DIFFUSION OF INDIUM ALONG [001] TWIST BOUNDARIES IN TIN: CONCENTRATIONAL β–γ' PHASE TRANSITION ON GRAIN BOUNDARIES

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Summary

Bulk and grain boundary diffusion of indium has been studied in the vicinity of the β–γ' transition in the Sn–In system. On [001] twist boundaries near the coincidence misorientation $\Sigma 17$ the β–γ' transition temperature is essentially lower than that in the bulk and depends on the misorientation angle. The observed decrease in the phase transition temperature on grain boundaries is explained in terms of the prewetting transition on the boundaries.

1. Introduction

Phase transitions on grain boundaries still attract much attention. Computer simulation of the grain boundary structure shows that the structure may depend both on the temperature and on the impurity content [1–7].

The "special grain boundary–general grain boundary" phase transition was first observed experimentally by Aleshin *et al.* [8] during studies on diffusion of nickel along the grain boundaries in copper in the vicinity of the coincidence misorientation $\Sigma 5$. The possibility of special grain boundary–general grain boundary transition with varying temperature and/or misorientation angle was analysed in detail by Shvindlerman and Straumal [9]. Lojkowski *et al.* [10] showed that the number of low energy special grain boundaries near the coincidence misorientations actually increased with decreasing temperature. In ref. 11 the region of existence of special boundaries near the coincidence misorientation $\Sigma 17$ in tin was experimentally constructed. Rabkin *et al.* [12] established how the surface-active impurity alters the temperature of the "special boundary–general boundary" phase transition in tin. Sickafus and Sass [13] showed experimentally that an increase in the impurity concentration in grain boundaries gives rise to a structural phase transition.

These experimental data and the computer simulation results pertain to phase transitions occurring only in grain boundaries and not followed by any phase transformations inside the grains which form the boundary. However, of equal interest are the grain boundary phase transitions occurring near the bulk phase transitions.

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In these experiments, however, the object of the investigations, *i.e.* the grain boundary with definite crystallographic parameters, must exist.

Therefore, these experiments involve second order bulk phase transitions or first order "weak" transitions, close to second order ones, with a small thermal and bulk effect. First order weak transitions are not usually accompanied by strong restructuring of the crystalline lattice; in the process of transition the atoms are shifted by a spacing smaller than the interatomic spacing. These transitions may, under certain conditions, proceed like second order transitions, without the formation and growth of nuclei. On the external surface near the weak first order phase transitions one may observe prewetting phenomena [14–16].

2. Experimental technique: Sn–In phase diagram

In order to study the bulk phase transitions in grain boundaries, we have taken the Sn–In system. From the data [17–19] the region of the solid solution of indium in β-tin goes as far as 6–8 at.%In with $T < 150$–170 °C, and between 10 and 23 at.%In there is the γ-phase with a simple hexagonal lattice. The γ-phase is formed according to a peritectic or a peritectoid reaction using different data: 75 °C [20], 250 °C [17], 224 °C [18, 19]. The width of the tin-based solid solution region, cited in these works, is unusually large: in the other tin-based systems the solubility limit drops rapidly at temperatures below 170–150 °C and does not exceed several tenths or hundredths of a percent [21]. Giessen [22] reports the existence at room temperature of the γ'-phase, the homogeneity region of which is located on the Sn–In diagram between the γ-phase and the β-tin-based solid solution. The γ'-phases with analogous structure also exist in the In–Bi and Tl–Sn systems [22] and the γ-phases in the Mg–Sn and Cd–Sn systems [23]. The position of the γ-phases on binary diagrams of B metals is determined by their electron concentration (3.9 electrons atom^{-1}). A simple hexagonal lattice of the γ-phase is formed from the β-tin structure by displacement of sublattices by a quarter-period along the c axis [24]. In the γ'-phase lattice the same displacement occurs by a less than a quarter-period along the c axis [22], the elements of the γ'-phase crystalline lattice remaining the same as in β-tin. Straumal *et al.* [25] determined the temperature dependence of the coefficient of bulk diffusion in the In–Sn system within the concentration range 1–8 at.%In. This temperature dependence exhibits two portions with different activation energies. On the high-temperature portion the activation energy agrees well with the published data on diffusion of isotopes, whereas on the low-temperature portion it is approximately twice as low. A drastic change in the activation energy was not followed by a noticeable jump in the diffusion coefficient. This is a characteristic feature of the second order phase transition or the first order transition close to second order transition.

Therefore, in the first part of our work we determined more accurately the Sn–In phase diagram in the range of high concentrations of tin. For this we determined the homogeneity regions of β-, γ- and γ'-phases by means of an analysis of the concentration distributions in the diffusional layers formed at the interdiffusion of tin and indium.

To investigate the grain boundary properties in the vicinity of the bulk phase transition, we selected [001] twist boundaries. As in the β–γ' transition, the atoms are displaced along the tetragonal [001] axis, whereas in the twist boundary plane with the [001] rotation axis the mutual location of the crystal lattice sites is unchanged. Bicrystals with misorientation angles of 27.6°, 28.1°, 29.1°, 30.2° and 31.1° (±0.5°) were grown. These angles are near the coincidence misorientation $\Sigma 17$. We have studied the "special grain boundary–general grain boundary" transition in the vicinity of the coincidence misorientation $\Sigma 17$ on tilt boundaries in tin [11]. It takes place at temperatures close to melting temperatures (190–215 °C), and the region of existence of $\Sigma 17$ special boundaries goes from 26° to 30°. Hence, four out of five twist boundaries are special at $T < 190$ °C, whereas a boundary with the misorientation angle of 31.1° is a general boundary. In order to study the behaviour of grain boundaries in the vicinity of the β–γ' transition in Sn–In alloys, we investigated the diffusion of indium on these twist boundaries in tin bicrystals.

Mono- and bi-crystals of tin were grown from tin grade OB4-0000, with a nominal purity of 99.9999 at.% Sn, by means of directed crystallization in a high-purity argon atmosphere. Very high purity graphite boats were employed for crystal growth. The misorientation parameters were assigned by monocrystalline seed crystals in the process of growing. These seed crystals were oriented by means of a laser light source directly on an electric-spark cutting machine [26]. The indium layer was electrolytically deposited on the surface of mono- and bi-crystals of tin. The layer thickness was 100–200 μm. The diffusional anneals were conducted in a high-purity argon atmosphere in a special furnace consisting of a thermostat and 12 small furnaces in which different temperatures were maintained [27]. The indium concentration distribution in the diffusional zones was determined by means of electron microprobe analysis from the intensity of the In Lα line at an accelerating voltage of 15 kV. The coefficients of the bulk diffusion D of indium in the alloy were determined from

$$\frac{C}{C_0} = 1 - \operatorname{erf}\left(\frac{Y}{2}(Dt)^{0.5}\right) \tag{1}$$

where C is the concentration of indium (diffusion into a semi-infinite sample via the surface with a constant concentration), Y is the diffusion distance and t is the annealing duration. The product of the grain boundary diffusion coefficient D' and the boundary width δ was determined by Fisher's method. The validity of utilization of Fisher's formula as an asymptotic form of Whipple's solution was analysed by means of LeClaire's criteria [28]. The two-phase regions on the Sn–In diagram were determined by abrupt changes on the concentration curves in the zone of the interdiffusion of tin and indium.

3. Results

Figure 1 presents the Sn–In diagram within the concentration range from 0 to 40 at.% In. An analysis of one- and two-phase regions in the zones of interdiffusion

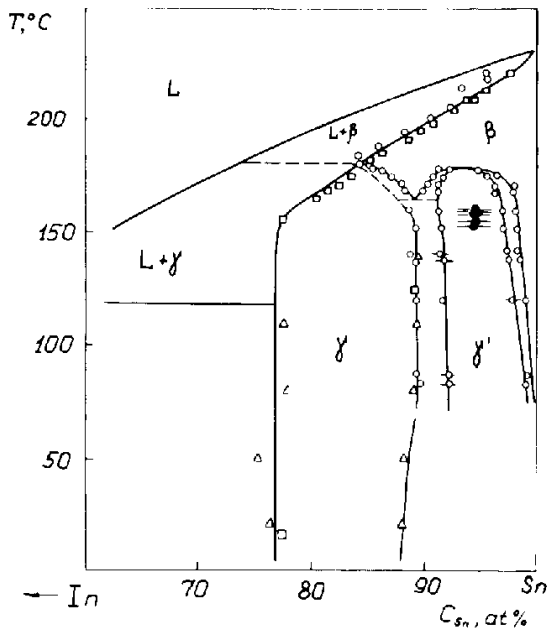


Fig. 1. Portion of the β–γ' diagram. (○) results of this work obtained from the concentration distribution in the region of bulk diffusion of indium in tin; (□) differential thermal analysis data [19]; (△) X-ray structural analysis data in a high-temperature chamber [18]; (●) β–γ' transition temperature on grain boundaries (see Figs. 4 and 5).

of tin and indium enabled us to plot the boundaries of β, $\beta + \gamma'$ and $\gamma' + \gamma$ regions. The diagram also demonstrates the experimental points from refs. 18 and 19 obtained by means of differential thermal analysis and X-ray structural studies in a high-temperature chamber. Our data for the solidus line and the boundary of the γ and $\gamma + \gamma'$ regions are seen to agree well with the published data. The data obtained by different authors for liquidus and eutectic are practically coincident and are shown in Fig. 1 as continuous lines. The studied portion of the Sn–In diagram exhibits similarity with the other diagrams where solid solution disintegration followed by the formation of an intermediate phase with a wide homogeneity region is observed: Fe–Ir (phase with about 40 at.% Ir), Fe–Ni (phase $FeNi_3$), Fe–Pd (phases FePd and $FePd_3$), V–Ga (phase V_3Ga) [21].

Figure 2 presents the temperature dependence of the coefficient of the interdiffusion of indium in tin for the range of indium concentrations from 2–4 to 8 at.% In. At a temperature $T_c = 178.7 \pm 0.5$ °C this dependence exhibits a discontinuity. Above and below T_c the diffusion coefficient obeys the Arrhenius law. The temperature T_c coincides with the temperature of γ'-phase formation from the solid solution of indium in β-tin (see Fig. 1). Approaching T_c from the region of elevated temperatures, the values of D deviate downward from the Arrhenius straight line. Approaching T_c from the side of lower temperatures, the diffusion coefficient deviates upward from the Arrhenius straight line.

Figure 3 presents a maximal concentration of indium in grain boundaries C_b as a function of depth in Fisher's coordinates. All these dependences have the form of straight lines. The coefficients of the boundary diffusion, like D values for the bulk diffusion, were determined in the concentration range from 2–4 to 8 at.% In. Figure 4 presents the temperature dependences of the product $D'\delta$ for five twist bound-

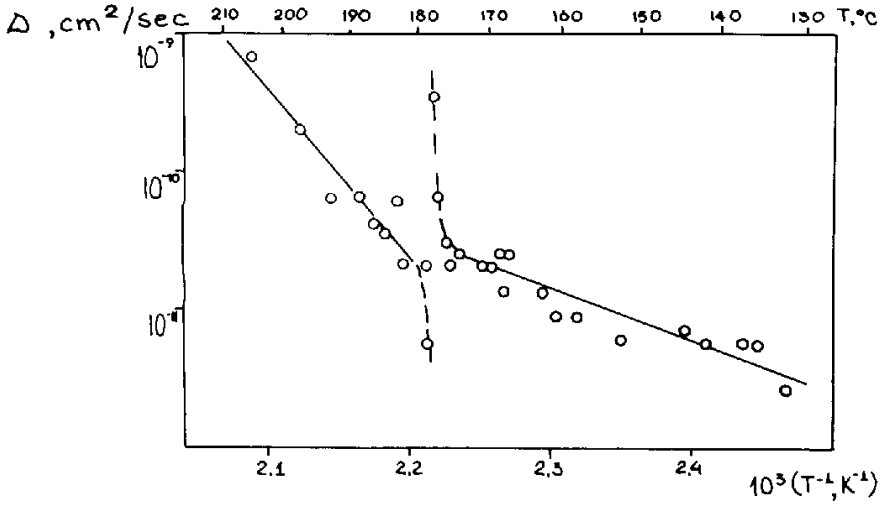


Fig. 2. Temperature dependence of the bulk diffusion coefficient of indium in the concentration range from 1 to 8 at.% In. The discontinuity on the temperature dependence at T_c corresponds to the β–γ' transition (see Fig. 1). Deviations from the Arrhenius law near T_c are seen.

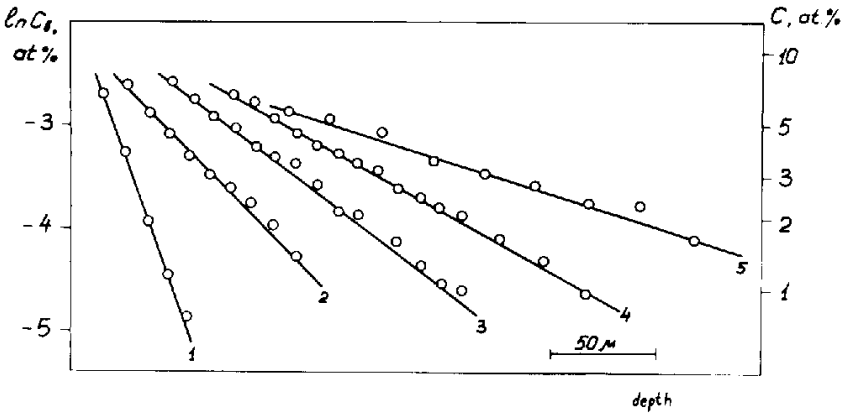
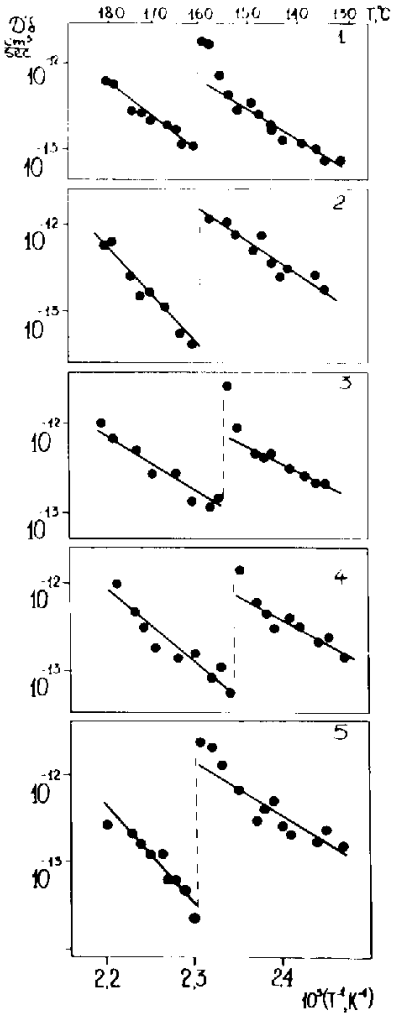


Fig. 3. Logarithm of the indium concentration on the grain boundary as a function of the depth. 1: $\varphi = 31.1°$, $T = 158.5$ °C, $t = 160$ h. 2: $\varphi = 30.2°$, $T = 175.2$ °C, $t = 160$ h. 3: $\varphi = 29.1°$, $T = 136.9$ °C, $t = 122$ h. 4: $\varphi = 27.6°$, $T = 144.8$ °C, $t = 122$ h. 5: $\varphi = 30.2°$, $T = 158.5$ °C, $t = 160$ h.

aries with different misorientation angles. Each of these dependences exhibits two regions: high- and low-temperature ones. At some temperature T_c^b the diffusion coefficient changes abruptly. Temperature T_c^b are different for different boundaries and are by 17–25 °C lower as compared with T_c for the bulk diffusion coefficient. At a temperature T_c of the bulk phase transition β–γ', where an abrupt change of D is observed, the dependences $D'\delta(T)$ for grain boundaries exhibit no singularities.

Figure 5 presents T_c^b as a function of the misorientation angle of grain boundaries. It is seen that when a misorientation angle φ deviates from the coincidence misorientation $\varphi_{\Sigma 17} = 28.1°$, the temperature drops. T_c^b decreases within the region of existence of special $\Sigma 17$ boundaries [9, 11]. Beyond this region, T_c^b increases again. Figure 5 also shows the values of the difference between the temperatures T_c in the bulk and T_c^b on the boundaries.



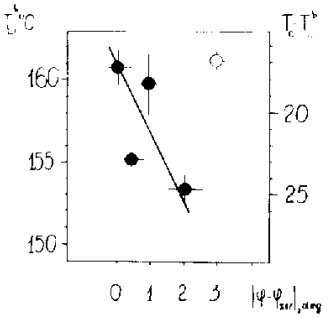


Fig. 4. Temperature dependences of the product of the grain boundary diffusion coefficient of indium D' and the boundary width δ for boundaries with different misorientation angles φ: 1, 28.1°; 2, 29.1°; 3, 27.6°; 4, 30.2°; 5, 31.1°.

Fig. 5. Dependence of the β–γ' transition temperature T_c^b on the grain boundaries on the misorientation angle φ. T_c is the β–γ' transition temperature in the bulk. (●) boundaries lying within the region of existence of special boundaries $\Sigma 17$, (○) a general boundary, $\varphi_\Sigma = 28.1°$ ($\Sigma 17$).

4. Discussion

A comparison of Figs. 1 and 2 shows that the formation of the γ'-phase from the β-tin-based solid solution is accompanied by a discontinuity in the temperature dependence of the bulk diffusion coefficient of indium and a decrease in the activation energy of diffusion. The discontinuities in the temperature dependences of the boundary diffusion coefficients are displaced from the bulk value of T_c towards lower temperatures and at T_c no singularities are seen on $D'\delta(T)$. In our opinion this suggests that the β–γ' transition on the grain boundaries occurs at a lower temperature compared with the bulk. In Fig. 1 the points in the homogeneity region of the bulk γ'-phase indicate the temperatures of the grain boundary β–γ' transition for the boundaries studied. It follows from the Gibbs rule for grain boundary phase transitions in two-component systems that the two-phase region $\beta + \gamma'$ is trans-

formed to a line on the grain boundary phase diagram. In Fig. 1 these lines are drawn through each point designating the temperature of the grain boundary β–γ' transition. In the studied range of concentrations the lines of the grain boundary phase equilibrium are nearly horizontal: with a small decrease in temperature (approximately 0.5 °C) the diffusion coefficient increases sharply as a result of transition from the grain boundary β-phase to the grain boundary γ'-phase. Should these lines curve noticeably, some of Fisher's straight lines (see Fig. 2) would exhibit two differently sloping portions at temperatures slightly below T_c^b. Therefore, the β–γ' phase transition on grain boundaries precedes the transition in the bulk. What causes this behaviour? How can one explain the observed dependence of the grain boundary transition temperature on the misorientation angle? In our opinion there is a certain analogy between the grain boundary phase transition, observed in this work, and so-called prewetting phase transitions [15]. We shall consider adsorption of liquid or gas on a solid substrate. If we approach the line of coexistence of gas and liquid on the phase p–T diagram from the side of the gas phase and the temperature is low enough, then there is a thin layer of a liquid-like phase of a microscopic thickness l. Let us go along the coexistence curve in the direction of high values of p and T. The l value will increase in this case. At some temperature T_w l becomes infinite. If this growth is continuous then we are dealing with critical wetting, and if l changes abruptly at T_w from a finite value to infinity then this is a first order wetting transition. This infinite abrupt change of the l value during the motion along the phase coexistence line at T_w is an ultimate form of abrupt changes of a finite quantity observable in the one-phase region of the p–T diagram. This phenomenon, *i.e.* when the thickness l of the liquid-like layer on the substrate increases abruptly, is called a prewetting transition. A phenomenological description of these transitions was first done by Cahn [14]. These presentations also hold for phase transitions of solids (*e.g.* see the review by Dietrich [16]). If at some temperature T_c the order–disorder transition takes place in the crystal bulk then the disordered phase wets the "crystal–vacuum" interphase as it approaches T_c. If the wetting transition occurs as a first order phase transition, then in this system one can also observe prewetting transitions. The experimental results in this field are, however, meagre [29–32]. We assume that in the system in question, near the bulk phase transition β–γ' on the grain boundaries, the β-phase can be abruptly transformed to the γ'-phase. In our discussion we shall use Cahn's presentations [14].

We shall consider a solid near a weak bulk phase transition of first order. We shall describe it by means of a scalar order parameter η. The quantity $\eta = 0$ corresponds to a high-temperature disordered phase and the quantity $\eta = \eta_v$ to a low-temperature phase. Let η_s be the quantity of the order parameter in the atomic layers directly adjoining the grain boundary. At a temperature lower than the bulk transition temperature T_c η_s is, generally speaking, not coincident with η_v owing to the disturbing action of the boundary. The corresponding contribution to the free energy of the system is given by some function of η_s. In this presentation our problem is equivalent to that in Cahn's model [14]. It should be borne in mind only that the η_s value is closely connected with the boundary structure. Correspondingly, any variation in η_s is connected with a boundary structure variation. Under appro-

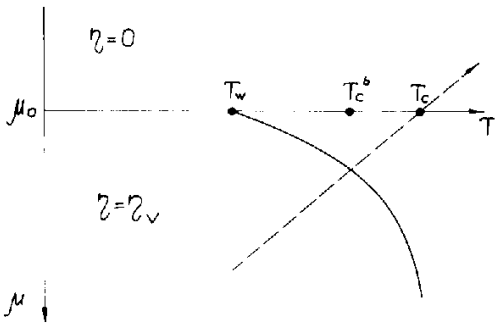


Fig. 6. Schematic presentation of the $\mu-T$ phase diagram of the Sn–In system. The dashed line shows a change in the chemical potential of tin atoms with increasing temperature (p-constant). The temperature T_c corresponds to the $\beta-\gamma'$ transition in the bulk. The temperature T_c^b corresponds to the prewetting transition on the boundary. T_w is the wetting temperature.

priate conditions the prewetting transition occurs in this system. This transition involves the η_s value decreasing abruptly. Figure 6 shows schematically the phase diagram analogous to that in the work by Dietrich [16] for gas adsorption on the substrate. It is seen that at a temperature $T_c^b < T_c$ η_s decreases in an abrupt manner and the grain boundary structure changes.

Therefore, such a simple analogy already suggests that phase transitions, similar from the thermodynamical view point to prewetting transitions, can occur on grain boundaries. However, the bulk phase transitions are not always preceded by the grain boundary transitions. There are two important limitations inherent in this model. Firstly, the phase transition must have a small thermal effect, *i.e.* it must be close to a second order transition. Only in this case can one use continual approximation. Secondly, an alteration of the order parameter at the phase transition need not affect the mutual arrangement of crystal-lattice sites of two grains in the boundary. Only in this case may the contribution of the boundary to the free energy of the system be present in a quadratic form, as in the work by Cahn [14].

Both these conditions are likely to be fulfilled for the $\beta-\gamma'$ transition in the Sn–In system. Firstly, the thermal effect of the $\beta-\gamma'$ transition is small: the boundaries of the two-phase regions ($\beta+\gamma'$) were not registered by the differential thermal analysis method in the studies of the equilibrium diagram of Sn–In, and the boundary of the γ' and $\gamma'+\gamma$ regions was interpreted as a solubility limit of indium in the β-phase [19] or it was not registered at all [18]. Secondly, during the $\beta-\gamma'$ transition one of the body-centred tetragonal sublattices is displaced along the tetragonal c axis at a spacing of less than $c/4$ [22]. The plane of a [001] twist boundary is perpendicular to the c axis along which these displacements occur. Therefore, during the $\beta-\gamma'$ transition, the geometry of crystal-lattice coincidence of two grains on the studied [001] twist boundaries does not change.

Thus, the observed abrupt changes in the temperature dependences of the grain boundary diffusion coefficient in the Sn–In system, observed by us, are attributed to the $\gamma'-\beta$ transition on the boundaries, which precedes the $\gamma'-\beta$ transition in the bulk. The small thermal effect of the transition and the geometry of the atomic displacements at the transition enable us to describe it in terms of a model analogous to that for the prewetting transition [14]. We shall consider the implications of this boundary transition model. How does the temperature T_c^b of the pre-

wetting transition on grain boundaries depend on the misorientation angle of the boundaries φ in the vicinity of the coincidence misorientation φ_Σ? How will T_c^b change on going out of the region of existence of special boundaries into the region of existence of general boundaries? It is known that at a small deviation of $\Delta\varphi$ from the coincidence misorientation φ_Σ a network of so-called secondary grain boundary dislocations (SGBDs) with a period $d = b_\Sigma/2 \sin(\Delta\varphi/2)$, b_Σ being the Burgers vector of SGBDs, is formed on special boundaries. We shall consider the simplest assumption that the core structure of SGBDs corresponds to the structure of the high-temperature grain boundary phase. Generally speaking, in this case, a two-dimensional problem $\eta = \eta(x, Y)$ must be solved. We shall, however, considerably simplify the calculations by considering the prewetting phase transition only in one grain boundary phase, irrespective of the grain volume.

The thermodynamic potential of the unit volume of the grain boundary phase as a function of the order parameter has the form shown in Fig. 7. η_s is the quantity of the order parameter in the layers adjoining the low-temperature grain boundary phase. As the temperature is increased the "thermodynamic gap" Δ is decreased, and with $T = T_c^{sp}$ it vanishes. Here T_c^{sp} is the temperature of the prewetting transition on the boundary with $\varphi = \varphi_\Sigma$. With account taken of our assumption on the core structure of SGBDs we minimize the function

$$\varphi_B = \int_0^d \left\{ \frac{1}{2} m \left(\frac{\partial \eta}{\partial x} \right)^2 + \Phi(\eta) \right\} dx \tag{2}$$

with the boundary conditions

$$\eta(0) = \eta(d) = 0; \qquad \left. \frac{\partial \eta}{\partial x} \right|_{x=0} = \left. \frac{\partial \eta}{\partial x} \right|_{x=d} = 0 \tag{3}$$

We approximate the $\Phi(\eta)$ function by the fourth order polynomial

$$\Phi(\eta) = (\eta - \eta_s)2 \left(\frac{\Delta}{\eta_s^2} + \frac{2\Delta}{\eta_s^3} \eta + \eta^2 \right) \tag{4}$$

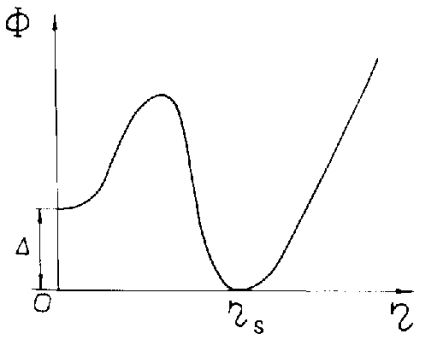


Fig. 7. Schematic dependence of the volumetric density of free energy Φ for a grain boundary on the order parameter η. At low temperatures the disordered phase ($\eta = 0$) is separated from the ordered phase by a "thermodynamic gap" Δ. As the temperature grows, Δ decreases and vanishes at the temperature of the grain boundary phase transition on the special boundary.

We seek for the solution in the form

$$\eta(x)=\begin{cases}\frac{1}{2}\eta_s\left(1-\cos\frac{\pi x}{h}\right); & 0\leqslant x\leqslant h\\ \eta_s & h\leqslant x\leqslant d-h\\ \frac{1}{2}\eta_s\left(1-\cos\frac{\pi(d-x)}{h}\right); & d-h<x\leqslant d\end{cases} \tag{5}$$

After the optimization with respect to h we obtain that with

$$\Delta=\frac{m\pi^2\eta_s^2+(m^2\pi^4\eta_s^4+\frac{35}{2}m\pi^2\eta_s^6d^2)^{0.5}}{8d^2} \tag{6}$$

the solution $\eta(x)=0$ corresponds to a minimum of the energy. With $\Delta\varphi\to 0$ $(d\to\infty)$ we have

$$\Delta T=T_c^{Sp}-T_c\approx\Delta\approx d^{-1}\approx|\Delta\varphi| \tag{7}$$

Therefore, with deviation of the misorientation angle of the grains from the coincidence misorientation φ_Σ by a small angle $\Delta\varphi$ the prewetting transition temperature on the boundary drops in proportion to $\Delta\varphi$. If $\Delta\varphi$ is such that the misorientation angle goes beyond the limits of the existence region of the given special boundary [9], then this boundary will no longer exhibit large inhomogeneities. The prewetting transition on such a boundary will occur at a higher temperature compared with a special boundary with a close misorientation angle. All of these reasons explain qualitatively our experimental dependence $T_c^b(\varphi)$.

It should be noted that although the above stated presentations are applicable only to weak first order transitions, computer simulation in terms of the lattice gas model [1] and molecular dynamic simulation [33, 34] show that at a temperature lower than the melting temperature a liquid-like phase layer is formed on the special boundary. Our model suggests that secondary grain boundary dislocations are wetted by the high-temperature phase. Then, at a certain temperature T_c, lower than the melting temperature, the SGBD network in the special boundary may disappear. It is one of the possible mechanisms of the "special boundary–general boundary" transition [9, 11, 12]. This approach explains why this transition occurs at a change of both the temperature and the misorientation angle: in either case the SGBD cores begin to overlap, in the former case owing to geometrical factors; in the latter case owing to thermodynamic causative factors.

Acknowledgments

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