THE INFLUENCE OF INTERFACES ON DISLOCATION MOBILITY

THE INFLUENCE OF INTERFACES ON DISLOCATION MOBILITY

By

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SCOPE AND CONTENTS:

A numerical method was developed to calculate the passing stress of pile-ups in a general internal stress field. This method was used to calculate the passing stress for a pile-up of like edge dislocations for a sinusoidal internal stress field, a simple tilt wall, and a Van der Merwe net of misfit dislocations. The passing stresses for single dislocation for these internal stress fields were also calculated.

The Burgers vectors of a common set of misfit dislocations at the interface of Ni_3Ge particles in a Ni matrix were determined. The surface slip lines of a directionally solidified sample of Ni-20% Ge alloy tested in compression were examined.

ACKNOWLEDGMENTS

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CHAPTER 1

INTRODUCTION

The correlation of the mechanical properties of materials and the detailed microstructure is the basis of many metallurgically important problems. This problem is essentially the calculation of the resistance of internal stresses to the motion of dislocations. The internal stress field can be due to dislocation debris or second phase particles or both. Theoretical estimates of the flow stress have been based on the sum of the internal stresses acting on a single dislocation. However, experimental observations show that large numbers of dislocations move on slip bands. Thus the calculation of flow stress should consider the resistance of internal stress fields to the movement of large numbers of dislocations rather than single dislocations.

To describe a slip band it is mathematically preferable to describe a slip band as a continuous distribution of dislocations of infinitesimal Burgers vector instead of a band of discrete dislocations. Considering only a single dislocation overestimates the flow stress and thus the objective of the present analysis, is a comparison of the mobility of single dislocations and pile-ups. A few idealized barriers to dislocation motion were considered.

The type of obstacles considered was chosen to exemplify the use of the method to a variety of metallurgically important problems. A periodic internal stress field was chosen to test the numerical solutions against the analytical method developed by Smith (1967).

Also such distributions of internal stress may result from accumulation of dislocations in work hardening, although it is unlikely that the idealized sinusoidal model discussed here is itself of any direct physical significance.

The motion of groups of dislocations through simple tilt boundaries was chosen because of the importance of simple tilt substructures in determining the yield characteristics of hot worked and thermal mechanically processed materials.

The model of the interphase interface dislocation network was chosen to show the value of the method in regard to two phase materials. It is recognized that the interface dislocations do not necessarily represent the most significant contribution to the flow stress of such materials.

To relate the theoretical concepts developed concerning the propagation of slip bands in two phase solids an experimental program was undertaken. The objective of this program was to determine the interphase interfaces developed in a directionally solidified two phase material and the mechanism of plastic deformation.

CHAPTER 2

LITERATURE REVIEW

The investigation of the motion of groups of glide dislocations in two phase materials necessitates a review of the mathematical methods of dealing with large numbers of like dislocations and the nature of the barriers resisting dislocation motion. Thus the literature review is divided into two sections which consider each of these aspects separately.

2.1 The Nature of Interphase Interfaces

In general any interface between two phases must contain two energy terms, structural and chemical. The energy due to the internal stress produced by the second phase is covered in the structural term. The energy due to the broken chemical bonds across the interface is covered by the chemical term.

In discussing the interaction of glide dislocations with second phase particles it must be recognized that in general a number of types of long and short range interactions are possible. Firstly for coherent particles, if the average atomic volume of the two phases is different, long range elastic stresses can be established in the matrix as discussed by Mott and Nabarro (1948). They identify the flow stress with a simple arithmetic mean of the internal stress produced by the coherent precipitates and they obtain the following equation for the flow stress,

$$\sigma = 2\mu \, \varepsilon \, f \qquad 2.1$$

$\varepsilon = 3K\delta/[3K + 2E/(1+\nu)]$, where K is the bulk modulus.

where μ is the shear modulus, f is the volume fraction of the precipitate, and E and ν are Young's modulus and Poisson's ration of the matrix. The atomic volume of the precipitate is $(1 + \delta)^3$ where the atomic volume of the matrix is unity.

Since the elastic moduli of the precipitate and matrix may be different, this also may produce a long range force on approaching dislocations as suggested by Fleischer (1960).

Second phase particles can have short range forces on dislocations other than those due to the stress field of the particles. If a dislocation cuts a particle, then the dislocation must do work to create the extra particle-matrix interface of the cut particle. If the particle is ordered, then, if the Burgers vector of the matrix is not equal to the repeat distance of the ordered matrix of the particles, then work must be done by the dislocation to create the disordered interface across the slip plane. These short and long range forces of particles on dislocations are reviewed in detail by Kelly and Nicholson (1963).

For large particles, the misfit between the lattices of the matrix and the particles can be accommodated by a network of dislocations. If the misfit is completely accommodated by interface dislocations, then the short and long range elastic stresses produced by the second phase particle can be attributed to the interface dislocations.

Frank and Van Der Merwe (1949) were the first to suggest that the misfit between the two lattices of crystals grown epitaxially on one another, is accommodated by a grid of dislocations in the interface of the two crystals. Because these dislocations accommodate misfit, the stress field of these dislocations is less than a dislocation of the same Burgers vector produced by deformation. Van Der Merwe (1950) calculated the energy and stress of a set of dislocations which accommodate the misfit between two semi-infinite crystals which have a misfit in only one direction. The equations are linear, and thus solutions can be superimposed to obtain stress fields of more complicated misfit dislocation networks.

When the misfit is completely accounted by dislocations, the spacing of the dislocations is (Brooks 1952)

$$d = |b| \frac{(d_1 + d_2)}{2(d_1 - d_2)} \qquad 2.2$$

where $|b|$ is the magnitude of the Burgers vector and d_1 and d_2 are the spacings of lattice planes for the two crystals.

Mathews (1961) observed the first misfit dislocation network in the interface between a thin film of PbSe deposited epitaxially on a thin film of PbS. The dislocation network was a square grid of edge dislocations with Burgers vectors of the a/2 [110] type. The dislocation spacing agreed with the misfit between the two lattices.

As in epitaxially grown specimens, coherent precipitates can become semi-coherent at large sizes and contain a dislocation network to accommodate the misfit. This was first observed by Merrick and Nicholson (1962). They observed misfit dislocations on plate like precipitates of Ni_3Ti in a Ni-20% Cr - 6% Ti alloy. Phillips (1966) observed misfit dislocations for spherical, cobalt rich precipitates in a Cu - 3.12% Co alloy. Weatherly and Nicholson (1967) observed a hexagonal network of dislocations with Burgers vectors of a/2 [110] type in spherical precipitates of Ni_3 (Ti, Al) in a Nimonic 80A alloy. They also observed a square grid of dislocations with a [100] type Burgers vector for disc-shaped precipitates in Al-Cu alloy and also they observed arrays of loops with Burgers vector of the type a/2[110] spaced along the length of lath shaped precipitates in a Al-Cu-Mg alloy. Weatherly and Nicholson found that the spacing of the misfit dislocations agreed with the Brooks formula (equation 2.2).

Laird and Aaronson (1967) investigated Widmanstatten γ plates in an Al-15% Ag alloy and found that the misfit between the plates and the matrix was accommodated by a hexagonal network of dislocations with Burgers vectors of the type a/6 [112] or a network of two sets of parallel dislocations with Burgers vectors of the type a/6[112].

In addition to the direct interaction of dislocations with the particles which occurs if the particle is sheared, indirect interaction may occur in the case of hard particles which don't deform. The matrix must produce large numbers of dislocations in the vicinity of these hard particles to keep the material continuous. This causes localized work-hardening of the matrix. The theory for this localized work-hardening has been developed by Ashby (1968).

2.2 Dislocation Pile-Ups

If the process of plastic yielding is considered as the motion of single dislocations, the applied stress required for dislocations to overcome an obstacle gives a maximum applied stress. If a pile-up of dislocations against the barrier is considered, the required applied stress for the leading dislocation to overcome the obstacle is lower since the other dislocations in the pile-up create a stress concentrate at the head of the pile-up aiding the leading dislocation to overcome the obstacle.

Cottrell (1949) made the first calculation of the stress concentration at the head of a pile-up, where the obstacle had only a very short range stress field. That is the stress field of the obstacle acts only on the leading dislocation of the pile-up. By means of a virtual work argument, he calculated the stress at the head of a pile-up to be $n\sigma$, where n is the number of dislocations in the pile-up and σ is the applied stress.

Suppose there are n parallel infinitely long dislocations on the slip plane $y = 0$ which are piled up against some obstacle. Let the axis of these dislocations be parallel to the z-axis and there be dislocations at points $x = x_i$. Let $P(x)$ be the stress acting on these dislocations due to the applied stress and due to the stress field of the obstacle. For these dislocations to be in equilibrium the following equation must be satisfied,

$$\sum_{\substack{i=1 \\ i \neq j}}^{n} \frac{A}{x_j - x_i} + P(x_j) = 0 \quad j = 1, 2, \text{---} n \qquad 2.3$$

where $A = \frac{\mu b}{2\pi}$ for screw dislocations and $A = \frac{\mu b}{2\pi(1-\nu)}$ for edge dislocations. Here μ is the shear modulus, b is the Burgers vector of the dislocations, and ν is Poisson's ratio. In equation 2.3, $P(x)$ is the appropriate component of shear stress, xy component for edge dislocations and yz component for screw dislocations. Eshelby, Frank and Nabarro (1951) developed a general method of solving this equation for the positions taken up by the dislocations in the pile-up and solved for the case of n-1 dislocations piled up against a locked dislocation. They also found that the stress concentration at the head of the pile-up to be $n\sigma$ where σ is the applied stress. If the length of the pile-up is L, the number of dislocations, n, can be expressed as:

$$n = \frac{L\sigma}{2A} \qquad 2.4$$

Thus the stress at the head of the pile-up is

$$\frac{L\sigma^2}{2A} \qquad 2.5$$

Although the method of Eshelby, Frank and Nabarro is exact, it is very difficult to put into practice and thus is rarely used.

Head and Louat (1955) considered the pile-up to be a group of smeared out dislocations of infinitesimal Burgers vector instead of discrete dislocations. This enables equation 2.3 to be approximated with the integral equation,

$$A \int_D \frac{f(x)}{x_i - x}\, dx + P(x_i) = 0 \qquad 2.6$$

where A and P(x) are as defined previously, and f(x) is the dislocation density in the pile-up at point x. This integral is taken over region D which contains all dislocations in the pile-up. Head and Louat developed a general method of solving for f(x) which is simplier than the exact method of Eshelby, Frank and Nabarro. Head and Louat's results agree with those of the exact method when a large number of dislocations are in the pile-up.

Smith (1967) extended the method of Head and Louat to consider the mobility of groups of dislocations in a variety of internal stress fields. Suppose a pile-up extends from A to B, then equation 2.6 becomes

$$A \int_{A}^{B} \frac{f(x)}{x_i - x} \, dx + P(x_i) = 0 \qquad 2.7$$

If there are no singularities in P(x) and thus in f(x), there is a solution for f(x) only if the following equation is satisfied (Head and Louat, 1955).

$$\int_{A}^{B} \frac{P(x)\, dx}{\sqrt{(x-A)(x-B)}} = 0 \qquad 2.8$$

But $P(x) = P_1(x) - \sigma$ where $P_1(x)$ is the internal stress acting on the pile-up and σ is the applied stress. Thus if the stress $P_1(x)$ is known (the stress field of some obstacle) then σ the applied stress to propagate a pile-up with a source at A to B in the internal stress $P_1(x)$, can be calculated from equation 2.8. This method can be applied to any situation where the analytical form of the internal stress field is known, but it will be most accurate when the internal stresses are not rapidly varying functions in the region AB.

Very recently Smith (1968) developed a method where he leaves the leading dislocation discrete and smears out the remaining dislocations in the pile-up. This should be strictly a more accurate approach in the case of rapidly varying internal stresses. However in the present work the smeared out pile-up has been used in an effort to contrast the behaviour of single dislocations and groups of dislocations with respect to a variety of obstacles.

CHAPTER 3

MOBILITY OF SINGLE DISLOCATIONS AND PILE-UPS

3.1 Theory on the Mobility of Pile-Ups

To treat the problem of a pile-up of dislocations in a general internal stress field, the method of Smith (1967) was adopted. Thus it is necessary to review in detail the solution of equation 2.6 as developed by Head and Louat (1955).

Equation 2.6 becomes on rearrangement:

$$A \int_D \frac{f(x)}{x-x_i}\, dx - P(x_i) = 0 \qquad 3.1$$

Suppose P(x) is a known function and f(x) an unknown function and that D consists of p finite segments of the x-axis (a_1,b_1), (a_2,b_2) ---, (a_p,b_p). Suppose that at q of the end points of the segments, denoted by C_1, C_2 ---, C_q, f(x) is to remain bounded, and that at the remaining 2p-q end points, denoted by C_{q+1}, C_{q+2}, ---, C_{2p}, f(x) may be unbounded. Let

$$R_1(x) = \prod_{k=1}^{q} (x - C_k), \quad R_2(x) = \prod_{k=q+1}^{2p} (x - C_k) .$$

Then if $p-q \geqslant 0$, solutions to equation 3.1, bounded at C_1, C_2 ---, C_p, always exist and are given by

$$f(x_i) = \frac{-1}{\pi^2 A} \sqrt{\frac{R_1(x_i)}{R_2(x_i)}} \int_D \sqrt{\frac{R_2(x)}{R_1(x)}}\, \frac{P(x)}{x-x_i}\, dx + \sqrt{\frac{R_1(x_i)}{R_2(x_i)}}\, Q_{p-q-1}(x_i) \qquad 3.2$$

where $Qp\text{-}q\text{-}1(x_i)$ is an arbitrary polynomial of degree not greater than p-q-1 (it is zero for p=q).

If p-q < 0, a unique solution, bounded at C_1, C_2, ---, C_q, exists if and only if P(x) satisfies the conditions

$$\int_D \sqrt{\frac{R_2(x)}{R_1(x)}}\ x^n\ P(x)\ dx = 0 \quad \text{for } n = 0, 1, ---, (q\text{-}p\text{-}1) \qquad 3.3$$

and if this is so the solution is given by equation 3.2, with $Qp\text{-}q\text{-}1(x_i) = 0$.

Smith (1967) continued this development. Consider a pile-up extending from A to B, in a stress field $P(x) = P_1(x) - \sigma$, where $P_1(x)$ is the internal stress field, and σ is the applied stress. If $P_1(x)$ has no singularities, then f(x) will be bounded in the region AB, and thus in order that f(x) have a solution equation 3.3 must be satisfied. Thus,

$$\int_A^B \frac{P(x)\quad dx}{\sqrt{(x-A)\ (B-x)}} = 0\ . \qquad 3.4$$

Since $P(x) = P_1(x) - \sigma$, then, the applied stress, σ, necessary to keep a pile-up extending from A to B, in an internal stress field $P_1(x)$, in equilibrium can be calculated. On substitution of $P(x) = P_1(x) - \sigma$, equation 3.4 becomes

$$\sigma = \frac{1}{\pi} \int_A^B \frac{P_1(x)\ dx}{\sqrt{(x-A)\ (B-x)}} = 0 \qquad 3.5$$

Thus if this integration can be performed, σ can be calculated. This integration is usually very difficult to perform analytically for most internal stress fields, $P_1(x)$, except relatively simple analytical forms of $P_1(x)$.

With a change of variable,

$$x = \left(\frac{B-A}{2}\right) X + \left(\frac{A+B}{2}\right) ,$$

equation 3.5 becomes

$$\sigma = \frac{1}{\pi} \int_{-1}^{1} \frac{P_1(X)\, dX}{\sqrt{1 - X^2}} \quad . \qquad 3.6$$

This equation can be converted to a summation plus a remainder (Kelly, 1967) which is

$$\sigma = \frac{1}{n} \sum_{k=1}^{n} P_1(X_i) \quad + \quad \frac{2}{2^{2n}\,(2n)!} \; f^{(2n)}\,(\varepsilon) \qquad 3.7$$

where $X_i = \cos\,[\frac{2i - 1)\,\pi}{2n}]$, $i = 1, 2, ---, n$, and $-1 < \varepsilon < 1$. The last term in equation 3.7 approaches zero for large n and thus equation 3.7 becomes for large n,

$$\sigma = \frac{1}{n} \sum_{k=1}^{n} P_1(X_i) \qquad 3.8$$

Equation 3.8 was tested for a suitable value of n. It was found on trying different values of n, that the results for equation 3.8, changed very little for n > 6. Thus a value of n = 14 was used for the remaining calculations.

All numerical calculations of σ were performed with the aid of the 7040 IBM computer.

Using equation 3.8, the applied stress required to propagate a pile-up to various points in an internal stress field $P_1(x)$ can be calculated by changing values of A and B. As a further test of equation 3.8, the results of σ for an internal stress field $P_1(x) = \sigma_1 + \frac{\sigma_m}{2} [1 - \cos (\frac{\pi x}{a})]$ were calculated and compared with those of Smith (1967) who calculated σ for this internal stress field analytically. As can be seen in Fig. 3.1 the results agree very well with those of Smith. Further examination of Fig. 3.1 indicates that the required applied stress for a pile-up to propagate through this internal stress field, is less than that for a single dislocation.

3.2 Slip Propagation Through a Simple Tilt Wall

Simple tilt walls are formed during polygonization and creep deformation and it is thus of value to examine the role of such substructural barriers in resisting the propagation of slip bands. The stress fields of a simple tilt wall have been developed by Cottrell (1952) and Li (1961). In all the following calculations on simple tilt walls, it is assumed that the dislocations of the tilt wall are pinned and thus can not move on being approached by either a single dislocation or a pile-up.

Let the tilt wall be in the x = 0 plane, the dislocations be parallel to the z-axis and the Burgers vectors of the dislocations be parallel to the x-axis. Let the spacing between the dislocations in the tilt wall be h, and one of these dislocations pass through the origin. Let a pile-up of like edge dislocations on the y = h/2 slip plane, with their axis parallel to the z-axis, and their Burgers parallel to the

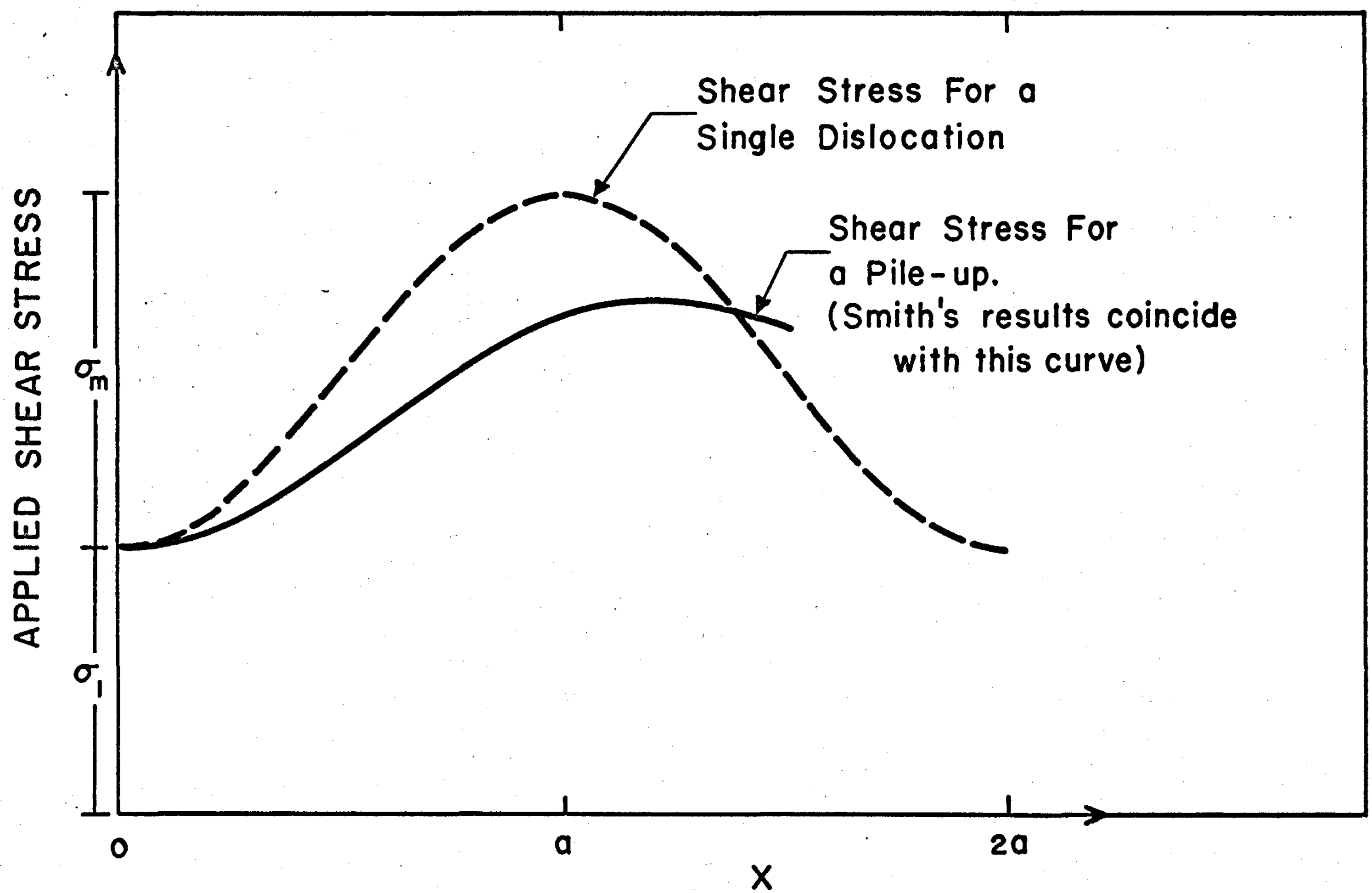


Figure 3-1. Applied shear stress required to propagate the leading dislocations of a pile-up to x, (full curve). Source of pile-up is at x=0 .

x-axis, approach this tilt wall. For this edge dislocation pile-up, $P_1(x)$ is the xy component of shear stress for the tilt wall and thus

$$P_1(x) = \frac{\mu b \alpha}{h(1-\nu)} [\cosh(2\alpha) \cos(2\lambda) - 1]/[\cosh(2\alpha) - \cos(2\lambda)]^2 \quad 3.9$$

where μ is the shear modulus, b is the Burgers vector of the tilt wall dislocations, ν is Poissons ratio, $\alpha = \frac{\pi x}{h}$ and $\lambda = \frac{\pi y}{h}$. Using equation 3.8, with A = - 10h, the applied stress required to propagate a pile-up with a source at -10h, to various positions with respect to the tilt wall were calculated by varying the value of B. These results are plotted in Fig. 3.2. Also in Fig. 3.2 are plotted the required applied stress to propagate a single edge dislocation with the same plane, same Burgers vector and same axis, to different positions with respect to the simple tilt wall. It is seen that the pile-up requires a lower applied stress to overcome this barrier.

If the pile-up length in the preceding discussion is increased, the passing stress, which is the applied stress required for a pile-up to overcome a barrier, will decrease. Calculations similar to those of the previous paragraph were carried out for various pile-up lengths. The passing stress versus pile-up length is plotted in Fig. 3.3. It is seen as the pile-up length is increased that the passing stress decreases when the pile-up length is less than 50h. For pile-up lengths greater than 50h, the passing stress decreases very little with an increase in pile-up length because the dislocations at the end of the pile-up have very little affect on the leading dislocation, for pile-up lengths greater than 50h.

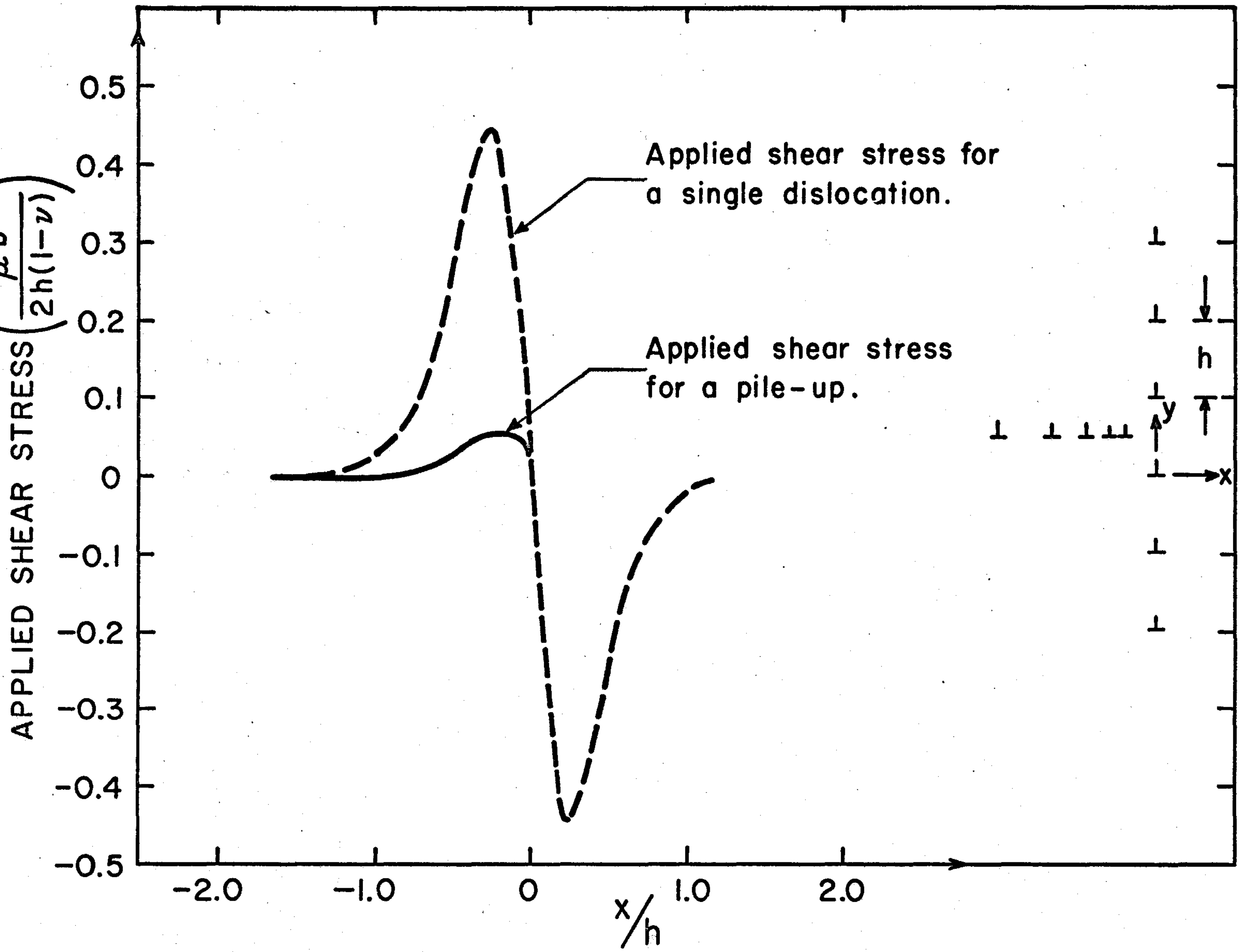


Figure 3−2. Applied shear stress required to propagate the leading dislocation of a pile-up to x,(full curve). Source of pile-up is at $x/h = -10$. There is a simple tilt boundary at $x = 0$.

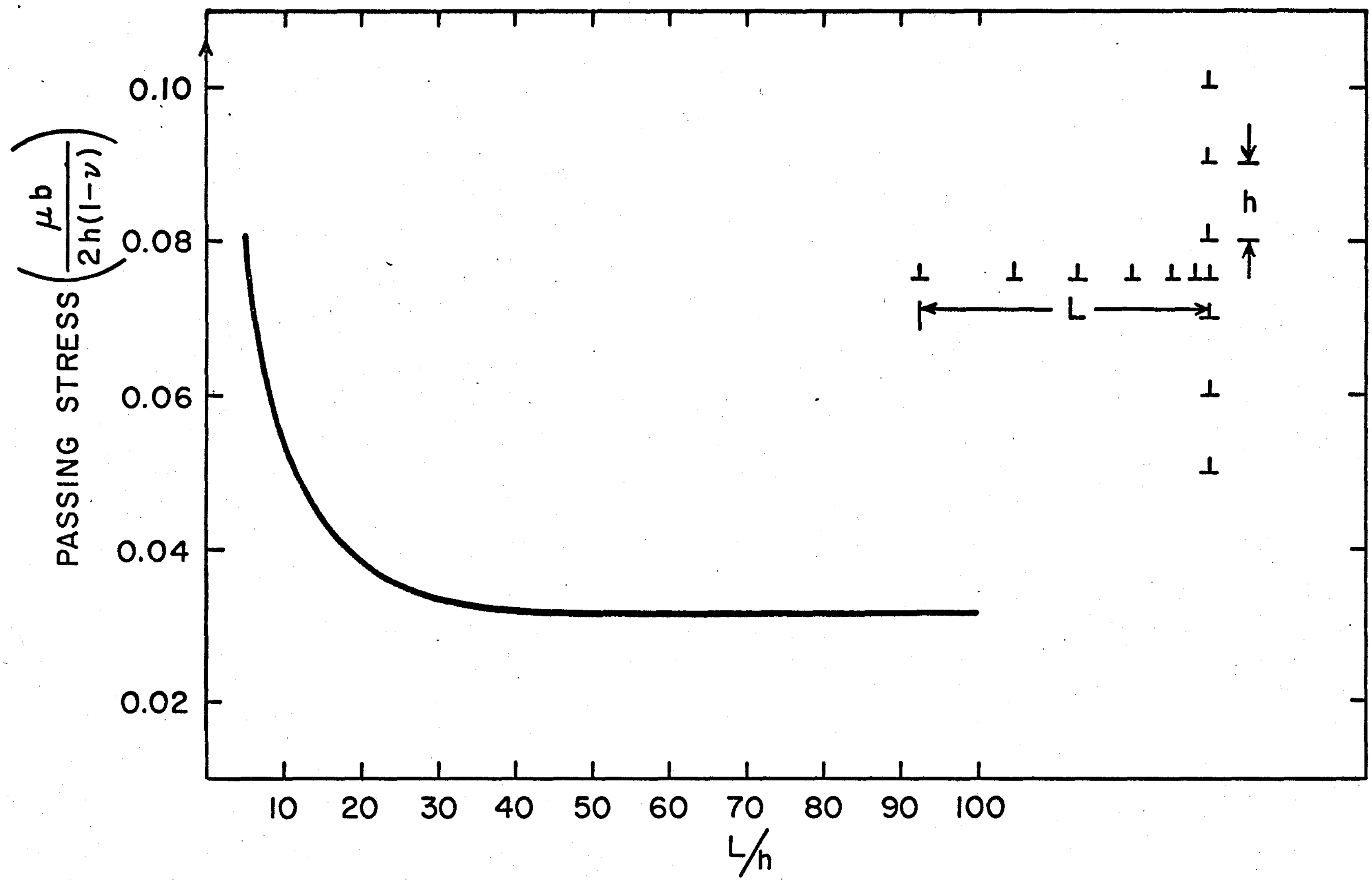


Figure 3–3. Passing stress for a pile-up of length L, through a simple tilt wall.

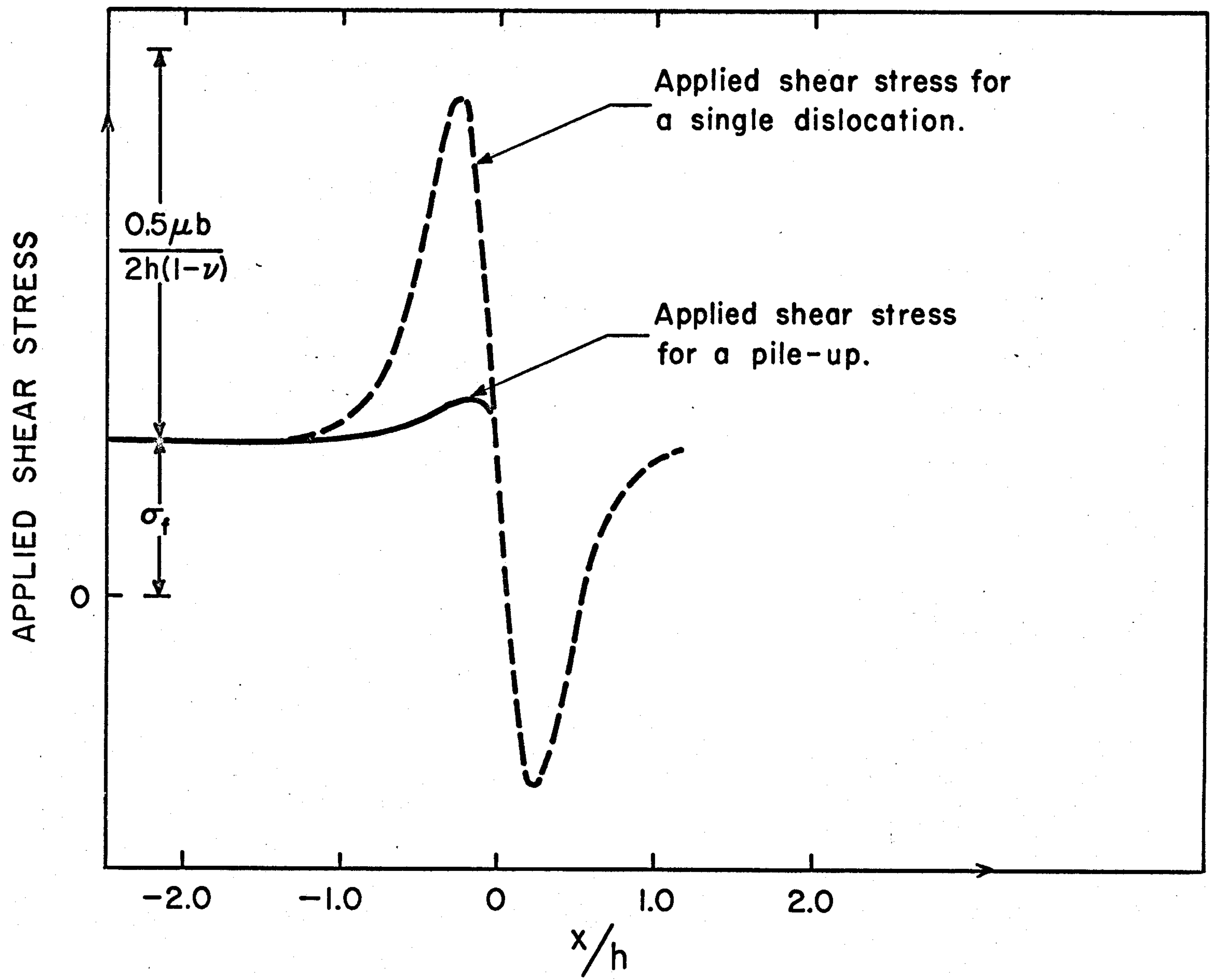


Figure 3-4. Applied shear stress required to propagate the leading dislocations of a pile-up to x, (full curve). Source of pile-up is at $x/h = -10$.

In addition to the effect of substructural barriers the resistance to deformation of a crystal may be increased by an overall increase in the friction stress. This situation may arise in interstitual or substitutional solid solutions. If a pile-up against a simple tilt wall is considered for which each of the dislocations in the pile-up must overcome a friction stress, σ_f, due to solute atoms in the matrix, equation 3.9 will have another term and becomes

$$P_1(x) = \sigma_f + \frac{\mu b \alpha}{h(1-\nu)} \; [\cosh (2\alpha) \cos (2\lambda)-1]/[\cosh(2\alpha)-\cos(2\lambda)]^2 \qquad 3.10$$

where the variables are as defined for equation 3.9. Fig. 3.4 shows the required stress to propagate a pile-up with a source at x = -10h, to various positions with respect to the tilt wall. Comparing Fig. 3.2 and Fig. 3.4 we see that the required applied stress to propagate a pile-up in the presence of a friction stress to any position is simply the sum of the stress required in the absence of a friction stress plus the value of the friction stress. Thus a pile-up of dislocations does not aid dislocations in overcoming a friction stress, or any stress that does not vary in magnitude with position along the pile-up because this stress acts equally on all dislocations.

3.3 Slip Propagation Through a Network of Misfit Dislocations in a Two Phase Material

The total resistance to the propagation of a slip band in a two phase material is determined by a number of factors including chemical composition and elastic constants of the second phase particles and the nature of the interphase interface. In regard to the role of the interface it is of value to examine the role of misfit dislocations in resisting the propagation of a slip band. In order to examine this problem two approaches have been taken. An experimental investigation has been undertaken of a simple binary nickel based system containing particles of Ni_3Ge and is described in Chapter 4. In addition the effect of an idealized network of misfit dislocations on the mobility of a dislocation pile-up was examined using the numerical method outlined previously.

The interface model used, assumes an infinite network of edge dislocations which form a square grid on the (001) plane. The Burgers vectors of these dislocations are a [100] and a [010]. To calculate the stress field of this idealized model of misfit dislocations, the stress fields of the two sets of parallel dislocations were calculated separately (Van der Merwe, 1950) and the total stress field was obtained by summing these two stress fields. The coordinate system used for this calculation is: x-axis parallel to [100], y = axis parallel to [010] and the z-axis parallel to [001]. The components of stress for this idealized model of misfit dislocations for $z \leq 0$ are:

$$P_{xx} = -Q\ \{Z[(1 + C^2\ e^{2Z})\cos X - 2Ce^Z] + 2\ R^2\ (\cos X - Ce^Z)\}$$

$$P_{yy} = -Q_1\{Z[(1 + C^2 e^{2Z})\ \cos Y - 2Ce^Z] + 2\ R^2\ (\cos Y - Ce^Z)\}$$

$$P_{zz} = QZ\{(1 + C^2 e^{2Z})\cos X - 2\ Ce^Z\}$$

$$+ Q_1 Z\{(1 + C^2 e^{2Z})\ \cos Y - 2\ Ce^Z\}$$

$$P_{xy} = 0$$

$P_{yz} = - Q_1 \sin Y \{T^2 + Z (1 - C^2 e^{2Z})\}$

$P_{xz} = - Q \sin X \{R^2 + Z (1 - C^2 e^{2Z})\}$

where $X = \frac{2\pi x}{h}$, $Y = \frac{2\pi y}{h}$, $Z = \frac{2\pi z}{h}$, $C = (1 + \beta^2)^{\frac{1}{2}} - \beta$,

$\beta = \pi/82\ (1-\nu)$, $R^2 = 1 + C^2 e^{2Z} - 2Ce^Z \cos X$,

$T^2 = 1 + C^2 e^{2Z} - 2Ce^Z \cos Y$, $Q = \mu Ce^Z/[82\ (1-\nu)\ R^2]$,

$Q_1 = \mu Ce^Z/[82\ (1-\nu)\ T^2]$, μ is the shear modulus and ν is Poisson's ration. For the above calculations the shear stress and Poisson's ratio were assumed to be equal for both phases.

A pile-up of edge dislocations having Burgers vectors a/2 $[\bar{1}01]$ which are on the slip plane $(1\bar{1}1)$, in the stress field of the tilt wall will be considered in the stress field of the network of misfit dislocations. For this pile-up the appropriate component of the stress field, $P_1(x,y,z)$ acting on these edge dislocations is:

$$P_1(x,y,z) = - 0.408\ P_{xx} + 0.408\ P_{zz} - .408\ P_{yz}$$

The interface is divided into squares by the misfit dislocations. The stress field of the misfit dislocations is periodic with respect to these squares. Thus the applied stresses required to propagate pile-ups and single dislocations toward this network, were calculated with respect to various positions in one square. This is illustrated schematically in Fig. 3.5. The positions chosen were those along line EF, and these positions will be denoted by the y coordinate of the point. For example the middle of the square is y = 0.5. The distance of the

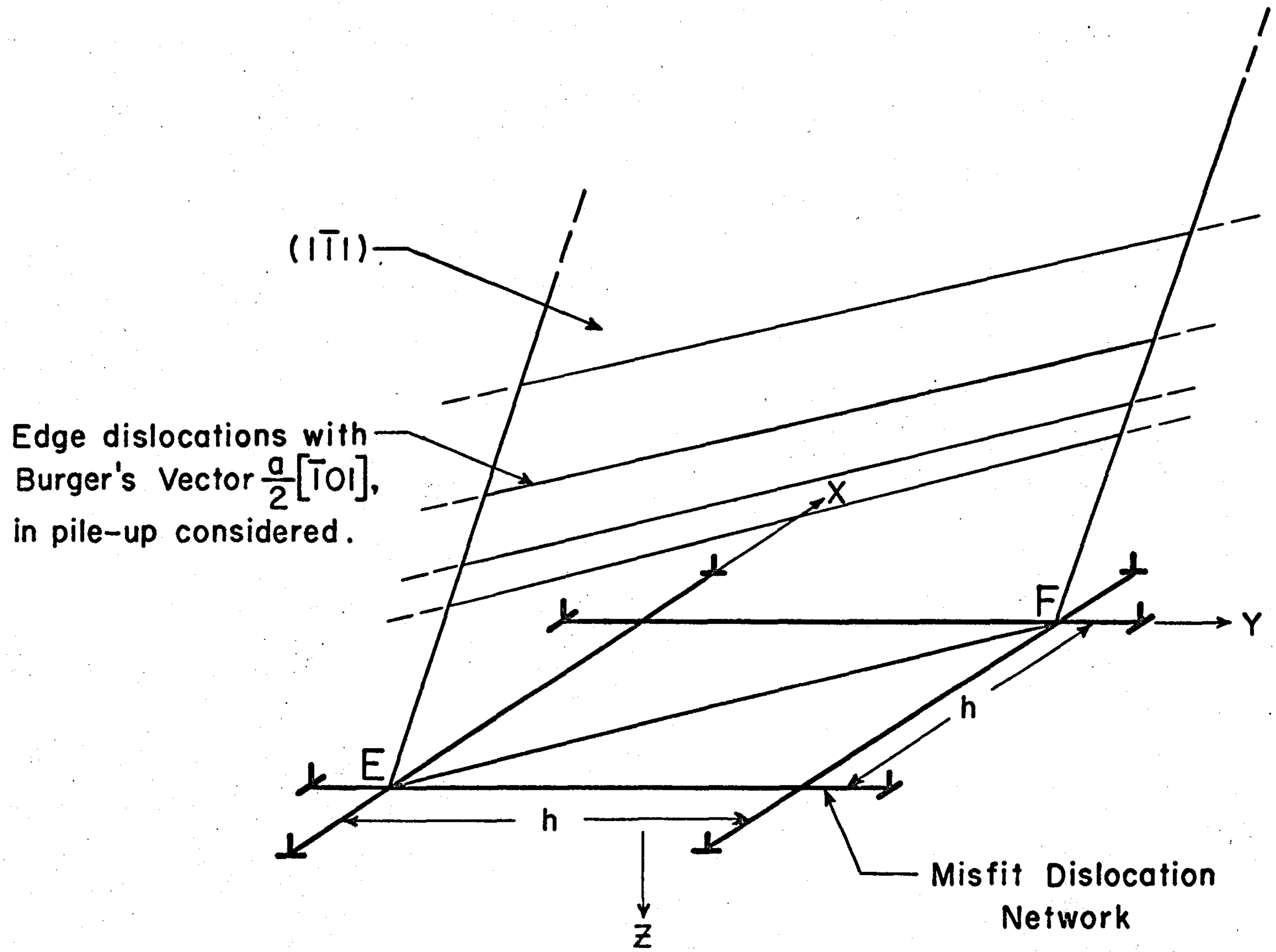


Figure 3–5. Schematic diagram of the dislocation pile-up considered, approaching a misfit dislocation network.

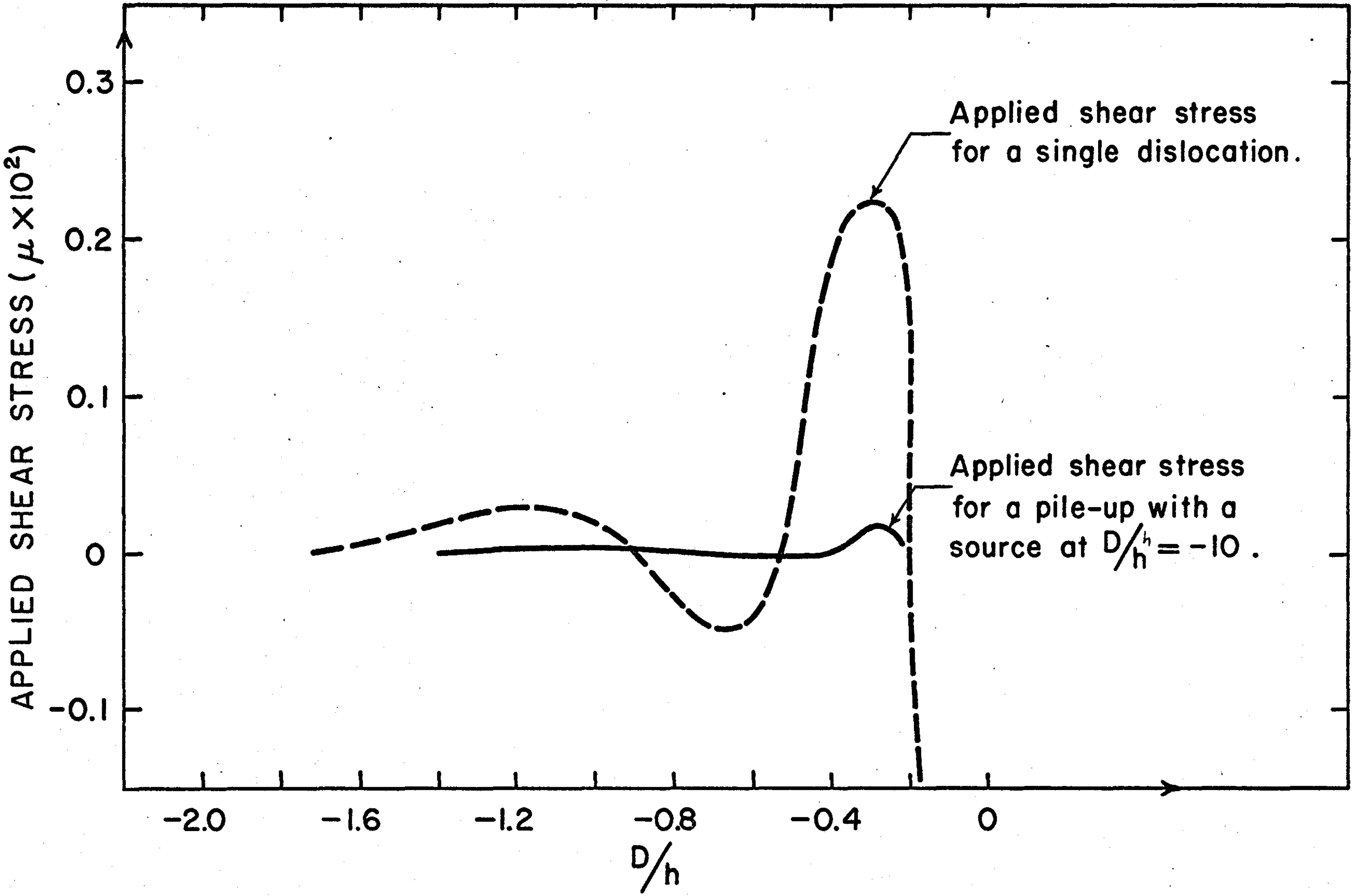


Figure 3-6. Applied shear stress reguired to propagate the leading dislocations in a pile-up, to a distance D from a misfit boundary.

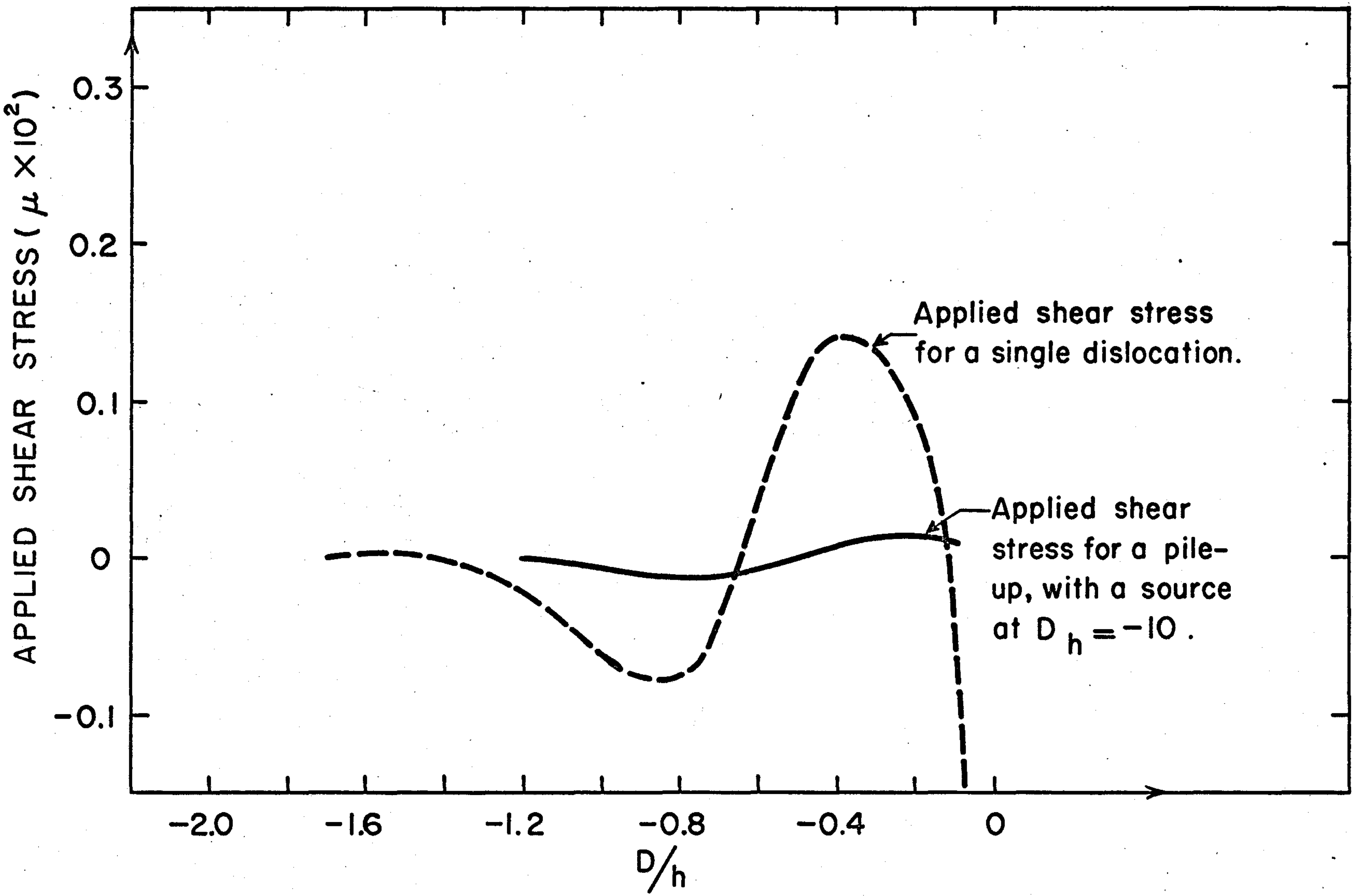


Figure 3-7. Applied shear stress required to propagate the leading dislocations of a pile-up to a distance D from a misfit boundary.

approaching dislocation from the misfit boundary will be denoted by D, and this distance will be measured in the $[10\bar{1}]$ direction from the point on line EF which is being considered. Figs. 3.6 and 3.7 are graphs for applied shear stress required to propagate pile-ups with a source at D/h = -10, and single dislocations versus D, for points y = 0.5 and y = 0.3, on line EF, respectively. It is seen from these figures that the passing stress for a pile-up for this internal stress field is lower than for a single dislocation.

CHAPTER 4

EXPERIMENTAL PROCEDURE AND RESULTS

A nickel-germanium binary alloy was selected because the Ni rich solid solution and the ordered intermetallic compound Ni_3Ge have the same lattice (f.c.c.) with a misfit of about 1%. Thus if the interphase interface is semi-coherent there should be a network of misfit dislocations to accommodate the misfit at the interface of the large Ni_3Ge particles. These misfit dislocations were observed and characterized by transmission electron microscopy as described in this chapter.

4.1 Experimental Procedure

Nickel has an f.c.c. lattice with a lattice parameter, a = 3.512Å (Pearson, 1958) and Ni_3Ge is an ordered alloy of the $L1_2$ type with a lattice parameter, a = 3.560Å (Pearson, 1958). The phase diagram of Ni-Ge (Hanson, 1958) is given in Fig. 4.1.

An alloy of Ni - 20 weight percent Ge was used since this alloy will contain about 50% Ni_3Ge. Samples of this alloy, weighing 35 grams were melted several times in the argon arc furnace and then cast in ingot form. These samples were then directionally solidified to produce large particles of Ni_3Ge. To directionally solidify the samples, they were melted in an inert atmosphere of argon by a Toccotron unit, and then the sample was lowered through the coil at a rate of 0.5" per hour. The apparatus for directionally solidifying the samples is shown

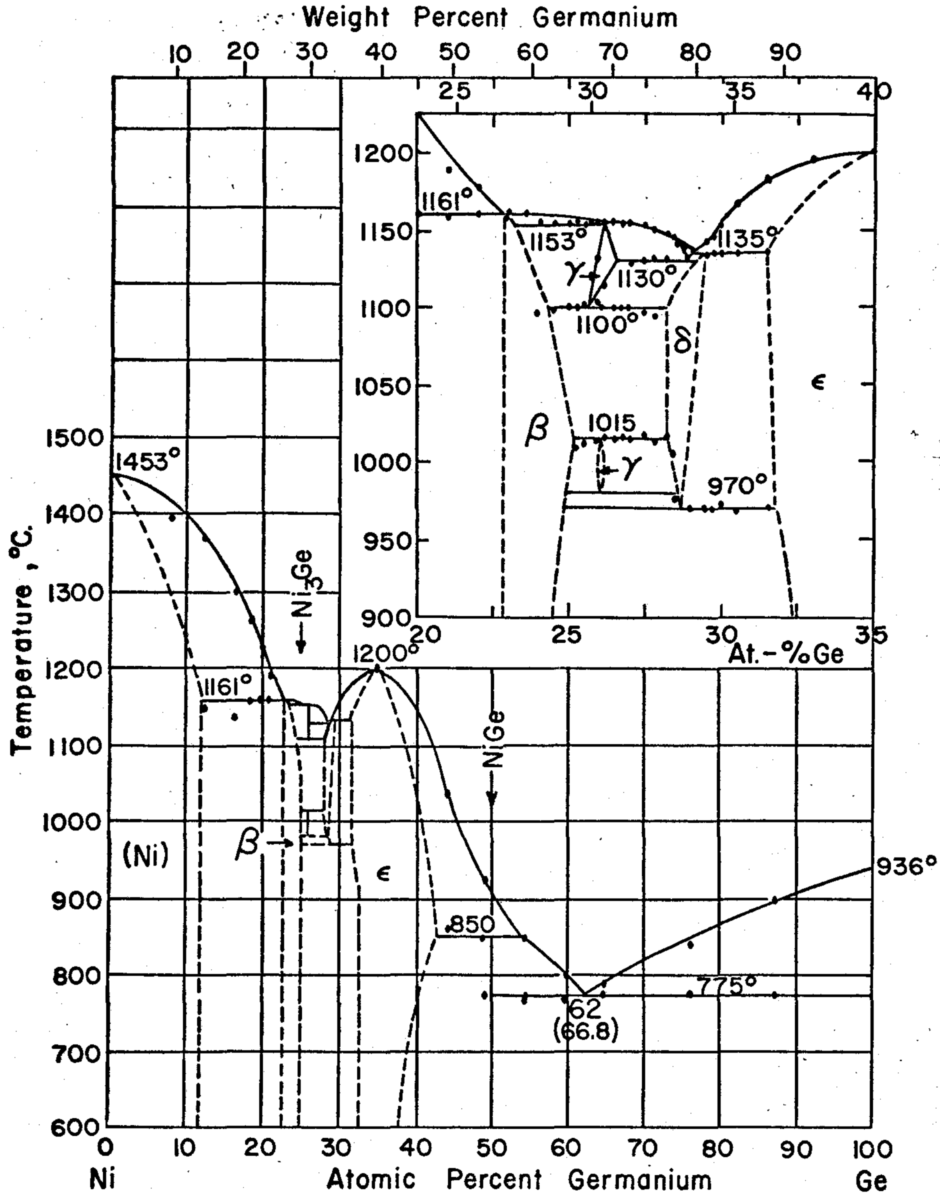


Figure 4-1. Phase diagram of Nickel-Germanium.

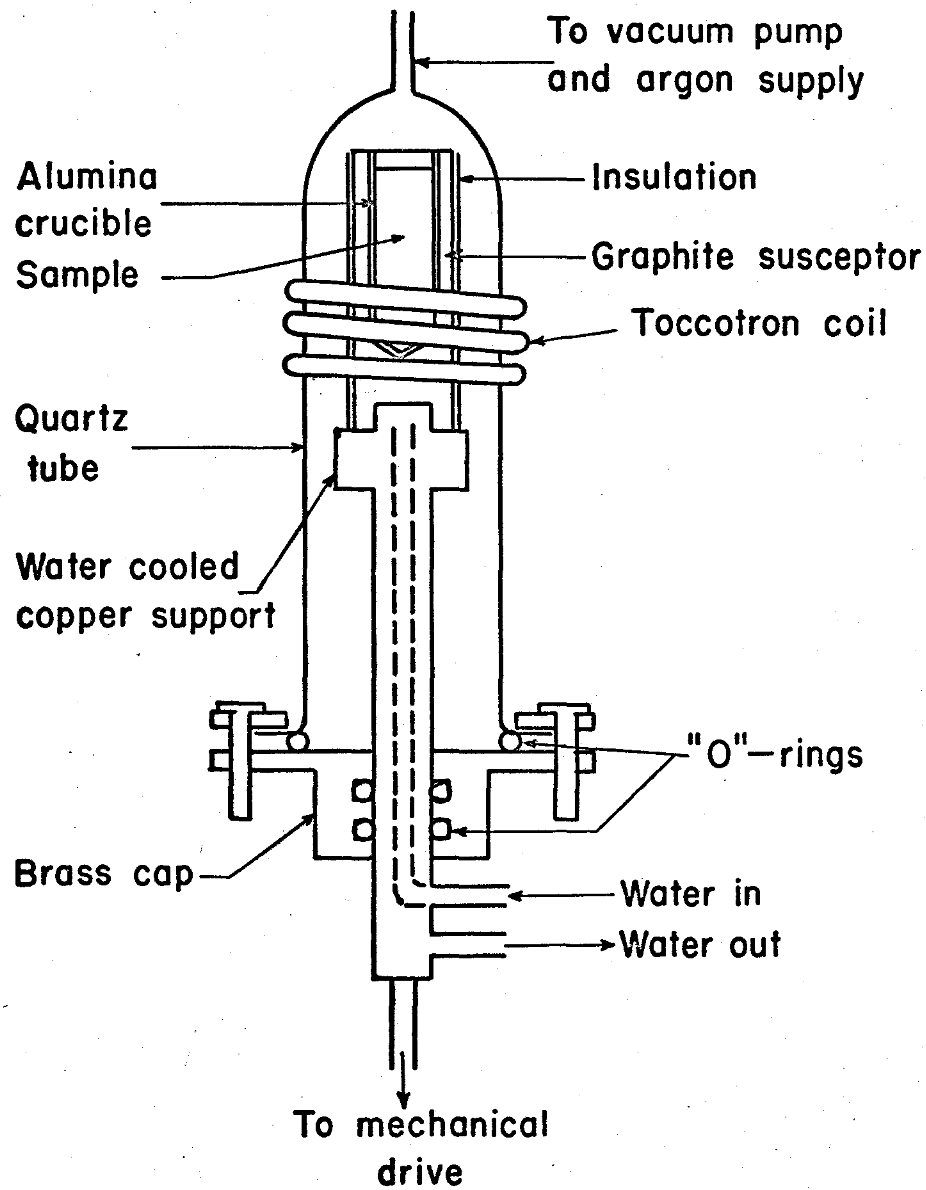


Figure 4-2. Directional solidification apparatus.

schematically in Fig. 4.2. The inner diameter of the crucible is 0.5" and the diameter of the graphite susceptor is 1.0".

Thin films of the directionally solidified alloy were prepared in order to examine the detailed nature of the interphase interface in the electron microscope. Transverse sections about 0.050" thick were cut from the directionally solidified sample with a cut-off wheel. These samples were then mechanically polished to a thickness of approximately 0.003". The edges of these samples were then masked with microstop, and then the samples were electropolished in a solution of ethanol-10% perchloric acid (volume) at a potential of 24 volts using the window technique. A stainless steel cathode was used. The best results were obtained with the polishing solution at a temperature between -20^{o}C and -30^{o}C. The thin films were examined in a Siemens Elmskop I electron microscope using a double tilt stage. The Burgers vectors of the misfit dislocations were determined by observing the dislocation arrays under various contrast conditions. The theory of electron diffraction contrast is described comprehensively by Hirsch, Howie, Nicholson, Pashly, Whelan (1965).

A compression sample was machined from the directionally solidified sample. This compression sample was 0.5" long, and had a diameter of 0.25". A flat, 0.1" wide was ground along the length of the sample to facilitate the observation of surface slip lines. To remove the damage due to machining, the compression sample was electropolished, using the solution and potential described in the previous paragraph, reducing the diameter by 0.002". The flat of the specimen was lightly etched with a solution of ethanol-10% bromine (volume). This sample was then tested in compression using the Instron TTC-L , removing the load periodically during the test to observe the slip lines. The slip lines were examined directly on the flat of the compression sample.

4.2 Experimental Results

Some areas of the directionally solidified alloy contain a high volume fraction of large Ni_3Ge particles which have a fibrous nature, while other grains contain a relatively low volume fraction of large Ni_3Ge particles. This inhomogenity is illustrated in the optical photographs, plates 4.7, 4.8 and 4.9 which also contain slip lines. Plate 4.1 which is a composite of a region with many large Ni_3Ge particles, shows the interconnecting particles and also their interconnecting network of misfit dislocations. Plate 4.2 is also of a similar region at a lower magnification. Plate 4.2 shows the fibrous nature of this region.

To determine the Burgers vectors of dislocations the criterion that a dislocation will be out of contrast when $\bar{g}.\ \bar{b} = 0$ was used (Hirsch, Howie, Nicholson, Pashley, Whelan, 1965). In this equation $\bar{g}$ is the reciprocal lattice vector of the operating reflection, and $\bar{b}$ is the Burgers vector of the dislocation. In region A, of plate 4.3, there is a square grid of misfit dislocations. All the misfit dislocations are in contrast because there are many operating reflections which are [200], [020] and [220]. In the same region A, in plate 4.4, only one set of dislocations are in contrast and the others set are out of contrast. The operating reflection for this plate is [200]. In plate

Plate 4.1

Composite of a region of a high volume fraction of incoherent Ni_3Ge particles.

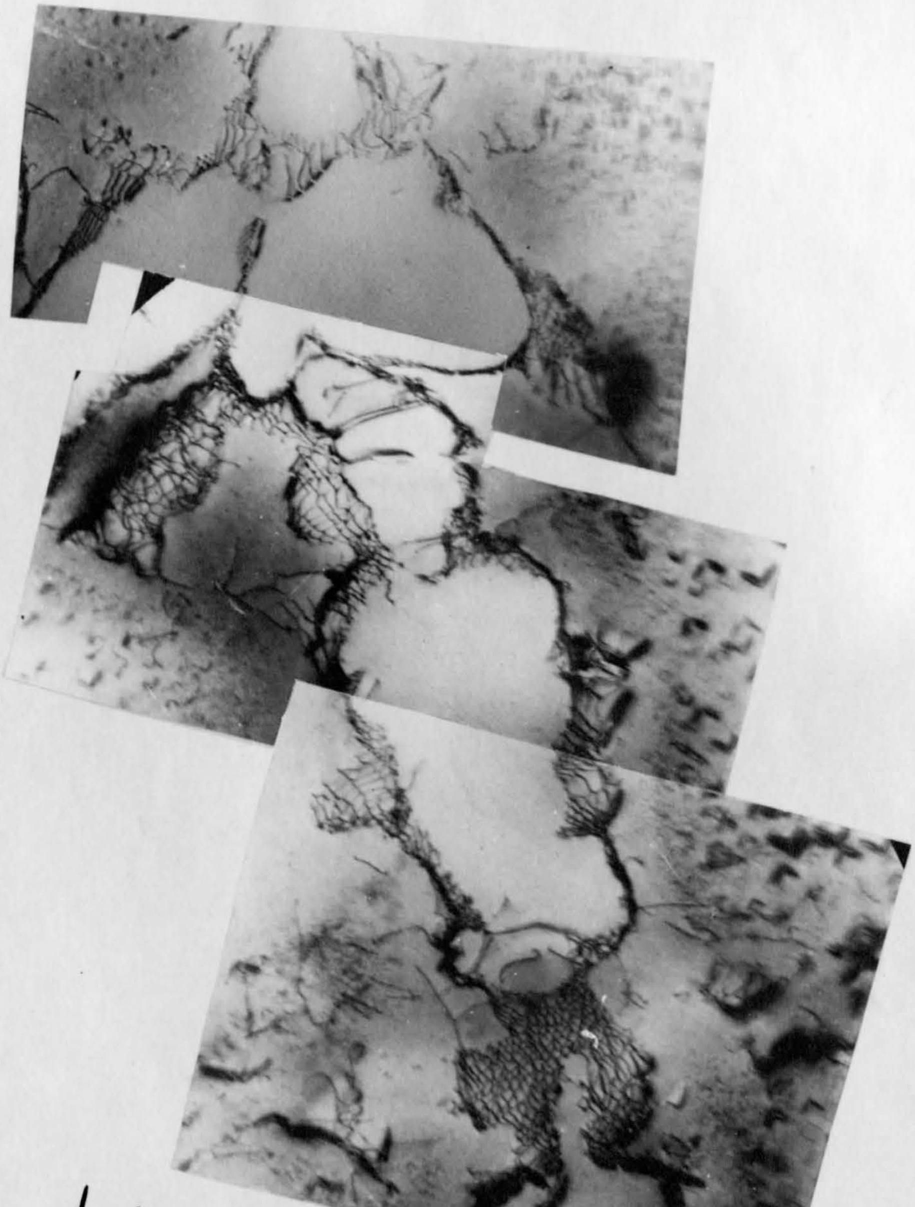

1μ

Plate 4.2

Fibrous nature of incoherent Ni_3Ge particles in a region of high volume fraction of Ni_3Ge particles.

1 μ

Plate 4.3

Region A shows a square grid of misfit dislocations.
There are many operating reflections.

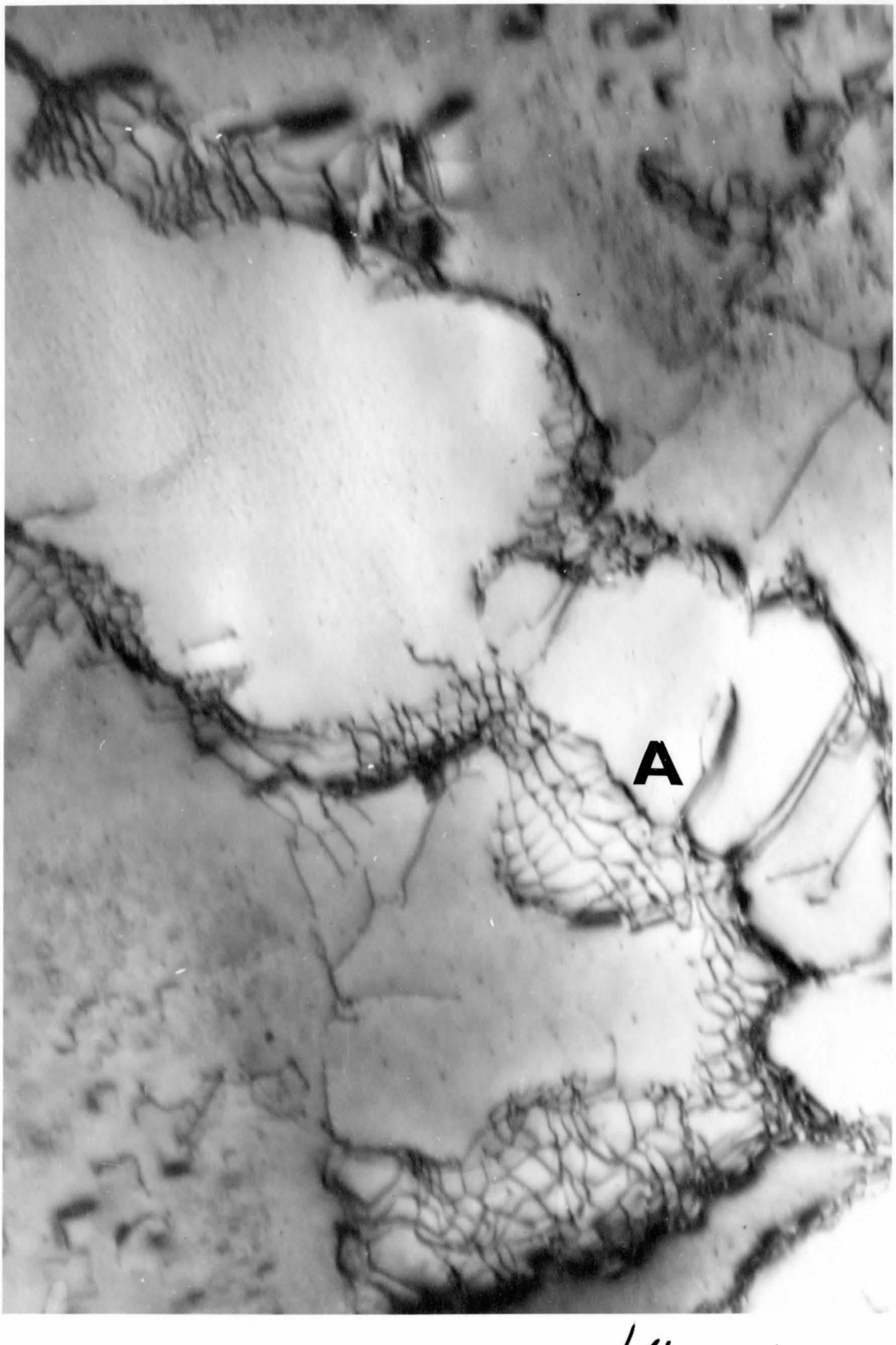

A
1μ

Plate 4.4

Region A shows one set of misfit dislocations out of contrast. Operating reflection is [200].

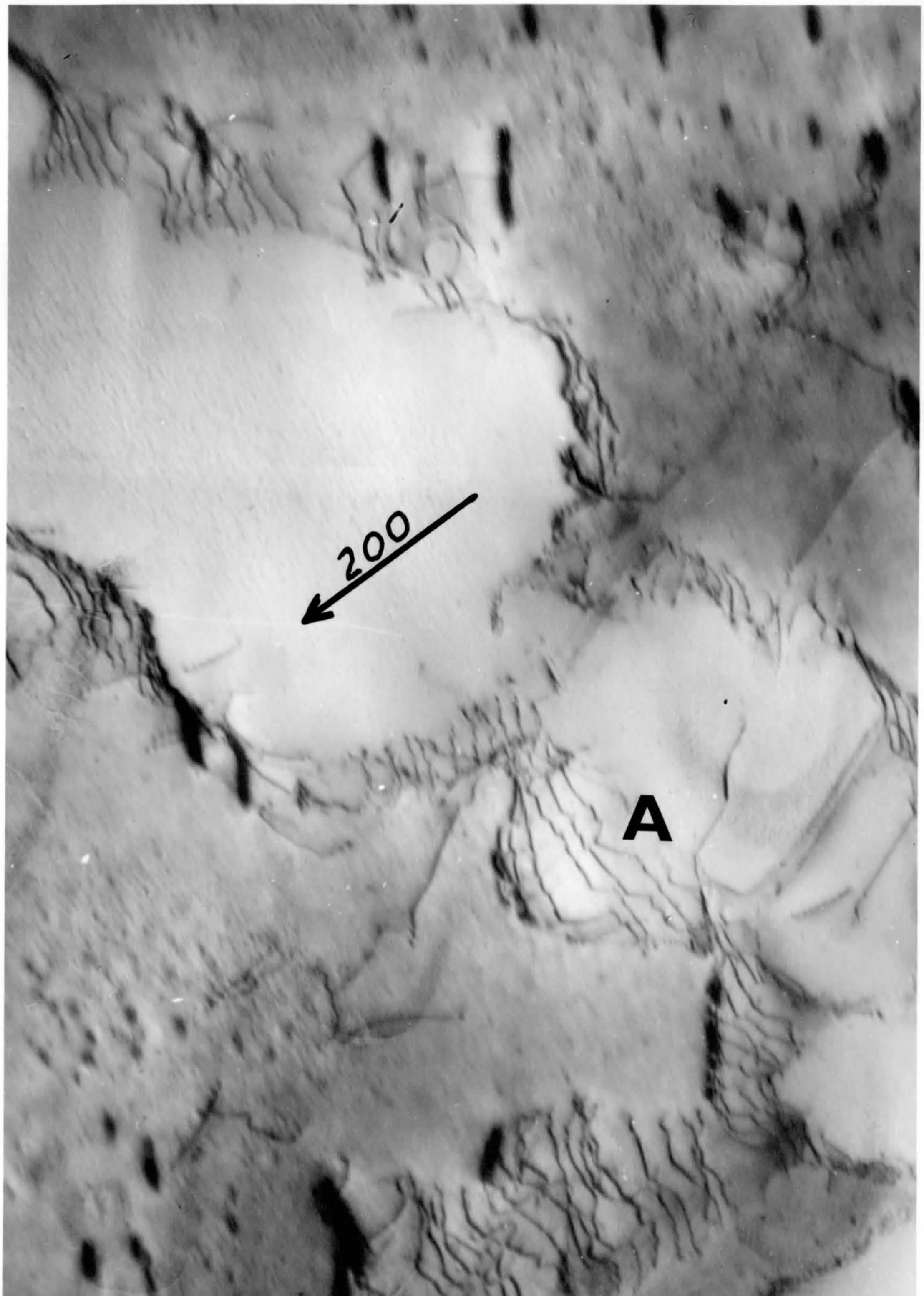
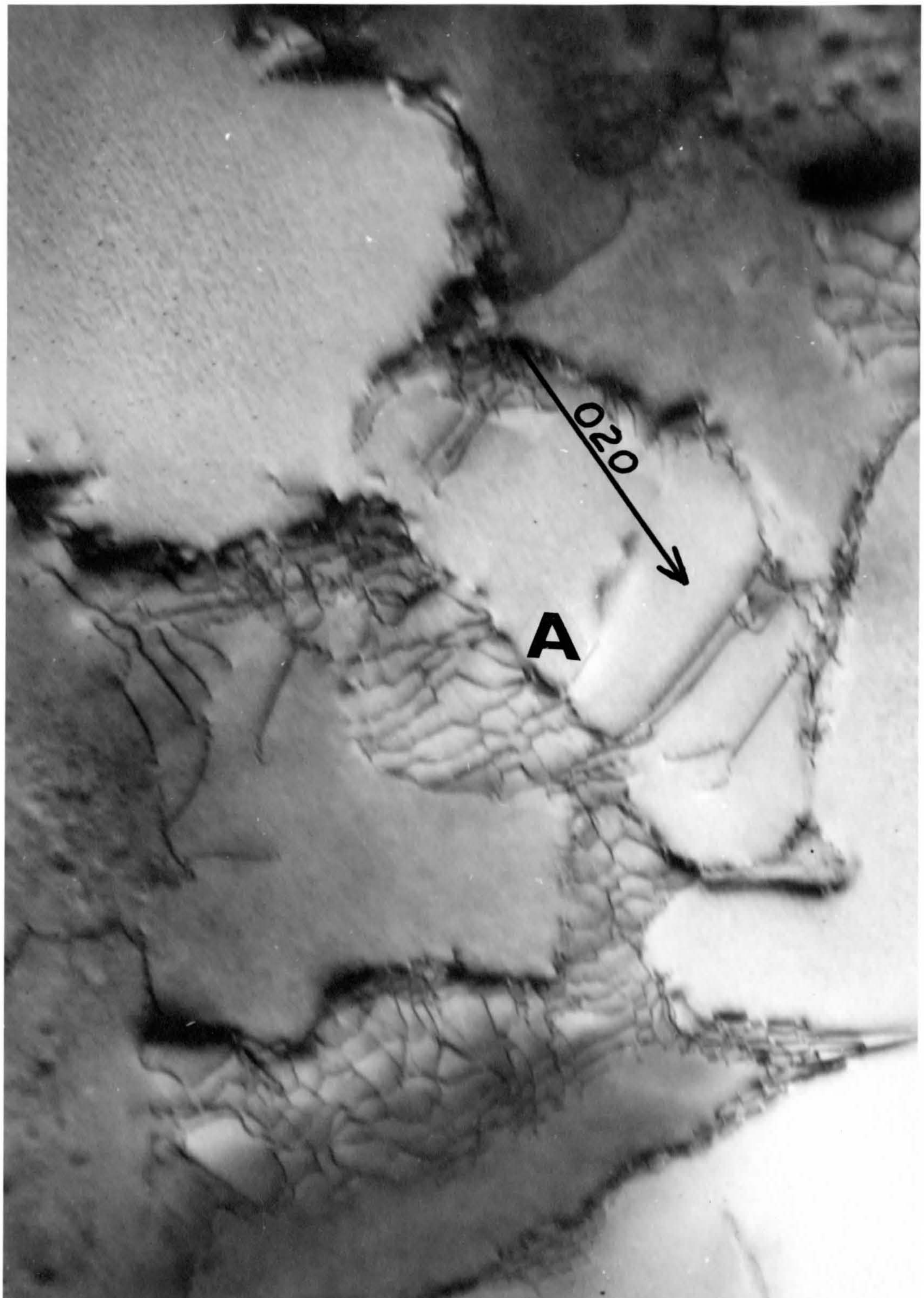

200
A
1 μ

Plate 4.5

Region A shows the other set of misfit dislocations out of contrast. Operating reflection is [020].


020
A
1μ

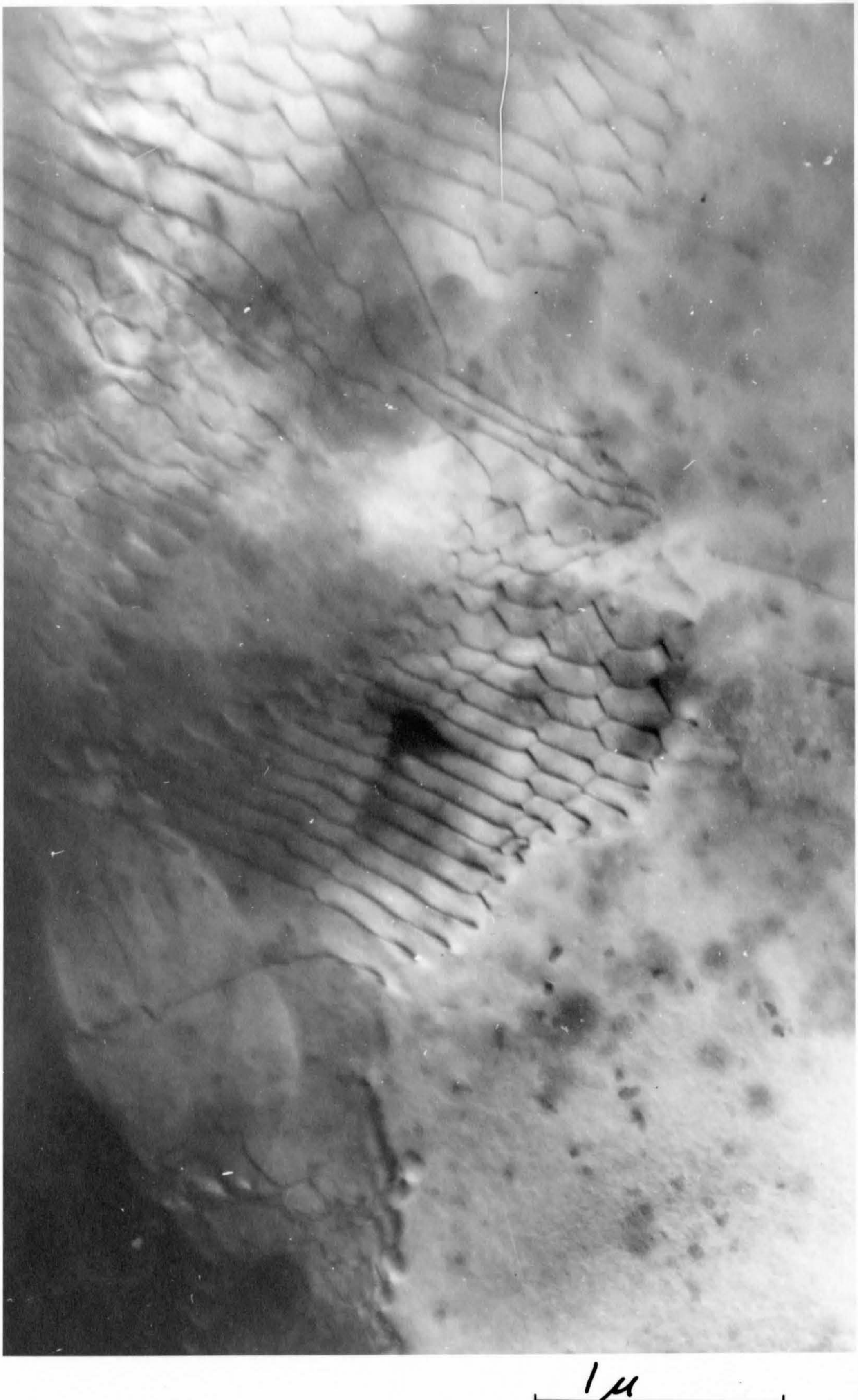
4.5, with an operating reflection of [020], the second set of dislocations is in contrast and the first set extinguished. There are two types of possible Burgers vectors: $\pm$ a/2 [011], $\pm$ a/2 [01$\bar{1}$] and $\pm$ a/2 [101], $\pm \frac{a}{2}$[10$\bar{1}$] or $\pm$ a [100] and $\pm$ a [010]. The normal to the foil surface is close to the [001] direction. If the set of dislocations have Burgers vectors of the type a/2 [110], then the interface plane would be of the (111) type and thus would make an angle of about 54^{o} with the surface of the foil. This region was near the edge of the foil and thus a maximum possible thickness is 2000Å. If the interface foil was a plane of the (111) type, the thickness of the foil would have to be about 2.0 x tan (54) x 10^{8}/40,000 = 7000Å. This is an impossible foil thickness. Thus the interfacial plane must be (001). Thus the Burgers vectors of the two sets of misfit dislocations appear to be $\pm$ a [100] and $\pm$ a [010]. This is discussed in detail in a later chapter.

Other regions of the foil have a hexagonal network as is shown in Plate 6. The Burgers vectors for this plate have not been calculated. There are different misfit dislocation arrangements, because the Ni_3Ge particles are irregular, and thus the misfit must be accommodated on different types of interfacial planes producing different arrangements of misfit dislocations.

Misfit dislocations have a smaller stress field than an edge dislocation of the same Burgers vector produced by deformation (Van der Merwe, 1950). As further evidence, it was found that to bring misfit dislocations into contrast, the misfit dislocations had to be much closer to the Bragg contours than for ordinary glide dislocations.

Plate 4.6

Illustrates the hexagonal misfit dislocation network sometimes observed at the interface of the Ni_3Ge particles.


1μ

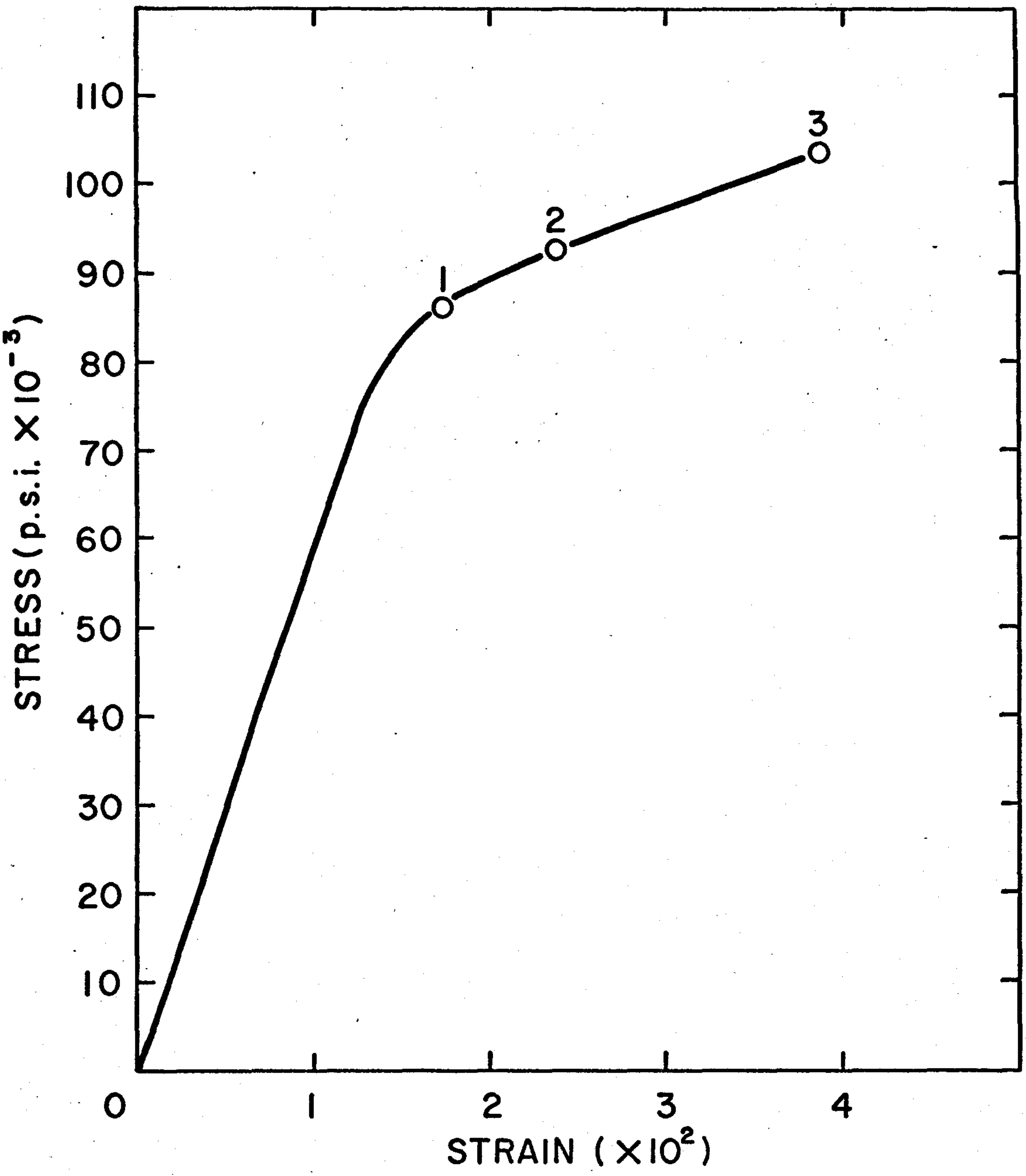


Figure 4-3. A compression stress-strain curve for a directionally solidified Ni-70 weight percent Ge alloy.

A stress-strain curve for this directionally solidified Ni-20 weight percent Ge alloy tested in compression is given in Fig. 4.2. This alloy had a high yield stress of about 79,000 p.s.i.. Also the work hardening rate was about G/150, where G is the shear modulus for pure nickel. This value is high compared to the usual values of between G/200 to G/300, where G in this case is the shear modulus of the metal concerned.

The load was removed at points 1, 2, and 3 shown in Fig. 4.2 and the slip lines were studied. Plate 4.7 is taken at point 1. In this plate the large particles are Ni_3Ge and there is a relatively low volume fraction of Ni_3Ge particles as compared to the volume fraction of Ni_3Ge particles in Plate 9. Slip lines can be seen in Plate 7, but extremely few slip lines cut the Ni_3Ge particles. Plates 8 and 9 are taken after the specimen had been strained to point 2. The slip lines are more numerous, but in plate 8, very few of the Ni_3Ge particles are cut. In plate 9, the particles have a fibrous nature and thus it is very difficult to tell if the Ni_3Ge particles are cut because of the close spacing of the particles. It is difficult to tell whether the slip lines are continuous or consist of numerous small segments. At point 3 the slip line configuration was similar to that at point 2 except the slip lines are more numerous.

Plate 4.7

Slip lines when the compression sample has been strained to point 1. This region contains a low volume fraction of Ni_3Ge particles.

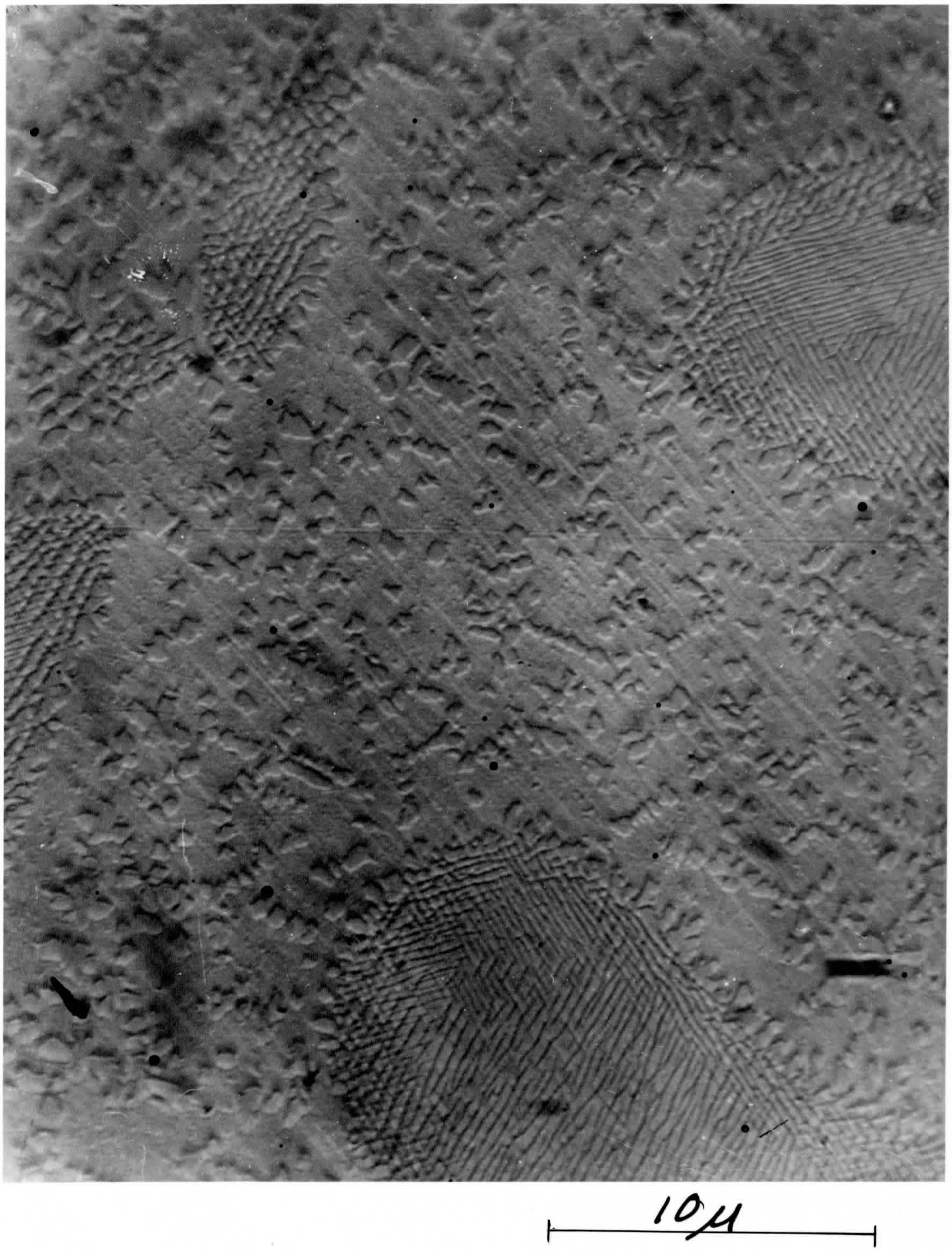
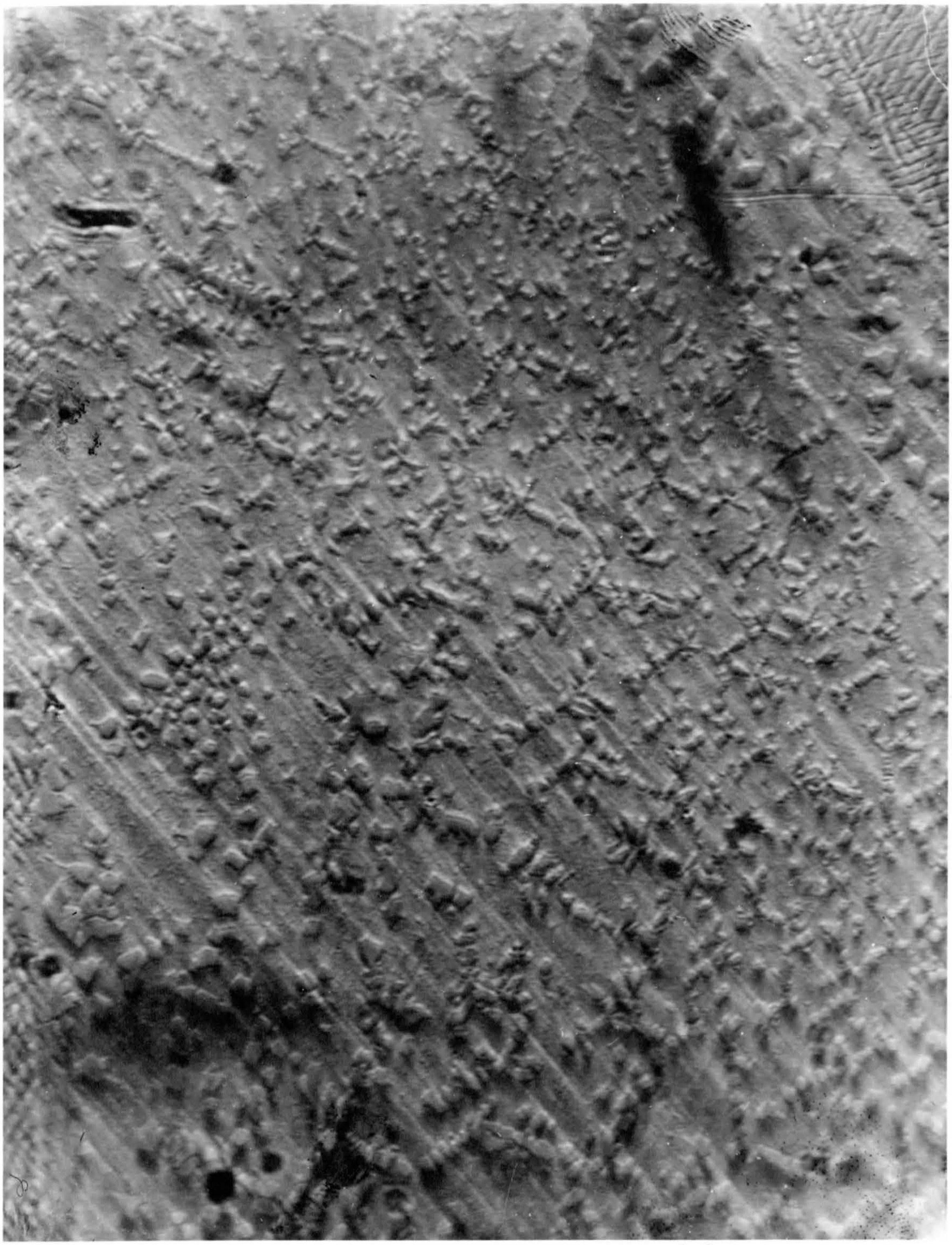

10μ

Plate 4.8

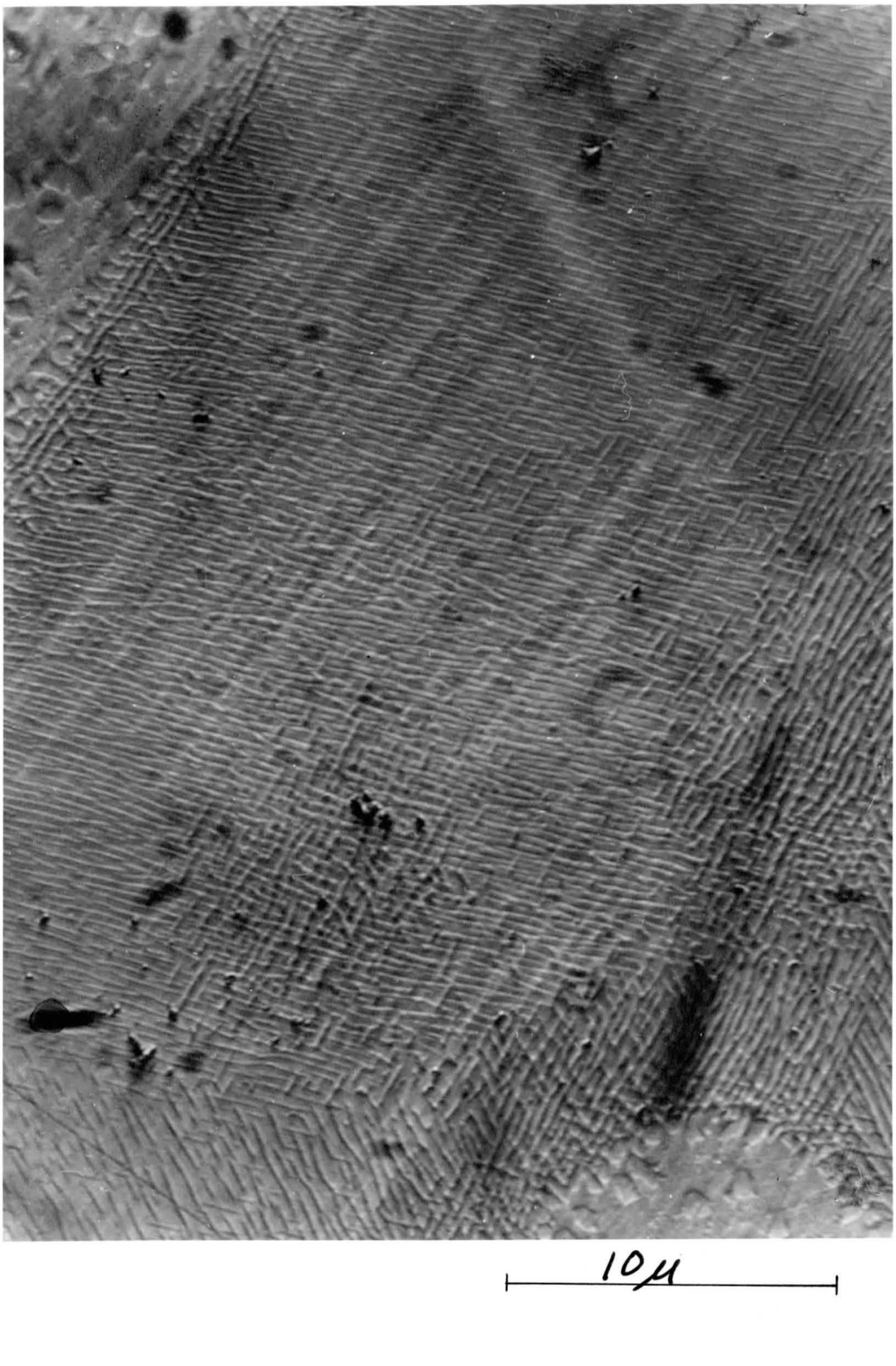
Slip lines when the compression sample has been strained to point 2. This region contains a low volume fraction of Ni_3Ge particles.



10 μ

Plate 4.9

Slip lines when the compression sample has been strained to point 2. This region contains a high volume fraction of Ni_3Ge particles.


10μ

CHAPTER 5

DISCUSSION OF RESULTS

For clarity the discussion is divided into separate sections dealing with the experimental observations and numerical computations.

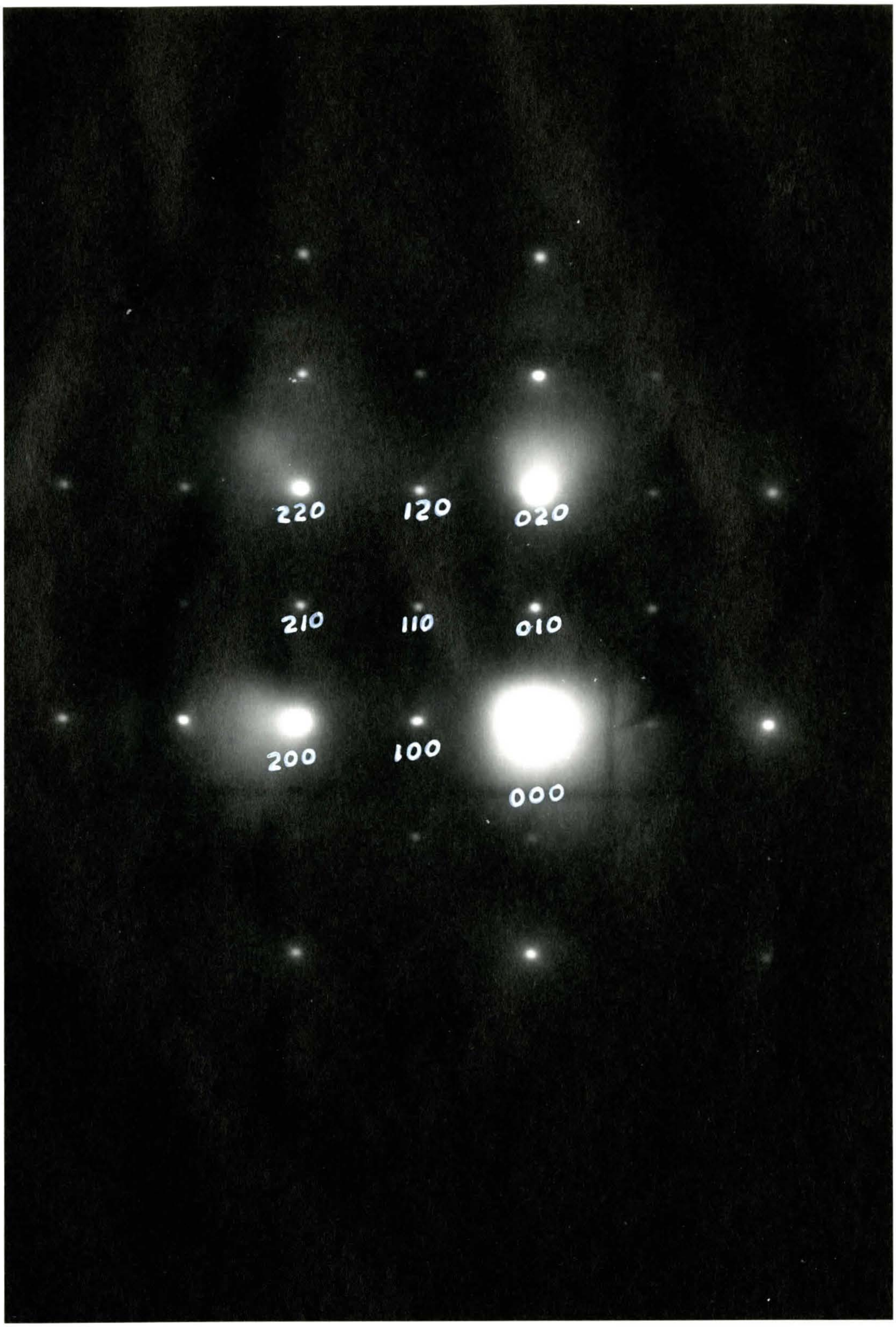
5.1 Burgers Vectors of Misfit Dislocations

The misfit dislocations at the interface of the large Ni_3Ge particles should accommodate the lattice misfit between the Ni_3Ge lattice and the matrix. If the lattices of Ni_3Ge and the Ni matrix are aligned such that the (hkl) planes of the particle are parallel to those of the matrix then the accommodating strain will be pure dilation, and this type of strain can be accommodated completely by edge dislocations. The diffraction pattern in plate 5.1 is of the region seen in plates 4.3, 4.4, and 4.5. It can be seen that the ordered spots (mixed indices) are oriented such that the lattices of the two structures are simply aligned and $(hkl)_{matrix}$ is parallel to $(hkl)_{particle}$. Thus the misfit between the matrix and the Ni_3Ge particles can be accommodated by a set of edge dislocations, which accommodates the misfit in two perpendicular directions on the interface with the Burgers vectors of the misfit dislocations in the plane of the interface.

The square grid of misfit dislocations in section A of plates 4.3, 4.4, and 4.5, are edge dislocations since the operating reflection as can be seen in plates 4.4 and 4.5 are parallel to the dislocations that are out of contrast, and since $\bar{g}.\bar{b} = 0$ for these dislocations, $\bar{b}$, the Burgers vector, is perpendicular to the line length. The only set

Plate 5.1

Diffraction pattern showing the alignment of $(hkl)_{particle}$ with $(hkl)_{matrix}$. The ordered spots (mixed indices) are due to the ordered Ni_3Ge particles.


220
120
020
210
110
010
200
100
000

of simple Burgers vectors that are consistent with plates 4.3, 4.4 and 4.5 are $\pm$ a[100] and $\pm$ a[010] which lie in the (001) plane. The normal to the foil in section A, is close to the [001] direction and the width of the interface seen in plates 4.3, 4.4 and 4.5 is consistent with the interface being the (001) plane.

This square grid of edge dislocations can accommodate the misfit but the dislocation spacing should be 293Å, using the lattice parameters of pure Ni and pure Ni_3Ge. The measured spacing is about 500Å. The matrix is a solid solution of Ni-Ge and the particles may differ from stoichiometry, and thus the lattice parameters of the matrix and the particles may differ from those pure Ni and Ni_3Ge. This could account for the difference in the measured and calculated misfit dislocation spacing. A difference of 0.02Å, would cause an error of 100% in the dislocation spacing. Thus the spacing of dislocations on the interphase interface is extremely sensitive to the exact lattice parameter of the solid solution. To date no data on the lattice parameter of nickel-germanium solid solution have been reported and thus no direct comparison can be made with the experimental results. However the observations described above indicate that the interphase interface in as grown Ni-Ni_3Ge consists of a simple Van der Merwe net of a[100] dislocations. This is at variance with the interface structures reported in Ni-Ni_3(Al, Ti) by Weatherly and Nicholson (1968) for particles in which coherency was lost at long aging times. These differences may reflect the mechanism of formation of the interface array in the two cases because interfaces occurring at long aging times may arise from the presence of glide dislocations in the matrix.

5.2 Cutting of Incoherent Ni_3Ge Particles

If a large Ni_3Ge particle is cut, then either the glide dislocation must cut the misfit dislocations at the interface or the glide dislocation can be bowed out between the misfit dislocations in the same manner as a Frank-Read source operates. These two possibilities are now considered.

The required applied stress to propagate a single dislocation up to a misfit dislocation is about 0.25μ or 2,800,000 p.s.i. Since this stress acts only on the leading dislocation, the exact method of Eshelby, Frank and Nabarro (1951) was used to estimate the required applied stress to propagate the leading dislocation of a pile-up of length 10h to the misfit dislocation. This stress is 130,000 p.s.i.

If the glide dislocation is blocked by the misfit dislocations, the glide dislocation may be able to extend between the misfit dislocations as occurs with a Frank-Read source. The applied stress required to extend a single glide dislocation between the misfit dislocations, not considering the antiphase boundary formed is

$$\sigma = \frac{2\mu b}{h} .$$

Thus using the shear modulus of Ni and a value of 500Å for h, the misfit dislocation spacing, then

$$\sigma = \frac{2 \times 11.5 \times 2.46 \times 10^6}{500} = 110,000 \text{ p.s.i.}$$

This stress could be reduced somewhat if the glide dislocations were dissociated into partial dislocations of smaller Burgers vector.

Because the Ni_3Ge particles are ordered, a single dislocation passing through the particle creates an antiphase boundary. The dislocation must do work to form this antiphase boundary. The work done per unit length by a dislocation in moving a distance L is σbL. If this dislocation is in an ordered particle, it will create an antiphase boundary with an area L per unit length of the dislocation. Thus if the antiphase boundary energy density is ρ, then $\sigma bL = \rho L$ and $\sigma = \rho/b$. A typical value of antiphase boundary energy density for an $L1_2$ ordered alloy is about 150 ergs/cm^2. Thus the required applied stress to propagate the dislocation is about

$$\sigma = \frac{150}{2.46 \times 10^{-8}} = 6.1 \times 10^{9} \text{ dynes/cm}^2 = 88{,}000 \text{ p.s.i.}$$

The stress field of a square grid of misfit dislocations doesn't offer much resistance to dislocation motion except at the misfit dislocations. From Figs. 3.6 and 3.7 it is seen that the required applied stress to propagate a single dislocation and pile-up to the interphase (away from a misfit dislocation) is about 0.002μ and 0.0002μ respectively. Using the shear modulus for Ni, this gives values of 23,000 p.s.i. and 2,300 p.s.i. for single dislocations and pile-ups respectively.

The applied stress required to bow a glide dislocation between the misfit dislocations is an order of magnitude lower than that required to cut the misfit dislocations. Thus if the Ni_3Ge particles are sheared it appears probable that this will involve the bowing of glide dislocations between the misfit dislocations at the interface.

In regions of low volume fractions of Ni_3Ge particles, few Ni_3Ge particles seem to be cut as seen in plates 4.7 and 4.8. Although the slip lines stop at the particles the process of deformation can not be ascertained from these observations. The discontinuities in the slip lines may arise because the slip line heights in the particles are beyond the resolution of the optical microscope. In order to clearly delineate the role of the Ni_3Ge particles further X-ray and electron microscopy observations are needed.

5.3 Single Dislocations and Pile-Ups

The numerical method used to calculate applied stresses required to propagate pile-ups is a very general method and can be used for any internal stress field that does not have singularities. This method is only accurate for slowly varying stress fields, that is the internal stress does not vary significantly between the first and second dislocations in the pile-up. It is realized that this method is not strictly accurate for the internal stress fields considered but the method illustrates some significant differences between the mobility of a single dislocation and a pile-up, and some important properties of a pile-up.

Using a single dislocation to calculate a passing stress over-estimates the strength of barriers. Also when considering a pile-up, the maximum required applied stress does not occur when the head of the pile-up is at the maximum internal stress, but after the head of the pile-up has passed the maximum internal stress, if this maximum internal stress is not a singularity in the internal stress field. Also as the length of the pile-up is increased the passing stress decreases, until a point is reached when a further increase in pile-up length doesn't decrease the passing stress. When this occurs, the dislocations at the end of a pile-up have very little effect on the head of the pile-up.

Basinski (1959) had already suggested that the flow stress calculated from the resistance to motion of a single dislocation in an internally stress solid gives an upper limit for the flow stress. He suggested that plastic flow proceeds by dislocation source operation in favourably stressed regions, followed by slip band propagation through an internally stressed solid.

CHAPTER 6

CONCLUSIONS

The numerical method developed to calculate the applied stress required to propagate a pile-up in an internal stress field is very general, and can easily be applied to any internal stress field which has no singularities. The required applied stresses calculated using this numerical method are accurate for slowly varying internal stress fields.

The method has been used to calculate the stress necessary to propagate a dislocation pile-up through a sinusoidal internal stress field, a simple tilt boundary, and a Van der Merwe net of interface dislocations. These examples were chosen to show the relevance of the method to a variety of problems in crystal plasticity including work hardened, recovered, and two phase materials. In all cases the flow stress calculated for the propagation of a pile-up of dislocations is lower than the flow stress required to propagate a single dislocation for the same internal stress field. The maximum applied stress required (passing stress) to propagate a pile-up through an internal stress field does not occur when the head of the pile-up is at the maximum internal stress, but when the head of the pile-up has passed the position of the maximum internal stress. The passing stress of a pile-up decreases with an increase in pile-up length until a pile-up length is reached for which the dislocations at the end of the pile-up have little affect on the head of the pile-up, and at this point increases in pile-up length do not decrease the passing stress significantly.

The misfit dislocations at the interface of Ni_3Ge in a matrix of Ni-Ge solid solution, have a variety of different types of networks to accommodate the misfit at the interphase interface depending on the orientation of the interface. A common type of network observed, for which the dislocations were characterized, is a square grid of edge dislocations with Burgers vectors of the a[100] type on a cube plane. This observed arrangement is in good agreement with the theoretical network necessary to form a Van der Merwe net. The misfit dislocations offer little resistance to glide dislocation motion, except in the localized region of the misfit dislocations. If the incoherent Ni_3Ge particles are sheared by glide dislocations of the matrix the most probable mechanism is by the bowing of glide dislocations between the misfit dislocations. To date deformation by this mechanism has not been proven experimentally.

Experimental work should be carried out on single crystals with incoherent second phase particles, or single crystals with some type of barrier with a known stress field, to test the numerical calculations of flow stress for pile-ups. In these single crystals the appropriate component of applied stress on the various slip systems would be known, and thus the experimental results could be compared directly with the calculated results.

More electron microscopy and X-ray work should be performed on the deformed alloy of Ni-Ge to determine the role of the incoherent Ni_3Ge particles in the deformation process. Such investigations may provide direct experimental evidence concerning the interaction of glide dislocations and interphase interfaces.

BIBLIOGRAPHY

Ashby, M. F., 1968, United States Navy Contract No. N00014-67-A-0298-0010.

Basinski, Z. S., 1959, Phil. Mag., 4, 393.

Brooks, H., 1952, Metal Interfaces (A.S.M.), p.20.

Cottrell, A. H., 1949, Progress in Metal Physics (London: Butterworths Scientific Publications), p.105; 1952, Dislocations and Plastic Flow in Crystals, (Oxford).

Eshelby, J. D., Frank, F. C., Nabarro, F. R. N., 1951, Phil. Mag., 42, 351.

Fleischer, R. L., 1960, Acta. Met., 8, 598.

Frank, F. C., and Van der Merwe, J. H., 1949, Proc. Roy. Soc. A, 198, 216.

Hanson, M., 1958, Constitution of Binary Alloys, (McGraw-Hill).

Head, A. K., and Louat, N., 1951, Aust. J. Physics, 8, 1.

Hirsch, P. B., Howie, A., Nicholson, R. B., Pashley, D. W., Whelan, M. J., 1965, Electron Microscopy of Thin Crystals, (Butterworth).

Kelly, A., and Nicholson, R. B., 1963, Progress in Materials Science, 10, 149.

Kelly, L. G., 1967, Handbook of Numerical Methods and Applications, (Addison-Wesley).

Laird, C., and Aaronson, H. I., 1967, Acta Met., 15, 73.

Li, J. M., 1961, Acta. Met., 9, 384.

Matthews, J. W., 1961, Phil. Mag., 6, 1347.

Merrick, H. F., and Nicholson, R. B., 1962, Fifth International Congress for Electron Microscopy, Philadelphia, edited by J. S. Breeze Jr., Academic Press, 1, paper K8.

Mott, N. F. and Nabarro, F. R. N., 1948, Rep. Conf. Strength of Solids, p.1, Physical Society.

Pearson, W. B., 1958, A Handbook of Lattice Spacings and Structure of Metals and Alloys, London: Pergamon.

Phillips, V. A., 1966, Acta. Met., 14, 271.

Smith, E., 1967, Phil. Mag., 16, 1285; 1968, Ibid, 18, 1067.

Van der Merwe, J. H., 1950, Proc. Phys. Soc. (London), A63, 616.

Weatherly, G. C., and Nicholson, R. B., 1967, Phil. Mag., 17, 801.