

NOTES ON DEFECTS IN SOLIDS

For 3.46 Optical and Optoelectronic Materials
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1. HISTORICAL PERSPECTIVE

Perfection in the structure of matter is not an obvious concept. In the late 1600's and 1700's sketches were first published which associated the external geometry of natural geologic crystals with the concept of a regular array of identical building blocks.^{1,2} A "second" structural method was developed some one hundred years later when in 1912 Laue presented his theory of x-ray diffraction from a periodic array of atoms.³ In 1913 W. L. Bragg reported the first structural determinations by x-ray diffraction using crystals of the alkali halide system.⁴ At this time all materials were considered to be perfect. The proof of perfection was the structure determination.

In 1914 the first observation of ionic conductivity in the silver halides was reported.⁵ At the time various explanations for the phenomenon were proposed. One was that there were cracks in the material and the ions were flowing down the cracks; another suggested that the surfaces were dirty and the conductivity represented motion along the surfaces. The curiosity remained until 1926 when Frenkel proposed that internal lattice defects were created and carried the conductivity.⁶ This mechanism represented the first role (property) for imperfection in an otherwise perfect lattice.

Frenkel's proposal was as follows: silver ions which are interwoven within the chloride lattice could be excited into interstitial sites and migrate as interstitials leaving behind vacant sites on the silver sub-lattice. Three chemical species are involved: the silver ion on its own lattice site; the vacant lattice site, which is left behind when the silver ion is excited, and the interstitial silver ion which is moving through the lattice carrying the ionic conductivity. Frenkel proposed further that chemical relationships governed an equilibrium among these species with associated equilibrium constants relating their formation and interaction.

The formation reaction is expressed as



as silver on a substitutional site reversibly transforms to silver in an interstitial site plus a vacancy on the silver site.

In 1931 Wagner and Schottky refined Frenkel's concept to firmly establish that point defects should be treated as chemical species in equilibrium.⁷ They pointed out that the interstitial and the vacancy could be considered as separate entities with unique and independent energies of formation and, hence, equilibrium concentrations.

2. IMPERFECTION AS A CHEMICAL ENTITY

The introduction of an imperfection in a condensed system is accomplished by a change in the free energy ΔG of the system. This free energy change is represented by an enthalpy and an entropy change. The enthalpy term consists of the formation energy or the net internal energy which is required to produce the defect. If this term were the only contribution, one would rarely encounter imperfections in crystalline systems. However, this term is balanced by the entropy change associated with the introduction of imperfection into the system.

$$\Delta G_f = \Delta H_f - T(\Delta S_v + \Delta S_c) \quad (2)$$

There are two components to the entropy: the formation entropy ΔS_v which is relatively small and reflects the changes in the vibrational modes of the atoms which surround the imperfection; and the configurational entropy, ΔS_c which expresses the number of possible lattice arrangements. For the case of vacancies, the configurational term dominates and represents the number of ways a given quantity of vacancies can be placed on a certain quantity of lattice sites. This contribution can become fairly large, so that, at a given temperature, the total free energy of a system is minimized by increasing its imperfection.

The enthalpy of formation ΔH_f of an imperfection may be estimated in the following way. Consider the example of a vacant lattice site. A model reaction path could involve the removal of an atom from its lattice site in the internal part of the crystal and the placement of it on a lattice site at the surface. The energy associated with such a process is, identically, the latent heat of vaporization -- the energy required to remove an atom from the surface and place it at infinity. This equivalence is derived from the local coordination of the various sites. For fourfold bulk coordination, four bonds must be broken to remove the atom from its internal lattice position. Two bonds, however, are reformed at the surface. Thus, the formation enthalpy is, to first order, the energy required to break two bonds or the heat of vaporization.

The formation enthalpy can be reduced by any local relaxation of structure near the imperfection. The degree of directional bonding in a system determines, to a large extent, the amount of relaxation to be expected. In crystals such as solid argon, very little relaxation is observed. However, in covalent, semiconductor crystals, relaxation effects can be substantial. Table 1 lists some representative vacancy formation enthalpies for different solid types.

Table 1. VACANCY FORMATION ENTHALPIES

Solid Type	ΔH_f (eV)
noble gas	0.05-0.1
alkali metal	0.2-0.5
transition metal	2.0-3.0
semiconductor	2.5-4.5

We have, thus, considered the totality of the structure of an imperfection in a crystalline system-- the identity of the imperfect site(s) and the structure of the 'perfect' lattice surrounding the imperfect region. The nature of the local lattice

relaxation is, in particular, a very challenging problem. One can well understand why the study of the microscopic structure of imperfection has become one of the most active areas of research in modern physics and chemistry.

One can categorize the various classes of imperfection which exist in condensed matter. The smallest perturbation to perfection is present in all condensed matter as *zero point lattice vibrations*. As the temperature is raised, the vibrational amplitude increases and the modal distribution becomes more complex. The magnitude of this type of imperfection can become very large as in a diffusion jump between adjacent lattice sites.

One can also consider *free electronic carriers* as imperfection. Normally an electron will exist in a low energy, bound state. If it is excited that to a higher energy state in which it is free to migrate as an entity, the electron can be regarded as a point imperfection.

Chemical impurities as a class of imperfection can be located on either substitutional sites -- that is, regular lattice sites, or interstitial sites, between the regular lattice sites. The preference of an impurity for 'substitutionality' or 'interstitiality' depends on its electronegativity, size, valence, and preferred coordination relative to the host material. In addition, a significant difference in any of these properties relative to the host matrix usually results in a reduced solubility for the impurity. One frequently encounters difficulty in studying imperfections because they are present only in very small concentrations.

In addition to the lattice vacancy and interstitial atom, a third type of lattice imperfection can exist in a pure, but multicomponent (compound) structure. This class is known as *antisite* or *antistructure* imperfection. For example, in an ordered lattice consisting of A and B atoms an A atom might exist on a B site or a B atom might occupy an A site. This local disorder is significant in compounds of low ionicity where the A and B atoms are similar in properties.

Larger, macroscopic forms of imperfection are considered in terms of dimensionality. *One dimensional* imperfections, such a *dislocations* will be

considered in some detail later. *Two dimensional*, planar structures exist, for example, as *stacking faults* – an error in the stacking sequence of the lattice planes, or as *grain boundaries*– the boundaries between perfect, but misoriented crystals. *Three dimensional* imperfections include *precipitates*, *excess volume* which occurs in noncrystalline materials, and *the antiphase domains* which are a form of extended antisite structure.

Table 2. IMPERFECTION IN CONDENSED MATTER

CLASSIFICATION	DEFINITION	EXAMPLES
- VIBRATING ATOM	TEMPORARY, SMALL DISPLACEMENT FROM IDEAL POSITION	ZERO POINT, THERMAL
- ELECTRONIC CHARGE	CHARGE CARRIER EXCITED FROM GROUND STATE BONDING CONFIGURATION	ELECTRON (-), HOLE (+) EXCITON
- CHEMICAL IMPURITY	FOREIGN ATOM OF DIFFERING SIZE,	SUBSTITUTIONAL, INTERSTITIAL VALENCE, ELECTRONEGATIVITY, AND/OR STRUCTURE RELATIVE TO HOST ATOMS/ STRUCTURE
- POINT LATTICE DEFECT	MISSING HOST ATOM EXTRA HOST ATOM ATOM OCCUPYING WRONG LATTICE SITE NON BRIDGING BOND	LATTICE VACANCY SELF INTERSTITIAL ANTISITE DEFECT (COMPOUNDS) (NONCRYSTALLINE MATERIALS)
- ONE DIMENSIONAL DEFECT	ROW OF ATOMS AT EDGE OF EXTRA HALF PLANE OF ATOMS	DISLOCATION (EDGE AND SCREW)
- TWO DIMENSIONAL DEFECT	BOUNDARY SEPARATING AN ERROR IN STACKING SEQUENCE OF ATOMIC PLANES BOUNDARY BETWEEN TWO CRYSTALS OF DIFFERING RELATIVE ORIENTATION	STACKING FAULT GRAIN BOUNDARY
- THREE DIMENSIONAL DEFECT	MACROSCOPIC REGION OF DIFFERING DENSITY, CHEMICAL CONTENT, COORDINATION ETC. FROM HOST	VOID FREE VOLUME (NONCRYSTALLINE), DISORDER, DEFECT CLUSTER, PRECIPITATE

3. POINT DEFECT EQUILIBRIA

The configurational entropy change of defect formation is given by

$$\Delta S_c = k \ln W - k \ln W^* \quad (3)$$

where W^* and W are the number of ways to configure the perfect and imperfect solid, respectively. W^* equals unity for a perfect, periodic solid. For a lattice of n_1 identical atoms and n_2 identical defects on $(n_1 + n_2)$ sites

$$W = \frac{(n_1 + n_2)!}{n_1! n_2!}, \quad (4)$$

which represents the number of ways to arrange all sites divided by the number of ways to arrange the atoms or defects among themselves.

Consider the *vacancy formation* reaction



where A_A is an A atom on an A lattice site and V_A is a vacancy on an A lattice site. Thus, a vacancy is created in the bulk and the displaced atom is placed on the surface. The equilibrium constant for this reaction is

$$\begin{aligned} K_V &= \frac{[V_A][A_A]}{[A_A]} = [V_A] \\ &= \exp \left[-\frac{\Delta G_f}{kT} \right] \\ &= \exp \left[-\frac{(n_2 \Delta H_f(V_A) - T \Delta S_c - T n_2 \Delta S_v)}{kT} \right]. \quad (6) \end{aligned}$$

Figure 1 graphically displays the competition of terms which determines the equilibrium vacancy concentration.

A *Frenkel pair* is an interstitial-vacancy pair created by the reaction



The equilibrium constant of the reaction is given by

$$K_{FP} = \{ [A_I] [V_A] \} / \{ [A_A] \} \quad (8)$$

The activity of $[A_A]$ in a nearly perfect crystal is unity. Notice that equation 8 constrains the product of the concentrations of interstitials and vacancies to be a constant. Typically, one expects $\Delta H_f (V) \sim \Delta H_f (I)$ in group IV, covalent semiconductor materials because of the open nature of the lattice and because both defects have the same number of unsatisfied bonds. In these materials $[V]$ is typically $\sim 10^{-6}$ at the melting point. In metals, $\Delta H_f (V) \ll \Delta H_f (I)$ because of the close packed nature of the structure and the large strain energy associated with interstitial atoms. In these materials, $[V]$ is about 10^{-3} at the melting point. .

4. DEFECT IONIZATION EQUILIBRIA

In semiconductors, electronic interactions are uniquely important. The role of free carriers as chemical entities has been established. Ionization equilibria affect the equilibrium defect concentrations and, consequently, processes, such as diffusion, which depend on these concentrations in nonmetallic solids. Consider a system in which vacancies act as acceptors (become negatively charged).

The total vacancy concentration is given by

$$[V_T] = [V_i] \cdot n / n_i \quad (9)$$

where $[V_T]$ is the total vacancy concentration in the doped material and $[V_i]$ is the intrinsic vacancy concentration in pure material. Diffusion by a substitutional mechanism is dependent on the availability of vacant sites for an atomic jump to occur. Therefore,

$$D_S = D_S (\text{intrinsic}) \cdot n / n_i$$

5. ASSOCIATION REACTIONS

Defects and impurities will pair, cluster, or leave solution when the free energy of the associated system is lower than that of the isolated entities. Some association interactions are the relief of strain energy, Coulombic interactions, and local bonding.

The fraction of associated species AB in a system of equal concentrations of A and B is determined by the negative free energy change.

$$\begin{aligned}\frac{[AB]}{[A][B]} &= \frac{x}{(1-x)^2} \\ &= \exp[\Delta G_{AB}/kT] \\ &= \exp[(\Delta H_{AB} - T\Delta S_{AB})/kT]\end{aligned}\quad (10)$$

Since association induces order, the entropy change $\Delta S(AB)$ is negative. The enthalpy of association is the strength of the interaction. When imperfections of opposite strain associate, the elastic work on the lattice, H_e , is reduced by partial cancellation of the strain fields.

(11)

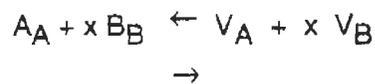
$$\begin{aligned}\Delta H_e(AB) &= 1/z H_e \\ &\equiv -0.1 \text{ to } -1.0 \text{ eV}\end{aligned}$$

where z is the coordination number of the pair. These interactions are long range for a dislocation ($\propto 1/r$) and short range for a point defect ($\propto 1/r^3$).

6. IMPERFECTION IN MULTICOMPONENT SYSTEMS

6.1 POINT DEFECTS IN COMPOUND MATERIALS

In compound solids defect equilibria include relations among the various atomic sublattices. The primary defect of an AB_x compound is the *Schottky defect*, a simple vacancy pair.



$$K_S = [V_A][V_B]^x \quad (12)$$

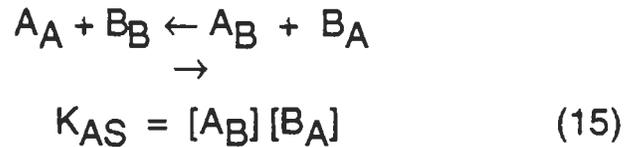
The expression for K_S , the Schottky equilibrium constant, includes the unity activity assignment to the perfect lattice (the left side of the formation equation). Note that the vacancy concentrations on each sublattice are interdependent. This result follows from the reaction path which places an interior atom on the surface. Since the perfect crystal lattice must be continued at the surface, defects on one sublattice must be created in cooperation with the other sublattice.

Frenkel pair equilibria are unique to each sublattice and are, therefore, independent.

$$K_{FP}(A) = [V_A][A_i] \quad (13)$$

$$K_{FP}(B) = [V_B][B_i] \quad (14)$$

Compounds whose constituents possess similar electronegativities can exhibit *antistructure* or *antisite defects*. For compound AB

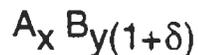


Since both sublattices are involved in defect formation, the product of the defect concentrations on each sublattice must be constant.

6.2 NONSTOICHIOMETRY IN COMPOUND SOLIDS

A stoichiometric compound $A_x B_y$ is one in which the constituents are present in precisely the concentration ratio $x:y$. Slight deviations from this condition can produce large effects on materials properties. For example, one part per million excess of one constituent could produce 10^{16} cm^{-3} excess free carriers in a semiconductor. In the more ionic materials, impurity diffusivity can vary by orders of magnitude with nonstoichiometry.

The degree of nonstoichiometry is given by the expression



where $\delta(+)$ means excess B and $\delta(-)$ means excess A. Section 6.1 discussed defect equilibria in stoichiometric compounds. Thus, stoichiometry does not imply perfection in a solid. Stoichiometry, however, requires that the defect concentrations on each sublattice are equal. Nonstoichiometry implies inequivalent concentrations of conjugate defects.

7. ELECTRONIC PROPERTIES

The electronic properties of imperfection results from perturbations of the local chemical bonding. An imperfection which is chemically similar to the matrix in which it is contained introduces a small perturbation to the system. This perturbation is diffuse, over a large volume, and will not severely affect the properties. This behavior is typical of isoelectronic substitutions in metals and insulators and dopant atoms in semiconductors. For instance, arsenic, phosphorous, and antimony (Group V elements) are added to silicon (Group IV) to increase the free electron concentration. In the ground state the orbit of the extra electron is 16 Å in radius in silicon and even larger in germanium.

An imperfection such as a lattice vacancy or a dislocation represents a very strong perturbation to the system. This class of imperfection displays a more localized potential associated with the defect region, and the electronic state representing it is tightly bound. In chemical terms, the valence electrons of the imperfection participate, primarily, in the local bonds around the affected region and not in extended states of the host crystal. The equilibrium structure of the imperfection is, therefore, influenced by the defect state electronic configuration as well as the host lattice. As electrons are added or subtracted in the region of the imperfection, modification of both structure and properties may occur.

Lastly, a local region of difference in the lattice will create a local vibrational mode. The mode may be a true local mode or a resonance mode which has a frequency in the range of the vibrations of the host lattice. In this respect a localized imperfection can be considered in terms of a *defect molecule*. One can often ignore the crystal matrix and regard the imperfect region as a defect molecule to be characterized in terms of its local properties.

The IV-VI compounds exhibit small, direct bandgaps and have applications as long wavelength detectors and lasers. They possess rock salt structures (CN=6). The rock salt structure signifies large ionicity. For excess M (metal, Pb^{++}), both n- and p-type behavior can result. For example, when an N (non-metal, Te^{--}) atom is removed to form a V_N , the electrons which were associated with the N^{-n}

ion in the crystal (or with the bond in covalent compounds) are left behind. The vacancy ionizes as a donor



Conversely, V_M should act as an acceptor. Table 3 summarizes the expected electrical activity of defects in compound semiconductors.

Table 3. ELECTRICAL ACTIVITY OF NONSTOICHIOMETRIC DEFECTS IN COMPOUND SEMICONDUCTORS

COMPOUND $MN_{x(1+\delta)}$	
EXCESS M (δ^-)	EXCESS N (δ^+)
V_N (donor)	V_M (acceptor)
M_i (donor)	N_i (acceptor)
M_N (acceptor)	N_M (donor)

Note, when antistructure is not important the degree and sublattice of nonstoichiometry can be determined (for a pure material) from the sign and magnitude of the extrinsic carrier concentration.

8. DISLOCATIONS

The onset of plastic deformation in solids (yield point) typically occurs at 0.1-0.2% strain. Atomic models of perfect crystals fail to explain how such a small deviation from the equilibrium lattice position can produce permanent relative shear motion of adjacent planes. The relative extension to reach a saddle point configuration in such models exceeds 10%. In addition, the level of stress

required to move one plane of atoms uniformly over another is extremely high, $>10^5 \text{ kg/mm}^2$, whereas observed values are the order of 1 kg/mm^2 . A special type of lattice imperfection, the dislocation, resolved these paradoxes.

Dislocations are characterized by a net displacement, the Burgers vector, \mathbf{b} and a vector \mathbf{l} , which is parallel to the dislocation line. Dislocations are classified by two primary types: edge, $\mathbf{b} \cdot \mathbf{l} = 0$ and screw, $\mathbf{b} \cdot \mathbf{l} = \mathbf{b}$. An edge dislocation is shown in Figure 2. Motion is confined to a *glide* plane which contains both \mathbf{b} and \mathbf{l} . Climb motion is normal to the glide plane and is nonconservative, requiring the addition or subtraction of point defects. Climb results in an increase of the dislocation line length, whereas glide does not.

The dislocation line energy is the strain energy along the dislocation line, $E_t \propto |\mathbf{b}|^2$. A dislocation introduces negligible configurational entropy because the disorder is concentrated along a single line. Dislocations may dissociate into components with Burger's vectors which are less than lattice translation vectors. These *partial* dislocations bound a two dimensional, stacking fault imperfection.

In 1934, the structures of dislocations were modeled by Taylor⁸, Orowan⁹, and Polanyi¹⁰, and classified as *edge*, Figure 3a, and *screw*, Figure 3b, *dislocations*. The edge dislocation is simply the extra half plane considered above, whereas the screw dislocation represents a twisting motion about a central axis. The structure of a dislocation is most easily revealed by a Burger's circuit. The circuit is constructed by circumscribing the imperfection with an equal number of lattice translations in each direction. The vector joining the end point to the beginning point is the Burger's vector. Electron and x-ray diffraction methods which resolve the orientation of \mathbf{b} relative to \mathbf{l} are routinely employed for structural determination.

In 1947, Shockley and Heidenreich¹¹ suggested that the lowest energy configuration of a dislocation is one which is dissociated into lines of partial lattice translations. Interestingly, all of these models were formulated before a dislocation had been observed. Only the discrepancy between the theoretical and observed mechanical properties of materials had been reported.

A turning point in the study of dislocations was the first direct observation of moving dislocations during the deformation process by Hirsch¹² in 1956. The structural models at the time were indeed powerful and could account for most of the observed properties. Now, however, dislocations could be visualized in three dimensions and the deformation process was no longer schematized in terms of a single straight dislocation. Concepts such as dislocation climb (the interaction of point defects with the dislocation) soon developed.

About the same time, another method of viewing dislocations was demonstrated by Dash¹³. Copper impurities were employed to decorate the dislocations creating regions of enhanced absorption of the infrared light. Following diffusion of copper into the silicon single crystal, the enhanced reactivity of the dislocation induced preferential precipitation along the dislocation line.

The work of Ray and Cockayne¹⁴ on silicon pioneered the weak beam technique in transmission electron microscopy. Under normal diffraction conditions the electron beam is oriented to diffract from the perfect crystal. Therefore, the observed contrast outlines the imperfection of this region. In the weak beam technique, the process is inverted. The electron beam is carefully tilted off the perfect crystal diffraction conditions, so that diffraction occurs only in a small region of the very severely bent planes near the dislocation. In this way the resolution can be enhanced by an order of magnitude in some cases. The work confirmed the predictions of Shockley and Heidenreich by revealing regions along the dislocation line where *splitting* occurs. The dark field image shows that the splitting bounds a plane of imperfection which can be characterized further as a stacking fault.

Figure 4 by A. Bourret and Desseaux¹⁵ depicts the current state of the art in structural diffraction methods. Shown is a dislocation imaged by a high resolution, direct lattice image technique using the transmission electron microscope. The unique aspect of this method relative to the other TEM methods is the use of all diffraction information. Conventional techniques portray

differences in diffracted beam *intensity*. The lattice image technique utilizes, in addition, the phase information produced by the interference of the direct and diffracted beams traveling through the crystal. By adding this phase information to the intensity information, one can image rows of atoms in the crystal.

In Figure 4 a dislocation is located at the center and is identified by construction of a Burger's circuit which requires an extra lattice translation for completion. A {100} plane of a germanium crystal is shown. The direct verification of the simple structural models of Figure 3 is striking. However, the modern questions remain unanswered. What is the microscopic bonding structure at the core?

9. GRAIN BOUNDARIES

A dislocation model¹⁶ of a tilt boundary in a simple cubic lattice is shown in Figure 5. The crystals on either side of the boundary are tilted at an angle with respect to a common cube axis [001] normal to the plane of the drawing. For the low-angle boundary of Figure 5, the misfit in general is accommodated in two ways: (1) by elastic deformation and (2) by atomic rearrangement at the boundary. Elastic deformation can accommodate much of the deformation but not all of it, and some of the vertical atomic planes must terminate at the boundary, thus forming edge dislocations normal to the plane of the drawing. Etch pit observations of similar tilt boundaries in germanium by Vogel *et al.*¹⁷ simultaneously confirmed the dislocation model of a grain boundary and the one-to-one correspondence between etch pits and dislocations. Work by Bourret and Desseaux¹⁸ using high-resolution TEM demonstrates that even in boundaries with tilts around 1° the dislocation structure is complex. The boundary is not straight on the scale of 100 to 1000 Å, and various dislocation dissociations are observed separated by stacking faults. At larger misorientations certain lattice sites on either side of the boundary that coincide are referred to as coincidence site lattice (CSL) boundaries. When this occurs, the boundaries' facet and twinning regions are formed. For still-higher misorientations in which dislocation spacing is of the order of the lattice spacing, the dislocation model is no longer

applicable and the misorientation is accommodated by a region of disorder at the boundary.

There is ample evidence from high-resolution TEM of the amazing variety and complexity of dislocation structures even in simple tilt boundaries. For boundaries with both tilt and twist components, the dislocations must possess screw components, and atomic descriptions of the structures of boundaries become even more complex. Since the electrical activity of grain boundaries is intimately associated with structural misfit at the boundary, the influence of all disorder must be considered and not just that due to dislocations.

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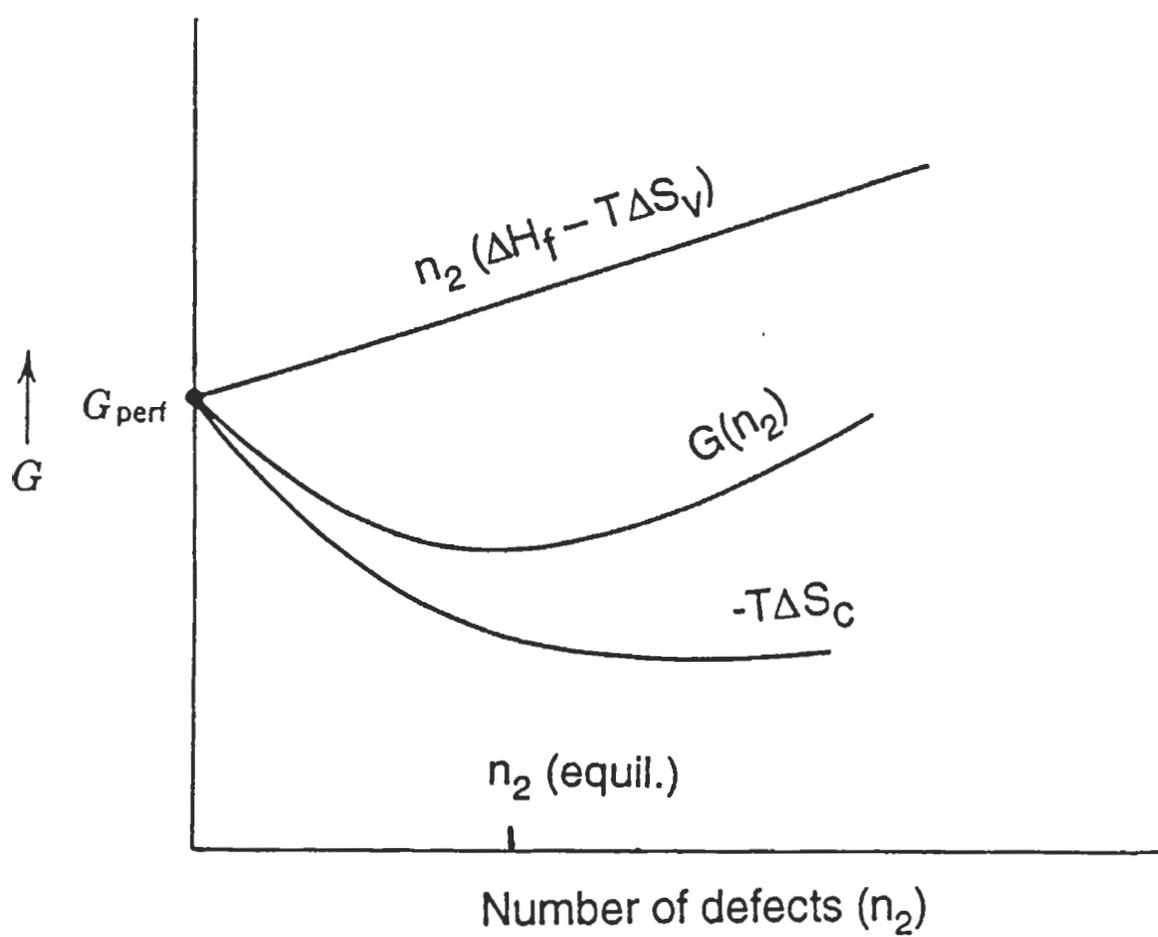


Figure 1: Free energy of a crystal as a function of point defect concentration.

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Figure 2: Deformation of a crystalline substance under an applied shear stress by dislocation motion.

Figure 3: Schematic models of (a) edge dislocation and (b) screw dislocation.

Figure 4: [Image of crystal structure].

Figure 5: Model of a low-angle grain boundary in a simple cubic lattice.