that the values $N = 1.00$ – 1.07

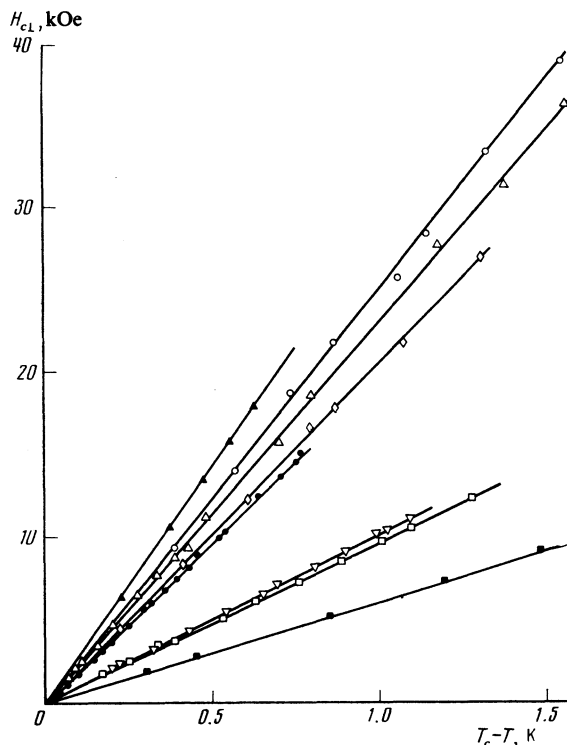


FIG. 2. Dependence of perpendicular critical magnetic field on $T_c - T$. Amorphous films: \diamond —25 at. % C, \bullet —29 at. % C, \blacktriangle —55 at. % C; cubic phase: \circ —25 at. % C, \triangle —29 at. % C; hcp solid solution: \blacksquare —12 at. % C, \square —22 at. % C, ∇ —29 at. % C.

states/eV · at. obtained with its aid⁵ for technetium films 320–1600 Å thick are close to the values determined for bulky samples by measuring the heat capacity ($N = 0.91$),¹⁰ the thermodynamic critical magnetic field ($N = 1.03$),¹¹ and H_{c2} ($N = 1.19$ states/eV · at.).¹²

The values of $|dH_{c1}/dT|_{T_c}$, ρ_0 , N and some other quantities that characterize the investigated films are listed in Table I.

We consider first the properties of the cubic phase. Interestingly, in the cubic phase large values of $|dH_{c1}/dT|_{T_c} \approx 22$ – 25 kOe/K exist together with high $T_k \approx 13.5$ K. These values lead to the estimate

$$H_{c\perp}(0) = 0.69T_c |dH_{c1}/dT|_{T_c} \approx 210$$
– 240 kOe.

The density of electronic states, estimated from 0 and $|dH_{c1}/dT|_{T_c}$, for this phase, also has high values (1.20–1.30 states/eV · at.), exceeding the values for other Tc-C phases (Fig. 3). It appears that the high values of the density of the electronic states are some of the cases of the high T_k of the cubic phase.

The electron-diffusion coefficients determined from H_{c1} are smaller in the films of this phase than in amorphous films having the same concentration, and considerably smaller than in hcp-phase films of the same concentration, although the resistance ratio R_{300}/R_n is larger for the cubic-phase films. This seems to indicate that the low values of the diffusion coefficients in the cubic phase are due not to the smallness of the electron mean free path, but to the small values of the Fermi velocity.

TABLE I.

| Phase | x, at. % | Heat treatment | $d, \text{\AA} \pm 6\%$ | $T_k, \text{K} \pm 0.02\text{K}$ | $\Delta T_k, \text{K}^*$ | $R_{300}/R_n \pm 2\%$ | $\rho_0, \mu\Omega \cdot \text{cm} \pm 7\%$ | $\rho_{300}, \mu\Omega \cdot \text{cm} \pm 7\%$ | $ dH_{cl}/dT _{T_c}, \text{mOe} \cdot \text{K} \pm 6\%$ | $N, \text{states} \pm 8\% / \text{eV} \cdot \text{at}$ | $D_{cm} / \text{sec} \pm 4\%$ |
|---|----------|--------------------|-------------------------|----------------------------------|--------------------------|-----------------------|---|---|---|--|-------------------------------|
| Amorphous | 25 | non-annealed | 420 | 7.77 | 0.15 | 0.98 | 139 | 135 | 20.1 | 0.50 | 0.547 |
| | 29 | non-annealed | 430 | 7.71 | 0.10 | 0.97 | 128 | 124 | 18.5 | 0.48 | 0.594 |
| | 55 | non-annealed | 550 | 5.15 | 0.11 | 0.95 | 175 | 166 | 27.9 | 0.43 | 0.394 |
| Cubic | 25 | annealed at 500 °C | 420 | 13.40 | 0.11 | 1.38 | 65 | 89 | 24.5 | 1.30 | 0.449 |
| | 29 | annealed at 500 °C | 430 | 13.53 | 0.25 | 1.37 | 63 | 86 | 22.5 | 1.20 | 0.489 |
| | 12 | annealed at 600 °C | 400 | 6.68 | 0.17 | 1.44 | 31 | 45 | 6.03 | 0.73 | 1.82 |
| hcp (supersaturated solid solution of carbon in technetium) | 22 | annealed at 600 °C | 410 | 3.07 | 0.76 | 1.10 | 75 | 82 | 9.53 | 0.45 | 1.15 |
| | 29 | annealed at 600 °C | 430 | 3.11 | 0.24 | 1.18 | 74 | 87 | 9.95 | 0.46 | 1.11 |
| | — | non-annealed | 640 | 7.52 | 0.03 | 3.15 | 11.1 | 35.0 | 2.92 | 1.07 | 3.77 |
| hcp (pure technetium films [Ref. 5]) | — | non-annealed | 1600 | 7.60 | 0.03 | 1.97 | 25.0 | 49.3 | 6.15 | 1.00 | 1.79 |
| | — | non-annealed | 320 | 7.14 | 0.03 | 2.99 | 12.1 | 36.2 | 3.06 | 1.03 | 3.59 |

*Width of transition between the levels $0.1R_n$ and $0.9R_n$ in a zero magnetic field.

The presented hcp-phase data pertain to a supersaturated solid solution with concentrations 12 at.% C (non-annealed films), 22 and 29 at.% C (annealed at 600 °C), as well as to films of pure technetium.⁵ When the carbon concentration is increased in this phase from zero to 12 at.% (the maximum concentration at which the hcp phase exists in nonannealed films), T_c and the resistance ratio R_{300}/R_n decrease

while ρ_0 , ρ_{300} and $|dH_{cl}/dT|_{T_c}$ increase. The density of the electronic states decreases in this case.

Hcp-phase films with still larger resistivity and $|dH_{cl}/dT|_{T_c}$ and with smaller resistance ratio R_{300}/R_n were obtained by annealing amorphous samples with concentrations 22 and 29 at.% C at 600 °C. The parameters

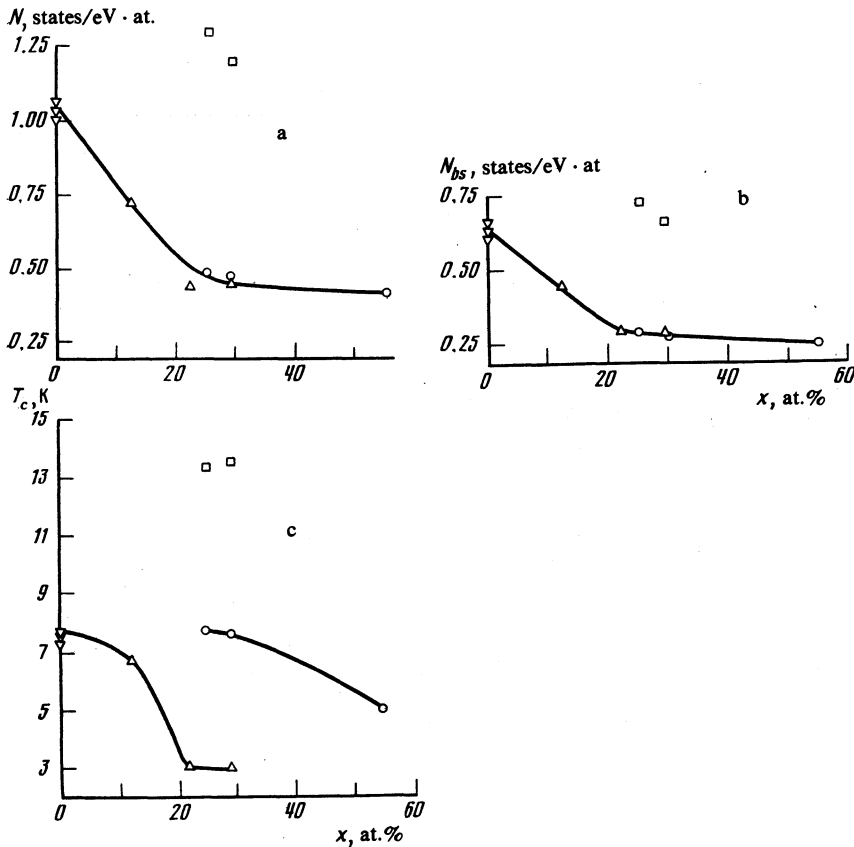


FIG. 3. Dependences on the carbon concentration: a—of the density N of the electronic states, b—of the band density $N_{bs} = N/(1 + \lambda)$ of the electronic states, and c—of the critical temperature. Symbols: \triangle —hcp solid-solution films, ∇ —pure technetium films (hcp structure),⁵ \circ —amorphous films, \square —films in the cubic phase.

characterizing the properties of these films are close to one another. The density of the electronic states in these samples assumes the smallest values for the hcp phase.

We consider now the results for amorphous films. We investigated amorphous films with concentration 25, 29, and 55 at. % C. With increasing carbon concentration in this composition region, the value of T_c of the amorphous phase decreases (from 7.77 to 5.15 K), the resistivities ρ_0 and ρ_{300} as well as $|dH_{cl}/dT|_{T_c}$ increase (from 139 to 175 $\mu\Omega \cdot \text{cm}$, from 135 to 166 $\mu\text{J} \cdot \text{cm}$, and from ~ 20 – 30 kOe/K, respectively), while the density of the electronic states decreases insignificantly (from 0.50 to 0.43 state/eV \cdot at.). The resistance ratio R_{300}/R_n remains approximately constant, somewhat smaller than unity, as is typical of amorphous samples.

We compare now the properties of the amorphous and crystalline films. We begin with a comparison of the amorphous and hcp phases. Examining the aggregate of these films, we can track the change of the density of the electronic states with increasing carbon concentration (Fig. 3a). The dependence of N on the carbon concentration for these two phases is described by a single curve. In the concentration region up to ~ 20 at. % C the density of the electronic states decrease, while in the region from 20 to 5 at. % C it remains practically unchanged.

An approximately similar dependence is observed for the band density of the electronic states $N_{bs} = N/(1 + \lambda)$, where λ is the electron-phonon interaction constant (Fig. 3b). In the calculation of λ we used McMillan's formula for T_c (Ref. 1)

$$T_c = \frac{\Theta}{1.45} \exp \left[- \frac{1.04(1+\lambda)}{\lambda - \mu^*(1+0.62\lambda)} \right] \quad (5)$$

under the assumption that the Coulomb pseudopotential is $\mu^* = 0.13$ and that the Debye temperature is $\Theta = \Theta_0 = 454$ K (the value determined for bulky technetium from measurements of the heat capacity, Ref. 10).²⁾

The observed dependence of N_{bs} on the carbon concentration in the hcp solid-solution films and in amorphous films might be attributed to smearing of the fine structure¹⁴ in the electronic spectrum of Tc on account of the disordering of the lattice by the carbon impurity. For such an explanation to hold, however, it is necessary that the Fermi level coincide with the peak of the plot of the density of the electronic states against the energy. Band calculations^{15,16} show, however, that the Fermi level in technetium is located on the slope or on the pedestal of the peak. We must therefore resort to a different interpretation of the variation of N_{bs} .

A similar change of N_{bs} with increasing nitrogen concentration was observed in Ref. 17 in films of another interstitial solid solution, Nb-N. The density of the electronic states was determined from x-ray electron spectroscopy data. On the basis of the spectra obtained the authors of Ref. 17, invoking results of band calculations for NbN, conclude that with increasing nitrogen concentration the electrons depart from the d -state region in the conduction band into the region of the hybrid states N_{2p} - Nd_{4d} , which is located several eV lower than the Fermi level, and as a result the density of states on the Fermi level is decreased. At nitrogen concen-

trations close to 10 at. % the decrease of N_{bs} stopped. It is possible that in the solid-solution phase of the Tc-C films a similar mechanism changes N_{bs} .

We consider now the possible causes of the change of T_c of Tc-C films on going from the hcp to the amorphous phase. We have seen that the band density of the electronic states (just as the one renormalized with allowance for the electron-phonon interaction) is not changed in this transition, so that the change of N_{bs} (or of N) with changing carbon concentration is described by a single curve for the hcp and the amorphous phases. On the contrary, the dependence of T_c on the carbon concentration (Fig. 3c) is not described by a single curve. In the concentration region close to 25 at. % C, in which both crystalline (hcp solid-solution phase) and amorphous films can be obtained, there is a difference between the values of T_c that characterize these two phases. The curve for T_c in the amorphous phase lies much higher, a reflection of the fact that the superconducting-transition increases by 2.5 times when the hcp solid solution becomes amorphous in the region 25–29 at. % C.

The increase of T_c on going from the hcp to the amorphous phase is apparently connected with the increase of the electron-phonon interaction constant λ . It is most unrealistic to assume that the observed increase of T_c can be due only to the pre-exponential factor in the McMillan equation (5) for T_c (this assumption requires that the average frequencies of the phonon spectrum more than double upon amorphization).

Thus, since the increase of λ , which can be expressed according to McMillan¹ in the form $\lambda = N_{bs} \langle I^2 \rangle / M \langle \omega^2 \rangle$, is not due to the increase of N_{bs} , the cause of the increase of the electron-phonon interaction constant is the increase of the ratio $\langle I^2 \rangle / \langle \omega^2 \rangle$ of the mean squared matrix element of the electron-ion scattering to the mean squared frequency of the phonon spectrum. This fact does not agree with the notion that upon amorphization of alloys and compounds based on transition metals the value of λ is changed on account of the density of the electronic states. This notion is based on the conclusion² that the changes of $\langle I^2 \rangle$ and $\langle \omega^2 \rangle$ cancel each other to a considerable degree and their ratio depends only on the width of the d band and on the overlap of the d -wave functions at the neighboring sites at which the transition-metal atoms are located. The last two quantities, in the opinion of the authors of Ref. 2, should not be noticeably changed by the amorphization. Although such an approach is justified in a number of cases,¹⁸ our results show that it cannot be regarded as universal when the changes accompanying the amorphization are considered. We can point out also another example where the increase of λ upon amorphization is not due to an increase of the density of the electronic states. The increase of T observed¹⁹ upon amorphization of $\text{La}_{78}\text{Zn}_{22}$ is accompanied by a decrease of the value of N_{bs} determined from the heat capacity (see also Ref. 20, where the contributions of different electron and phonon factors to the change of λ is analyzed).

At the same time, a transition from the amorphous to the cubic phase is accompanied (Figs. 3b, c) by an increase of both N_{bs} and T_c (and hence also λ). It must be noted that the

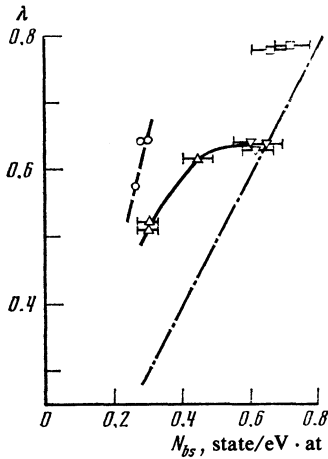


FIG. 4. Dependence of λ on N_{bs} . The symbols are the same as in Fig. 3. The values of λ were calculated from McMillan's formula for T_c under the assumption that $\theta = \theta_0 = 454$ K. The dashed and dash-dot lines correspond to the relation $\lambda = \alpha N_{bs}$ at $\alpha = \text{const} = 1.39$ and 0.59 (states/eV · at.)⁻¹, respectively.

values of λ for the cubic phase lie higher than the $\lambda(N_{bs})$ curve for the hcp phase (Fig. 4). We have already seen that the amorphous phase also has higher value of λ than the hcp phase in one and the same region of carbon concentration and of electronic-state densities (see also Fig. 4). Thus, the amorphous and cubic phases exhibit similar properties with respect to the strength of the electron-phonon interaction.

The plot of λ vs N_{bs} for the hcp phase, shown in Fig. 4, indicates a certain deviation from direct proportionality of these quantities. It must be borne in mind, however, that the values of λ shown in this figure were determined under the assumption that $\theta = \theta_0 = 454$ K. Since we have no data on the phonon spectrum, no conclusions can be drawn concerning the exact values of the deviations.

At the same time, the data obtained for amorphous films do not contradict the relation $\lambda \propto N_{bs}$. The possible reason is that the approximations made in Ref. 2 and on which the cancelation of the changes of $\langle I^2 \rangle$ and $\langle \omega^2 \rangle$ was deduced, are more justified for the amorphous state, in which, in view of the maximum smearing of the electronic structure, all its details whose influence was neglected vanish.

In conclusion, we summarize briefly the principal results.

In the cubic phase, which exists in a narrow range of carbon concentrations and annealing temperatures, high T_c coexist with high critical magnetic fields and are partially due apparently to a high density of the electronic phase. In this phase the density of the electronic states is higher, and the Fermi velocity lower, than in other Te-C phases.

In hcp films and in the amorphous phase, the dependence of the density of the electronic states on the carbon concentration is described by a single curve. The values of N for the hcp phase lie both on the descending part of this curve and on the almost horizontal part (up to 30 at. % C), while for amorphous films (in the region from 25 to 55 at. % C) they lie on the almost horizontal part of the curve. A similar behav-

ior is exhibited by the band density N_{bs} of the electronic states. No change takes place in N and N_{bs} on going from the hcp to the amorphous state. The decrease of N and N_{bs} in the initial concentration region is possibly due to the fact that when the carbon concentration is increased the electrons go off from the region of d states near the Fermi levels to a region of higher energies, owing to formation of hybrid states of Te-C.

For crystalline hcp solid solution films, the changes of T_c and of the density of the electronic states correlate: a decrease of N (and N_{bs}) with increase of the carbon concentration is accompanied by a decrease of T_c , and these quantities saturate at the same concentration (~ 20 at. % C). Nonetheless, a deviation from direct proportionality is noted in the plots of λ against N_{bs} for these films.

On going from the hcp to the amorphous phase, T_c increases considerably (by ~ 2.5 times) at constant N_{bs} (and N). The increase of T_c is due to the increase of the electron-phonon interaction constant λ , which is due to the increase of $\langle I^2 \rangle / \langle \omega^2 \rangle$. This fact contradicts the notion that the change of λ upon amorphization of transition metals is determined mainly by the change of the density of the electronic states.

At the same time, this notion agrees with the decrease of N_{bs} and T_c (and hence also λ) on going from the cubic to the amorphous phase. The electron-phonon interaction constants in the cubic and amorphous phases are higher than in the hcp phase (Fig. 4). In this sense the cubic phase can be regarded as the crystalline analog of the amorphous phase. It should be noted that electron-diffraction data do not indicate unambiguously which of the crystalline phases is more closely related to the amorphous phase.

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¹To show that our samples are type-II superconductors, we estimate the Ginzburg-Landau parameter κ using the formula $\kappa = \kappa_0 + 7.5 \cdot 10^3 \gamma^{1/2} \rho_0$ that follows from Gor'kov's theory. In pure Te, κ_0 is somewhat larger than $1/\sqrt{2}$.¹¹ With increasing carbon concentration in the Te, it is not excluded that κ_0 can decrease. But for the Te-C films investigated by us, the term $7.5 \cdot 10^3 \gamma^{1/2} \rho_0$ ensures values $\kappa > 10$. In this estimate we used the relations $1/\rho_0 = (2/3)e^2 N v_F l$ and $\gamma = (2/3)\pi^2 k^2 N$ as well as the experimental values of ρ_0 subject to reasonable restrictions on the values of v_F and l ($v_F < 10^8$ cm/sec and $l < l_p = 100$ Å, where l_p is the mean free path in a film of pure technetium 640 Å thick.⁵

²To estimate the extent to which the assumption $\theta = \theta_0$ can distort the true value of N_{bs} on account of the factor $1 + \lambda$, we assume that the relative change of θ is the same as observed in the interstitial solid solution Nb-O from heat-capacity data,¹³ obtained for oxygen concentrations less than 3.5 at. %, namely $\theta = \theta_0(1 + 0.054x)$, where x is the concentration of the interstitial impurity (in at. %). We shall assume that this law is valid up to 29 at. % of carbon in crystalline hcp-phase films. Even though it leads to a very strong increase of θ (by 2.4 times at 20 at. % C), the deviations of N_{bs} from the values calculated at $\theta = \theta_0$ are insignificant (only 7% at the same carbon concentration).

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