QUANTUM SIZE EFFECT IN SUPERCONDUCTING TIN FILMS

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The dependences of the critical superconducting transition temperature and energy gap on the thickness of tin films obtained by sputtering in vacuum are investigated. The energy gap was measured by the tunnel technique. The dependences have an oscillatory nature as the result of quantization of the quasiparticle energy spectrum. Two types of oscillations, with periods ~7.5 and ~15 Å are observed. The Fermi momenta corresponding to such oscillation periods are respectively 4.4×10^{-20} and 2.2×10^{-20} g-cm/sec.

1. INTRODUCTION

A number of theoretical papers[1-6] have dealt with the question of the possible influence of the quantum size effect on the magnitude of the energy gap and the critical temperature of the superconducting transition in thin films. Owing to the limited dimension in thin films, the quasimomentum component corresponding to the motion of the electrons across the film turns out to be quantized. As the result, the spectrum of the electrons in the film breaks up into several subbands, the number of which decreases with decreasing thickness of the film[7]. This results in a jumplike change of the density of states when the film thickness changes, owing to the change of the number of subbands. According to calculations by Blatt and Thompson[1], allowance for this circumstance should lead to an oscillatory dependence of T_c on the film thickness. A similar result was obtained also in[2-6]. Kresin and Tavger[6] considered the problem with allowance for the possible difference between the electron-phonon interaction for electrons in different subbands.

In the simplest interpretation, the result is due to the fact that in the relation

$$kT_c = 1.14\hbar\, \omega_D \exp\left(-1/nVN_0\right) \tag{1}$$

the argument of the x exponential contains both the two-dimensional density of the states of the electrons in the subbands

$$N_0 = m/2\pi\hbar^2 d, \tag{2}$$

and the number of subbands

$$n \approx (2m\varepsilon_F)^{1/2} d/\pi\hbar \tag{3}$$

(m—effective mass in the direction of the small dimension, n = 1, 2, 3, ...). Within the limits of the constancy of n, the dependence of N_0 on d leads to a monotonic decrease of the critical temperature with increasing film thickness, but an increase from n to n + 1 at a certain thickness causes a jumplike increase of T_c. This leads to the occurrence of a sawtooth-like $T_c(d)$ dependence; the amplitude of the oscillations increases with decreasing film thickness. At the same time, the decrease of n leads to an increase of the electron-phonon interaction constant[6], i.e., the monotonic component of the $T_c(d)$ dependence should increase with decreasing film thickness.

To realize the quantum size effect in thin films, special conditions must be satisfied[7]. The most significant requirements are specular reflection of the electrons from the film surface and a sufficiently long relaxation time of the electrons, ensuring a small smearing of the edges of the subbands compared with the distances between neighboring subbands:

$$\frac{h}{\tau} \ll \varepsilon_{n+1} - \varepsilon_n, \quad \varepsilon_n = \frac{\hbar^2\pi^2}{2md^2} n^2. \tag{4}$$

Estimates show that in a film for which d = 200 Å, $m = m_0$, and $\epsilon_F = 1$ eV the shift of the subbands near the Fermi level, $\Delta\epsilon_F = (\pi\hbar/d)\sqrt{2\epsilon_F/m}$ amounts to $\sim 6 \times 10^{-2}$ eV, from which it follows that the mean free path of the electrons must be $l \gg 10^{-6}$ cm (at $v_F \sim 10^8$ cm/sec). The latter requirement can be satisfied only by sufficiently perfect and pure films.

The requirement of specular electron reflection is satisfied to a complete degree only by films of semimetals and semiconductors in which the carriers have a large de Broglie wavelength. With respect to normal metals, the question of the probability of specular reflection of the electrons on the surface remains on the whole open, but apparently electrons that "glance" against the surface can be specularly reflected[8].

The difficulty in observing the effect predicted by Blatt and Thompson lies in the fact that the period of the oscillations of the gap and of the critical temperature, with varying film thickness, is equal to half the de Broglie wavelength of the carriers, which amounts to several Angstroms for normal metals. In this connection, the theoretical papers discuss more frequently the question of the possibility of observing a new effect in films of superconducting semiconductors[4,9], for which the de Broglie wavelength of the carriers is sufficiently large. However, such objects are either unobtainable in the form of thin films, or else have an exceedingly low critical temperature. In films of ordinary superconducting metals, it seems that oscillations of the critical temperature and of the gap cannot be observed, and all that can be noted is a total growth of the average value of T_c with decreasing film thickness[6].

Such an explanation of the dependence of the critical temperature on the film thickness observed for a number of metals[10] is quite interesting. In this connection, Alekseevskiĭ and Vedeneev[11] attempted to investigate the passage of monochromatic light through a film of aluminum at helium temperature, and observed an oscillatory dependence of the intensity of the transmitted light on the film thickness. They connect these oscillations with the size quantization of the spectrum of the electrons in the films, although the period of the oscillations in aluminum films (~200 Å) turned out to be not characteristic of the known carriers in aluminum. In[12] it was reported that an oscillatory thickness dependence was observed in the relative resistance of tin films at the superconducting-transition temperature, together with the associated oscillations of T_c.

We present here the results of a further study of the new effect, performed, in particular, with the aid of a tunnel procedure.

2. SAMPLE PREPARATION PROCEDURE

The thin films were prepared by evaporating pure (99.999%) tin in a vacuum of $\sim 10^{-6}$ Torr from a tantalum vessel of 3 mm diameter. The distance from the evaporator to the substrate was ~150 mm. The evaporator was placed eccentrically relative to the substrate, in order to obtain films of variable thickness. On a film 70 mm long and 0.2 mm wide, the thickness varied by approximately 30%. The thickness variation along the film corresponded to a distribution from a point-like evaporator. The thickness was determined by measuring the optical density S of the films. The dependence of S on the film thickness was plotted beforehand by an independent method described in[13]. The accuracy of the method is ~10%, and the sensitivity is less than 1 Å (at thicknesses not exceeding 250 Å). The films were deposited on a glass substrate at $T \sim 200°K$,

The substrate was cooled in order to decrease the critical thickness of the film, although this led to a certain deterioration of the structure characteristics. The structure of the films prepared under the same conditions on acetate laquer was investigated by electron diffraction and with an electron microscope. The films had a texture: the [100] direction was normal to the plane of the film. The disorientation angle of the texture axis did not exceed ~20°. The dimension of the crystallites in the plane of the film exceeded the film thickness. Films of very small thickness were cut up by channels, which were filled with metal as the film thickness increased. The fraction of the area occupied by the channels was an insignificant part of the total area of the film.

An Sn-I-Sn tunnel system was produced with the films prepared in this manner. The first (thin) film was oxidized in an oxygen atmosphere (~1 mm Hg) for one half-hour. Transverse ("thick," $d \sim 500$ Å) films were then condensed over the oxide layer under the same conditions as the first film. The distance between the transverse films was 2 mm. These films subdivided the sample into sections. They served as potential contacts for the measurement of the electric resistance.

The thickness of the film on each section is not strictly constant. In addition, the film has microscopic irregularities. The faces of the crystallites forming the outer surfaces of the film are not absolutely flat, but have steps (vicinals), representing atomically-smooth sections much longer than the period of the lattice. When the quantum size effect is produced, the characteristics of the electronic states are naturally different on different vicinals, and for the greater part of the film they become averaged out and should be referred to some average effective thickness, which can assume arbitrary values, not necessarily multiples of the lattice period. The deviation of the true thickness from the mean value should affect unfavorably the possibility of observing the quantum size effect (see the Appendix), and apparently only the use of the method of variable-thickness samples, which ensures undeviating monotonic variation of the effective thickness along the sample, makes it possible to construct reliably the dependence on the thickness with small thickness steps.

3. MEASUREMENTS OF THE ELECTRIC RESISTANCE OF THE SAMPLES

Using the described samples, we investigated the dependence of the electric resistance of films of different thicknesses on the temperature (near the critical temperature of the superconducting transition). The temperature was determined by measuring the vapor pressure over the He^3 and was stabilized with a manostat. The accuracy with which the temperature was measured was not worse than 1×10^{-3}°K. All the measurements were made while the temperature was lowered monotonically from one cycle of measurements along the sample to the next cycle.

Small changes of the critical temperature are best characterized by the change of the relative resistance of the film R/R_n (R—resistance of the section of the film at the given temperature, R_n—resistance in the normal state), since very small shifts of T_c lead to noticeable changes of R/R_n. This "enhancement" is due to the rapid rate of transition into the superconducting state. Indeed, the temperature interval of the transition, for tin films, amount on the average to ~0.02°K, and a change of T_c by 0.002°K (i.e., by 5×10^{-4} of absolute magnitude) leads to a 10% change of R/R_n. In addition, by varying the temperature as a parameter, and plotting a series of curves $R/R_n = f(d)$ it is possible to reveal with sufficient reliability minimal points on these curves, corresponding to maxima of T_c. These points are reproducible at different temperatures, and this enables us to discard random deviations.

Typical curves of $R/R_n = f(d)$ are shown in Fig. 1, from which it is seen that these functions have an oscillating character; the period of the oscillations is practically independent of the thickness and amounts on the average to 7–8 Å. From these curves it is easy to obtain the dependence of the critical temperature on the thickness. To this end it is necessary to find those thicknesses for which R/R_n assumes definite values at the given temperatures (for example, $R/R_n = 0.5$ or $R/R_n = 0.1$, depending on the chosen criterion). Such a construction is in essence a method of interpolating

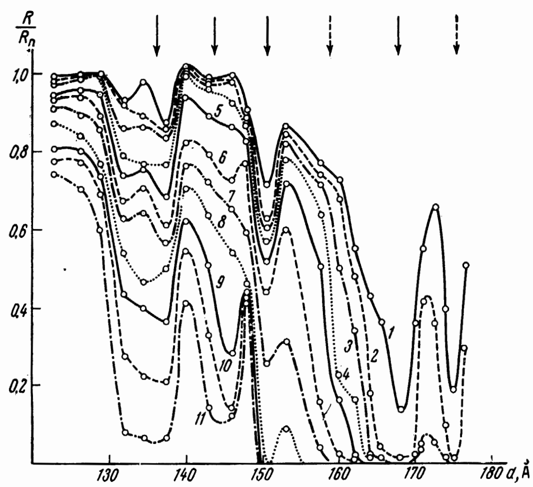

FIG. 1. Relative resistance of tin films as a function of the thickness. Temperature (°K): 1–4.041, 2–4.038, 3–4.035, 4–4.031, 5–4.026, 6–4.022, 7–4.016, 8–4.010, 9–4.002, 10–3.996, 11–3.992.

the thicknesses, and the number of points obtained on the $T_c(d)$ curve can be greatly increased by decreasing the interval between the temperatures at which the measurements are carried out. The points obtained in this manner are designated by light circles in Fig. 2b. The second independent method of obtaining the $T_c(d)$ curve consists of constructing plots of R/R_n against T for all the sections of the sample, i.e., for all the represented thicknesses, and finding the temperatures at which $R/R_n = 0.5$ or $R/R_n = 0.1$ (this is a method of interpolating the temperature on a transition curve plotted point by point). The points obtained in this manner are designated by dark circles in Figs. 2a and 2b. It is seen from Fig. 2b that both methods give practically identical curves.

Two types of extrema are observed on the $T_c(d)$ curves: clearly pronounced maxima with a period 15 Å, and weaker maxima with a period ~7.5 Å, observed after subtracting the monotonic component of the $T_c(d)$ dependence (they are marked in Figs. 2a and 2b by vertical arrows of different heights).

In Fig. 1, the vertical arrows denote those thicknesses at which maxima of the critical temperature were observed in the given sample. It should be noted that the plots of R/R_n against d have a much larger number of singular points (inflection points or curvature changes) than designated by the arrows[1)]. This points to the possible existence on the $T_c(d)$ curves of singularities with a smaller period than 7.5 Å, which, however, cannot be observed in films 150–200 Å thick, since they should be due to subbands with very high quantum numbers.

4. MEASUREMENT OF THE ENERGY GAP

To measure the width of the energy gap in the excitation spectrum of the superconducting tin films, we used a tunnel procedure. We plotted the current-voltage (I–V) and the dV/dI–V characteristics with a two-coordinate automatic-recording potentiometer. The measurements were carried out at $T \sim 1.6$°K. The accuracy with which the gap is measured depends to a strong degree on the quality of the tunnel junctions. The current-voltage characteristic of a high-grade tunnel junction consisting of two superconductors with close gap values is practically vertical at a voltage $(\Delta_1 + \Delta_2)/e$ (Δ_1—energy gap in the thin film, Δ_2—energy gap in the thick film, e—electron charge). Plotting dV/dI as the function of V, we obtained a very sharp minimum, the position of which determined $\Delta_1 + \Delta_2$. To obtain the derivative dV/dI, we used the standard modulation procedure[14]. To increase the accuracy with which the voltage corresponding to the minimum was read, the recording was obtained in a scale that was strongly magnified with respect to V (the full scale of the automatic potentiometer corresponded to 150 μV). To this end, a voltage $U_0 \approx 1.1$ mV was applied to the potential circuit in opposition to the voltage picked off the tunnel junction. One of the plots of the section of the dV/dI vs. V curve near the minimum is shown in Fig. 3. The position of the extremal point was determined accurate to 1 mm, which corresponds to a voltage of ~0.5 μV. This value was not exceeded in repeated plots. The amplitude of the modulation signal at the minimum was ~3 μV, but in view of the absence of a noticeable asymmetry of the curve near the minimum, this should not lead to noticeable errors.

The production of samples suitable for such measurements entails great difficulties, since it is necessary to obtain in a sample of variable thickness a large number of high-grade tunnel junctions. To obtain the quantum size effect it is desirable to have the thickness of the investigated film as small as possible. However, in films with thickness close to critical, the

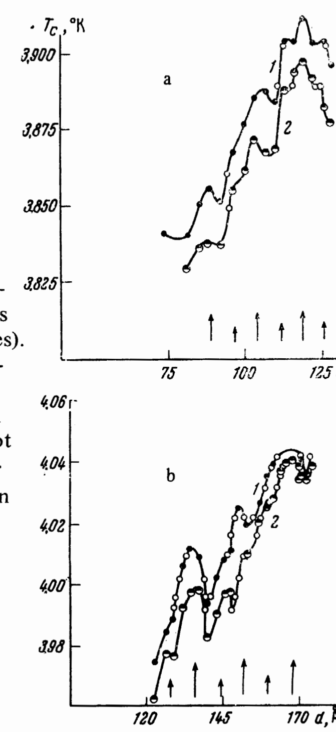

FIG. 2. Dependence of the critical temperature of the superconducting transition of tin films on the thickness (for two samples). O—points obtained by interpolation of the thickness, O, O—points obtained by interpolation of the temperature. Curve 1—plot of T_c against the thickness in accordance with the criterion $R/R_n = 0.5$; curve 2—$R/R_n = 0.1$.

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1)No account should be taken of the configuration of the curves for very small values of $R/R_n < 0.1$, and for large values close to unity, since random distortions of the shape of the curve of the transition into the superconducting state may be observed here, for example under the influence of the edge region, etc.

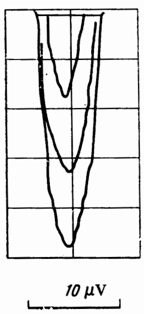

FIG. 3

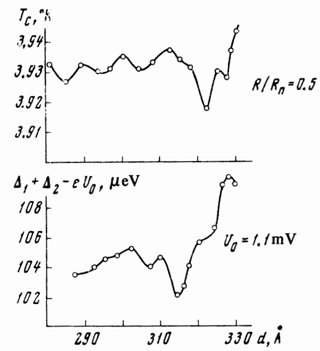

FIG. 4

FIG. 3. One of the plots of dV/dI = f(V) near the minimum.

FIG. 4. Dependence of the critical temperature and of the superconducting gap on the thickness of tin film. T_e is determined from the criterion $R/R_n = 0.5$. The initial bias on the tunnel junction is $U_0 = 1.1$ mV.

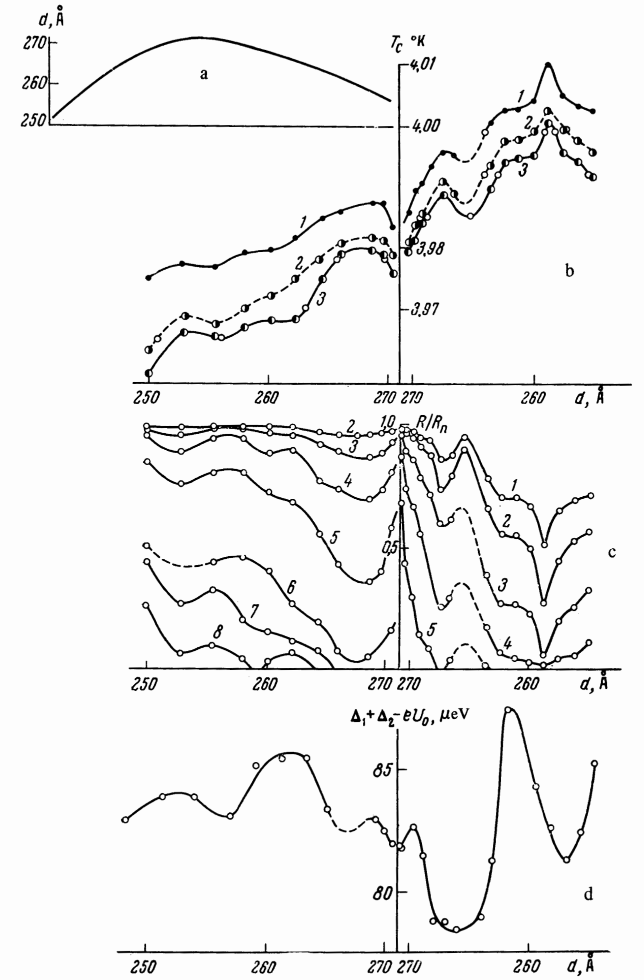

FIG. 5. Dependence of the critical temperature and of the gap on the thickness of tin films for a sample having a hill-type distribution of the thickness. a) Change of thickness along the length of the sample. b) Dependence of T_c on the thickness in accordance with the following three criteria: $1-R/R_n = 0.5$, $2-R/R_n = 0.2$, $3-R/R_n = 0.1$. c) Dependence of R/R_n on the thickness. Temperature (°K): 1–4.011, 2–4.004, 3–3.999, 4–3.992, 5–3.985, 6–3.978, 7–3.970, 8–3.965. d) Dependence of the superconducting gap on the thickness, $U_0 = 1.12$ mV.

tunnel junctions, as we have verified, are technically unattainable. With increasing thickness, the structure of the films improves, and this determines the success in producing high-grade tunnel junctions, but the amplitude of the oscillations and of the gap in this case decreases, owing to the increasing number of subbands. In addition, at the chosen thickness gradient, the thickness interval between the experimental points increases. These circumstances, naturally, make it difficult to detect the fine points of the dependence of the gap on the film thickness. We succeeded, however, in preparing a number of samples with a large number of high-grade tunnel junctions. Measurements performed on these samples demonstrated quite clearly the oscillatory character of the dependence of the energy gap on the thickness of the investigated film (Fig. 4) (we assume that for the thick film the gap Δ_2 is practically independent of the thickness).

In all cases, we observed oscillations with a period of ~7.5 Å. The position of the maxima on the $T_c(d)$ and on the $(\Delta_1 + \Delta_2 - eU_0)$ vs. d curves do not coincide on the thickness scale. This is apparently connected with the fact that the thin film on the section of the tunnel junction is covered by a second film, whereas the sections between the tunnel contacts are open and can become additionally oxidized during the period between the production of the sample and its placement in the cryostat. Our measurements of the thickness pertain to the open sections of the sample, and consequently the true thickness of the film on the sections of the tunnel junctions is somewhat larger than the indicated one. The plots of $(\Delta_1 + \Delta_2 - eU_0)$ vs. d, generally speaking, should be shifted to the right on the thickness scale.

Interesting conclusions can be drawn on the basis of results obtained for a sample with a thickness distribution in the form of a hill (Fig. 5). In this sample, the interval between the experimental points is quite small, and in addition, equal thicknesses occur in this sample twice. The sample was obtained by successive sputtering from two evaporators, one of which was located symmetrically and the other asymmetrically with respect to the substrate. The structure characteristics of the sample are not equivalent, and deteriorate from right to left, owing to the increase of the angle between the direction of the molecular beam and the normal to the plane of the film.

It is seen from Fig. 5 that the position of the maxima on the plots of T_c and of $(\Delta_1 + \Delta_2 - eU_0)$ vs. d, and accordingly the positions of the minima on the plots of $R/R_n = f(d)$, are strictly symmetrical with respect to the maximum thickness. The distance between maxima, as in the other cases, is ~7.5 Å. However, oscillations of the gap on the right slope are more strongly pronounced and, more significantly, the monotonic component of the $T_c(d)$ plot decreases continuously from right to left with deterioration of the sample structure. Thus, the course of the monotonic component of the $T_c(d)$ plot is determined in our samples mainly by factors not connected with the quantum size effect. For a structurally-symmetrical sample, the monotonic component of $T_c(d)$ is, naturally, symmetrical.

5. DISCUSSION OF RESULTS

It is seen from the foregoing experimental results that the thickness dependences of the critical temperature of the superconducting transition and of the energy gap in tin films have an oscillatory character. Two types of oscillations with different periods are observed, ~7.5 and ~15 Å. The values of the extremal momentum p_{extr} corresponding to these periods in the direction of the small dimension, determined from the relation $\Delta d = \pi\hbar/p_{extr}$[15], are 4.4×10^{-20} and 2.2×10^{-20} g-cm/sec, in agreement with the published data for tin[16]. At film thicknesses ~150 Å, a band with $p_{extr} = 4.4 \times 10^{-20}$ g-cm/sec contains approximately 20 subbands, and a band with $p_{extr} = 2.2 \times 10^{-20}$ g-cm/sec only approximately 10. The amplitude of the oscillations with the period corresponding to the band with the smallest number of subbands is of course much higher than on the presented curves. The obtained momentum values pertain to the [100] direction, which in textured films is normal to the film.

Interesting conclusions can be drawn by comparing the present data with those of Gantmakher[17], who measured by the method of the radio-frequency size effect the extremal Fermi momenta in different directions of single-crystal samples of tin. For the [100] direction, he obtained the following values: 2.1×10^{-20}, 4.5×10^{-20}, 6.3×10^{-20}, and 11.6×10^{-20} g-cm/sec. Our results agree very well with the first two values. They correspond to the extremal Fermi momenta in bands 5 and 4 (c). The character of the surface in the fifth band is such that a change in the direction by several degrees changes insignificantly the extremal momentum, so that the imperfection of the structure of the investigated films should not greatly hinder the observation of the effect. The two other values of the momentum given in[17] correspond to closed trajectories in surfaces open along [100]. We observed no oscillations corresponding to such values of the extremal momentum. The surfaces that are open in the direction of the small dimension should apparently not make an appreciable contribution to the oscillatory effects that are connected with the spatial quantization. In addition, the corresponding periods should be ~5 and ~3 Å. Observation of oscillations by such periods seems to be already impossible in principle.

Such a good agreement between the characteristic dimensions of the Fermi surface in very thin films and in bulky metal is in general surprising. It offers evidence that a crystal dimension of several dozen atomic layers suffices for the formation of a band structure close in its principal parameters to the structure of the bulky metal in the given direction.

Yet the small dimension of the crystal can, on the other hand, be the cause of the deviation of the parameters of the electronic spectrum. It is known that the customarily employed methods of investigating the energy structure of solids (resonance and oscillatory effects) are not very suitable for thin films. The quantum size effect affords new possibilities, which make it applicable, in principle, to the study of the energy spectrum of quasiparticles in thin films. Such information is important both from the purely theoretical point of view, and for the understanding of the physical nature of the phenomena in thin layers and for the explanation of the anomalies and singularities in the physical properties of thin films.

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APPENDIX

The amplitudes of the oscillations of the critical temperature and of the gap, obtained in our experiments, are quite small and amount to 10^{-3} (for the oscillations with the period ~15 Å) and ~10^{-4} (for the oscillations with the period ~7.5 Å) of the absolute magnitude, whereas in the calculations of Blatt and Thompson[1] the amplitude of the oscillations was of the order of the quantity itself. A circumstance strongly reducing the amplitude of the effect is the surface imperfection of the film, or the presence of steps of atomic dimensions on the surface.

Let us assume that the probability of deviation of the thickness from the mean value are described by a Gaussian distribution

$$p(x) = \frac{1}{\sqrt{2\pi}\,\sigma} \exp\left\{-\frac{(x-x_0)^2}{2\sigma^2}\right\},$$

where $p(x)$ is the density of the probability that the thickness will assume a value x; x_0 is the average (most probable) film thickness.

Let the oscillating term in $T_c(d)$ (or in $2\Delta_0(d)$, etc.) be described by the relation

$$\Omega(x) = A_0 \sin\frac{2\pi x}{\Delta d},$$

where A_0 does not depend on the thickness and Δd is the period of the thickness oscillation. Then the oscillating term in the dependence of T_c on d, with allowance for the smearing of the thickness, can be written in the form

$$\Omega'(x) = A_0 \frac{1}{\sqrt{2\pi}\,\sigma} \int_0^\infty \sin\frac{2\pi y}{\Delta d} \exp\left\{-\frac{(y-x)^2}{2\sigma^2}\right\} dy.$$

After integration we obtain

$$\Omega'(x) = A_0 \exp\left\{-\frac{2\pi^2\sigma^2}{\Delta d^2}\right\} \sin\frac{2\pi x}{\Delta d}.$$

The relative decrease of the amplitude is

$$\Omega'/\Omega = \exp\{-2\pi^2\sigma^2/\Delta d^2\}.$$

For the oscillations with the period 7.5 Å, we get $\Omega'/\Omega \sim 10^{-4}$.

The relative amplitude obtained in our experiments corresponds to a parameter σ equal to ~5 Å in the Gaussian distribution. At such a value of the parameter, the character of the distribution is such that the thickness of the greater part (~80%) of the investigated section of the film differs from the mean value by not more than two interplanar distances (±6 Å), i.e., the thickness oscillates approximately ±5%.

The obtained estimates yield a perfectly reasonable order of magnitude of the deviation of the film thickness from the mean value.

The obtained estimates yield a perfectly reasonable order of magnitude of the deviation of the film thickness from the mean valve.

In accordance with the calculation, the amplitude of the oscillations with the period ~15 Å should be much higher, but in this case, apparently, the anisotropy of band 4 (c), which is responsible for the oscillations with such a period, comes additionally into play. The imperfection of the texture in the case of strong anisotropy of the band can greatly decrease the amplitude of the effect.

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