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Generation and Distribution of Dislocations by Solute Diffusion

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The stress introduced by solute lattice contraction of boron and phosphorus in silicon is shown to be sufficient to generate dislocations in silicon wafers. Such dislocations were observed. Expressions for the density distribution and for the total number of dislocations generated are derived and a mechanism of dislocation generation and distribution during the diffusion process is postulated. The dislocation distribution is shown to be highly dependent upon the nature of the diffusion process, expressions being derived for the conditions of (1) constant surface concentration of solute and (2) diffusion from a finite source. An expression for and means to determine the residual stresses in diffused silicon wafers are given.

I. INTRODUCTION

STUDIES of diffusion in semiconductor materials over the past decade have been stimulated by the development of solid-state devices. The influence of dislocations on the properties of such devices has recently been reviewed in the literature.¹ The possible introduction of dislocations into crystals by solute concentration gradients set up during solidification has been described.^{2,3} The introduction by diffusion processes of stresses sufficient to generate dislocations in silicon could have been calculated then, but observation of this phenomenon required the development of dislocation-free silicon⁴ as well as of new techniques.

In its simplest form, the diffusion process utilized in silicon device fabrication consists in subjecting a wafer of single-crystal silicon, 0.5 to 1.0 mm thick, to a gaseous, liquid, or solid source of diffusant at elevated temperatures. Under the proper conditions, we can maintain a constant solute concentration at the surface. In such a situation, the solute concentration profile can be described by a complementary error-function distribution, i.e.,

$$C = C_s \operatorname{erfc}[(a-y)^2/4Dt]^{\frac{1}{2}}, \quad (1)$$

where C is the solute concentration at y and t , C_s is the surface concentration, a and y are shown in Fig. 1, D is the diffusion coefficient, and t is time after initiation of diffusion.

We could also use a two-step diffusion process as follows. In the short first step, the prediffusion, the desired amount of solute is absorbed at the wafer

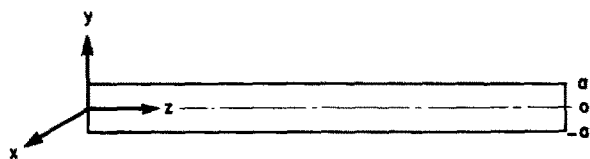


FIG. 1. Schematic diagram of the cross section of a wafer whose surfaces are $y=a$ and $y=-a$.

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¹ W. Bardsley, *Progress in Semiconductors*, edited by A. F. Gibson (John Wiley & Sons, Inc., New York, 1960), Vol. 4, p. 155.

² F. C. Frank, *Advances in Phys.* 1, 91 (1952).

³ A. J. Goss, K. E. Benson, and W. G. Pfann, *Acta Met.* 4, 332 (1956).

⁴ W. C. Dash, *J. Appl. Phys.* 30, 459 (1959).

surface. In the second step, the redistribution, no additional diffusant is introduced, but the solute near the surface diffuses in to the desired depth. The resulting solute concentration can be described by a Gaussian distribution, i.e.,

$$C = C_s \exp[-(a-y)^2/4Dt]. \quad (2)$$

II. STRESSES GENERATED BY DIFFUSION IN A WAFER

The stresses caused by concentration gradients are similar to those caused by temperature gradients in an otherwise unstressed body. Both are based on linear strain coefficients. For concentration gradients we have the relation

$$e_x = -\beta C, \quad (3)$$

where e_x is a component of strain and β is the solute lattice contraction coefficient.

Timoshenko⁵ has derived an expression for the stresses in a thin plate bounded by the planes $y=\text{constant}$ when the temperature distribution is independent of the x and z coordinates. By analogy we find the stresses developed in a wafer by diffusion in from the surfaces to be

$$\sigma_x = \sigma_z = \frac{\beta CE}{1-\nu} - \frac{\beta E}{2a(1-\nu)} \int_{-a}^{+a} C dy - \frac{3\beta Ey}{2a^3(1-\nu)} \int_{-a}^{+a} Cy dy, \quad (4)$$

where E is Young's modulus, and ν is Poisson's ratio.

The last term in the expression gives the stress relief by buckling when the stresses developed are non-symmetrical about the central plane, $y=0$. For a symmetrically diffused wafer we can drop this term and obtain the following:

$$\sigma_x = \frac{\beta E}{1-\nu} \left[C - \frac{1}{2a} \int_{-a}^{+a} C dy \right]. \quad (5)$$

For the erfc concentration distribution given by

⁵ S. Timoshenko, *Theory of Elasticity* (McGraw-Hill Book Company, Inc., New York, 1934), 1st. ed., p. 203.

Eq. (1), Eq. (5) becomes

$$\sigma_x = \frac{\beta C_s E}{1-\nu} \left[\operatorname{erfc} \frac{a-y}{(4Dt)^{1/2}} - \frac{2(\pi Dt)^{1/2}}{a\pi} \right]. \quad (6)$$

For the Gaussian concentration distribution given by Eq. (2), Eq. (5) becomes

$$\sigma_x = \frac{\beta C_s E}{1-\nu} \left[\frac{2}{\pi} \exp - \left[\frac{(a-y)^2}{4Dt} \right] - \frac{2(\pi Dt)^{1/2}}{a\pi} \right]. \quad (7)$$

For both concentration distributions, we note that the same maximum stress develops at the beginning of the diffusion process, $t=0$. For both,

$$(\sigma_x)_{\max} = (\beta C_s E / 1 - \nu). \quad (8)$$

The earliest values of β were obtained by Pearson and Bardeen⁶ in 1949 for boron and phosphorus in polycrystalline silicon. Later evaluations for boron by Horn⁷ and Epel'baum *et al.*,⁸ gave consistently higher values. This was attributed to boron precipitation in the grain boundaries of the polycrystalline samples. All investigators determined β by means of room temperature lattice constant measurements.

Saturation of the wafer surfaces by the diffusant is not an uncommon occurrence at the beginning of the diffusion process. Trumbore⁹ found solubility values at 1225°C of 1.5×10^{21} atoms/cm³ for phosphorus and 5×10^{20} atoms/cm³ for boron. Recent data for boron in single-crystalline silicon yielded a value for β of 5.6×10^{-24} cm³/atom at room temperature. The assumption that this value will hold for elevated temperatures is not unreasonable. The elastic constants of silicon and their temperature coefficients given by McSkimin *et al.*,¹⁰ yield a value of 21.56×10^{11} d/cm² for $(c_{11} + c_{12})$ at 1225°C. For diffusion into a {100} surface, $(c_{11} + c_{12})$ equals $E/1-\nu$. On substituting these values into Eq. (8), we obtain a value of 6.0×10^9 d/cm² for the maximum possible stress developed at the silicon surface as a result of the lattice contraction caused by substitutional boron atoms. Taking into account that the only available β value for phosphorus is probably too low by a factor of two to three as a result of grain boundary precipitation, the stress possible with phosphorus as the solute is even greater than with boron.

Pearson *et al.*,¹¹ give 5.5×10^9 d/cm² as the stress necessary to cause yield in silicon whiskers at 800°C. Extrapolating their data to 1225°C reduces the necessary stress by an order of magnitude. It would thus appear that, even without considering surface stress raisers, the stresses

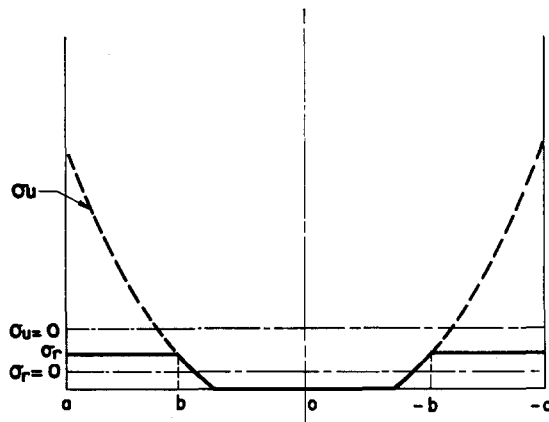


Fig. 2. Distribution of stresses over the cross section of a diffused wafer when the stress is relieved by the generation of dislocations, solid curve marked σ_r , and when the stress is unrelieved, curve σ_u , as given by Eq. (5). Each curve has its own datum given by $\sigma_r=0$ and $\sigma_u=0$.

developed by solute lattice contraction are sufficient to cause plastic deformation and thus generate dislocations in dislocation-free silicon. For less than perfect material, stresses of a lower order of magnitude would be required.

III. EXPERIMENTAL OBSERVATION OF DISLOCATIONS

The detection of dislocations in diffused layers proved impossible by decoration techniques. The solute atoms made the silicon opaque to the wavelengths of infrared radiation observable with an image converter tube. The following etch-pit evaluation technique was used instead. The diffused wafer was coated with Apiezon wax and cleaved. It was then placed in an agitated Dash etch solution¹² for a period ranging from 15 sec to several minutes. The attack was sufficient to bring out the dislocation etch pits in the diffused layer. Figures 5 and 6 illustrate the results obtained. The dislocation arrays were typical of a polygonized structure, which would be expected, considering that the material was subjected for relatively long times to a temperature of 1225°C.

The interior of the wafer was much less rapidly attacked than the diffusion layers. Thus, if the cleavage plane was etched sufficiently long to bring out dislocation etch pits in the interior, the diffused layers were heavily eroded, making it very difficult to discern the dislocation etch pits there.

IV. RELIEF OF STRESS BY THE GENERATION OF DISLOCATIONS

Equation (5) gives the stress in a wafer as equal to a constant multiplied by the difference between the concentration and an integral term. Thus, with appropriate ordinates, the stress distribution can be identical to the concentration profile from which it was derived, but

⁶ G. L. Pearson and J. Bardeen, *Phys. Rev.* **75**, 865 (1949).

⁷ F. Hubbard Horn, *Phys. Rev.* **97**, 1521 (1955).

⁸ V. A. Epel'baum, M. A. Gurevich, and M. S. Starostina, *Russian J. of Inorg. Chem.* **4**, 851 (1959), in translation.

⁹ F. A. Trumbore, *Bell System Tech. J.* **39**, 205 (1960).

¹⁰ H. J. McSkimin, W. L. Bond, E. Buehler, and G. K. Teal, *Phys. Rev.* **83**, 1080 (1951).

¹¹ G. L. Pearson, W. T. Read, Jr., and W. L. Feldmann, *Acta Met.* **5**, 181 (1957).

¹² 1 part hydrofluoric, 3 parts nitric, and 10 parts acetic acid.

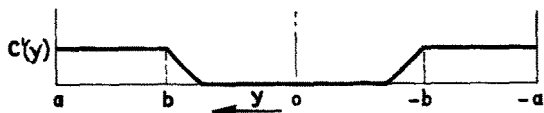


FIG. 3. Equivalent solute profile corresponding to the relieved stress distribution shown in Fig. 2.

will have a different datum, introduced by the integral term. This term, $(1/2a) \int_{-a}^a C dy$, is the average concentration across the wafer, the concentration at which the stress is zero. It satisfies the condition that the sum of the tensile forces is equal to the sum of the compressive forces. Where the datum concentration intersects the concentration profile, it defines the neutral x - z planes which separate the outside layers in the wafer which are in tension from the interior in compression.

In Fig. 2, the distribution of stresses across a diffused wafer induced by an arbitrary solute distribution is shown with the stress datum $\sigma_u = 0$. Curves above the datum represent tensile stresses, those below, compressive stresses.

The presence of dislocations in the diffused layers of originally dislocation-free silicon⁴ indicates that the dislocations were generated by the solute lattice contraction stresses and in turn partially relieved these stresses. Solid lines in Fig. 2 give the expected stress distribution if all tensile stresses above σ_r were reduced to this value. The new datum, $\sigma_r = 0$, is determined by the requirement that the sum of the compressive forces equals the sum of the tensile forces. As a result of stress relief, the neutral planes are moved farther in from the surfaces of the diffused wafer. The greater the stress relief, the closer do the neutral planes approach the depth of solute penetration. Since the datum must be positive when the applied stresses are in tension, it is impossible for a neutral plane to penetrate farther than the depth of solute penetration.

From the stress distribution resulting from partial stress relief, we can define an equivalent solute profile $C'(y)$. This hypothetical solute distribution is that which will produce the specific stress distribution on the basis of solute lattice contraction alone. Figure 3 illustrates the equivalent concentration profile corresponding to the relieved stress distribution shown in Fig. 2 by solid lines. We can describe the equivalent concentration distribution as follows:

$$\begin{aligned} C'(y) &= C(y) & 0 < |y| < b \\ C'(y) &= C(b) & b < |y| < a. \end{aligned} \quad (9)$$

We can now concentrate on the volume of the diffused layer lying between the planes $y = a$ and $y = b$, Fig. 2, in which the stress has been partially relieved. Let us examine an element of volume in the form of a thin plate parallel to the wafer surface. It is 1 cm in the x and z directions and dy cm thick. In Fig. 4 let C be the solute concentration at the bottom surface y of the element. The concentration at the upper surface $y + dy$ then becomes $C + (\partial C / \partial y) \cdot dy$.

Let the dislocation density (total length of dislocations per unit volume) be ρ . We note that each positive edge dislocation contributes one extra plane of atoms that exists at $y + dy$ that did not exist at y . The increase Δz in the z dimension of the element is equal to the total number of dislocations in the element ρdy multiplied by the contribution of each dislocation α , i.e.,

$$\Delta z = \rho \alpha dy.$$

In general, α is the component in the z direction of the edge component of the Burgers vector of the dislocation. It is a function of the slip system of the material and the crystallographic orientation of the diffused wafer. In the Appendix the value of α is calculated for silicon wafers with $\{111\}$ diffusion surfaces.

The solute lattice contraction between y and $y + dy$, $\Delta'z$, is equal to the product of the lattice strain coefficient β and the difference in concentration, i.e.,

$$\Delta'z = \beta \cdot (\partial C / \partial y) \cdot dy.$$

If dislocations relieve the solute lattice contraction strain within the element, we have an expression for the dislocation density in terms of the solute concentration gradient:

$$\rho = (\beta / \alpha) \cdot (\partial C / \partial y).$$

This expression gives the dislocation density between $y = b$ and $y = a$. The solute strain has been relieved here but the residual stress distribution given in Fig. 2 remains. The level of residual stress is directly dependent upon the depth in the diffused layer to which the dislocations penetrate. This determines b and thereby $C'(y)$.

We can now give the equivalent concentration at any point in the diffused layer as a function of the solute concentration and dislocation density distribution in the diffused layer:

$$C'(y) = \int_0^y \left(\frac{\partial C}{\partial y} - \frac{\alpha}{\beta} \cdot \rho \right) dy. \quad (10)$$

V. TOTAL NUMBER OF DISLOCATIONS

The dislocation density distribution in a diffused wafer may be described as follows:

$$\begin{aligned} \rho &= (\beta / \alpha) \cdot (\partial C / \partial y) & b < |y| < a \\ \rho &= 0 & 0 < |y| < b. \end{aligned} \quad (11)$$

The total number of dislocations N per unit length of diffused layer, the z dimension in Fig. 4, can be determined as follows:

$$N = \int_b^a \rho dy = (\beta / \alpha) \int_b^a (\partial C / \partial y) \cdot dy \quad (12)$$

$$N = (\beta / \alpha) [C_a - C(b)].$$

When $C_s \gg C(b)$ as often happens, N will be directly proportional to the surface concentration.

VI. MECHANISM OF DISLOCATION GENERATION

As has been described, the highest stress that develops is found at the surface at the very beginning of the diffusion process. As diffusion proceeds, this surface stress decreases as a result of increase of the datum, decrease of the surface concentration, or both. The initial stress, including the effect of surface stress raisers, may exceed the stress σ_0 necessary to generate dislocations. When this happens, the stress can be developed only to this level. The total number of dislocations generated, in number per unit length of cross section, is N , where

$$N = (\beta/\alpha)(C_s - C_0) \quad (13)$$

and where C_0 is determined by σ_0 according to Eq. (8).

The dislocations generated must lie in the surface and their Burgers vectors must have a positive edge component in the surface. Otherwise, they could not relieve the solute lattice contraction strain in the surface.

With increasing diffusion, the stress imposed by lattice contraction decreases at the surface. The transport of solute into the interior of the silicon wafer increases the stress there. These stresses are equalized by the movement of dislocations into the interior. The distribution of dislocations is given by Eq. (11).

After equalization, the residual stress is less than σ_0 . This removes the possibility of generating additional dislocations at the surface.

A. Constant Surface Solute Concentration Diffusion

When diffusion proceeds under conditions of constant surface concentration, the total number of dislocations remains fixed at $N = (\beta/\alpha)(C_s - C_0)$. The dislocations are dispersed over a greater depth as b increases with diffusion. The form of the dislocation distribution can be obtained by substituting the concentration erfc distribution, Eq. (1), into Eq. 11. This gives us for $b < |y| < a$

$$\rho = \frac{\beta}{\alpha} \cdot \frac{C_s}{(\pi Dt)^{1/2}} \cdot \exp\left[-\frac{(a-y)^2}{4Dt}\right] \quad (14)$$

Thus, for an erfc solute profile we have a Gaussian dis-

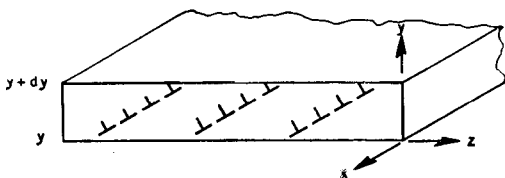


FIG. 4. Element of a diffused layer of a wafer whose solute strain has been partially relieved by the generation and distribution of dislocations.

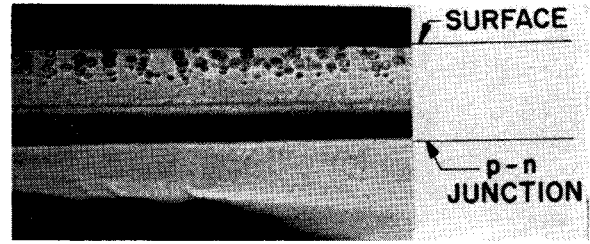


FIG. 5. Etched cleavage plane showing a diffused layer produced by the closed-tube diffusion of boron into n -type silicon. The source of diffusant, KBH_4 , was sealed in a quartz tube with the silicon wafers and the tube was then held at 1225°C for 75 hr. The junction depth as shown is $79\ \mu$. The wafer surface was prepared by chemical polishing.

location distribution from the surface in to point b , where the dislocation density drops to zero.

Figure 5 is a photomicrograph of a diffused layer produced by the closed-tube diffusion of boron into n -type silicon. This technique was chosen as it approximates constant surface concentration conditions. A significant feature of the etch-pit distribution is the fact that it penetrates only about halfway into the diffused layer.

B. Finite Surface Solute Source Diffusion

In the two-step diffusion process previously described, the pre-diffusion step can be considered as occurring under conditions of constant surface concentration. The redistribution step produces a Gaussian distribution of the solute, the surface concentration C_s decreasing as diffusion proceeds. From Eq. (12) we might expect that as C_s drops off there would be an excess of dislocations and $C(b)$ would drop off to zero, signifying complete relief of residual stress. However, in order to relieve the residual stresses, dislocations must move into the area of solute penetration. The stress necessary to move the dislocations through the crystal against resistances such as Peierls force and impurity locking is the new residual stress σ_0 . Since the temperature at which the dislocations move is high, the rate low, and the mechanism partly by climb, σ_0 will be relatively low.

For the residual stress to remain constant during the diffusion, excess dislocations must escape at the surface. Equation (11) would then describe the dislocation density distribution and Eq. (12) would give the total number of dislocations at any one time. On combining a Gaussian solute concentration with Eq. (11) we have for $b < |y| < a$

$$\rho(y) = (\beta/\alpha) \cdot [(a-y)/4Dt] \cdot C(y). \quad (15)$$

The maximum dislocation density occurs at a depth of $(2Dt)^{1/2}$ from the surface and is equal to $0.428(\beta/\alpha) \cdot [C_s/(Dt)^{1/2}]$ or approximately $0.428N/(Dt)^{1/2}$ when $C_s \gg C(b)$.

Figure 6 is a photomicrograph of a diffused layer produced by a two-stage open-tube diffusion of phosphorus into p -type silicon. One significant feature is

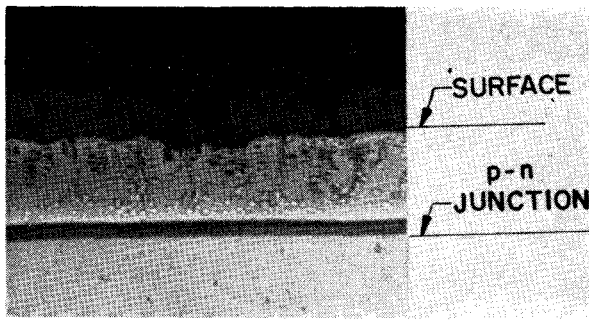


FIG. 6. Etched cleavage plane showing the diffusion layer produced by a two-stage open-tube diffusion of phosphorus into *p*-type silicon. The source of the diffusant, P_2O_5 , was passed over the heated wafers for a few hours to form a phosphorus-containing glass on the silicon surface. Phosphorus was absorbed by the wafers by heating them for 18 hr at 1225°C. Following this, the glass was stripped from the wafers with hydrofluoric acid and the wafers were returned to the furnace for an additional 125 hr at 1225°C. The junction depth as shown is 93 μ . The wafer surface was prepared by mechanical lapping.

that, unlike Fig. 5, the etch pits here penetrate almost up to the *p-n* junction. Another is that the etch-pit density reaches a maximum part way between the surface and the junction, as predicted for a Gaussian concentration distribution.

Studies of actual concentration distributions by measurements of sheet resistivities indicate that they are in the main neither exactly Gaussian or *erfc*.¹³ Determination of the dislocation distribution in the diffused layer would indicate what the actual concentration distribution is.

VII. RESIDUAL STRESS IN DIFFUSED WAFERS

The level of residual stress in a diffused wafer is of practical interest as it is certain to affect the wafer in further operations such as lapping and additional heat treatments. It is also of academic interest, being related to the stresses required to generate dislocations and to move them.

We can calculate this stress by introducing the equivalent concentration of Eq. (9) into Eq. (5). The calculation requires the same basic assumption as was made for the determination of the maximum unrelieved stress, namely, that the coefficient of thermal expansion is unaffected by the small concentrations involved. The desired relation is Eq. (16).

$$\sigma_r = \frac{\beta E}{1-\nu} C(b) \cdot \frac{b}{a} + \frac{2\beta E C_s (\pi D l)^{\frac{1}{2}}}{a(1-\nu)\pi} \times [1 + b\pi^{\frac{1}{2}} \operatorname{erfc} b - \exp(-b^2)]. \quad (16)$$

To apply this equation we need values for C_s , b , and $C(b)$. The first can be obtained by applying the method of Backenstoss,¹⁴ and the second by noting the depth

¹³ T. J. LaChapelle (personal communication).

¹⁴ G. Backenstoss, Bell System Tech. J. 37, 699 (1958).

TABLE I. Surface dislocations and their slip systems.

Slip system	Slip plane	Burgers vector ^a	Dislocation direction
1	(11 $\bar{1}$)	$a[011]/2$	[110]
2	(11 $\bar{1}$)	$a[101]/2$	$[\bar{1}10]$
3	(1 $\bar{1}1$)	$a[110]/2$	$[\bar{1}01]$
4	(1 $\bar{1}1$)	$a[011]/2$	$[\bar{1}01]$
5	($\bar{1}11$)	$a[110]/2$	[0 $\bar{1}1$]
6	($\bar{1}11$)	$a[101]/2$	[0 $\bar{1}1$]

^a a = lattice constant.

of penetration of the dislocation etch pits into the diffused layer. Finally, $C(b)$ can be obtained by substituting C_s and b into Eq. (1) or (2), whichever is applicable.

APPENDIX

In the derivation of Eq. (9), α was defined as the component in the z direction of the edge component of the Burgers vector of the dislocation. We can illustrate the calculation of this value easily for silicon, whose slip planes are {111} and whose slip directions are (110).

In Fig. 7, the base of the {111} tetrahedron, the (111) plane, represents the wafer surface. Since the dislocations must lie in the surface plane, only the six slip systems listed in Table I can be activated. The Burgers vector in slip system 1 can be resolved into screw and edge components. The edge component can be further resolved into a component lying in the surface plane and one normal to it.

$$\frac{a}{2} [011] = \frac{a}{4} [110] + \frac{a}{4} [112]$$

$$\frac{a}{4} [112] = \frac{a}{3} [111] + \frac{a}{12} [\bar{1}\bar{1}2].$$

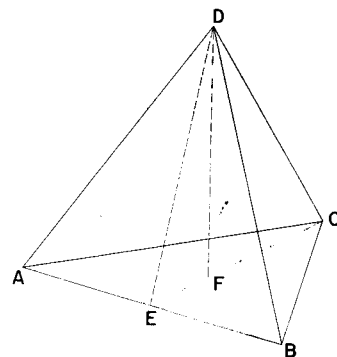


FIG. 7. The {111} tetrahedron showing the orientation of the various slip planes, dislocations, and Burgers vectors. These are as follow:

$$\begin{array}{lll} ABC = (111) & DE = [112] & AB = [\bar{1}10] \quad DA = [011] \\ ABD = (11\bar{1}) & DF = [\bar{1}11] & BC = [0\bar{1}1] \quad DB = [101] \\ BCD = (\bar{1}11) & EF = [\bar{1}\bar{1}2] & CA = [\bar{1}01] \quad DC = [110] \\ ACD = (111). \end{array}$$

Figure 8 illustrates the surface plane components of the edge components of the Burgers vectors of the six slip systems.

If our cross-sectional plane is the $(11\bar{1})$ cleavage plane, our z direction becomes $[\bar{1}\bar{1}0]$. The only contribution to strain in the z direction comes from the dislocations lying in the $[\bar{1}01]$ and $[0\bar{1}1]$ directions. Their surface edge components can be resolved further as follows;

$$\frac{a}{12}[\bar{1}2\bar{1}] = \frac{a}{24}[\bar{1}1\bar{2}] + \frac{a}{8}[\bar{1}10]$$

$$\alpha = \frac{a}{8}[\bar{1}10] = \frac{a(2)^{\frac{1}{2}}}{8}.$$

Similar calculations for a $\{100\}$ surface plane yield a value for α that is twice as large.

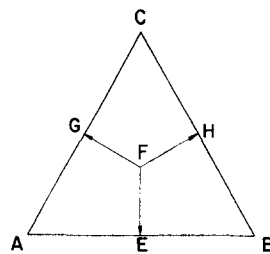


FIG. 8. The components of the edge components of the Burgers vectors lying in the $(11\bar{1})$ surface plane for the six possible slip systems.

$$FE = a[\bar{1}\bar{1}2]/12, FG = a[\bar{1}2\bar{1}]/12, FH = a[2\bar{1}\bar{1}]/12.$$

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