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MECHANISM OF INITIATION OF ACOUSTIC EMISSION IN CRYSTALLIZATION AND MELTING OF A SUBSTANCE. I.

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A theoretical model of the acoustic effect of crystallization is suggested based on the representation of a stepwise character of formation or disappearance of macrolayers and macroregions on a growing (or melting) surface. According to this model, the picture of oscillations reproduces in basic features the form of the signals observed in experiments. The oscillation frequency of the liquid is determined by the frequency of generation of jumps at the crystallization front, while the comparatively large values of peak pressures in acoustic waves are a consequence of the resonance phenomena.

As is known, with solidification of the majority of substances their relative volume decreases, with the exception of only a small group that includes water, cast iron, antimony, and some others, for which the crystallization process is accompanied by an increase in their relative volume. Usually the relative change in the volume with phase changes of substances is (3–10)%. Thus, practically for all substances the moving solid–melt interface is a source of disturbance for the medium density.

In this connection, as early as 1967 Prof. S. N. Zadumkin suggested that with phase transformations of substances the acoustic waves of compression and rarefaction must propagate in the surrounding medium. This suggestion was completely confirmed by numerous experiments, among which are also the experiments conducted at the Kabardinian-Balkar State University [1-5]. The phenomenon of propagation of acoustic waves emerging from the zone of substance crystallization and melting was named in [2] the acoustic effect of crystallization. It should be noted that the order of the high-frequency signals observed in our experiments coincides with the data of [6, 7]. At present the existence of the acoustic effect of crystallization is considered to be reliably established.

For different substances acoustic signals are characterized by a wide frequency range from 20 to 2000 kHz at characteristic heights of first emissions of $(10^2–10^3)$ Pa. The repetition frequency of the signals increases with the temperature gradient in a growing solid phase. Thus, in the case of water crystallization at $\Delta T/\Delta X \sim (2–8)$ deg/cm the acoustic signals are mainly observed at frequencies of 20–60 kHz, although sole signals with frequencies of up to 1000 kHz also take place; at $\Delta T/\Delta X \sim (10–15)$ deg/cm signals whose frequencies are within the range from 100 to 1000 kHz or more prevail. These frequencies substantially exceed the fundamental frequency of natural oscillations of a liquid as well as the frequencies of signals (~3–15 kHz) occurring due to side factors that accompany the crystallization process, namely, cleavage of a growing crystal, separation of the crystal from the wall of a crystallization cell because of the difference in the temperature coefficients of expansion. The velocities of the crystallization front, at which the high frequencies are observed, are (1–2) mm/min.

Interest in the acoustic effect of crystallization is explained by different reasons. Thus, for instance, investigating the structure of acoustic signals of the crystallization zone can provide new information about the elementary processes that occur in the crystallization zone of a substance at the molecular level. In [8] it has been established that if an ultrasound is applied to a substance under crystallization with the frequency of natural acoustic radiation of the crystallizing system, then the structural components of the solid substance formed

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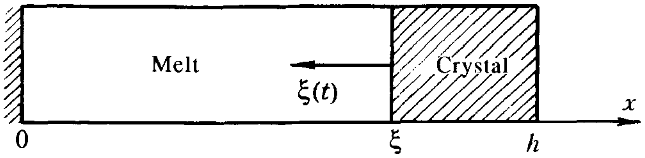

Fig. 1. Schematic of a cylindrical crystallization cell.

undergo drastic reduction in size. This makes it possible to select the optimum frequency when the process of melt crystallization is acted upon by ultrasound.

The acoustic waves emerging from the crystallization or melting zone serve to signal the onset of melting or crystallization of a substance inside closed volumes, for instance, in blast furnaces, which is of great practical interest.

However, the nature of initiation of acoustic waves associated with crystallization and melting of a substance is still unclear. In [2] the occurrence of acoustic signals with crystallization (melting) of a substance is related to the stepwise character of formation or disappearance of macrolayers and macroregions on a growing (or melting) surface. Not only does this view not contradict the kinetics of phase transformations of substances but in a sense is a direct consequence of it.

As is known, latent heat of melting of substances is usually small as compared to that of evaporation; for the majority of substances the ratio of these heats is 30–40; for water (8–9). From this it is inferred that in the melted state only a comparatively small portion of the bonds between atoms and molecules is broken, while the remaining ones stick together to form small groups of, approximately, 30–40 (for water 8–9) atoms and molecules with a size of $\sim 10^{-7}$ cm, inside which the distances between the atoms and molecules are the same as in the solid phase.

With crystallization of liquids the broken bonds between these groups or complexes are restored, i.e., atomic (molecular) blocks are incorporated into the crystal lattice of the solid phase. This process of lattice restoration proceeds via independent chains in the longitudinal direction of the crystal surface and is stochastic.

Each event of the addition of the atomic or molecular blocks (complexes) to the surface of the solid phase is accompanied by an abrupt change in the density or relative volume; this change in the density (volume) as the medium disturbance is imparted to the neighboring layers of the liquid in the form of high-frequency impulses, which initiates forced oscillations of the medium surrounding the crystallization (melting) front of the substance. Thus, the growing crystal acts as a piston that moves into (out of) a tube in momentary jerks. The duration of these jerks is a random quantity; therefore the process of generation of signals in the medium around the crystallization zone is stochastic. The investigation of such processes is a rather complicated problem; therefore below we restrict ourselves to the consideration of a simplified model that makes it possible to reveal some regularities in initiation of the acoustic effect of crystallization.

At first we will investigate acoustic waves which are generated in a liquid when the crystallization front performs stepwise motion in accordance with the above concept of crystal growth.

The crystallization cell will be considered as a tube placed horizontally (see Fig. 1). The left end of the tube is closed tightly, while from the right end the liquid begins to crystallize and the crystallization front $x = \xi(t)$ moves from right to left. The solid phase formed during melt crystallization serves as a piston: if, with crystallization of the liquid, the volume increases, we are concerned with the case of the piston moving into the tube; if the volume decreases, this indicates that the piston moves out of the tube. In both cases, at the moving interface the density and the pressure undergo disturbance; consequently, this interface is a source of compression and rarefaction waves. These waves are described, as is known, by the equation

$$\frac{\partial^2 u}{\partial t^2} = a^2 \frac{\partial^2 u}{\partial x^2} + \nu \frac{\partial^3 u}{\partial t \partial x^2}, \quad 0 < x < \xi. \tag{1}$$

Assuming that liquid crystallization starts from the instant of time $t = 0$, the initial and boundary conditions can be written in the form

$$u(x, 0) = 0\,; \quad u_t(x, 0) = 0\,; \tag{2}$$

$$u(0, t) = 0\,; \quad u(\xi, t) = -\beta\,(h - \xi)\,, \tag{3}$$

where $u_t = \partial u/\partial t$.

A solution of problem (1)-(3) will be sought as the sum of two functions

$$u(x, t) = \vartheta(x, t) + W(x, t)\,,$$

where

$$W(x, t) = -\beta \frac{x}{h}(h - \xi)\,,$$

and $\vartheta(x, t)$ is a solution of the problem

$$\vartheta_n = a^2 \vartheta_{xx} + \nu \vartheta_{xxt} - \beta \frac{x}{h} \ddot{\xi}(t)\,;$$

$$\vartheta(x, 0) = 0\,; \quad \vartheta_t(x, 0) = -\beta \frac{x}{h} \dot{\xi}(0)\,;$$

$$\vartheta(0, t) = 0\,; \quad \vartheta(\xi, t) = 0\,. \tag{4}$$

We will seek the function $\vartheta(x, t)$ as the series

$$\vartheta(x, t) = \sum_{n=1}^{\infty} \vartheta_n(t) \sin \frac{\pi n x}{\xi}\,, \tag{5}$$

where $\vartheta_n(t)$ are solutions of the equations

$$\ddot{\vartheta}_n + 2\gamma_n \dot{\vartheta}_n + \omega_n^2 \vartheta_n = f_n(t)\,, \tag{6}$$

in which

$$\omega_n = \frac{\pi n a}{\xi}\,, \quad \gamma_n = \frac{\nu \omega_n^2}{2a^2}\,, \quad f_n(t) = (-1)^{n+1} \frac{2\beta\xi}{\pi n h} \ddot{\xi}(t)\,,$$

and satisfy the initial conditions

$$\vartheta_n(0) = 0\,; \quad \dot{\vartheta}_n(0) = (-1)^n \frac{2\beta\xi}{\pi n h} \dot{\xi}(0)\,.$$

In writing Eqs. (5) and (6) we took into account the circumstance that usually the crystallization front moves very slowly, therefore $\xi(t) \approx h$ and a change in ξ under the sine can be neglected.

We will re-write the solutions of Eqs. (6) in the form

$$\vartheta_n(t) = \frac{2\beta\xi}{\pi h}(-1)^{(n+1)} \left[J_{1n}(t) - \frac{\gamma_n}{\omega_n^*} J_{2n}(t) \right], \tag{7}$$

where

$$J_{1n}(t) = \int_0 \dot{\xi}(\tau) \exp[-\gamma_n (t-\tau)] \cos \omega_n^* (t-\tau)\, d\tau\,; \tag{8}$$

$$J_{2n}(t) = \int_0^t \dot{\xi}(\tau) \exp[-\gamma_n (t-\tau)] \sin \omega_n^* (t-\tau)\, d\tau\,; \tag{9}$$

$$\omega_n^* = \sqrt{\omega_n^2 - \gamma_n^2}\,. \tag{10}$$

The pressure in an acoustic wave is determined by the formula

$$p(x,t) = -\frac{1}{K}\frac{\partial \vartheta}{\partial x} = \frac{2\beta}{Kh} \sum_{n=1}^{\infty} (-1)^n \left[J_{1n}(t) - \frac{\gamma_n}{\omega_n^*} J_{2n}(t) \right] \cos \frac{\pi n x}{\xi}\,, \tag{11}$$

where K is the compressibility factor.

As is seen from relation (11), any small changes in the solid–melt interface lead to initiation of acoustic waves in the liquid. The parameters of these waves substantially depend on the character of motion of the interface and, consequently, on the regime of liquid crystallization (solid melting). To evaluate the parameters of acoustic waves, we will consider, as an example, the regime of liquid crystallization at which the initial temperature of the liquid would be equal to the temperature T_0 of stable equilibrium of the liquid–crystal phases and the temperature of the growing-crystal surface would be maintained constant and equal to $T_s < T_0$. Then, as is known [9], the velocity of the crystallization front of the liquid in the chosen coordinate system is determined by the formula

$$\dot{\xi}(t) = -\alpha \frac{\sqrt{\chi}}{\sqrt{t}}\,, \tag{12}$$

where

$$\alpha = \left[c \frac{T_0 - T_s}{2L} \right]^{1/2}.$$

Substituting (12) into (11) and using the asymptotic formulas for the Fresnel integrals, which appear upon the expansion of the integrals (8) and (9), we will have

$$p(x,t) = -\frac{\beta}{Kh} \left[\frac{\chi_1 c_1 \xi (T_0 - T_s)}{aL} \right]^{1/2} \sum_{n=1}^{\infty} \frac{(-1)^n}{\sqrt{n}} \left(1 - \frac{\gamma_n^2}{\omega_n^{*2}} \right)^{3/2} \exp(-\gamma_n t) \times$$

$$\times \left[\left(1 + \frac{\gamma_n^2}{\omega_n^*} \right) \sin \omega_n^* t + \left(1 - \frac{2\gamma_n}{\omega_n^*} - \frac{\gamma_n^2}{\omega_n^{*2}} \right) \cos \omega_n^* t \right] \exp(-\gamma_n t)\,. \tag{13}$$

For the absolute majority of liquids the ratio $\gamma_n/\omega_n^* << 1$; consequently, $\omega_n^* \approx \omega_n$, i.e., due to the liquid viscosity the oscillation frequency of the acoustic waves changes insignificantly. The oscillation amplitude of individual harmonics decreases according to the law $p_n \sim \exp(-\gamma_n t)$. For water, for example, $\gamma_n = \nu\nu_n^2/8\pi^2 a^2 \sim 10^{-14}\nu_n^2$ sec^{-1}, where ν_n is the oscillation frequency of the n-th harmonic. It is seen that oscillations with frequencies $\nu_n \sim 10^6$ Hz will damp very slowly, in practice.

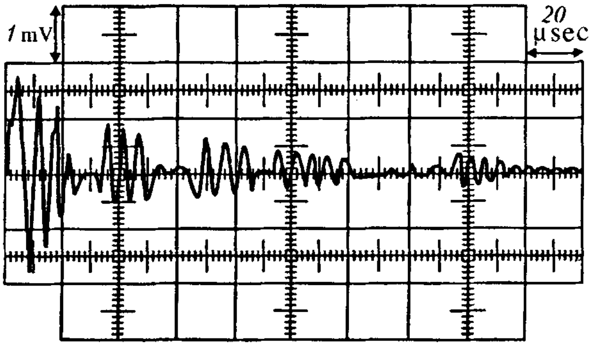

Fig. 2. Typical form of the acoustic signals observed during crystallization of substances.

The main contribution to $p(x, t)$ is made by the amplitude of the fundamental mode

$$p_{\max} \approx \frac{\beta}{Kh}\left[\frac{\pi\chi_1 c_1 \xi (T_0 - T_s)}{aL}\right]^{1/2} \exp(-\gamma_n t) . \tag{14}$$

For water at $h = 0.3$ m and $T_0 - T_s = \Delta T = 20$ K the preexponential factor in (14) is ~10^3 Pa. With allowance for the influence of the gate at the right end of the tube on the liquid-crystallization rate, this value of $p_{\max}$ decreases by a factor of 3–4.

Figure 2 shows one of the characteristic recordings of acoustic signals that accompany the process of water crystallization in the tube. The analysis of this representation shows that the theoretical value of $p_{\max}$ is in good agreement with experimental data. However, the values of oscillation frequencies show substantial disagreement between theory and experiment. The fundamental frequency of standing waves $\nu_1 \sim a/\xi$ formed in the liquid in the case considered above is lower by 1–2 orders of magnitude than the characteristic frequencies of (10^5–10^6) Hz which take place in the experiment. Moveover, substantial disagreement between theory and experiment is also observed in the oscillation picture itself.

The oscillations depicted in Fig. 2 usually occur at amplitude modulations in oscillatory systems with resonance. For the resonance phenomena to occur it is necessary that periodic forces act on the liquid or that the liquid boundary perform periodic motion. The latter will be observed in the case of a stepwise growth of the solid phase. In this case, the frequency of the steps (jumps) must be of the same order as the characteristic frequencies of the acoustic waves in crystallization and melting of the substance.

Thus, it follows from the calculations made that the high-frequency oscillations which are revealed in crystallization (melting) of the substance, cannot arise inside the liquid in smooth motions of the interface. The occurrence of the high-frequency oscillations is associated either with the stepwise motion of the liquid–crystal interface or with the influence of high-frequency oscillations of the medium with which the liquid is in contact. In both cases, large oscillation amplitudes of the order of 10^2–10^3 Pa can occur only under resonance conditions.

Here we restrict ourselves to consideration of the first possible mechanism of occurrence of the acoustic effect of crystallization.

We assume that the crystal–melt interface moves as is shown in Fig. 3a. The crystallization front performs a number of successive jumps of the same duration $\Delta\tau$; then it is at rest within the interval $\tau_0 - \tau_1$. In this interval a sufficient amount of heat is removed from the crystallization front; then the process is repeated, and so on. Here, the function $\xi(t)$ can be prescribed analytically in the form

$$\dot{\xi}(t) = \begin{cases} \vartheta_0 \sin^2 \omega_0 t & \text{at } \ 0 < t < \tau_1 \ , \\ 0 & \text{at } \ \tau_1 < t < \tau_0 \ , \end{cases}$$

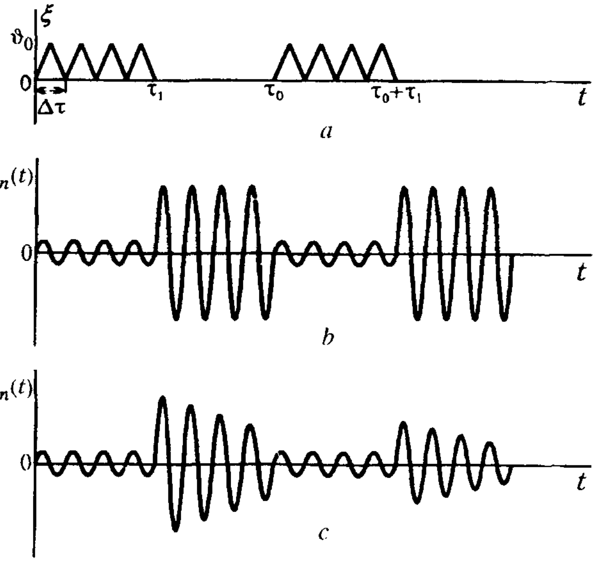

Fig. 3. Form of the signals near resonance frequencies obtained theoretically: a) of the input signal; b) of the output signal without allowance for viscosity; c) same with allowance for viscosity.

where $\omega_0 = \pi/\Delta\tau$ and $\vartheta \sim \delta_0/\tau_0$ (δ_0 is the mean linear dimension of molecular complexes incorporated into the crystalline structure). As for the order of magnitude, $\delta_0 \sim d\sqrt{L_1/L}$, here d is the diameter of the atoms or molecules and L_1 is the specific heat of evaporation of the substance. In evaluative calculations, it can be assumed that $\delta_0 \sim 10^7$ cm.

In the experiments, ϑ_0 was $(10^{-3}$–$10^{-2})$ cm/sec, to which $\tau_0 \sim 10^{-6}$–10^{-5} sec corresponds.

The ratio $\tau_1/\Delta\tau$ gives the number of maxima (flash-ups) in a wave package. A comparison of Fig. 3a with the picture of the flash-ups in Fig. 2 reveals that this ratio changes from 3 to 7.

To simplify further calculations, we will neglect the liquid viscosity. Then instead of Eq. (6) we will have

$$\ddot{\vartheta}_n + \omega_n^2 \vartheta_n = f_n(t)\,. \tag{6'}$$

In (6′) the function $f_n(t)$ is periodic with period τ_0. A solution of this equation must also be periodic. As is known [10], it can be found in the form

$$\vartheta_n(t) = \frac{C_n \cos \omega_n (t + \tau_0/2) + S_n \sin \omega_n (t + \tau_0/2)}{2\omega_n \sin(\omega_n \tau_0/2)} + $$

$$+ \frac{1}{\omega_n} \int_0^t f_n(\tau) \sin \omega_n (t - \tau)\, d\tau\,, \tag{15}$$

where

$$C_n = \int_0^{\tau_0} f_n(\tau) \cos \omega_n \tau\, d\tau\,; \quad S_n = \int_0^{\tau_0} f_n(\tau) \sin \omega_n \tau\, d\tau\,.$$

The integral in the right-hand side of (15) is calculated for two time intervals, i.e., $[0, \tau_1]$ and $[\tau_1, \tau_0]$.

After the corresponding calculations we arrive at

$$\vartheta_n(t) = (-1)^{n+1}\frac{2\beta\xi\vartheta_0}{\pi n h\omega_n}\left[\frac{C'_n\cos\omega_n(t+\tau_0/2)+S'_n\sin\omega_n(t+\tau_0/2)}{2\sin(\omega_n\tau_0/2)} - \right.$$

$$\left. - \left\{\begin{array}{l} a_n\sin\omega_n t - b_n\sin 2\omega_0 t \quad \text{at} \quad 0\le t<\tau_1 \\ \sin(\omega_n\tau_1/2)\cos\omega_n(t-\tau_1/2)+a'_n\cos[\omega_n(t-\tau_1/2)-\omega_0\tau_1]- \\ -b'_n\cos[\omega_n(t-\tau_1/2)+\omega_0\tau_1] \quad \text{at} \quad \tau_1<t<\tau_0 \end{array}\right.\right], \tag{16}$$

where

$$C'_n = \sin^2\frac{\omega_n\tau_1}{2} - \frac{\sin^2[(\omega_n/2)-\omega_0]\,\tau_1}{2(1-2\omega_0/\omega_n)} - \frac{\sin^2[(\omega_n/2)+\omega_0]\,\tau_1}{2(1+2\omega_0/\omega_n)};$$

$$S'_n = \frac{1}{2}\sin\omega_n\tau_1 - \frac{\sin(\omega_n-2\omega_0)\,\tau_1}{4(1-2\omega_0/\omega_n)} + \frac{\sin(\omega_n+2\omega_0)\,\tau_1}{4(1+2\omega_0/\omega_n)};$$

$$a_n = 1+\frac{\omega_0/\omega_n}{1-(2\omega_0/\omega_n)^2}; \quad b_n = \frac{1}{2[1-(2\omega_0/\omega_n)^2]};$$

$$a'_n = \frac{\cos[(\omega_n/2)+\omega_0]\,\tau_1}{2(1+2\omega_0/\omega_n)}; \quad b'_n = \frac{\cos[(\omega_n/2)-\omega_0]\,\tau_1}{2(1-2\omega_0/\omega_n)}.$$

Thus, at $\omega_n = 2\omega_0$ resonance is observed. Far from resonance the terms entering (16) are small.

At $\omega_n \approx 2\omega_0$, we have

$$\vartheta_n(t) \approx (-1)^{n+1}\frac{4\beta\xi\vartheta_0}{\pi n h\omega_n}\cos\omega_n(t+\Delta t) \quad \text{at} \quad 0<t<\tau_1,$$

$$\vartheta_n(t) = (-1)^{n+1}\frac{\beta\xi\vartheta_0}{\pi n h(\omega_n-2\omega_0)}\cos[\omega_n(t-\tau_1/2)+\omega_0\tau_1] \quad \text{at} \quad \tau_1<t<\tau_0, \tag{17}$$

where

$$\Delta t = \frac{\tau_0}{2} + \arctan\frac{C'_n}{S'_n}.$$

In Fig. 3b a plot of the function $\vartheta_n(t)$ is depicted schematically. In the interval $[0, \tau_1]$ the system performs weak sine oscillations with the frequency $\omega_n \sim 2\omega_0$, while in the interval $[\tau_1, \tau_0]$ it performs harmonic oscillations of the same frequency but with a large resonance amplitude. These oscillations slightly resemble trains of waves.

The functional dependence between the intervals τ_1 and τ_0 can be established in the following way. The amount of heat released at the crystallization front in one series of jumps is equal to $Q = L\vartheta_0\tau_1$; it must be removed from the crystallization zone before the next series of jumps, i.e., in the interval $\tau_0 - \tau_1$. Thus, the relation

$$L\vartheta_0\tau_1 = \lambda_1\left|\frac{\Delta T}{\Delta X}\right|(\tau_0-\tau_1),$$

must be fulfilled, where λ is the thermal conductivity of the crystal and $\tau_0 - \tau_l$ is the duration of the signals (flash-ups). Whence we obtain

$$\frac{\tau_l}{\tau_0 - \tau_l} = \frac{\lambda_l}{L\vartheta_0} \left| \frac{\Delta T}{\Delta X} \right| . \tag{18}$$

In the case of water crystallization, $L = 335 \cdot 10^3$ J/kg and $\lambda = 2.22$ J/(m·sec·deg). At a velocity of the crystallization front of 1 mm/min, $\vartheta_0 \approx 2 \cdot 10^{-3}$ cm/sec. Then at $\Delta T / \Delta X = 20$ deg/cm, according to formula (18) $\tau_l / (\tau_0 - \tau_l) = 0.7$, which is quite acceptable.

The resonance value of the pressure amplitude is

$$p_{\max} = \frac{\beta \vartheta_0}{hK(\omega_n - 2\omega_0)} . \tag{19}$$

As is known [7], due to the influence of viscosity in resonance

$$\frac{1}{\omega_n - 2\omega_0} = \frac{a^2}{2\nu\omega_0^2} = \frac{a^2}{8\nu\pi^2\nu_0^2} .$$

Moreover, $1/K = \rho a^2$. Then equality (19) acquires the form

$$p_{\max} \approx \frac{\rho_0 \beta \vartheta_0 a^4}{8\pi^2 h\nu\vartheta_0^2} .$$

Whence for water at $\vartheta \approx 10^{-2}$ cm/sec, $h = 0.1$ m, and $\nu_0 = 10^6$ sec^{-1} we obtain $p_{\max} = 5 \cdot 10^3$ Pa.

With allowance for a small damping the picture of oscillations in resonance will look as shown in Fig. 3c. Thus, the considered model of motion reproduces to some extent the picture of acoustic waves of crystallization photographed from the oscillogram by a movie camera: the occurrence of the high-frequency oscillations is explained, and the peak values of the oscillation amplitude are in good agreement with the experimental data.

Furthermore, the considered model of substance crystallization makes it possible to provide a qualitative picture of the effect of ultrasound on the crystallization process.

The molecular complexes (blocks) that form the melt perform oscillatory motions relative to certain centers or axes. Under the action of ultrasonic oscillations with a frequency coincident with the natural frequency of oscillations of these complexes, the latter strongly swing, thus facilitating their jump to the surface of the growing crystal; furthermore, large molecular blocks disintegrate into smaller ones. As a result of "spillage," the molecular blocks on the crystal surface are packed more tightly and uniformly.

A considerable drawback of the model under consideration is the fact that it is too idealized and extremely artificial. It is difficult to imagine that the layers of a crystal with a thickness of the order of the molecular complexes are strictly periodically added to the crystal surface, although on the average the possibity of some periodicity in formation of flat layers on the crystal surface is not ruled out.

In the next paper we will consider the second possible mechanism of occurrence of the acoustic effect of crystallization.

NOTATION

$\Delta T / \Delta X$, temperature gradient; $x = \xi(t)$, crystal–liquid interface; $u(x, t)$, longitudinal displacement of the liquid particles; a, velocity of sound in the liquid; ρ, liquid density; ν, kinematic viscosity of the liquid; t, time; β, volume expansion coefficient of the liquid; h, length of the crystallization cell; p, pressure in the acoustic wave; χ, thermal diffusivity of the crystal; c, heat capacity of the crystal; L, specific heat of liquid crystallization; T_0, temperature of stable equilibrium of the liquid–crystal phases; T_s, surface temperature of the growing crystal; ϑ_0, mean velocity of the crystallization front; ν_n, oscillation frequency of the n-th harmonic; $\xi(t)$, velocity of the crystallization front; τ_1, duration of the series of successive jumps of the crystallization front; $\Delta\tau$, duration of one jump; τ_0, repetition period of the jumps; ω_n, oscillation frequency of the liquid particle; $\omega_0 = \pi/\Delta\tau$. Subscripts and superscripts: m and n, numeration of the quantities; s, surface; point above a symbol, first derivative with respect to time; two points, second derivative with respect to time, etc.

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