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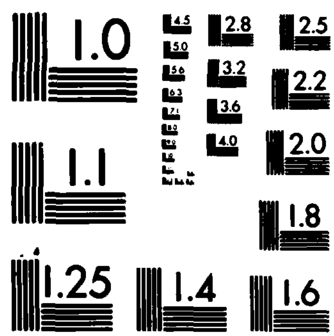

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ION IMPLANTATION METALLURGY:

A STUDY OF THE COMPOSITION, STRUCTURE AND CORROSION BEHAVIOR OF SURFACE ALLOYS FORMED BY ION IMPLANTATION AND ION BEAM MIXING

C. R. CLAYTON AND A. H. KING

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DIFFUSION INDUCED GRAIN BOUNDARY MIGRATION IN SURFACE MODIFICATION PROCESSES.*

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1. INTRODUCTION: DESCRIPTION OF THE PHENOMENON

Diffusion induced grain boundary migration (DIGM) is a phenomenon which was first reported in 1972 by den Broeder (1); it did not receive very much attention, however, until the observations by Cahn, Pan and Balluffi were published in 1979 (2). Since that time, several studies of the phenomenon have been reported (e.g. 3 - 8). DIGM is characterized by the sideways motion of grain boundaries in single phased materials during the diffusion of solute along them. As the grain boundaries migrate, solute is deposited in, or depleted from that volume through which a boundary sweeps, as illustrated schematically in Fig. 1. Since the process depends on grain boundary diffusion, it is observed most readily in temperature ranges for which lattice diffusion is essentially negligible over the period of the experiment: for most laboratory experiments, this limits the useful range of temperature to between 0.3 and 0.5 of the absolute melting temperature of the solvent material, although DIGM does occur outside this range (3).

Observations of DIGM have been made using techniques ranging from optical metallography through scanning electron microscopy to transmission electron microscopy, and encompassing various micro-analytical techniques. Types of specimens range from thin films, for which DIGM extends throughout the specimen, to bulk specimens in which it is restricted to the near surface region. Solutes have been delivered to, or removed from, the surfaces of specimens via vapor phases or by solid state diffusion along the interface with a thin film overlay.

Several aspects of the DIGM phenomenon are now well established, and appear to depend very little on the exact nature of the diffusion couple. For example, it is found that within any specimen the behaviour of individual boundaries is apparently random: somedo not migrate at all, some migrate uniformly and some migrate in a rather non-uniform fashion such as that shown in Fig. 2. There is, as yet no clear understanding of what causes these variations in behavior, although it may be expected that they are related to the boundary structures, and hence to the misorientations across the boundaries: no experiments attempting to correlate mobility with misorientation have yet been reported. The structural dependence is further supported by observations of facet development during DIGM (2), since the facet planes represent boundary

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orientations of low (or zero) mobility. The exact orientations of such planes of low mobility have not yet been determined experimentally so it is impossible to say, at this stage, whether or not they correlate with the planes of low mobility expected on the basis of the postulated mechanisms outlined below.

Grain boundary sliding is an important aspect of DIGM, revealed by the formation of steps on the specimen surfaces where migrating boundaries intersect them. These steps do not appear to be related simply to the change in volume of the specimen associated with the addition or subtraction of material to or from it, since extrusions are also formed in some cases (4). These correspond to material forced out of the specimen surface by the large compressive stresses built up when the net atom flux proceeds into the specimen. The formation of these extrusions indicate that the formation of surface steps by grain boundary sliding does not fully accomodate the change in specimen volume and thus that the sliding is controlled by other mechanisms.

An important question, both for the understanding of DIGM and for its utilization as a tool for the modification of surface properties, is the distribution of solute behind a moving boundary and whether or not it can be controlled: the fundamental aspects of this question will be the main theme of this paper. Preliminary work on the measurement of diffusion profiles behind migrated boundaries indicates that the solute is not necessarily uniformly distributed and that for many boundaries there is a concentration "spike" at the starting position of the boundary (5). This may be taken to indicate a "threshold effect" which is variable from boundary to boundary, and will be discussed below:

In most cases reported to date, DIGM is halted after a relatively small amount of migration has taken place, the maximum distance covered by a boundary being of the order of a few microns, and thus only a small proportion of the surface area of the specimen becomes alloyed, depending on the original grain size. This presents a serious problem in the technological application of DIGM to surface engineering, but the problem is not insurmountable since cases of complete surface coverage with a new layer of fine, alloyed grains have been reported in experiments utilizing large driving forces (6). This behavior is termed "diffusion induced recrystallization" (DIR), and is distinguished from DIGM by the fact that new crystal orientations are produced. It is most likely, however, that DIGM is the basic mechanism by which DIR proceeds. An interesting case of DIR is illustrated in Fig. 3 which shows the result of ion beam irradiation used to intermix a thin layer of iron with an aluminum substate. There is strong evidence that DIR has occurred, since it was also revealed by Auger depth profiling that the aluminum had diffused into the iron, even though the temperature was too low for bulk diffusion to have occurred. In this case, it appears that the point defects produced by the energetic ions may have also promoted the process, and this possibility will be discussed below.

2. UTILIZATION OF DIGM Diffusion induced grain boundary migration

DIGM is necessarily a near surface phenomenon, as may be visualized by artificially separating the process into discreet steps (which, it should be emphasized, occur continuously and simultaneously in practice). The first step is the diffusion of a small amount of solute into a stationary grain boundary, C_b, above that in the adjacent matrix. Immediately following this step, the boundary moves sideways by means of some atomic shuffling across the

boundary plane: This deposits the solute from the boundary into the volume of matrix through which it sweeps. After this second step, the concentration of solute in the boundary is restored to its original level, and so, therefore, is the driving force for grain boundary diffusion, assuming that the source concentration remains constant, and that there is no effect of strain in the alloyed region. The process should then continue, if the boundary continues to move in the same direction and the depth and concentration of the alloyed zone should both be related to the grain boundary diffusivity and the grain boundary mobility. Since the ability to control the depth and concentration of an alloyed zone is potentially useful for the purpose of tailoring surface properties to service needs, a study of the exact relationships is desirable~~; as shown below~~, however, much of the necessary information is not yet available, and complicated relationships may exist between grain boundary diffusivity and mobility.

2.1 Mechanisms of DIGM

In general, the rate of a kinetic process of this kind can be related to the diriving force by an equation of the type

$$R = M.\Delta G \tag{1}$$

where M is a mobility associated with the process driven by the "driving force", ΔG. The problem with DIGM is that the phenomenon is not characterized by a single atomic process, so it is not clear how the driving force is dissipated. Presumably some portion of the overall driving force (the change of free energy associated with alloying or a de-alloying a unit volume of the specimen) is used up in driving a rate-limiting step: the amount of energy and the mobility for this step are, then, critical to the formulation of a *predictive theory.* Clearly the starting point must be a mechanism for the phenomenon of DIGM. Detailed mechanisms have been proposed and discussed by Smith & King (9), Balluffi & Cahn (10) and King (11, 12): two distinct mechanisms with essentially similar features emerge and are discussed below.

2.1.1 Grain boundary dislocation climb models.

In any interdiffusion experiment, the diffusivities of the two species are generally different and this gives rise to the well known Kirkendall effect when the diffusion is by a vacancy mechanism. The different fluxes of the two components, say A and B, are generally balanced by a flux of vacancies so that, on average,

$$J_A + J_B + J_V = 0 \tag{2}$$

The required vacancy flux is supplied by means of dislocation climb, emitting vacancies where the vacancy chemical potential is negative and absorbing them where it is positive. These effects are also expected to occur for grain boundary diffusion experiments in the light of recent work suggesting that grain boundary diffusion proceeds, at least in some cases, by a vacancy mechanism (13). If the contribution of lattice diffusion is essentially negligible, the required vanancy fluxes must be produced by the climb of grain boundary dislocations (gbds).

The properties of gbds have been studied intensely over the past decade or so, and it is known that they have many properties which are different from those of regular crystal lattice dislocations: for example the Burgers vectors of perfect gbds may be smaller than those of crystal lattice dislocations, and depend on the misorientation of the two grains separated by the boundary. In addition, there may be a step in the boundary plane associated with a gbd (14). A diagram illustrating these essential features is shown in Fig. 4.

The essential features of this mechanism, then, are that the climb of the gbds is driven by a grain boundary Kirkendall effect; it is the concommitant motion of the step in the boundary plane which provides the grain boundary migration. The mechanism may also operate for gbds which have a component of their Burgers vector parallel to the grain boundary plane, in which case there is a component of glide in the motion of the gbd, and its motion therefore gives rise to some grain boundary sliding in addition to the migration of interest here. Grain boundary may be constrained by the contiguity of the grain boundary network within the specimen as well as by any net mass flow resulting from the Kirkendall effect: for cases of constrained sliding, it is envisaged that the process takes place by the motion of a suitable set of dislocations such that the net Burgers vector parallel to the boundary plane is as required. Near a surface, where DIGM occurs most readily, constraints on grain boundary sliding are also minimized, so that for boundaries intersecting free surfaces the sliding should be just that required for the DIGM mechanism and is controlled by the crystallography of the active dislocations. The motion of a general gbd, involving glide and climg and producing grain boundary sliding and grain boundary migration is shown in Fig. 5.

An interesting corollary of the proposed mechanism is that if DIGM does proceed in this way, then the migrating boundaries should develop facets parallel to the net step vectors of the gbds responsible for migration. Facets are indeed observed, for example in the work of Cahn, Pan & Balluffi (2) but it is not clear whether or not they lie parallel to the appropriate step vectors.

2.1.2 Grain boundary step migration models. A secondary mechanism by which DIGM may occur involves the interaction of a step in the boundary plane with a concentration gradient of either vacancies or solute along the boundary. A "pure" step on a grain boundary is considered to be one for which there is no closure failure of a suitable drawn Burgers circuit drawn completely around the step. Even though the total step may be dislocation free, there will, in general be a dislocation at each junction between different boundary planes, such that the step has a strain field like that of a dislocation dipole, as shown schematicaly in Fig. 6.

In presence of a uniform concentration of solute or vacancies the dislocations have equal but opposite forces applied to them by their interactions with the solution. In this case the net force applied to the step is zero. When the concentration of the solution is no longer constant, however, one of the dislocations will experience a greater force than the other one, and a net force is applied to the step: this will result in the directed motion of the step and thus the translation of the grain boundary plane. The concentration gradient required to drive such migration is generated by the initial grain boundary diffusion. A detailed analysis of this mechanism (11) reveals that it should contribute only a small amount of grain boundary migration compared to that generated by the gbd climb mechanism.

This mechanism, like the former one, should give rise to the formation of grain boundary facets, but the characteristic step vectors to which they should be parallel are different from the ones which are appropriate to gbd climb. Another difference between the two mechanisms is that there is no grain boundary sliding associated with the step mechanism, and therefore fewer constraints upon it.

2.2 Diffusion equations for DIGM

Since the gbd-climb mechanism currently provides the most complete explanation of the DIGM phenomenon, we shall base the derivation of a set of diffusion equations on the assumption that this is the only mechanism operating. It is important to recognize that for this mechanism, the forward velocity of the grain boundary, V_b, is controlled by the density of gbds, their velocity along the boundary plane, V_d, and the height of the steps associated with them, h. The dislocation velocity, in turn, is controlled by the driving force for dislocation climb, i.e. the vacancy supersaturation or undersaturation. Under these conditions the boundary velocity is given by

$$V_b = \frac{MhbkT}{\lambda\Omega} \ln(C_b^V/C_b^{Vo}) \tag{3}$$

where M is the gbd climb mobility, b is the Burgers vector magnitude, kT has its usual meaning, λ is the gbd spacing, Ω is the atomic volume and the subscript "b" indicates the concentration in the boundary of (superscript "V") vacancies. A superscript "o" indicates an equilibrium concentration.

Now we may consider a differential element of grain boundary, of length dx and width δ, equal to the width of the diffusive path provided by the boundary, as shown in Fig. 7. We choose to use an axis system which is embedded in the boundary and moves with it so that the axis system moves with velocity V_b relative to a stationary observer, and the boundary does not move relative to the chosen axis system. Under this choice of axis system, the matrix appears to flow through the boundary with velocity $-V_b$. The rate of change of concentration in the diffential element is then given by considering four fluxes across its boundaries: J_1 is a diffusive flux into the element, parallel to the boundary plane, and J_2 is the corresponding diffusive flux out of the element. J_3 and J_4 are fluxes into and out of the element, respectively, due to the flow of matrix through it. Under conditions of constant grain boundary diffusivity, D_b, this yields

$$\frac{\partial C_b}{\partial t} = D_b\delta \frac{\partial^2 C_b}{\partial x^2} + V_b(C_m - C_b)/\delta \tag{4}$$

which is essentially equivalent to Fick's second law. As it stands, with a suitable substitution for V_b from Eqn. 3, this expression is suitable for either component of the diffusion couple. In this unusual case, however, the concentration of vacancies is of critical importance, since it controls the magnitude of the boundary velocity. An expression for the rate of change of vacancy concentration is accordingly needed, and it can be seen that Eqn. 4 is not adequate because it takes no account of the production or removal of vacancies from the system by the dislocation climb which drives the entire process. The vacancies produced by the gbds climbing at a velocity V_d can be determined to produce a rate of concentration change

$$\frac{\partial C^V{}_b{}^1}{\partial t} = \frac{Mb^2kT}{\Omega^2\delta\lambda} \ln(C_b^V / C_b^{Vo}) \tag{5}$$

and this expression must be added to Eqn. 4 to yield a complete flux equation for the vacancies in the system.

The complete solution of the DIGM problem then requires the simultaneous solution of the three flux equations

$$\frac{\partial C_b^\alpha}{\partial t} = D_b^\alpha \, \delta \frac{\partial^2 C_b^\alpha}{\partial x^2} + (C_m^\alpha - C_b^\alpha) \frac{MhbkT}{\delta\lambda\Omega} \ln(C_b^V / C_b^{Vo}), \ \alpha = A, B \qquad (6,7)$$

$$\frac{\partial C_b^V}{\partial t} = D_b^V \delta \frac{\partial^2 C_b^V}{\partial x^2} + (C_m^V - C_b^V) \frac{MhbkT}{\delta\lambda\Omega} \ln(C_b^V / C_b^{Vo}) + \frac{Mb^2kT}{\Omega^2\delta\lambda} \ln(C_b^V / C_b^{Vo}) \qquad (8)$$

and the solution is subject to a further restriction since Eqn. 2 must be satisfied for the diffusive fluxes along the boundary plane, so we also have

$$0 = D_b^A \frac{\partial C_b^A}{\partial x} + D_b^B \frac{\partial C_b^B}{\partial x} + D_b^V \frac{\partial C_b^V}{\partial x} \qquad (9)$$

The solution of Eqns. 6-9 will enable us to determine the solute concentration profile behind a moving boundary as well as the shape of the boundary, and hence the depth of the alloyed region. In addition to these directly useful parameters, it should be noted that gbd climb velocity will vary with position in the boundary, so the solution will also enable us to predict the redistribution of gbds which should occur as a result of DIGM. All of these measurable quantities will have to compared with experimental observations in order 1) to confirm that gbd climb is the mechanism of DIGM, and 2) that the equations presented here represent a good model of the phenomenon; only then can the solutions be used to predict the results of a DIGM-based process and to control the phenomenon for useful application.

Eqns. 6-9 are not analytically soluble, but numerical methods for solving them are being developed.

At this stage the model still has various shortcomings, among the most serious of which is that it cannot deal with the case where lattice diffusion is not completely frozen out: this is likely to be a technologically important case simply because thermal processes which are carried out between 0.3 and 0.5Tm are usually considered to be too slow for profitable use. This is a serious shortcoming of the model which is not easily overcome. Other items which are not yet included in the model, but which could be without great difficulty, include the inclusion of solute segregation to the boundary and effects such as threshold driving forces below which gbds will not climb, as suggested by King & Smith (15). This latter possibility is interesting since it may provide the explanation for the concentration spikes discussed above, by allowing the boundary solute concentration to build up to a relatively high level before migration starts to occur, thus depositing this large concentration in the matrix at the boundary's starting position.

3. SUMMARY AND CONCLUSIONS

DIGM is a phenomenon which presents several interesting possibilities in the field of surface alloying; indeed, it may already contribute to certain processes such as pack cementation and ion beam intermixing. In the latter case, it appears that the vacancy supersaturation produced by the ion bombardment also contributes to driving the climb of gbds, and so to moving the grain boundaries. The fine grain size of vapor deposited layers also contributes, in this case, by ensuring that the entire layer is swept through by moving

boundaries before the process is halted. It is to be expected that DIGM may be identified as the cause of many surface engineering phenomena.

The conscious application of DIGM in surface engineering is, as yet, still in the future, since it is not yet established how to control the depth and concentration of the alloyed (or de-alloyed) zone formed by the use of the phenomenon. Work intended to resolve this problem is currently under way.

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Fig. 1. Schematic illustration of DIGM: solute is diffused into the specimen via the grain boundaries and their migration is induced. The dashed and full lines indicate the initial and final boundary positions respectively, and the shaded region is solute-enriched. Case (a) is a thin-film specimen and case (b) is bulk.

Fig. 2. Scanning electron micrograph (and schematic) of the surface of a pure copper specimen after exposure to zinc vapor at 350°C for 48 hours. The dashed and full lines of the schematic indicate the initial and final boundary positions respectively.

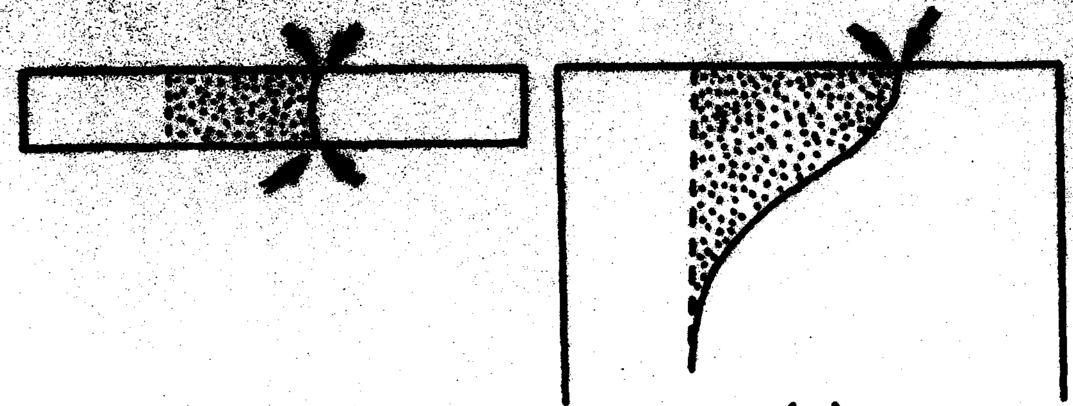
Fig. 3. Dark field transmission electron micrographs showing grain growth in a 400Å layer of iron on an aluminum substrate, as a result of 190keV helium ion beam intermixing. The left-hand micrograph is of an unirradiated specimen, and the right-hand micrograph is of an irradiated one. The iron contains dissolved aluminum in the latter case, and the grain boundary migration mechanism is thought to be DIGM.

Fig. 4. Schematic illustration of an f.c.c. grain boundary dislocation with the Burgers vector 1/11 113 in a grain boundary with a misorientation of 50.41° about an axis parallel to 110. The step in the boundary plane must move with the dislocation if it climbs.

Fig. 5. Ball model illustration of the motion of a grain boundary dislocation by combined glide and climb. The Burgers Vector is 1/10 013 and the misorientation is 36.87° about 100; the extra half-plane is indicated by a row of darker balls. The process involves the removal of atom "A" by the condensation of a vacancy, the shuffling of atom "B" across the boundary plane from the upper grain to the lower one, then the shear of the model restoring a configuration identical to the original one with atom "B" in a position equivalent to that originally occupied by atom "A". Boundary migration is achieved since the upper grian loses two lattice sites while the lower one gains a sincle site.

Fig. 6. Schematic illustration of a pure step in a grain boundary plane: the equilibrium translations on the two planes are $\underline{T}_1$ and $\underline{T}_2$ and the Burgers vectors of the dislocations are $\underline{b}_1 = \underline{T}_1 - \underline{T}_2$ and $\underline{b}_2 = -\underline{b}_1$. The step may therefore be considered as a dislocation dipole.

Fig 7. Differential element of a moving grain boundary illustrating the fluxes relative to the element needed to form a complete flux equation equivalent to Fick's second law.



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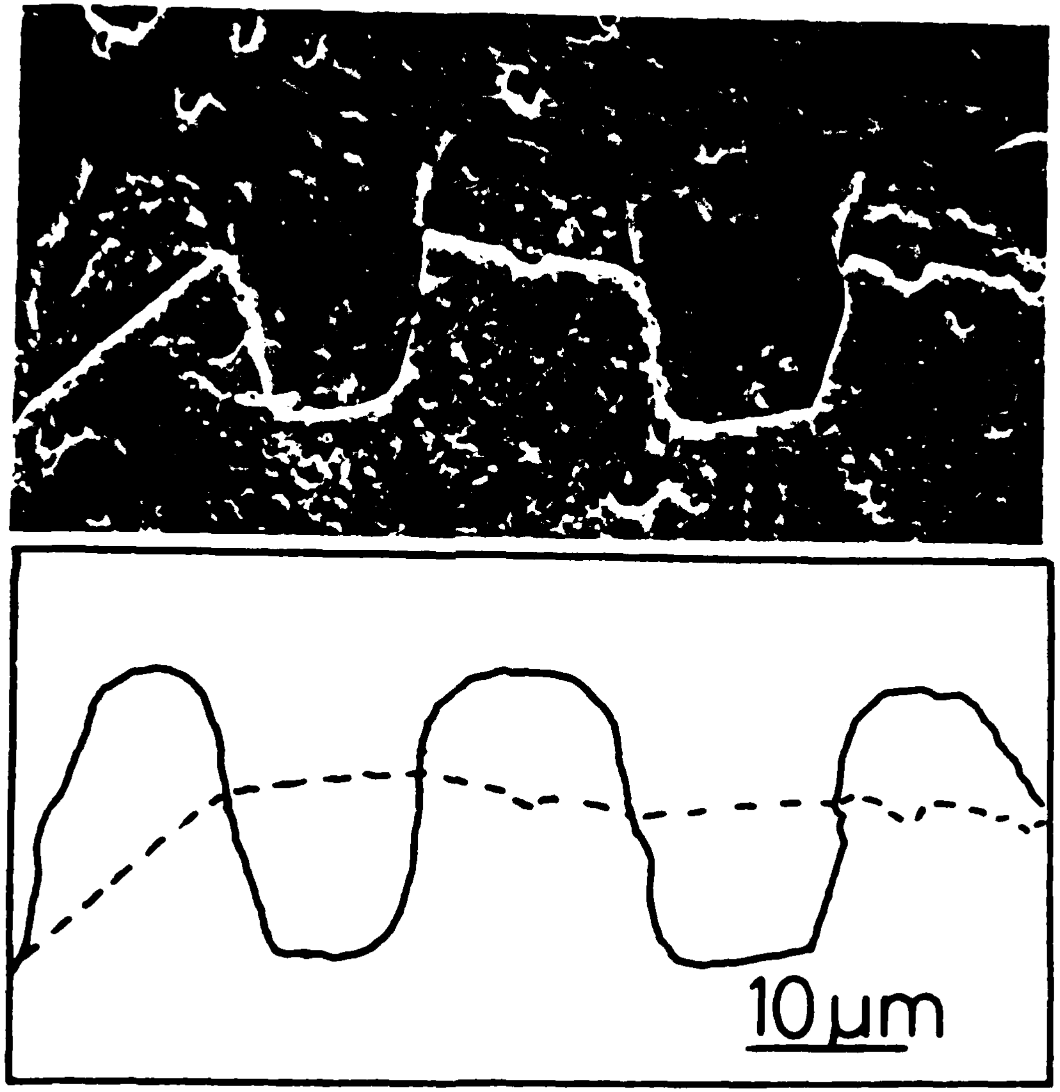
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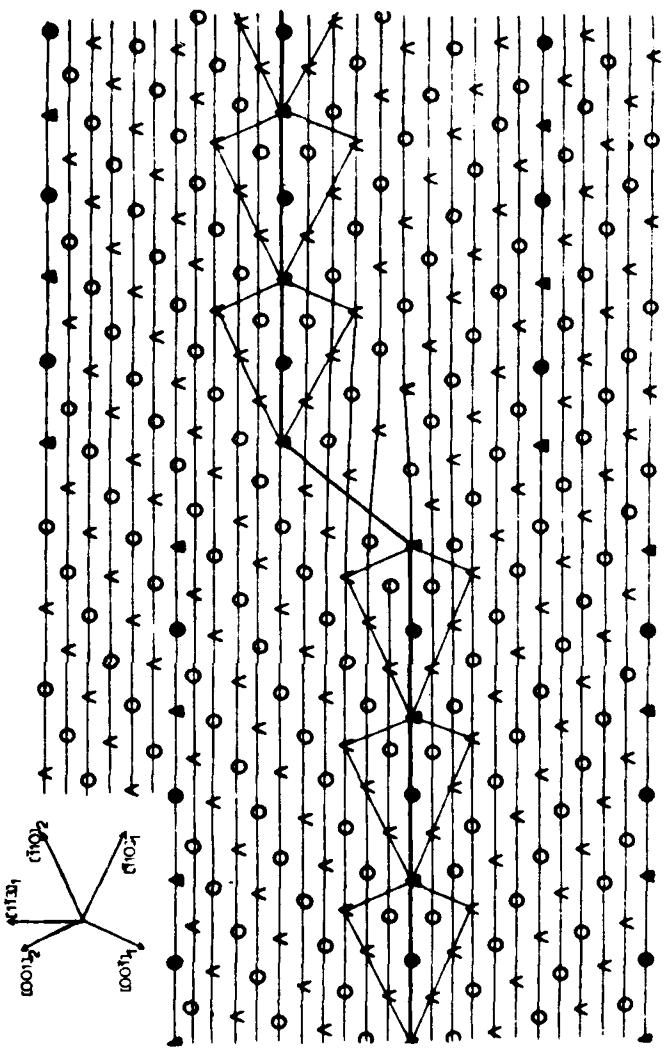

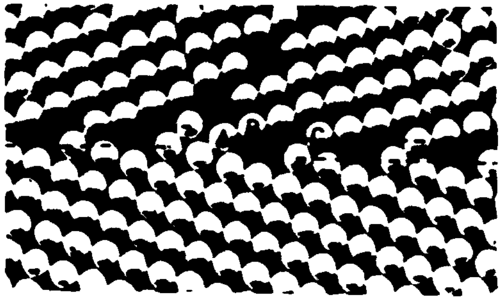

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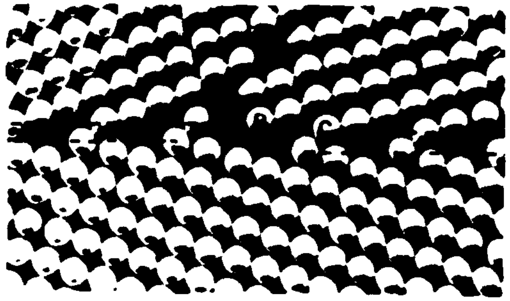

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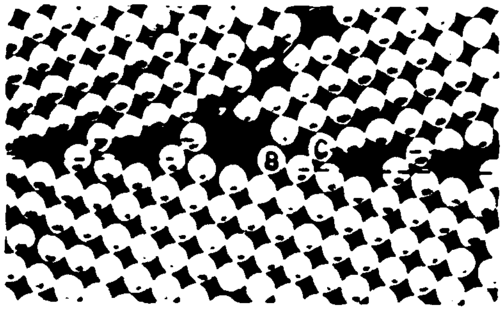

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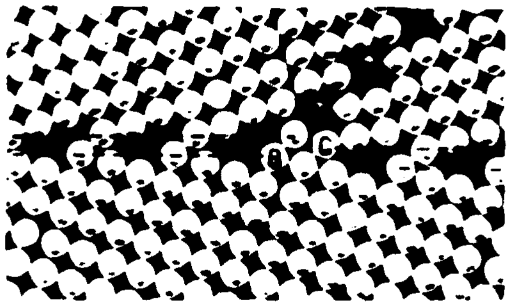

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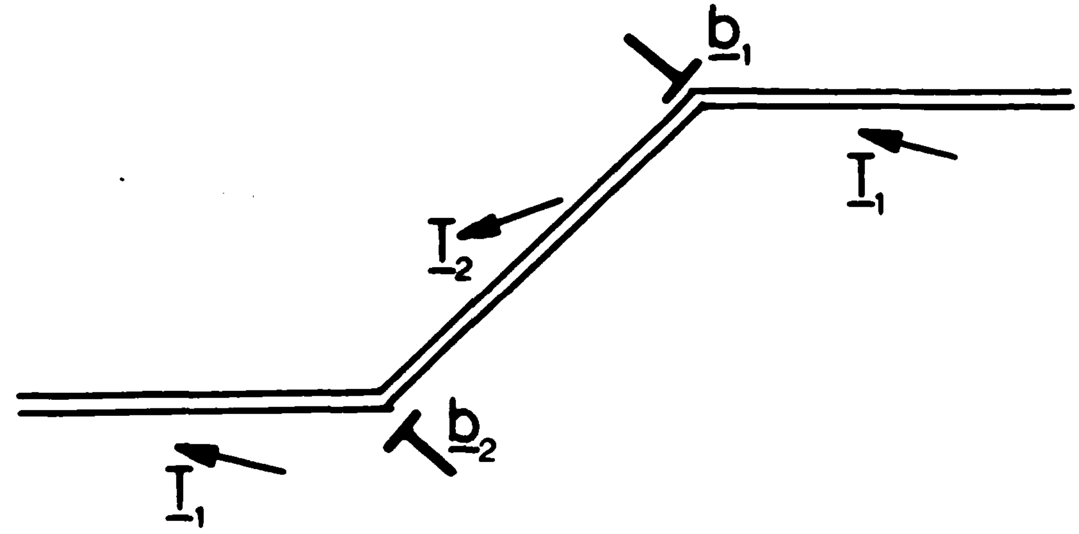

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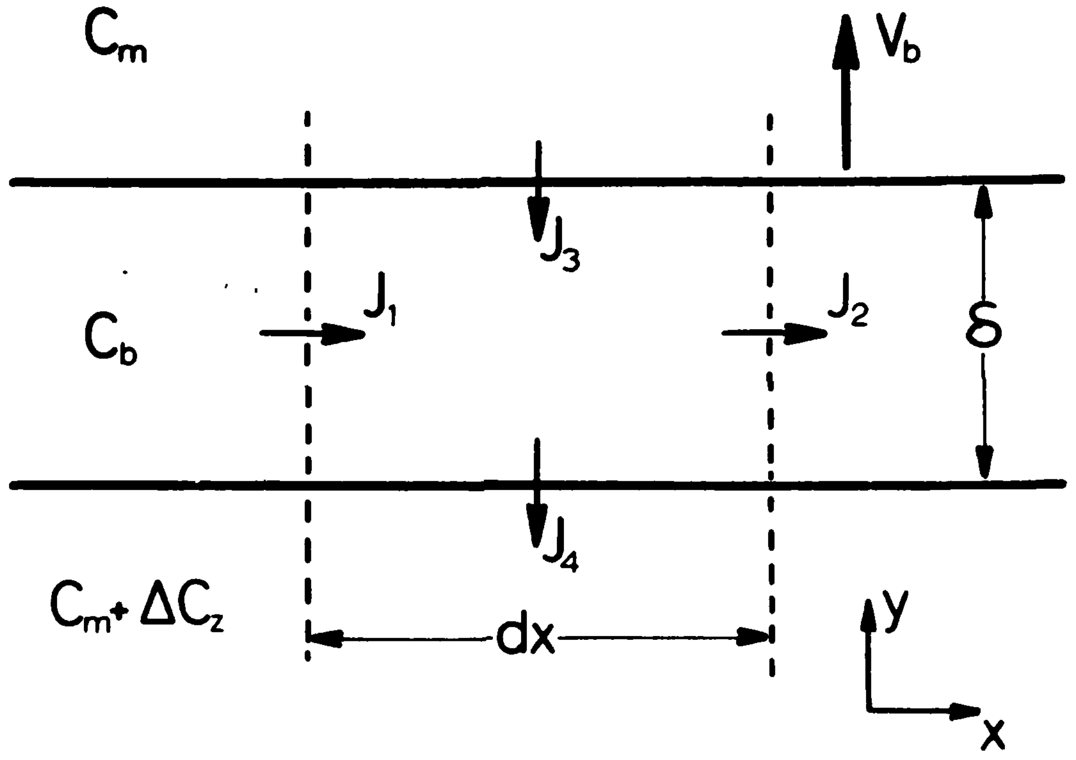
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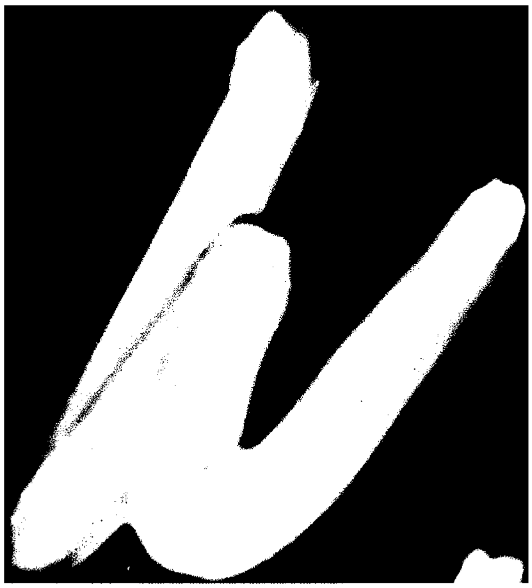

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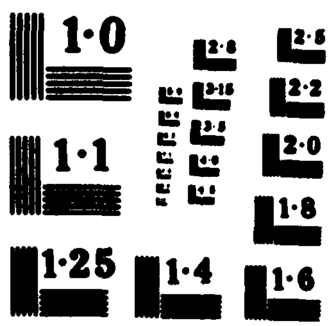
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