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LECTURE 3: NONEQUILIBRIUM DEFECTS IN METALS

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1. Introduction

Most of the properties of metals are strongly dependent on the type, concentration and distribution of defects which are present. Examples of these properties include mechanical behavior, diffusion, mechanisms and kinetics of phase transformations, and electrical properties including superconductivity.

The detailed behavior of crystal defects is very complicated but they can be studied directly using high resolution techniques as will be demonstrated in later lectures. The subject is extensive (see e.g. refs. 1-9) and here it is possible only to give a brief outline. Not only are individual defects of fundamental importance but also their interactions and associations. For example, accumulated vacancies can cluster together and nucleate dislocation loops and these can subsequently be nucleation sites for precipitates, and so on.

It is impossible to produce metals which are free from defects because of solidification constraints, growth accidents, non-equilibrium cooling rates, and defects introduced by processing treatments. As a result, point, line, surface and volume physical imperfections can be created in addition to chemical impurities such as foreign atoms, inclusions, concentration gradients, etc. These are listed in the summary. The minor electronic defects which are very important for elemental semiconductors are not important for metals so these will not be considered here. Examples of the main types of defects and some properties associated with them are given in the following.

2. Description of Defects

A. Point defects. A point defect is associated with a short range

interaction, i.e. within a few atomic diameters outside its position in the lattice. The most important are vacant lattice sites (fig. 1b). If these exist in excess of the equilibrium thermal concentration they can lower their energy by migrating and interacting with each other to form small voids or planar defects, e.g. loops, or by moving to sinks, e.g. dislocations, free surfaces, grain boundaries, etc. Clusters of di and tri vacancies may be more energetically stable than mono-vacancies at low temperatures. The motion of vacancies accounts for diffusional processes, such as recovery, dislocation climb, creep and aging reactions in alloys. The concentration c of thermal vacancies is proportional to $A \exp^{-Q_f/kT}$ where the entropy factor A is 1-10, the formation enthalpy $Q_f \sim 1\text{eV}$ for fcc metals, k is Boltzman's constant and T the absolute temperature. Thus, for aluminum at the melting point $c \approx 10^{-4}$ and at 20°C , $c \approx 10^{-13}$. Rapid cooling $\sim 10^4\text{C/min}$ is sufficient to retain most of the vacancies so that very large supersaturations can exist. These high concentrations account for the very rapid rates of clustering and zone formation as in Al-Cu and other alloys after solution heat treatments.

Vacancies can also be created during irradiation and plastic deformation, particularly fatigue.

In a pure metal, there is no necessity for the number of vacancies to equal the number of interstitials,* fig. 1c. In close packed metals the formation energy for interstitials is 4-7 times that of vacancies, so that the fractions of vacancies to interstitials is two to four orders of magnitude. Thus interstitials are not present in important

* Unlike ionic solids, where charge neutrality must be maintained.

concentrations under normal conditions. They can be generated by knock-on events when high energy particles are bombarded into crystals such as during neutron radiation but even then association into clusters and collapse into dislocation defects occurs, since the single interstitial is a high energy defect which produces enormous local strains (fig. 1c).

It is difficult to study vacancies directly although they can be resolved by field ion microscopy. Electron microscope studies have been made of dislocation loop shrinkage and growth, i.e. dislocation climb, from which estimates can be made of the vacancy migrational energies and other parameters⁸⁻¹¹. Indirect methods of study include measurements of physical property changes¹⁰ such as electrical resistivity, density and lattice parameters¹² from which estimates of formation and migration energies can be made.

Although single point defects cannot be resolved by electron microscopy (see chapter 4) clustered point defects and effects arising from point defect interactions can, and examples of some defects resulting from vacancies are shown in fig. 2 (see refs. 6, 9, 10 for reviews). The stacking fault tetrahedra (fig. 2b) were not predicted by theory and were first observed in the electron microscope by Silcox and Hirsch. [Phil. Mag. 4, 72 (1959)] Vacancies are also produced when inter-metallic compounds deviate from stoichiometry (e.g. NiAl, FeAl, etc.). Chemical point defects are impurity atoms which can be substitutional or interstitial and which themselves alter the mechanical and physical properties. These alterations are not necessarily undesirable, but it is essential to control them through control of composition and microstructure. Certain kinds and distributions of impurities can be very detrimental, e.g. inclusions and surface films along boundaries.

B. Line defects. If a plane of atoms extends only part way inside a crystal the edge of this plane forms a line defect, viz. a dislocation. Dislocations are the most important defects in controlling mechanical properties. They are also important in determining the precipitation morphology in age-hardening alloys. Whilst vacancies can exist as thermodynamically stable defects in certain (high) temperature ranges, dislocations, due to their high strain energies, cannot exist as a thermodynamically stable defect. The self energy of a dislocation line is $\sim 5\text{eV/atom distance}$. Thus stresses of order $G/30$ are necessary to nucleate dislocations homogeneously (G is the shear modulus). Nevertheless, they do exist. The concept of dislocations was introduced 40 years ago by Orowan, Polanyi and Taylor in order to explain the softness of metals, e.g. pure metals can deform plastically at stresses less than 10^{-5} times the shear modulus ($\sim 50 \text{ gm/mm}^2$). Dislocations were not actually observed directly until thin foil transmission electron microscopy techniques were developed in the late 1950's. Examples are given in lecture 4. Although Professor Parker will review the theory of dislocations and the literature is extensive (e.g. the books of refs. 2 to 5) some basic features will be described here.

A dislocation is a line that forms the boundary between a sheared (slipped) and unsheared parts of a crystal. The usual shear planes are the close packed planes such as $\{111\}$ fcc, $\{110\}$ bcc and $\{0001\}$ hcp. A dislocation must form a closed loop or terminate at a surface or another dislocation and the displacement across the dislocation line is constant. This latter property characterises the dislocation in terms of the magnitude and direction of shear movement associated with

it and is called the Burgers vector \vec{b} . This is normally equal to a lattice translation vector \vec{T} such as $a/2\langle 110 \rangle$ in fcc, $a/2\langle 111 \rangle$ in bcc, etc. Figure 3 shows the variation in orientation of a dislocation with \vec{b} . When \vec{b} and the line are parallel the dislocation is defined as pure screw; when \vec{b} and the line are mutually perpendicular the dislocation is defined as pure edge, and at other orientations, mixed.

Dislocations can interact to form networks and the links in the network may be sessile such as Lomer-Cottrell locks. There is also a tendency for dislocations in close packed metals to dissociate into partials, e.g. as illustrated in fig. 4 for fcc metals. The partials form a ribbon bounding a faulted region, e.g. in fcc metals the first partial changes the stacking sequence from the normal ABCABC to ABCACABC sequence and the second partial restores the correct sequence. The separation of these so-called Shockley partials depends on the energy of the fault between them - the stacking fault energy, which is quite an important property. In the example of fig. 4 the stacking fault has the hexagonal close packed structure. In addition to the importance of these dissociations in plastic flow they are also important in effecting phase transformations, e.g. the fcc \rightleftharpoons hcp cobalt transformation can be accomplished by dissociation of whole dislocations into Shockley or Frank partials.

The stacking fault energies in crystals can be measured in several ways, e.g. by electron microscopy measurements of radii of dissociated nodes in dislocation networks (fig. 5), or the separation of partials if resolvable,^{13,14} and indirectly from x-ray diffraction spectra, twin boundary energies or the stress at which thermally activated cross slip

occurs (see refs. 14, 15 for reviews). For metals stacking fault energies range from about 20 erg/cm^2 (Ag) to about 200 erg/cm^2 (Al). In alloys the stacking fault energy generally decreases with solute content, e.g. stainless steel and α -brasses (fig. 6). In non-metals and layered structures the stacking fault energies can be very low, e.g. graphite $\sim 0.05 \text{ erg/cm}^2$.

The higher the stacking fault energy the narrower are the ribbons and more easily forced together by stresses. This means that cross slip is easier and the dislocation configuration changes (fig. 7). Thus the values of γ are important in understanding plastic flow -- particularly work hardening.

Other important partial dislocations include the stair-rod dislocation which cannot glide nor climb. The stair-rod is a "connecting" dislocation across two intersecting slip planes, but the Burgers vector lies in neither, e.g. in fcc metals:

Shockley in $(1\bar{1}1)$ plane + Shockley in $(\bar{1}11)$ plane - stair-rod in (111) or $(1\bar{1}\bar{1})$

$$a/6[\bar{1}\bar{2}\bar{1}] \quad + \quad a/6[211] \quad \rightarrow \quad a/6[\bar{1}\bar{1}0]$$

The Frank partial dislocation also bounds a stacking fault and in this case the Burgers vector is normal to the plane of the dislocation line (fig. 4c). Such dislocations are called prismatic dislocations. A summary of the dislocations that can exist in the fcc structure is shown in fig. 8. Similar possibilities exist for other metallic structures.

Dislocation loops (fig. 2a, 4c) can be formed by the aggregation and collapse of point defect clusters, e.g. vacancy loops or interstitial loops. In quenched metals many vacancy loops can be generated and both vacancy and interstitial loops are formed after extensive radiation damage particularly by neutrons. Loops can also form when a

moving dislocation line contains a jog (a segment which lies out of the slip plane so that it cannot glide easily), or after interactions of moving dislocations and obstacles, e.g. precipitates. Prismatic loops can also be generated to relieve strains due to precipitation, and loops can be punched out as a result of differential thermal expansion of particles and the surrounding matrix. Figure 9 shows an example where loop punching was observed directly in the electron microscope.

When an alloy becomes long range ordered, the dislocations which were whole dislocations (i.e. $\vec{b} = \vec{T}$) in the disordered state now become partial dislocations. These tend to move in pairs called superdislocations so that order is not destroyed. The portions of crystal between the superdislocation are antiphase domains.

Besides interactions with each other, dislocations interact with all other kinds of defects, e.g. with solute atoms which can slow dislocations down during plastic flow and can lead to heterogeneous precipitation. Both of these interactions can cause yield point phenomena. The interactions can arise from elastic, chemical, or electrical origins.

Dislocations also interact with surface defects such as stacking faults, grain boundaries, etc., and with volume defects such as voids, precipitates, inclusions. These interactions all tend to restrict dislocation motion and can lead to strengthening but also embrittlement (lecture 6).

Dislocation climb involves dislocation motion normal to the Burgers vector (whereas glide occurs in the direction of \vec{b}). This process involves removal of atoms, e.g. by vacancies or addition of atoms, e.g. by interstitials at the half plane. Screw dislocations are converted

into helices by climb (fig. 2a) and the climb process is a mechanism for dislocations to move to lower energy configurations upon annealing (recovery). The climb process can be observed directly by hot stage electron microscopy techniques.¹⁷

C. Origin of Dislocations.

Since the self energy of dislocations is very large ($\sim 5\text{eV/atom}$ distance) dislocations must be heterogeneously nucleated, e.g. by condensation of point defects forming loops. Observations have shown that during plastic deformation the dislocation density increases by several orders of magnitude. This means that multiplication processes such as from Frank-Read sources must occur continuously as slip proceeds. The following sections outline sources of the possible dislocation origins and multiplication mechanisms.

D. Dislocations formed during Solidification.

It now seems to be generally agreed that formation of dislocations from collapsed vacancy aggregates does not occur during solidification so that dislocations must be nucleated by other mechanisms. However, if dislocations already exist in the crystal, the total length of dislocation line can be increased by climb or glide during cooling. For example, as a result of climb, edges become jogged and screws transform into helices.

Since a nucleus of solid is formed homogeneously only when the liquid is supercooled sufficiently, a nucleus once formed is surrounded by supercooled liquid and will grow dendritically provided a reverse temperature gradient or supercooling persists. The tip of the dendrite

may be bent by mechanical disturbances, gas evolution, convection currents, or as a result of strains due to segregation of solute atoms to the liquid which solidifies last. Consequently, this dendrite can acquire slight misorientations with respect to its neighbors.

When the dendrites meet each other these misorientations can be matched by the formation of edge dislocation tilt boundaries, screw dislocation twist boundaries, or subboundaries containing mixed dislocations. Such networks give rise to the so-called lineage or mosaic structure in solidified crystals. An example of a twist boundary is shown in Fig. 10.

Thus, dislocations can be formed as soon as the first solid is nucleated, and a perfect crystal results only if these dislocations can be eliminated and the formation of subsequent dislocations prevented.

If segregation of impurities occurs, the last traces of liquid to solidify will have a different composition from the initially solidified dendrite. This usually means that the lattice parameters of both will also be different. As a result of the gradual change in lattice parameter across the freezing solid, a system of edge dislocations may be set up at the interfaces. Similar substructures can be formed around particles when precipitation occurs in the solid state, or as a result of diffusion induced stresses.

In any case of a solid growing on a solid already containing dislocations, then at the growing face, dislocations existing in the substrate may propagate into the new crystal, since a dislocation must terminate at a free surface or in other dislocations of different directions

(at nodes). In principle, it is possible to grow dislocation-free crystals, if the formation of new dislocations during and after growth is prevented. So far, only the covalent crystals of silicon and germanium have been grown perfectly, and no success has yet been reported for metallic and ionic crystals.

If the surface of a seed crystal or substrate is stepped, the discontinuity may be accommodated upon subsequent growth by the generation of dislocations or stacking faults. This is particularly true if impurity atoms are adsorbed on the surface. Recent high voltage electron microscopy experiments on vapor deposition of silicon have shown the impurity particles which are mainly responsible for the growth faults commonly observed in epitaxial deposits.²⁰

If two widely misoriented dendrites meet, a large-angle grain boundary will be formed. Such a boundary can contain a large number of dislocations, particularly at steps in the boundary. If the local stress is high enough, these dislocations can be emitted from the boundary, thereby eliminating the step.

The stresses necessary for nucleation and multiplication during cooling probably originate from (1) Local stresses due to dislocation arrays already formed in the solid; these stresses are probably the main cause for propagation of a small angle boundary into newly solidified layers; (2) Stresses due to segregation and atomic misfit, e.g. by differential thermal contraction; and (3) Thermal stresses due to non-linear temperature gradients -- particularly if cooling is done rapidly. These latter stresses are likely to cause multiplication of the existing dislocations. For example, if the thermal stress at the interface is deliberately increased, an increase in dislocation density is observed.

It seems that stress is often the most important cause of dislocations formed during growth from the melt; so, the critical stress to move a dislocation must be an important factor. This is borne out by the fact that crystals of silicon and germanium can be produced free of dislocations. Bonding in these crystals is covalent and very strong; so, the Peierls stress is very high, and dislocations in the diamond lattice are, thus, relatively immobile.

E. Heterogeneous Nucleation of Dislocations from Impurities.

A precipitate may act as a small punch inside the crystal in two ways: (1) By differential thermal contraction as the solid cools; (2) As a result of chemical stresses arising from a localized excess (or depletion) of point defects associated with the removal of solute atoms from the matrix around the particle. The classical example of punching was provided by the experiments of Jones and Mitchell, where prismatic dislocation loops and helicoidal dislocations were shown to emanate from glass spheres purposely introduced into AgCl crystals. Since this work, many examples of heterogeneous nucleation of dislocations from impurities have been observed.

In a metal containing precipitates or inclusions, e.g. Fig. 9, the coefficients of thermal contraction of inclusions are usually less than those of the metal; so, the dislocation loops resulting from punching should be of the interstitial prismatic kind.

Consider now the case of partially coherent precipitates, i.e. precipitates whose interface with the matrix consists of a grid of dislocations. If the local stress is high enough, some of these interfacial dislocations may be emitted from the particle. Since the

dislocations become held up at the obstacle until they start bowing around it and until an appreciable screw orientation can be attained. The screws then cross-slip to above the obstacle, eventually leading to the formation of a prismatic loop behind the particle. This loop can glide on its prismatic surface. When screws encounter the obstacle, cross-slip may eventually lead to the operation of a Frank-Read source. Typical obstacles that are observed to give rise to multiplication are precipitates, subboundaries, and strongly pinned, grown-in dislocations.

When the obstacles are not randomly distributed, parts of the dislocation may eventually meet. If both segments are on the same glide plane, they will annihilate each other, since the Burgers vectors are opposite in sense. If the segments are on parallel planes, those in screw orientation will tend to cross-slip together and be annihilated. However, if the segments are of edge character, a dipole will be formed. The dipole is stable when the two glide planes are separated by less than a critical value d_c , but if $d > d_c$, the dislocations can cross over one another. The critical separation is given by

$$d_c = \frac{Gb}{8\pi(1-\nu)\tau}$$

where τ is the local resolved shear stress, ν is Poisson's ration, and G and b have their usual meanings.

The continuation of cross-over can lead to multiplication. It is thought that jogs on screw dislocations resulting from intersections or by absorption of point defects are mainly responsible for the cross-over mechanism. The interaction between gliding dislocations, subboundaries, or immobile dislocations is another possible origin of cross-over and

multiplication. The double cross-slip mechanism is important only if the stacking-fault energy of the material is not too low. When the stacking-fault energy is low, each dislocation is dissociated into two partials separating a stacking fault. Cross-slip or bowing of a dislocation out of its original plane can occur only if the two partials come together by constriction, and the more widely the partials are separated (i.e. the lower the stacking-fault energy) the more energy this requires. Consequently, dislocations in these materials (e.g. copper alloys and some stainless steels) rarely leave their original slip planes (Fig. 7d,e).

In most pure metals, the stacking-fault energies are not low and the dislocation arrangements during deformation are complicated. Tangling and formation of densely populated dislocation cell walls (Fig. 7a) are common features in the deformation of both bcc and fcc crystals. As the strain increases, the separation between dislocations in the tangles decreases as the dislocation density increases. In favorable situations some segments in the tangle may bow out and be released or may act as a Frank-Read source.

Dislocation generation can also occur during phase transitions particularly in martensite. In low carbon and low alloy steels the dislocation densities in the martensite grains are very high ($> 10^{10}$ per cm^2).

G. Surface Defects. Of these, the greatest number are the internal surfaces such as grain boundaries, twin boundaries, subgrain boundaries, stacking faults, antiphase domain boundaries in ordered alloys (Fig. 13) and interphase interfaces. The external "free" surfaces are particularly important in terms of chemical activity (oxidation, corrosion), diffusion,

fatigue. The internal surfaces control the initial kind of interaction with dislocations. This can be illustrated by considering the state of coherency across the interfaces between phases as illustrated two-dimensionally in Fig. 14. As can be seen, if the strength of the material is the same on either side of the boundary the plasticity depends only on the nature of the interface, viz. whether dislocations can cross the interface or not.

Actually the crystals on either side of the interface can differ in orientation, structure and composition and when all three differ, coherency is not likely. In a system where only misorientations occur, e.g. grain/twin boundaries, the boundary can be considered to contain a stepped or ledge structure or planar interfacial dislocation networks depending on the misorientation involved.

There is considerable current interest in the dislocation structure of grain boundaries and their effect on boundary migration and sliding.¹⁸ Observations by field ion microscopy and transmission electron microscopy indicate that the width of grain boundary is only a few atomic spacings. The coincident site lattice theory¹⁹ describes a general boundary in terms of superposition of a dislocation small angle boundary on the appropriate coincidence site boundary. This theory requires that the grain boundary follows a plane in the coincidence boundary containing a high density of coincident sites. Deviations from this plane are accommodated by steps. Actually, the ledge structure is just a special case of the random dislocation interfacial structure. These boundaries allow diffusion to occur more rapidly than in the matrix and are thus sites of easy precipitate nucleation. Also, boundaries are sinks for vacancies and special morphological problems such as precipitate free

zones may develop as a result of these diffusions (see Fig. 7 lecture 6).

H. Volume Defects. These are three dimensional microscopic defects compared to the atomic dimensions of the previous categories. They include second phase particles such as non-metallic inclusions (e.g. oxides/sulphides in steel), voids, cracks, undesirable second phase particles (e.g. Bi in Cu). Control of the particles in this class depends on composition and processing.

Voids and cracks can develop by diffusion (e.g. Kirkendall effect) by the association of vacancies, and during deformation, especially in fatigue and creep. Cracks start off by the separation of surfaces, e.g. at intermetallic particle/matrix interfaces. Their propagation can be slow or catastrophic depending on stress distribution, magnitude, and properties of the material. All of these defects play a major role in service performance of metals particularly strength, ductility and toughness.

3. Summary of Lattice Defects

Point Defects

- a. Vacant Lattice Sites
- b. Substitutional Solute Atoms
- c. Interstitial Solute Atoms
- d. Point Defect Aggregates/Clusters of any or all of the above.

Line Defects

- a. Whole dislocations (Burgers vector $\vec{b} = \text{Lattice Vector } \vec{T}$)
- b. Partial dislocations (Burgers vector $< T$)
- c. Superdislocations (Burgers vector = superlattice vector)

18. H. Gleiter, et al., Acta Met. 16, 1053, 1968.
19. D. G. Brandon, et al., Acta Met. 12, 813, 1964. See also Field Ion Microscopy, Plenum Press, New York, 1968.
20. G. Thomas, Phil. Mag. 17, 1097 (1968).

Some Examples of General Reading

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See also references to next chapters.

FIGURE CAPTIONS

Fig. 1 (a) Lattice sites all occupied - perfect crystal

(b) Atom #1 has been removed leaving mono-vacancy, and local collapse occurs. A vacancy pair (divacancy) is created if any of the nearest neighboring atoms 2, 3, 4, or 5 is also removed. Other complexes are possible. (c) Interstitialcy created by an atom being forced between atoms on normal lattice sites.

a → b → c can occur by irradiation damage: atom 1 is ejected leaving a vacancy and creating an interstitialcy.

Similar distortions can occur from solute impurities, e.g.

(b) represents slightly smaller atom than solvent (c) interstitial atom such as C, N, O, H, B.

Fig. 2. Defects resulting from vacancies a) perfect dislocation loops and helices in Al-5% Mg quenched from 500°C, b) stacking fault tetrahedra in quenched gold. Transmission electron micrographs (100kV).

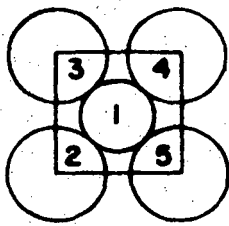
Fig. 3 (a) Slip plane containing a dislocation loop. The screw (S), edge (E), and mixed (M) components are marked.

(b) view normal to the edge dislocation line; there is an extra half plane associated with it. Slip causes the line to move by successive local bond changes across 1-2-3-4-5.

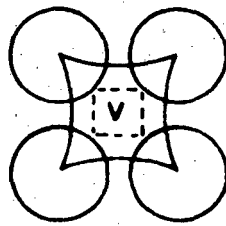
Fig. 4. (a) The close packed plane in fcc (and hcp) metals. The normal lattice translation $\vec{T} = \vec{AB} = \vec{BC} = a/2\langle 110 \rangle$ and this can also occur by partial translations $a/6[1\bar{2}1]$ followed by $a/6[2\bar{1}\bar{1}]$ which enclose a stacking fault (b). (c) shows a Frank dislocation loop surrounding a fault - note that in (b) the Burgers vectors lie in the fault plane whereas in (c) it lies normal to it.

- Fig. 5 Cu-33-1/2% Zn deformed 5% in tension the dislocation network contains extended nodes which are used to determine stacking fault energies (e.g. at A). Electron micrograph.
- Fig. 6 Stacking fault energies of α -brasses determined from nodes. The types of dislocation configuration after 10% tensile strain are indicated (see fig. 7).
- Fig. 7 Changes in dislocation substructure in α -brasses deformed 10% in tension at 273°K a) 4% Zn, b) 8% Zn, c) 16% Zn, d) 24% Zn, e) 33.5% Zn. Notice change from cells to planar groups as % Zn increases and SFE decreases (fig. 6). Electron micrographs.
- Fig. 8 Showing the possible dislocations in the (111) fcc plane. Three other equivalent {111} planes have similar possibilities.
- Fig. 9 Direct observations of dislocation loop nucleation in Al-5% Mg. The precipitates at S and T cause interstitial prismatic loops to be punched out. Electron micrograph, from Eikum and Thomas Acta Met 12, 537 (1964).
- Fig. 10 Intersecting screw dislocations in niobium forming a twist boundary in a plane nearly parallel to the page. Electron micrograph.
- Fig. 11 A Bardeen-Herring (climb) source showing the creation of dislocation loops from an edge dislocation pinned at AB. The slip plane is normal to the page.
- Fig. 12 Example of a climb source in Al-5% Mg after quenching. More and more prismatic loops are generated during aging.
- Fig. 13 Antiphase domain boundaries on {100} planes in ordered Cu₃ Au. Electron micrograph

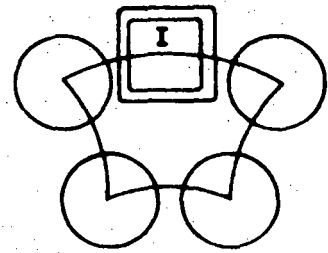
- Fig. 14 (a) Coherent interface - there may be some elastic strains at the interface, e.g. G.P. zones, spinodals, antiphase boundaries.
- (b) partial coherency, structural dislocations exist in the interface plane AB. Interface AC is coherent. e.g. intermediate phases small angle boundaries.
- (c) non-coherent, e.g. grain boundaries, inclusions.



(a)



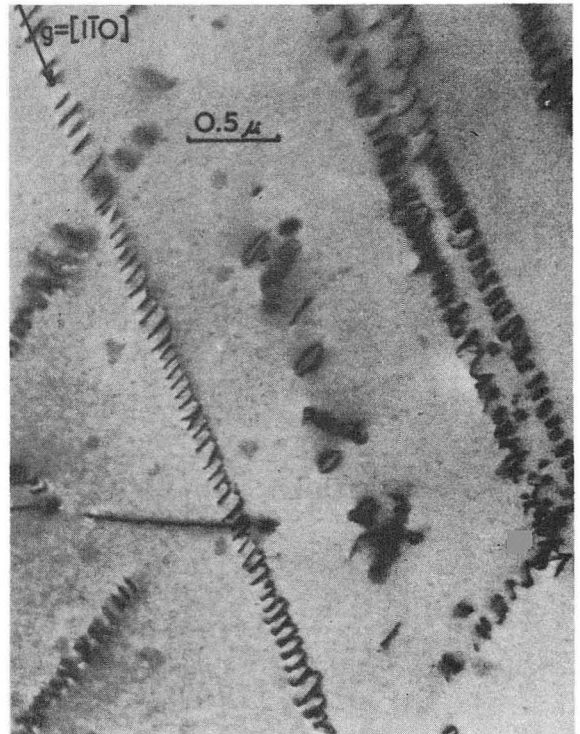
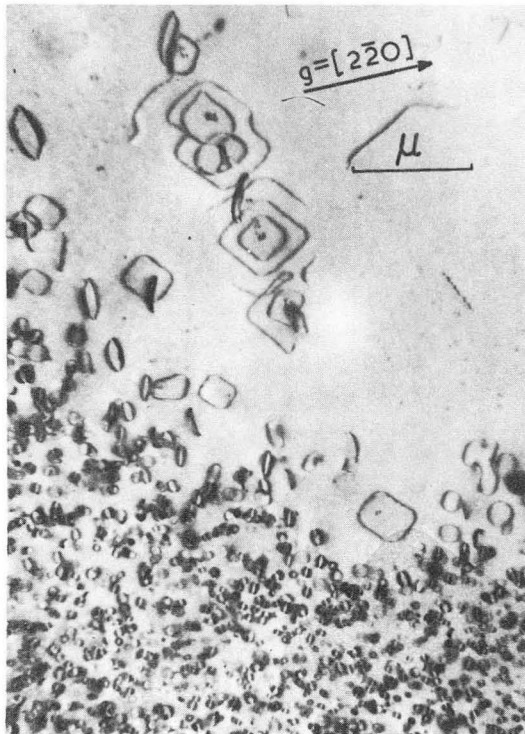
(b)



(c)

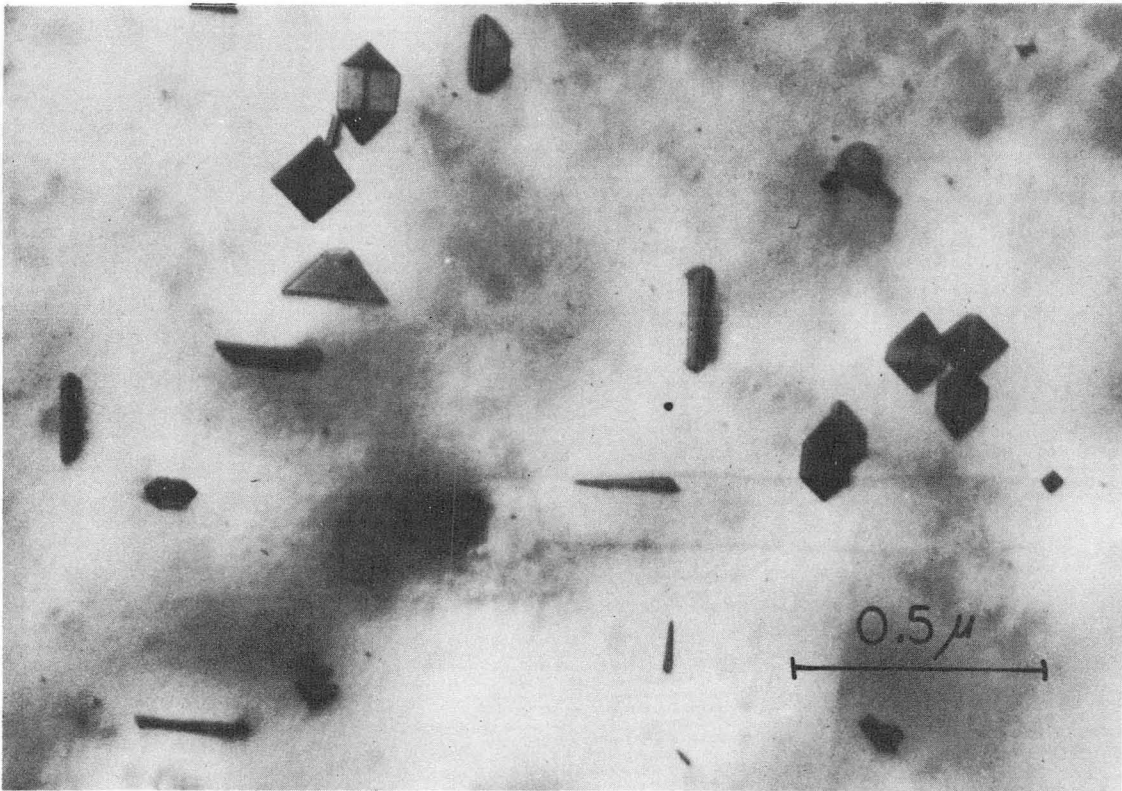
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Figure 1



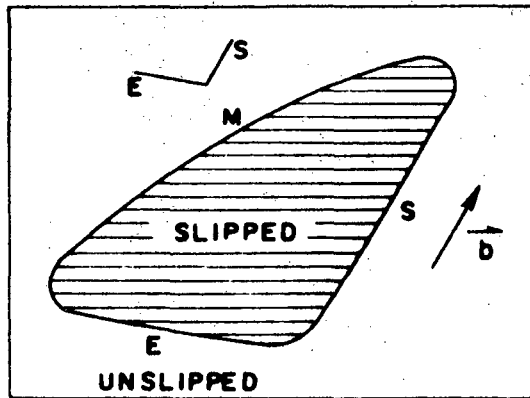
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Figure 2a

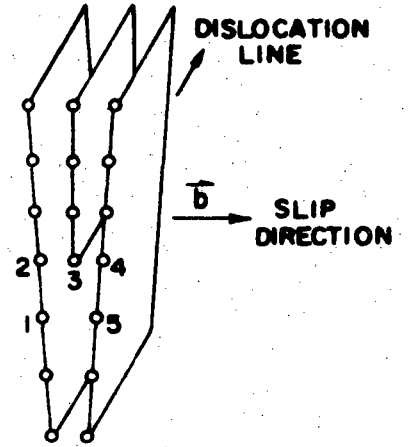


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Figure 2b



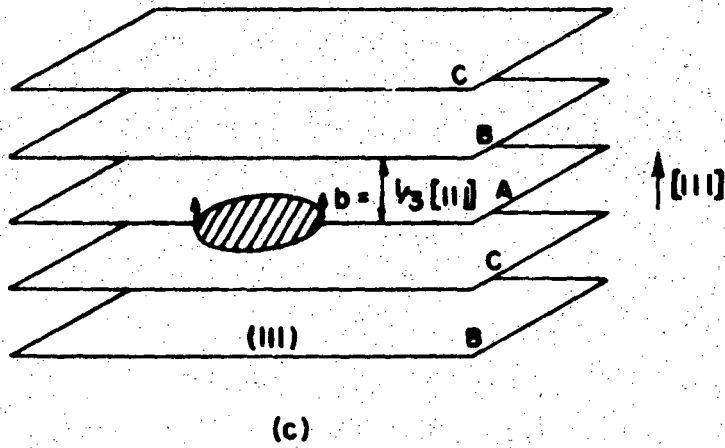
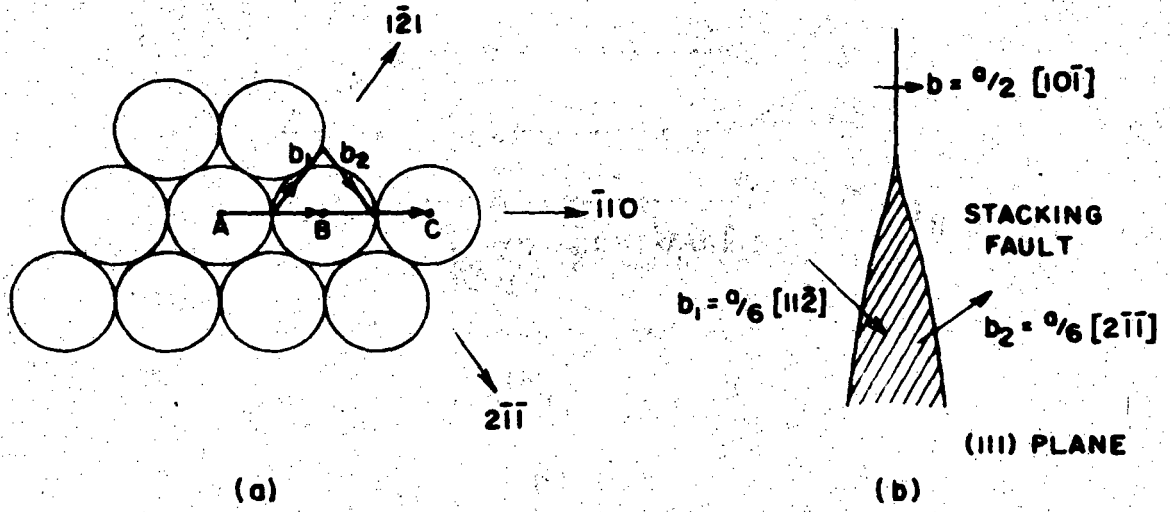
(a)



(b)

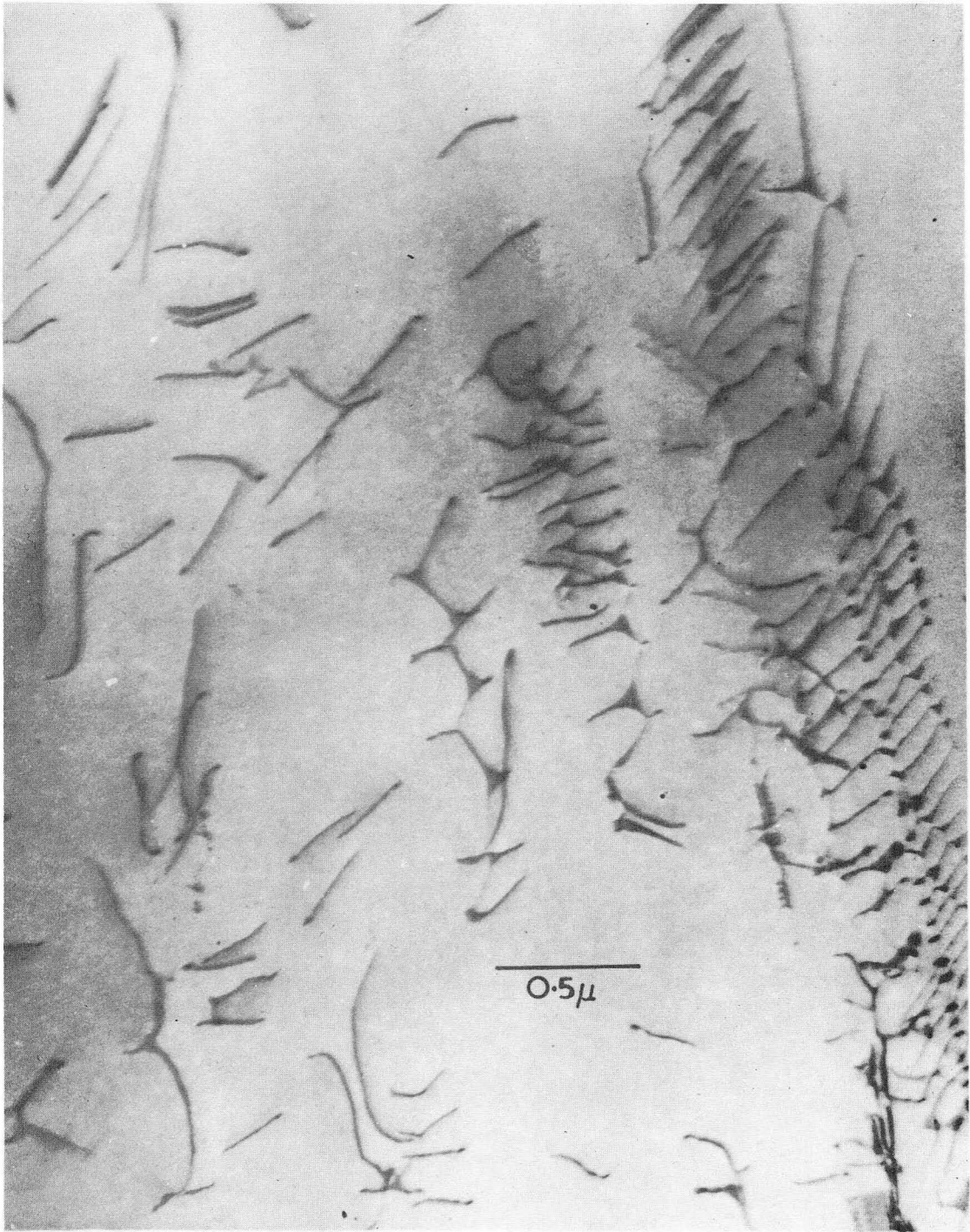
XBL 7011-6930

Figure 3



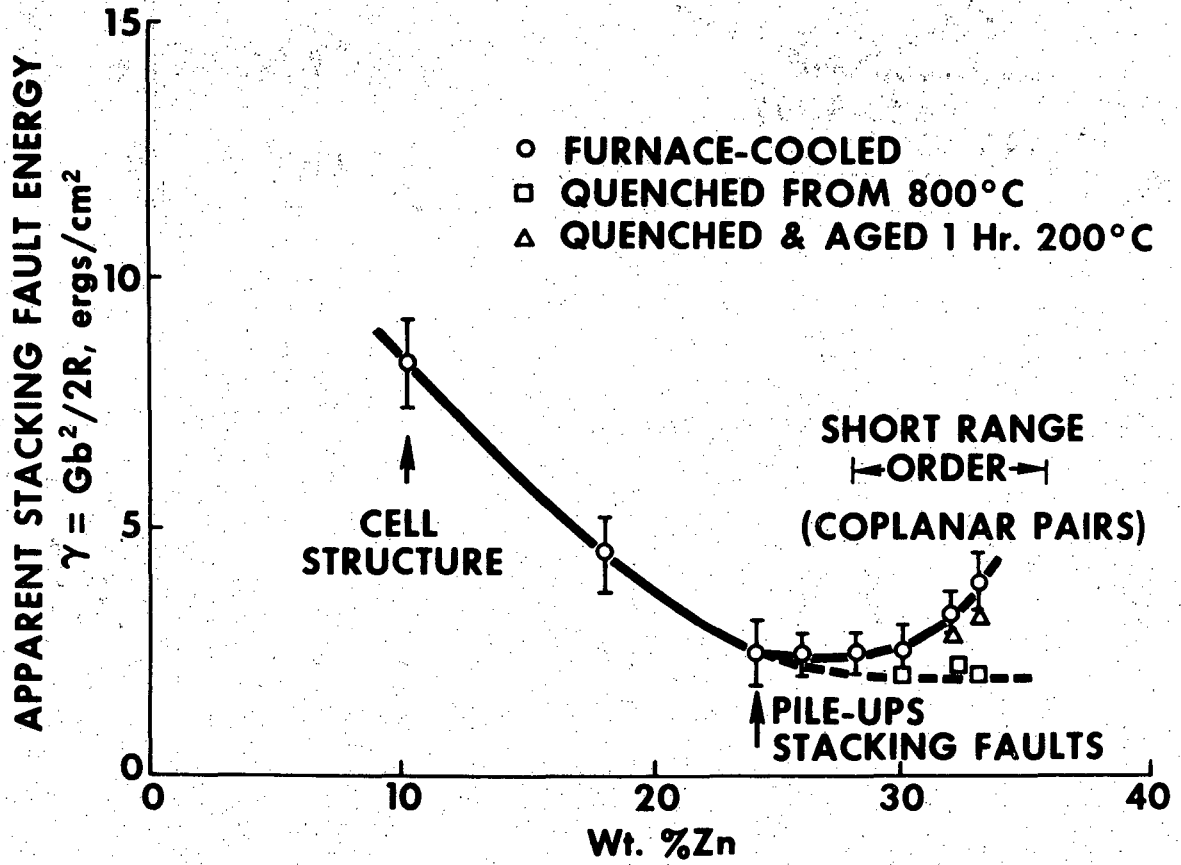
XBL 7011-6931

Figure 4



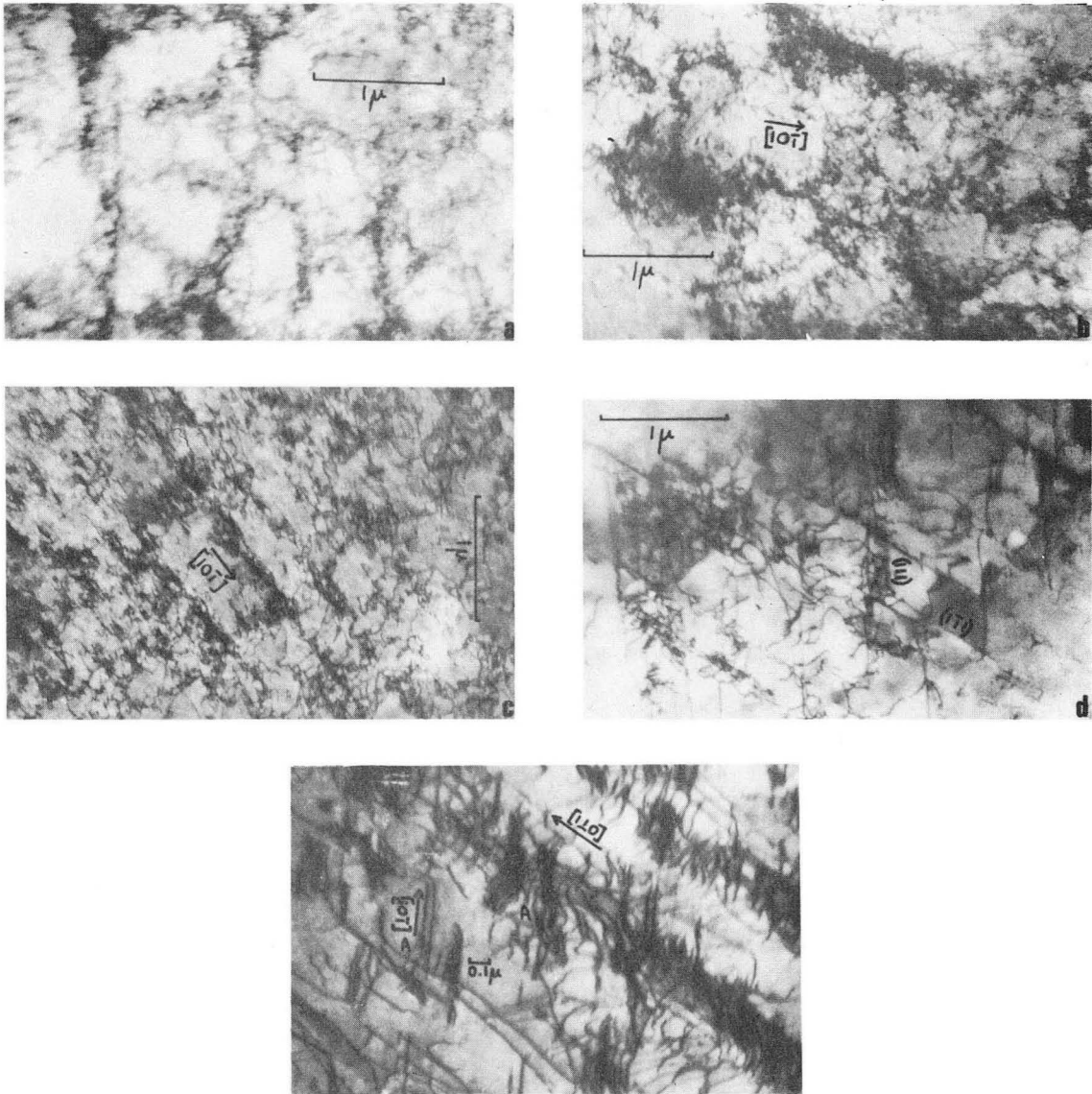
IM 863

Figure 5



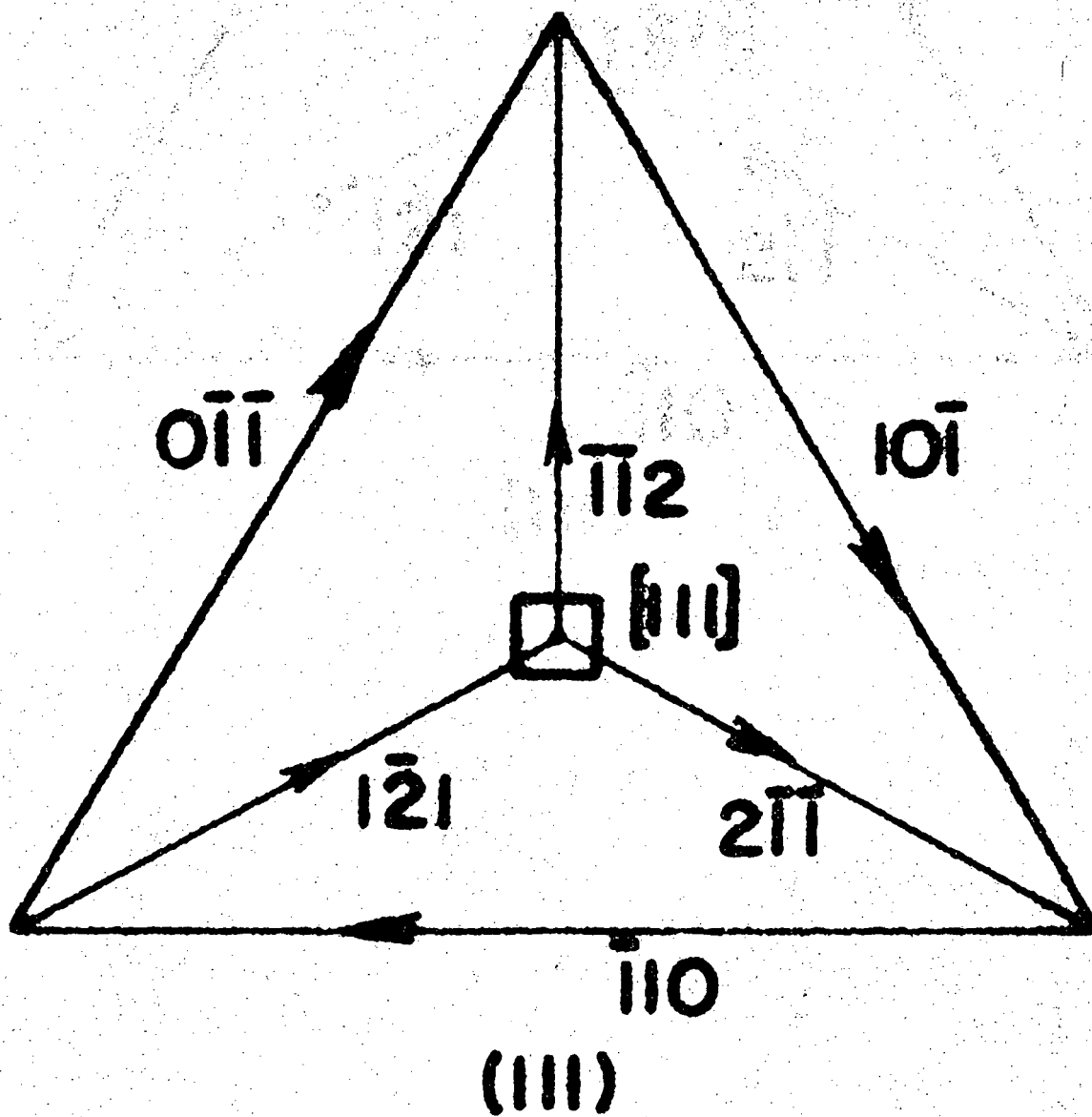
MU-30116

Figure 6



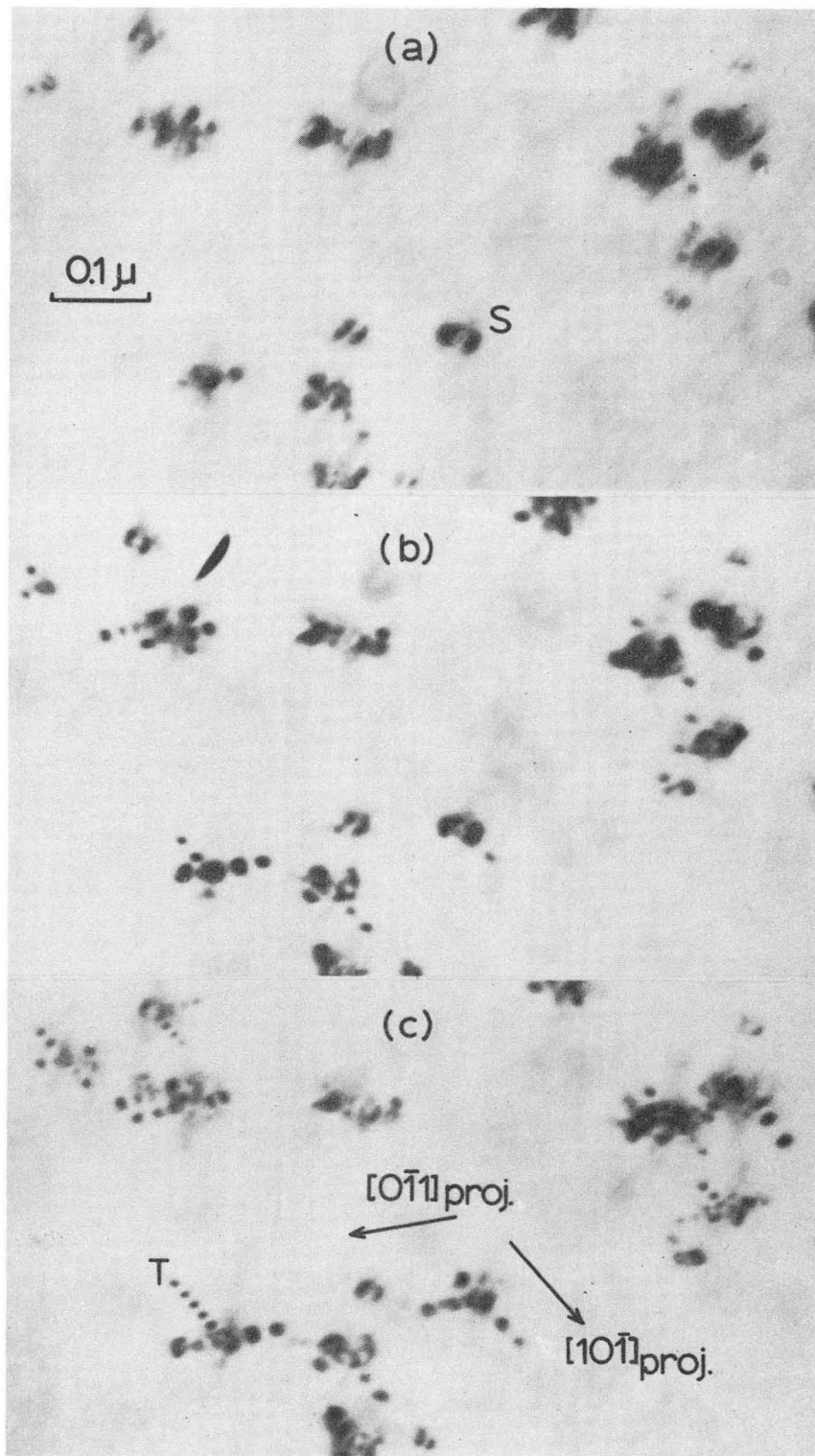
XBB 708 3533

Figure 7



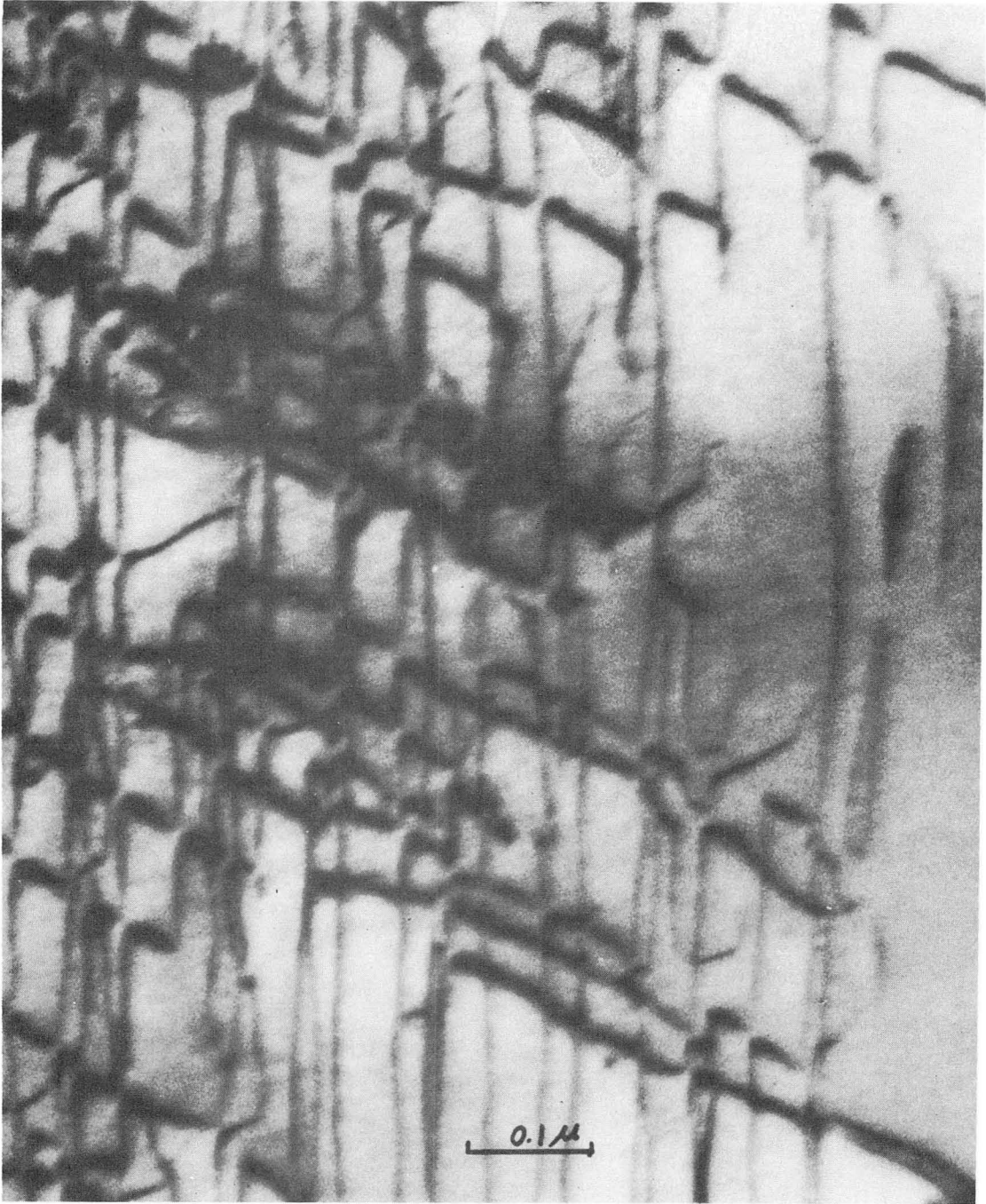
XBL 7011-6928

Figure 8



IM 724

Figure 9



XBB7011-5177

Figure 10

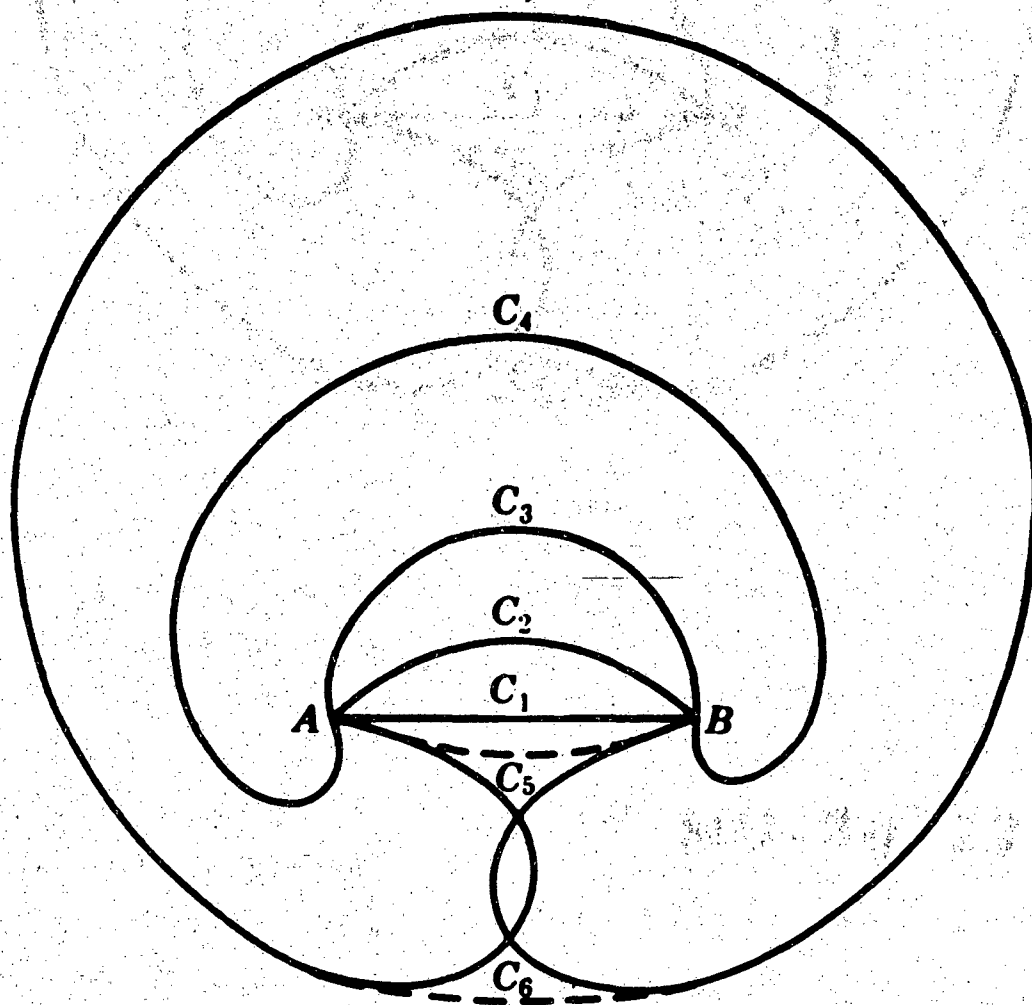
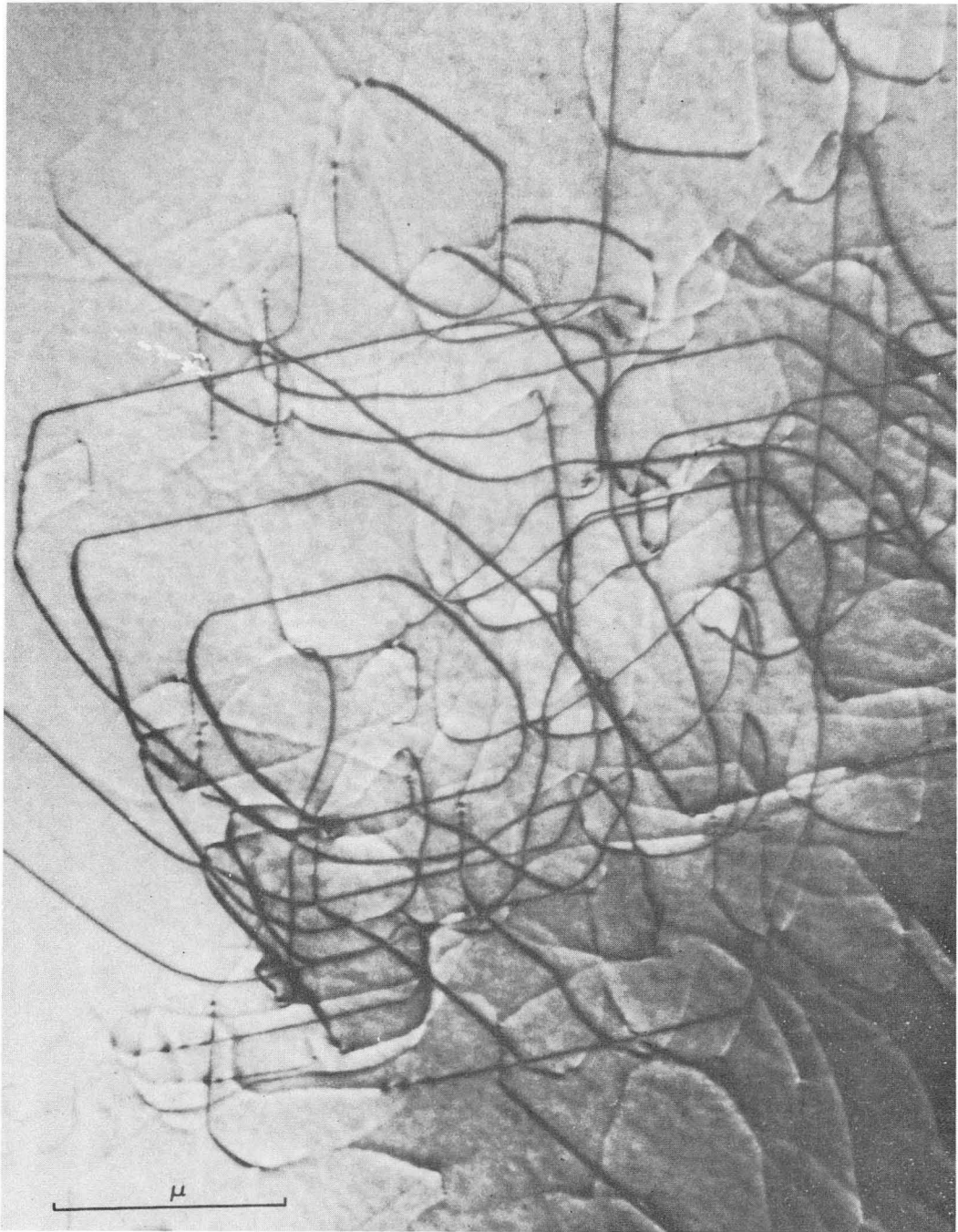


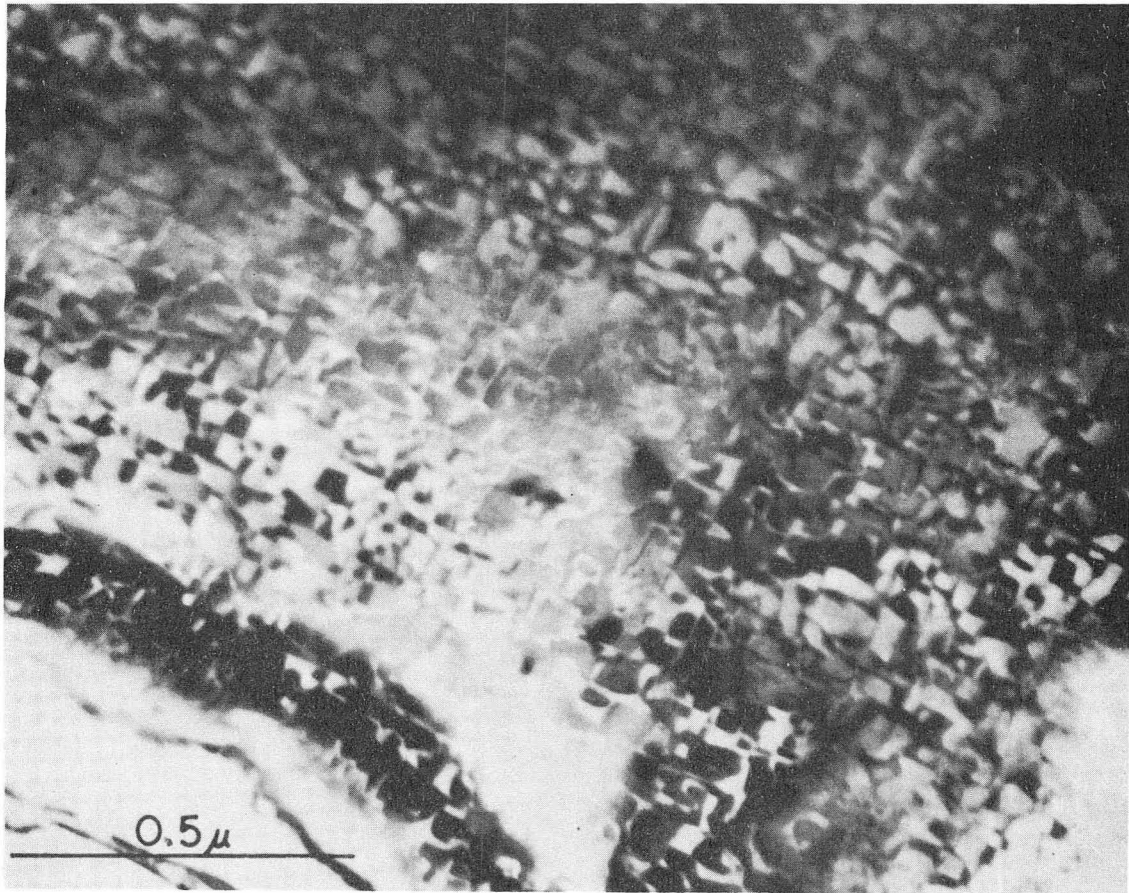
Figure 11

MU-30122



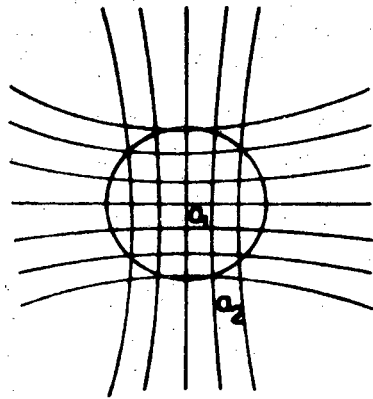
XBB7011-5100

Figure 12

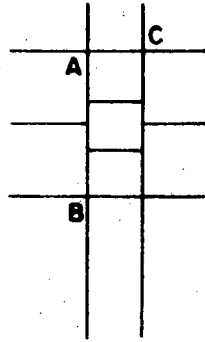


IM 1659

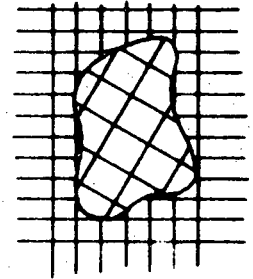
Figure 13



(a)



(b)



(c)

XBL 7011-6932

Figure 14

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