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Scientific Paper 62-944-116-P6
Proprietary Class 3

December 18, 1962

THE MIGRATION OF A LIQUID ZONE THROUGH A SOLID: PART I

W. A. Tiller

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Pittsburgh 35, Pennsylvania

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Abstract

The migration of slab, cylindrical and spherical zones through a block of solid under the influence of a temperature gradient has been analyzed theoretically. The migration rate is found to be directly proportional to the temperature gradient in the system and to depend upon orientation - dependent atomic kinetics of the melting and freezing processes. The magnitude of the atomic kinetic effect depends upon the size of the molten zone. Stable zone migration occurs only in certain crystallographic orientations. The theory is discussed in terms of the limited experimental results available.

THE MIGRATION OF A LIQUID ZONE THROUGH A SOLID: Part I

W. A. Tiller

Since the initial paper by Pfann⁽¹⁾ on "Temperature Gradient Zone Melting" (TGZM), only a few publications have appeared utilizing or studying this technique.⁽²⁾⁽³⁾⁽⁴⁾ However, the work of Wernick⁽²⁾⁽³⁾ has shown that the migration rate of the zone is not primarily diffusion controlled but depends upon the zone-size and zone-orientation as well. Further, the work of Mlavsky and Weinstein⁽⁵⁾ and others indicate that this might be a useful technique for preparing small crystals of certain complex materials.

The author feels that the migration of a liquid zone through a solid under either a thermal field or an electric field driving force can be developed into a very potent technique for both the preparation of crystals and the determination of fundamental information. Therefore, the present paper is an attempt to evaluate the scope of the technique through a theoretical analysis of zone migration in a temperature gradient; in a companion paper,⁽⁶⁾ the electric field effects are treated.

Theory

The starting geometrical and thermal configurations for a slab zone in a block of solid may be as illustrated in Figure 1. The upper portion of the block consists of solid solvent and perhaps some solute. The zone region consists of a substantial alloy of solute and solvent and the lower block usually consists of a single crystal seed of the solvent.

The ensemble is placed in a temperature gradient such that the temperature of the zone region is above the lowest melting temperature, T_c , of the system, and such that the highest temperature, T_h , in the system is below the melting point of the upper or lower blocks. Under these conditions, the zone region melts and the zone adjusts its length until the zone concentration is between C_1 and C_2 (Figure 1). Reactions will occur at the two solid-liquid interfaces to produce the equilibrium concentrations C_1 and C_2 at T_1 and T_2 respectively. A solvent concentration gradient is set up in the zone and the combination of solution reaction at T_2 , diffusion in the zone and freezing at T_1 causes the molten layer to migrate through the block.

In this treatment, the direction of the maximum temperature gradient will be taken as the Z axis. The migration of slab, rod, and spherical zones, having dimensions ℓ cms, as illustrated in Figure 2 will be considered. The zones are considered to have a concentration between C_1 and C_2 and to be located at $Z = Z_0$ at time $t = t_0$. The assumptions to be made in the treatment are: (i) we are dealing with a binary or pseudo-binary system, (ii) no exchange of either solute or solvent exists between any portion of the sandwich and the gas phase, and (iii) the zone migration occurs at such a slow rate that the influence of the interface motion on diffusion in the zone can be neglected. Let us first consider the case where the atomic kinetics are isotropic so that migration occurs in the $\theta = 0$ direction.

1. Isotropic Atomic Kinetics

The motion of the zone occurs as a result of (a) interface reaction to produce local equilibrium and (b) solvent transport across the zone as a result of the solvent distribution produced by (a). Thus, expressions need to be developed only for the rate of solvent transport across the zone, this rate being properly correlated with the rates of melting and freezing at the interfaces. Further, the atomic kinetics of these interface reactions relating the reaction rate to the departure from equilibrium must be considered. We shall treat the three cases (i) uniform interface motion, (ii) layer formation by screw dislocations, and (iii) layer formation by two-dimensional nucleation. For these mechanisms, the atomic kinetics are described by the following equations⁽⁷⁾⁽⁸⁾⁽⁹⁾

$$(i) \quad V = \mu(T^* - T) = m \mu(C^* - C) \quad (1)$$

where V is the freezing velocity in the Z direction, T^* and T are the equilibrium and actual temperatures at the zone face, μ is the atomic kinetic coefficient ($1 < \mu < 10^3$ cm/sec/ $^{\circ}$ C usually), m is the liquidus slope for the solute (m is negative) and C and C^* are the equilibrium and actual solvent concentrations respectively at the zone face.

$$(ii) \quad V = \mu(T^* - T)^2 = m^2 \mu(C^* - C)^2 \quad (2)$$

with μ negative for melting and with $10^{-5} < |\mu| < 10^{-1}$ generally and for a diffuse layer edge, $|\mu|$ may be as large as unity.⁽¹⁰⁾

$$(iii) \quad V = \mu e^{-A/(T^* - T)} = \mu e^{-A/m (C^* - C)} \quad (3)$$

with μ and A negative for melting and where $|\mu|$ is the product of the interfacial area, the layer step height and the pre-exponential rate term for two-dimensional nucleation. ($10 < |\mu| < 10^4$ usually) and $|A|$ is given by

$$|A| = \frac{S \lambda \sigma_e^2 V_m T^*}{k T \Delta H} \quad (4)$$

In eq. 4, S is a geometrical factor ($S \sim \pi$), λ is the layer step height, σ_e is the layer edge surface energy, V_m is the specific volume, k is Boltzmann's constant and ΔH is the latent heat of fusion ($1 < |A| < 10^4$ usually). In Figure 3, the general equilibrium temperature-concentration relationship is given for the case where atomic kinetics have been considered.

Since the migration of the zone occurs at such a slow rate, both the temperature and solute fields in the zone are very well described, at any moment of time, by the solution to Laplace's equation. Using straightforward procedures, the temperature for all three zone shapes is found to depend only on Z . Assuming the same thermal conductivity outside the zone and constant parameters in the two phases, the temperature gradient in the zone, G , may be related to the temperature gradient in the bulk solid, G^* .

$$(i) \quad \text{Slab} \quad G = \frac{K_S}{K_L} G^* - \frac{V_m \Delta H}{K_L} \quad (5)$$

$$(ii) \quad \text{Cylinder} \quad G = \frac{2K_S G^*}{(K_S + K_L)} - \frac{V_{\pi} \Delta H}{(K_S + K_L)} \quad (6)$$

$$(iii) \quad \text{Sphere} \quad G = \frac{3K_S G^*}{(2K_S + K_L)} - \frac{V_{\pi} \Delta H}{(2K_S + K_L)} \quad (7)$$

Where V_{π} is the velocity of the freezing face, K_L is the thermal conductivity of the zone and K_S is the thermal conductivity of the solid. From Figure 4, we can see that, in the velocity range of our interest, G is practically unaffected by the magnitude of V_{π} ($V_{\pi} < 10^{-4}$ cm/sec).

The solvent concentration, C , may be readily shown to depend only on Z (since the boundary conditions depend only on Z) for all zone geometries. This is

$$C = A Z + B \quad (8)$$

where A and B are constants that vary slowly with time. Let C^1 represent the initial solvent concentration throughout the upper block of solid and let subscripts refer to the value of θ under consideration where θ refers to the outward normal to the zone, i.e. for the slab zone, $\theta = 0$ and $\theta = \pi$ refer to the upper and lower faces respectively. The conservation of solute conditions give

$$V_{\pi} (1 - k_o) C_{\pi} = -D \left(\frac{dC}{dZ} \right)_{\pi} = \frac{-D}{\ell} (C_o - C_{\pi}) \quad (9)$$

$$V_o (C_o - C^1) = \frac{+D}{\ell} (C_o - C_{\pi}) \quad (10)$$

where k_o is the phase diagram solvent partition coefficient ($k_o > 1$) and D is the solvent diffusion coefficient in the melt. We can see immediately from eqs. 9 and 10 that, since C_o and C_π are functions of time, V_o and V_π will also be time dependent. Further, since V_o and V_π will generally be slightly different in magnitude, l will be time dependent also. The ratio of V_π/V_o is

$$\frac{V_\pi}{V_o} = \frac{-1}{(1 - k_o)} \frac{(C_o - C^i)}{C_\pi} \quad (11)$$

In Figure 5, the ratio $-V_\pi/V_o$ is plotted versus $(C^i - C_o)/C_\pi$ for several values of k_o .

From eqs. 1, 2, and 3 written in terms of V_π and V_o , both C_o and C_π can be determined, i.e. $V_o = m \mu_o (C_o^* - C_o)$, $V_\pi = m \mu_\pi (C_\pi^* - C_\pi)$. Inserting $C_o - C_\pi$ into eq. 9, we find the following relationships

(i) Uniform interface advance

$$V_\pi = \frac{-[\epsilon G/m]}{\left[1 - \frac{\epsilon}{\lambda_m} \left(\frac{1}{\mu_\pi} - \frac{1}{\mu_o^*}\right)\right]} \quad (12)$$

(ii) Screw dislocation

$$V_\pi = \frac{-[\epsilon G/m]}{\left[1 - \frac{\epsilon}{\lambda_m} (V_\pi)^{-1/2} \left(\sqrt{\frac{1}{\mu_\pi}} + \sqrt{\frac{1}{\mu_o^*}}\right)\right]} \quad (13)$$

(iii) Two-dimensional nucleation

$$V_\pi = \frac{-[\epsilon G/m]}{\left[1 - \frac{\epsilon}{\lambda_m} (V_\pi)^{-1} \left(\frac{A_\pi}{\ln(\mu_\pi/V_\pi)} - \frac{A_o}{\ln(\mu_o^*/V_\pi)}\right)\right]} \quad (14)$$

where $G = (T_o - T_{\pi})/\ell$, $\epsilon = -D/(1 - k_o) C_{\pi}$ and $\mu'_o = \mu_o V_{\pi}/V_o$. Here, ϵ is almost a constant since $C_{\pi} \approx C_{\pi}^*$ which is not likely to change by more than ten per cent in a single experiment. In general, melting kinetics are much faster than freezing kinetics and we can often neglect terms containing μ'_o relative to terms containing μ_{π} . Since C_{π} varies slightly over the curved zones, these zones will become slightly elliptical with the Z-axis as the semiminor axis of the ellipse. From eqs. 12, 13, and 14, we can see that the effect of the atomic kinetics is to slow down the zone motion and make V_{π} a function of zone size, ℓ . In Figure 6, eqs. 12 and 13 have been plotted in the form of V_{π} versus ℓ for several values of the atomic kinetic parameter $\Gamma = (1/\mu_{\pi} - 1/\mu'_o)$ for eq. 12 and $\Gamma' = (1/\sqrt{\mu_{\pi}} + 1/\sqrt{\mu'_o})$ for eq. 13. We see that for uniform interface kinetics, V_{π} is linear with ℓ for small ℓ , whereas for screw-dislocation kinetics, V_{π} is sigmoidal with ℓ . In Figure 7, eq. 14 has been plotted as V_{π} versus ℓ for several values of A_{π} and μ_{π} assuming that $-A_o/\ln(\mu'_o/V_{\pi}) \ll A_{\pi}/\ln(\mu_{\pi}/V_{\pi})$. Here we see an even greater sigmoidal relationship between V_{π} and ℓ and that the atomic kinetic effects are observed at an order of magnitude larger values of ℓ .

In general, the zone length will vary with time and, utilizing an overall conservation of solvent condition, we can develop an approximate expression for ℓ . Denoting $\ell(o)$ and $\ell(t)$ as the zone lengths at $t = o$ and at t and the zone travel distance $Z(t) = \int_o^t \frac{(V_o + V_{\pi})}{2} dt$, we find

$$\ell(t) = \left\{ \frac{Z(t) \left[C^i - k_o (C_M(o) - G^* Z(t)/2m) \right] + \ell(o) C_M(o)}{C_M(o) - G^* Z(t)/m} \right\} \quad (15)$$

where $C_M(0)$ is the initial concentration of the mid-point of the zone. Since $\ell(t)$ refers to the dimensions in the Z-direction, the circular cylinder will become elliptical and the sphere ellipsoidal due to changes in ℓ with time.

2. Convection Effects

The foregoing treatment has dealt with the case of zero convective mixing in the zone. If the convective mixing were complete, then the concentration would be constant throughout the zone. In this case, we have for uniform interface advance kinetics

$$V_{\pi} = \frac{G\ell}{\left(\frac{1}{\mu_{\pi}} - \frac{1}{\mu_0}\right)} \quad (16)$$

and

$$-V_0 - V_{\pi} = \frac{d\ell(t)}{dt} \quad (17)$$

For the case of partial mixing, we must replace ℓ by $\alpha \delta_c$ in eqs. 9 and 10 where $\alpha \approx 1/2$ and δ_c is the convectionless layer thickness defined in Figure 8. In this case, eq. 12 becomes

$$V_{\pi} = \frac{- \left[\epsilon G/m \right] \left[2\ell/\delta_c \right]}{\left[1 - \frac{2\epsilon}{m\delta_c} \left(\frac{1}{\mu_{\pi}} - \frac{1}{\mu_0} \right) \right]} \quad (18)$$

From eq. 18, we can see that convective mixing enhances V_{π} and may be extremely helpful in the determination of the atomic kinetic coefficients for the cases there $-\epsilon/\ell m \mu_{\pi} \ll 1$ by making $\delta_c/\ell \ll 1$. For the other

atomic mechanisms, the appropriate equations replacing eqs. 13 and 14 may be readily derived by substituting $\delta_c/2$ for ℓ in the denominator and multiplying by $2\ell/\delta_c$ in the numerator.

3. Anisotropic Atomic Kinetics

Our interest in the effects of anisotropic atomic kinetics may be directed in two directions, (i) do they introduce microscopic shape changes into the zone contour and (ii) do they cause macroscopic shape changes in the zone contour.

When the atomic kinetics are isotropic, microscopic undulations in the zone surface contour are unlikely to occur since any changes in curvature, of either sign, will alter the equilibrium temperature in such a way as to force the surface back to its original contour. Further, no macroscopic shape changes can occur since the fastest melting direction will be the $\theta = 0$ direction ($G = \text{maximum}$ in this direction) and this is the only direction in which every element of the zone can migrate at the same velocity.

When the atomic kinetics are anisotropic, the situation is quite different than the foregoing. Anisotropic μ implies anisotropic solid-melt surface energy, γ . In fact, for any particular crystallographic system, a plane which exhibits a low value of γ generally also exhibits a low value of μ . When the anisotropy of γ is sufficient, stable microscopic facet formation will occur on certain areas of the zone surface to minimize the

free energy of the system.⁽¹¹⁾ Since the anisotropy of γ is expected to increase as the temperature difference between the zone surface and the melting temperatures of pure solvent increases, we can generally expect a large anisotropy in μ in studies of alloy zone migration. Thus, the zone surface can be expected to be composed of segments of the "equilibrium form," the size of the individual facets decreasing as G increases.

To decide whether the zone can migrate, with stable shape, in a different direction than the $\theta = 0$ direction, it is sufficient to consider a slab zone and a spherical zone moving in some other θ direction. Under the assumption that the temperature distribution remains unchanged (only true for the spherical zone), V_θ is given by eqs. 12, 13, and 14 with G , μ and ϵ replaced by $G \cos \theta$, μ_θ and ϵ_θ respectively. However, the slab zone is easily seen to be unstable since ϵ is a function of C_θ and C_θ is a function of position on the slab surface. Thus, the slab zone should break up into segments. The sphere is also unstable because the isotherms are still normal to the Z direction and must be normal to the θ direction for $V_\theta = \text{constant}$ at all points of the surface ($V_\theta = \text{constant}$ is the condition for zone shape stability).

Since the zone surface will exhibit facets of the equilibrium form faces and since the macroscopic shape is unstable if the zone moves in other than the $\theta = 0$ direction, the zone is only stable if the $\theta = 0$ direction is the outward direction - normal of a face of the equilibrium form. It must be the most stable face (most singular)⁽¹²⁾ to assure complete stability. If a cylindrical or spherical zone is constrained to

move in a plane, then a similar stability condition applies; i.e., the zone is stable if the θ direction is the outward direction - normal of one of the lines of the two-dimensional equilibrium form in this plane.

Discussion

The experimental work of Wernick⁽³⁾ on the migration of molten Al-Ge wires along a Ge surface and through a Ge block reveals a V vs ℓ relationship for this system that is best described by eq. 13. This data, which shows that atomic kinetics were important in his experiments, is reproduced in Figure 9 along with the best-fit theoretical eqs. 13 and 14. Eq. 12 was not drawn on this graph since a value of $\Gamma \approx 2 \times 10^4$ would be needed for any appreciable fit and this value is much too large to be reasonable for a uniform interface advance mechanism. Reasonable values of Γ produce a V vs ℓ dependence only in the range $\ell \sim 10^{-6} - 10^{-5}$ cm. Likewise when one investigates the value of A that one might expect for this system using eq. 4, one finds $A = 500$, a value much larger than that needed to give a possible fit in Figure 9. Reasonable values of A produce a V vs ℓ dependence in the range $\ell \sim 10^{-1}$ cms.

The growth mechanism in these experiments seem definitely to be a screw dislocation mechanism with $\Gamma' \approx 88$. The determination of this value of Γ' required the selection of $G = G^*$ and $\epsilon/m = 0.75 \times 10^{-6}$ using $T = 667^\circ\text{C}$,⁽³⁾ $m_L = -8^\circ\text{C/At\%}$,⁽¹³⁾ $C_{\text{Al}} = 50 \text{ At\%}$,⁽¹³⁾ $k_0 = 2$,⁽¹³⁾ and choosing $D = 3 \times 10^{-4} \text{ cm}^2/\text{sec}$. This value of D may seem to be slightly larger than

expected; however, it is not inconsistent with other solvent diffusion studies in liquid alloy systems.⁽¹⁴⁾ We can compare the measured value of $\mu_w \approx (2/\Gamma')^2 = 5 \times 10^{-4}$ with the theoretical value from Hillig and Turnbull's equation⁽⁸⁾

$$|\mu| = \frac{3D (\Delta S)^2}{4 \pi \gamma R T_i V_m} \quad (19)$$

where ΔS is the molar entropy, R is the universal gas constant, T_i is the temperature of the system and V_m is the molar volume. Eq. 19 gives $\mu \approx 1.5 \times 10^{-2}$ using the above value of D . The two results can only be reconciled by choosing a much smaller value of D in eq. 19 than in the evaluation of ϵ , by assuming that $G > G^*$ so that D can be reduced in ϵ , or by allowing that the layer edges may be diffuse.⁽¹⁰⁾

The effect of zone orientation on zone stability was also investigated by Wernick.⁽³⁾ His results are reproduced in Table I. In this table, the plane in which the wire is constrained to move, the wire direction in this plane and the direction of G^* max (Z-axis) has been tabulated. The direction of the densest packed lines (equilibrium-form lines) are also tabulated. It may be seen that, except for the (111): $[\bar{1}10]$ experiment, the zone is always unstable unless the $\theta = 0$ direction was the outward direction - normal of the equilibrium form. It is interesting to note that, for zone motion on the (111) plane with a $[\bar{1}10]$ wire direction, the zone should be unstable if the $\theta = 0$ direction is the $[\bar{1}12]$ direction but should be stable if the $\theta = 0$ direction is the $[11\bar{2}]$ direction. This is because the (111) plane equilibrium form is triangular rather than

hexagonal due to the "pseudo-polar" bonding nature on this plane. Wernick⁽³⁾ found that, in one case, such a wire zone was stable and this leads one to speculate as to whether the $\theta = 0$ direction was the outward or the inward direction normal in this particular experiment.

Conclusions

The migration rate of a molten zone through a slab of material generally depends upon orientation-dependent atomic kinetics of the melting and freezing processes. The magnitude of the atomic kinetic effect depends upon the size of the molten zone and this size dependence of the migration velocity may be used as a method for studying the atomic kinetics of alloy melting and freezing. The migration velocity is found to be directly proportional to the temperature gradient in the system. Stable zone migration occurs only for migration in certain crystallographic orientations in a particular system.

Acknowledgments

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Table I—The effect of zone orientation on zone stability for Al-Ge wire zones

Plane	Wire Direction	$G_{\max}^*$ Direction	Most Dense Packing Directions	Wire Stability
(111)	$[1\bar{1}\bar{2}]$	$[\bar{1}10]$	$[\bar{1}01], [01\bar{1}], [1\bar{1}0]$	Unstable - Zone moves to lie in $[\bar{1}01]$ and $[01\bar{1}]$ directions
(111)	$[\bar{1}10]$	$[11\bar{2}]$	$[\bar{1}01], [01\bar{1}], [1\bar{1}0]$	Unstable - Zone breaks up and moves to lie in $\langle 110 \rangle$ directions
(001)	$[010]$	$[100]$	$[\bar{1}10], [110]$	Unstable - Zone breaks up and moves to lie in $\langle 110 \rangle$ directions
(001)	$[\bar{1}10]$	$[110]$	$[\bar{1}10], [110]$	Stable - Zone travels as a unit in $[110]$ direction
(110)	$[001]$	$[\bar{1}10]$	$[\bar{1}10]$	Unstable - Zone breaks up and moves to lie in $\langle 112 \rangle$ directions
(110)	$[\bar{1}10]$	$[001]$	$[\bar{1}10]$	Stable - Zone travels as a unit in the $[001]$ direction

FIGURE CAPTIONS

- Figure 1. Portion of phase diagram plus the solid and liquid charge used in temperature gradient zone melting.
- Figure 2. Spatial orientation of slab, cylindrical or spherical zone.
- Figure 3. Portion of phase diagram illustrating both the equilibrium and actual temperature and solvent concentration at the zone surfaces for $\theta = 0$ and $\theta = \pi$.
- Figure 4. Thermal parameter plot for different zone shapes allowing the determination of G from a knowledge of the thermal parameters of the system and the gross experimental conditions.
- Figure 5. Plot of the relative zone-edge migration rate $-V_{\pi}/V_0$ as a function of the dimensionless concentration parameter $(C^1 - C_0)/C_{\pi}$ for several values of the solvent partition coefficient, k_0 .
- Figure 6. Plot of zone migration velocity, V_{π} , as a function of zone size, ℓ , for the diffuse interface mechanisms ($1/\ell = 1/\mu_{\pi} - 1/\mu_0$) and the screw dislocation mechanism ($1/\ell = 1/\sqrt{\mu_{\pi}} + 1/\sqrt{\mu_0}$).
- Figure 7. Plot of the zone migration velocity, V_{π} , as a function of zone size, ℓ , for the two-dimensional nucleation mechanism assuming $A_0/\ln(\mu_0/V_{\pi}) \ll A_{\pi}/\ln(\mu_{\pi}/V_{\pi})$.
- Figure 8. Solvent distribution in the zone, C , as a function of distance, Z , for the conditions of (i) infinite atomic kinetics with no mixing and (ii) finite atomic kinetics with no mixing, partial mixing and complete mixing.
- Figure 9. Fitting of Wernick's data⁽³⁾ on the migration of Al-Ge wires through Ge with eq. 13 and eq. 14 choosing best-fit parameters.

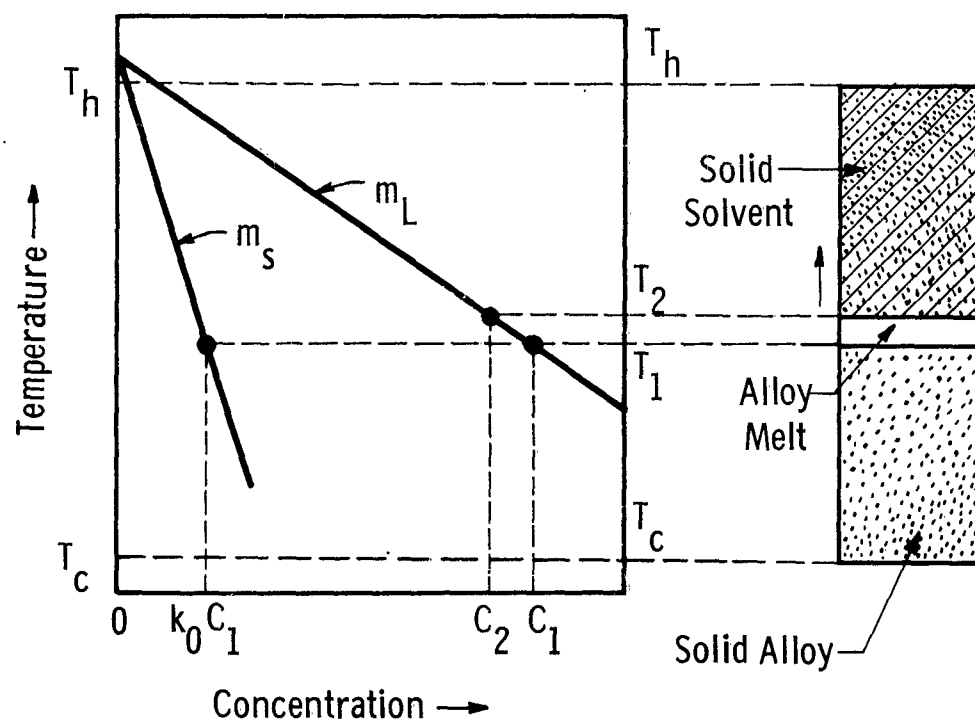


Fig. 1

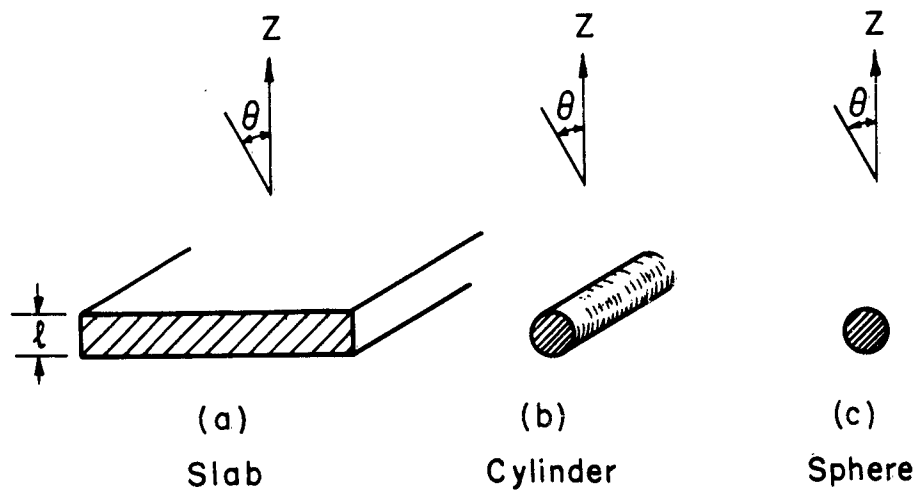


Fig. 2

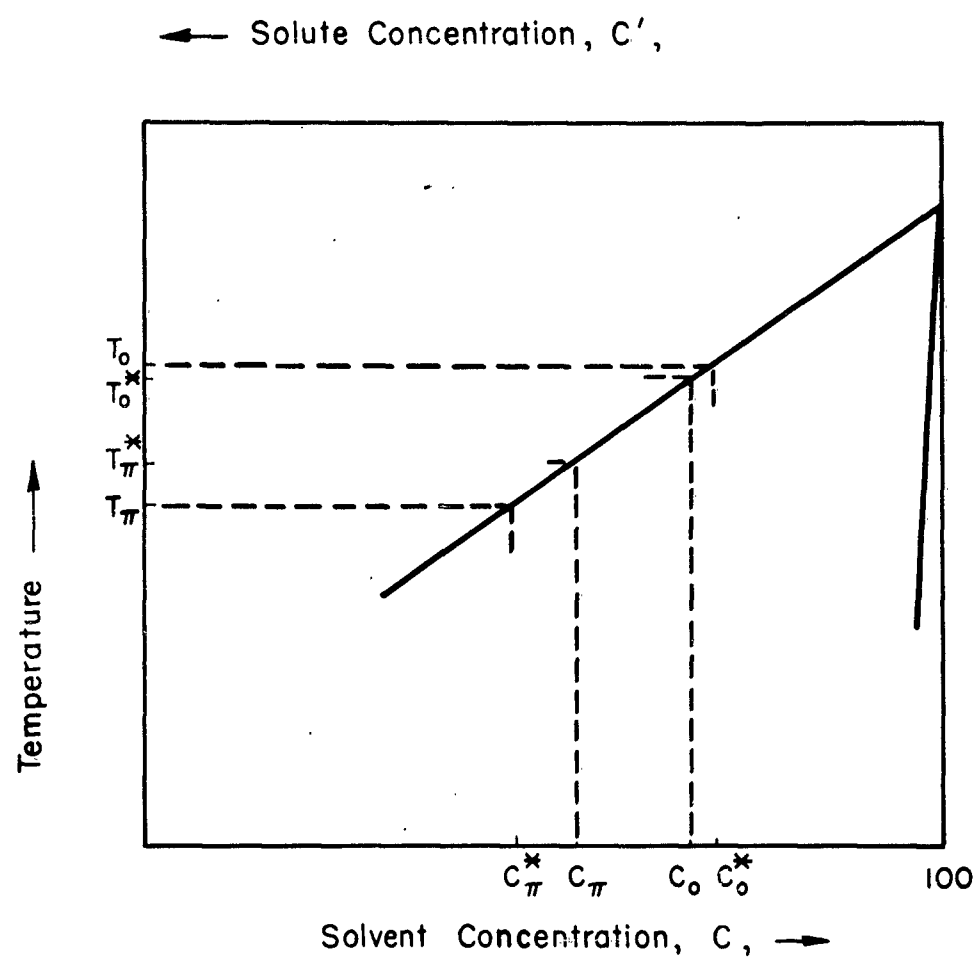


Fig. 3

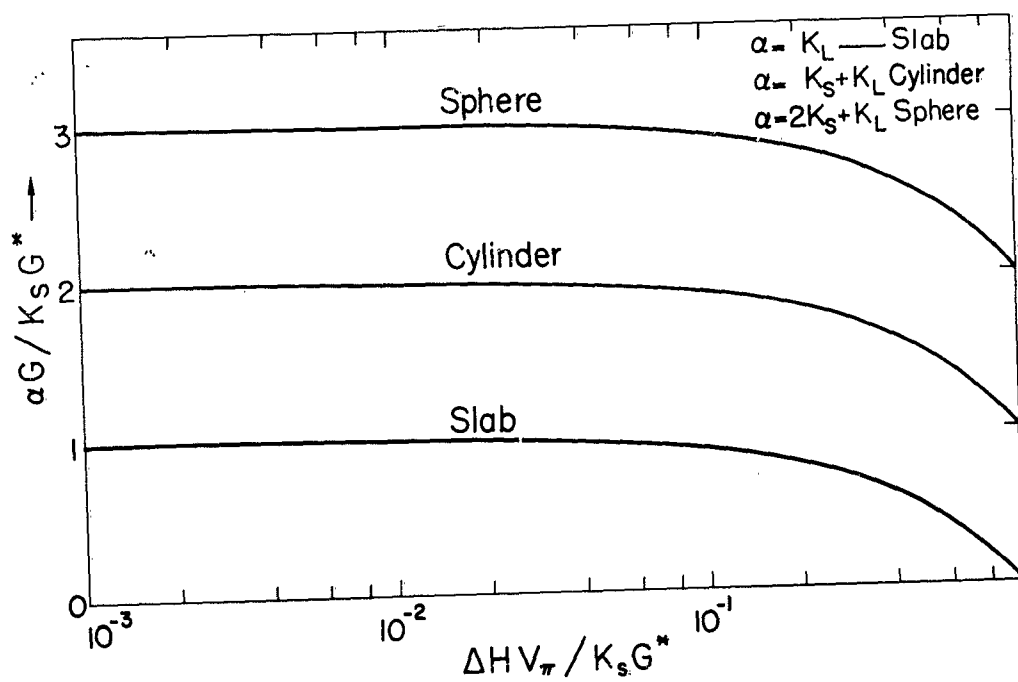


Fig. 4

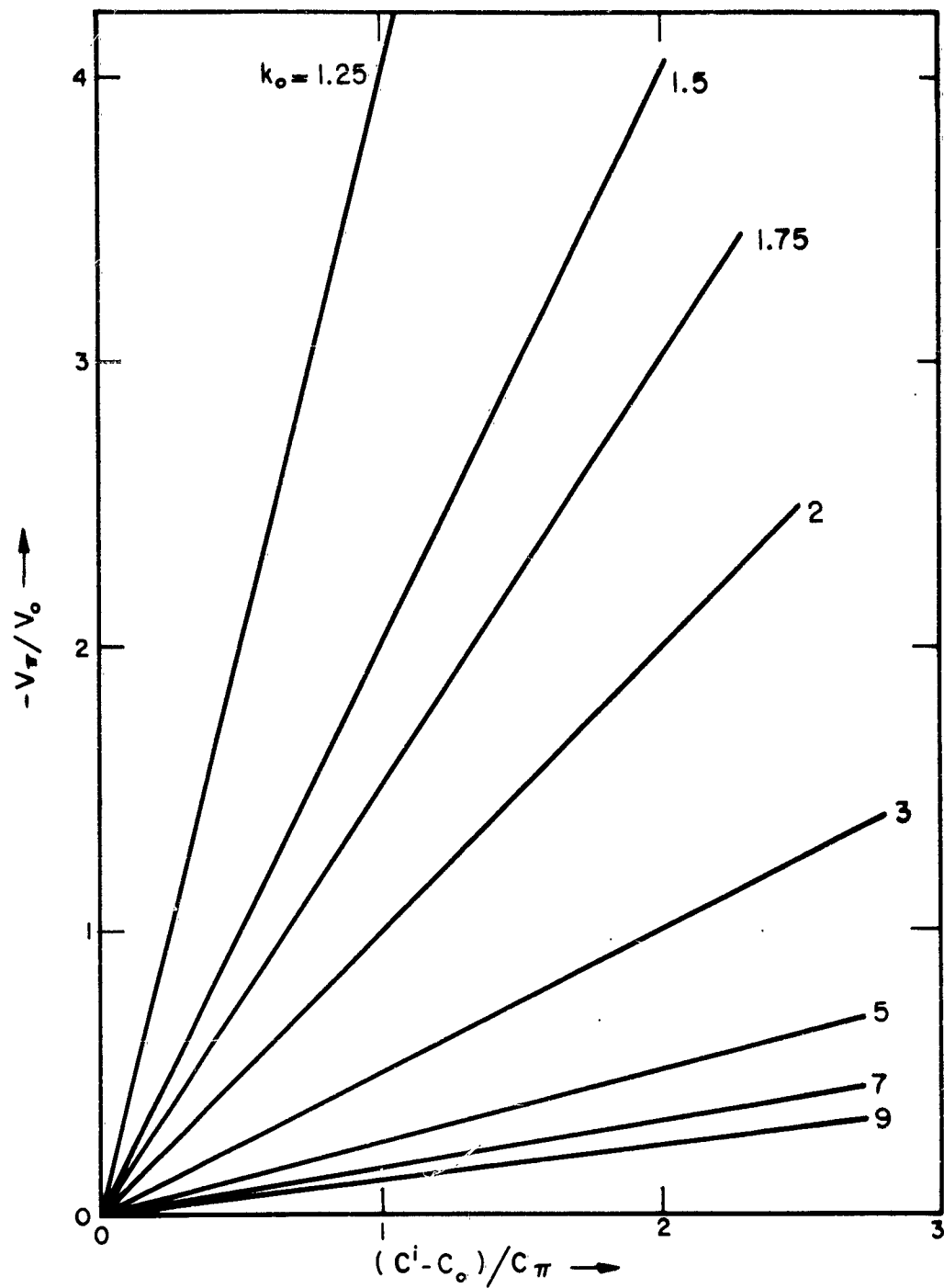


Fig. 5

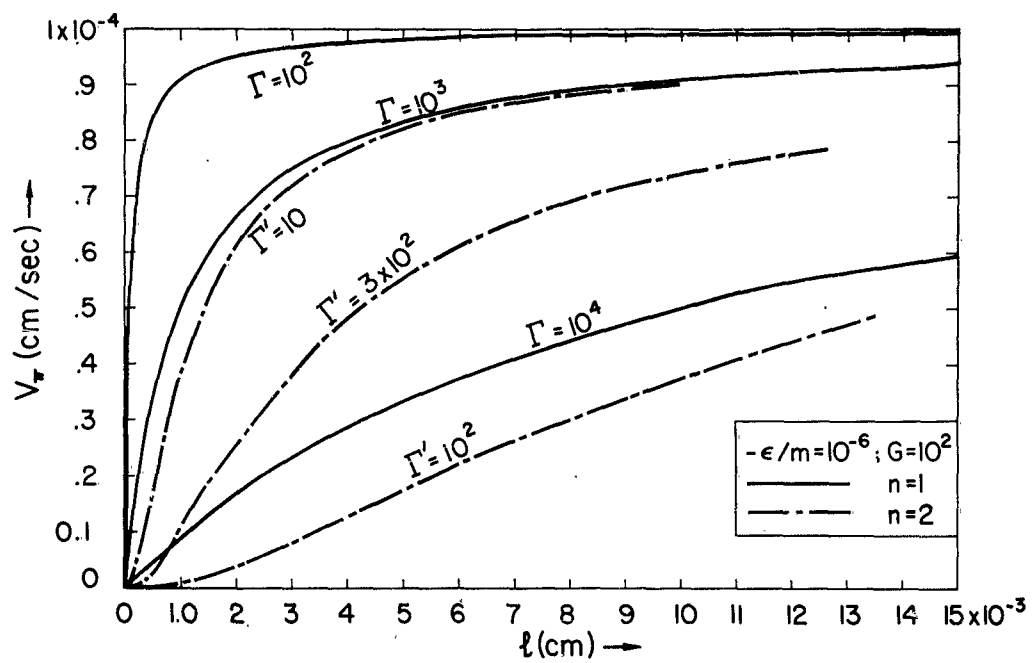


Fig. 6

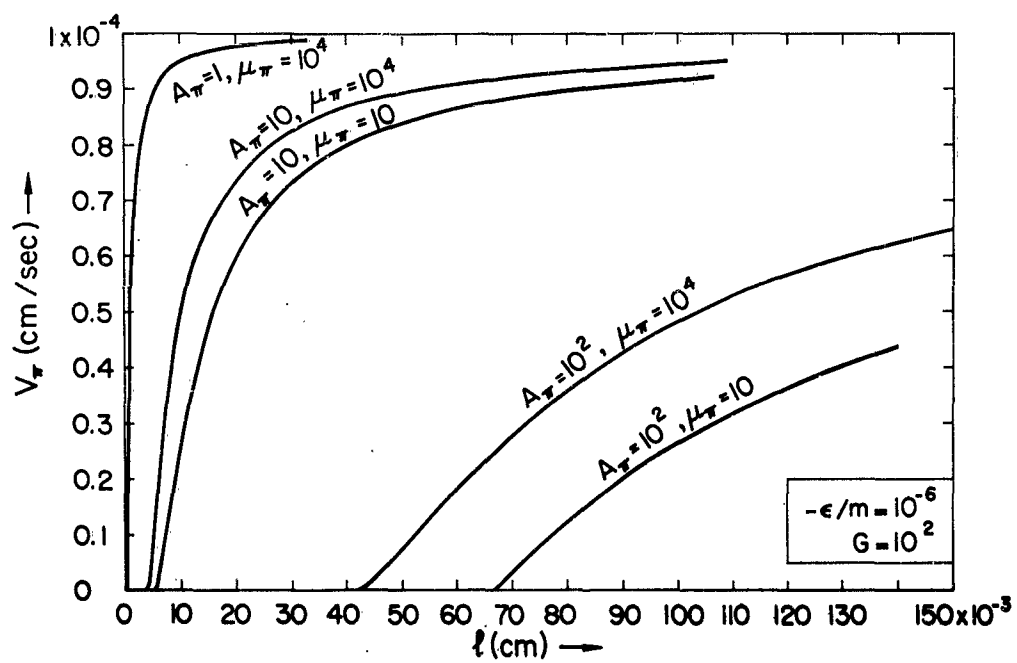


Fig. 7

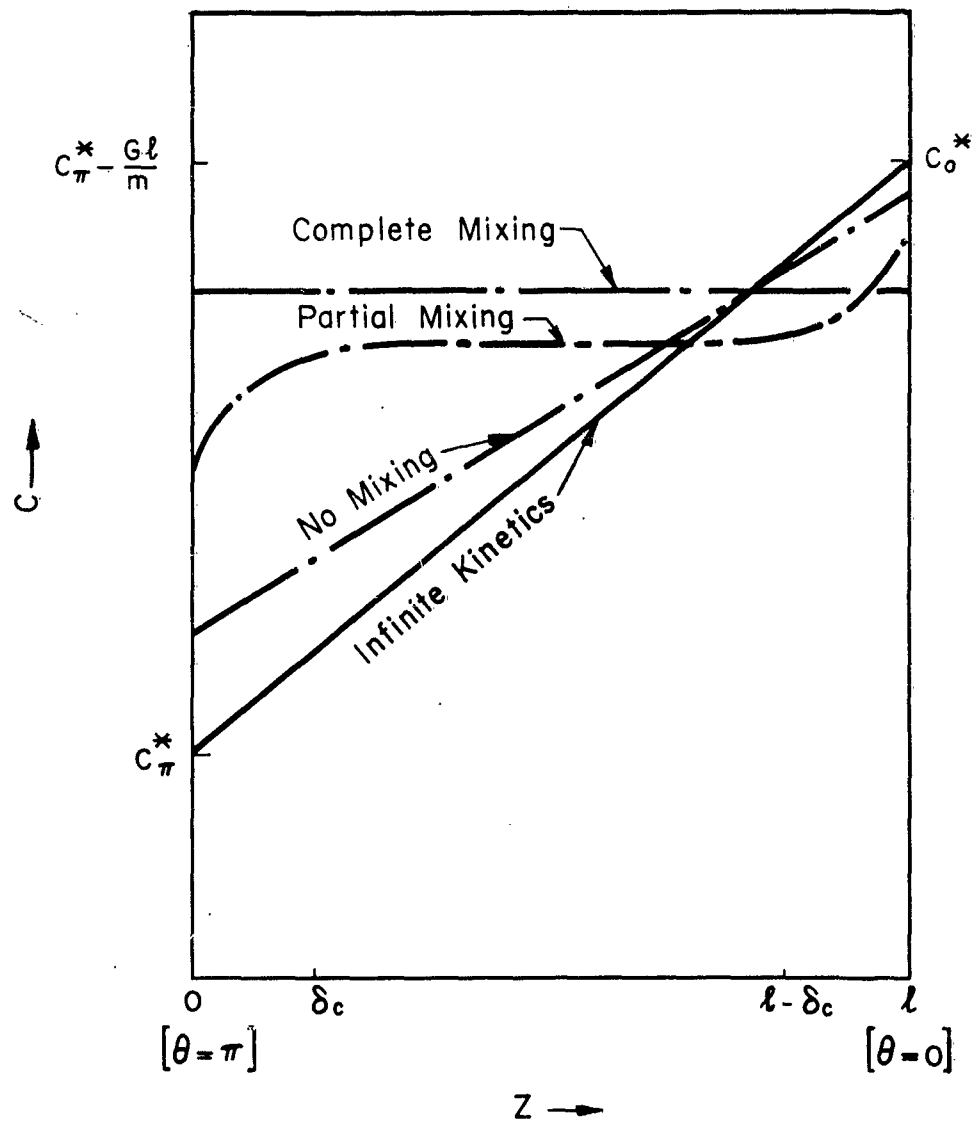


Fig. 8

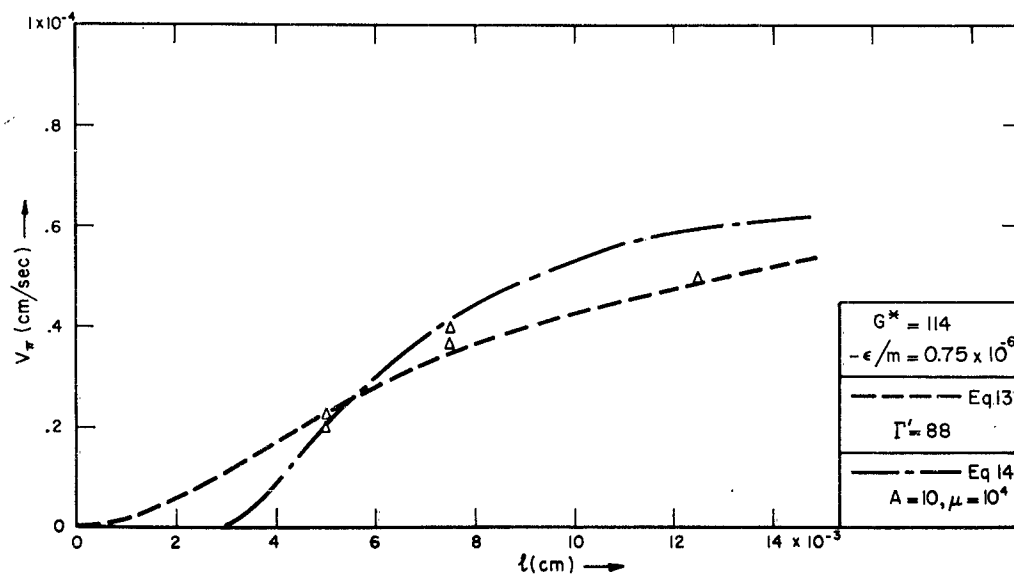


Fig. 9

Distribution List

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