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Material Microstructure and Phase Equilibria

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Abstract: In the solid state, atoms combine in a manner that ensures minimal internal energy by formation of a regular, long-range repetitive space lattice known as a crystal. Regular space lattices do not occur in amorphous structures as the atoms tend to be arranged in non-repeating units. The structural arrangements of the non-crystalline solids represent high internal energies. Phase diagram provides information about the phase structures of a particular system. There are three parameters that control and affect phase structure which are: temperature, pressure, and composition. Phase diagrams are constructed when various combinations of these parameters are plotted against one another.

Keywords: microstructure; phase diagram; Gibbs rule.

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I. Material Microstructure:

I.1. Microstructure of Metals:

Most metals used in dentistry belong to the cubic system such as iron (body centered cubic cell) and Copper (face-centered cubic cell). Other types of space lattices of dental interest such as hexagonal close-packed is observed in titanium, zinc, and zirconium.

I.1.1. Microstructure of Cast Metals:

I.1.1.1. Homogeneous Nucleation:

Metals are formed from multiple crystals because there are usually many nuclei of crystallization scattered throughout the molten metal. These nuclei are formed when atoms lose sufficient thermal energy and become able to form a unit cell. These unit cells will grow as more metal atoms reach a low enough energy to join and hence crystal formation occurs [1, 2].

I.1.1.2. Heterogeneous Nucleation:

Solidification may be initiated by the presence of impurities in the melt. As the temperature drops below the melting point, metal atoms will deposit on these impurities and crystals begin to form [1]. In both nucleation mechanisms, the crystals (grains) will continue to grow until complete solidification of the metal. Crystals impinge on one another during their

growth, giving rise to boundaries between the crystals where the atoms are irregularly arranged.

This boundary is called the grain boundary which is considered a defect in the crystal structure of the metal [1]. A fine grain size is usually desirable in a metal because it raises the yield strength. Yield strength varies inversely with the square root of grain size. Fine grain size can be obtained by:

- **Rapid solidification:**

Fine grain sizes could be obtained by rapid solidification conditions because the time during which solidification occurs is inadequate for the growth of large crystals. Rapid rate of cooling could be obtained by keeping the mold temperature below the melting temperature of the alloy [2].

- **Presence of many nucleating sites:**

Noble metal casting alloys have equiaxed fine-grain microstructures because they contain small concentrations of iridium (Ir), ruthenium (Ru), or rhenium (Re) as grain-refining elements [1, 2].

I.1.2. Microstructure of wrought alloys:

The microstructure of wrought alloys is fibrous and entangled internal structure. Wires or other wrought forms produced by cold working of cast structures have increased tensile strength and hardness when compared with corresponding cast structures. Wrought structures recrystallize during heating operations, where the fibrous microstructure is converted to a grain structure. The recrystallization depends on the time, temperature, alloy composition, and degree of cold working by which the wire was fabricated [3].

Recrystallization increases with increasing both the heating time and temperature. For example, in most noble dental wires, a short heating cycle during the soldering operation is not sufficient to recrystallize the wire, even though the temperature approaches the fusion temperature. However, a prolonged heating period of 30 to 60 seconds or longer may cause recrystallization, Recrystallization results in a reduction in mechanical properties in proportion to the amount of recrystallization [3].

I.1.3. Effect of method of manufacturing on the microstructures of metals and alloys:

Casting technique produces coarse microstructure and solidification defects because of the low cooling rate. Moreover, metals produced by powder metallurgy have much finer grain sizes compared to those produced by casting [4]. Powder metallurgy includes mechanical milling and hot consolidation. The grain size after powder sintering is close to the initial powder particle sizes. Therefore, the grain size of the final product could be adjusted by tailoring the initial powder size [5].

Selective laser sintering assisted by CAD-CAM technology is a layering additive manufacturing procedure that manufactures metal components directly from 3-dimensional CAD data by melting

fine layers of metal powder with a high-powered, focused laser beam. It produces fewer internal defects and porosities leading to formation of a dense material with finely homogeneous grains leading to production of metals with improved mechanical properties [6].

I.1.4. Examination of metal microstructure:

- **Light-reflecting optical microscope:**

The polished metal surface is etched with a suitably chosen chemical (e.g., Kalling's reagent) that attacks certain regions of the metal surface. These areas are the grain boundaries, where there is imperfect packing of the atoms. A groove is produced and it will scatter the incident light appearing as a dark line [1, 7].

- **X-ray diffraction:**

XRD is used to identify phases composition and to determine the relative amount of each phases present [4]. It provides information about crystal orientations, average grain size, crystallinity, and crystal defects [8].

- **Scanning Electron Microscope:**

Provides qualitative and quantitative information related to the makeup of crystals. SEM is used to measure and identify the grain size and crystal structure. The crystallographic orientation could be examined using electron back-scattered diffraction (EBSD) pattern [7].

- **Transmission electron microscope:**

It is used for examination of phase structures and phase compositions. For TEM observations thinning of specimens is mandatory. It also provides information about the formation mechanism of the developed phases and the volume fractions of the phases of interest. TEM coupled with EDS analysis provides analysis of phases chemical components [9].

I.2. Microstructure of Polymers:

Polymers consists of macromolecules with chainlike molecular structure. In the carbon-chain polymers, the backbone of each chain is a line of carbon atoms, each of them bonds to two adjacent carbon atoms on either side. In some polymers the chains are randomly coiled and entangled in a very disordered or random pattern known as an amorphous structure. In others, the chains align themselves to form a highly ordered, or crystalline structure. Most polymeric materials combine these two forms of organization [2].

I.2.1. Factors which control structure of polymers:

I.2.1.1. Molecular Weight:

During the polymerization process, not all polymer chains grow to the same length resulting in a distribution of chain lengths or molecular weights.

Average molecular weight determination:

➤ **The number-average molecular weight (Mn):**

It is obtained by dividing the chains into a series of size ranges and then determining the number fraction of chains within each size range. The number-average molecular weight is obtained from this equation:

$M_n = \sum x_i M_i$, where M_i is the mean molecular weight of size range i , and x_i is the fraction of the total number of chains within the corresponding size range.

➤ **Weight-average molecular weight M_w :**

It is based on the weight fraction of molecules within the various size ranges. It is calculated by the following equation:

$M_w = \sum w_i M_i$, where w_i denotes the weight fraction of molecules within the same size interval.

➤ **The degree of polymerization (DP):**

DP is related to the number-average molecular weight M_n by the equation:

$DP = M_n / m$, where m is the repeat unit molecular weight.

Polydispersity: it is the ratio M_w/M_n that is considered as a measure of the range and distribution of chain sizes. If the values M_w and M_n are the same (Polydispersity equals 1), the polymer is termed monodisperse (uniform). This occurs when all molecules have the same weight. When the values M_w and M_n are different, this polymer is termed polydisperse (non-uniform). Polydisperse polymer contains a mixture of molecules with wide molecular weight distribution. Polymers with equal value of M_w but different values of polydispersity will exhibit different properties. For example, polymers of higher polydispersity (Polydispersity more than 1) will begin to melt at a lower temperature and have a larger temperature range of melting and vice versa [2, 10].

Effect of molecular weight on polymer microstructure:

Crystallization is not favored in polymers that are composed of chemically complex repeat units. Crystallization occurs in chemically simple polymers with low molecular weight even for rapid cooling rates [10].

I.2.1.2. Chain Configuration:

- **Linear Polymers:**

Linear polymers are those in which the repeat units are joined together end to end in single chains. For linear polymers, there may be extensive van der Waals and hydrogen bonding between the chains.

- **Branched Polymers:**

Polymers may be synthesized in which side-branch chains are connected to the main ones. The branches may result from side reactions that occur during the synthesis of the polymer. The chain packing efficiency is reduced with the formation of side branches, which results in a lowering of the polymer density.

- **Crosslinked Polymers:**

In crosslinked polymers, adjacent linear chains are joined one to another at various positions by covalent bonds, crosslinking is achieved either during synthesis or by a nonreversible chemical reaction. Crosslinking is accomplished by additive atoms or molecules that are covalently bonded to the chains.

- **Network Polymers:**

Multifunctional monomers forming three or more active covalent bonds make three dimensional networks and are termed network polymers. Highly crosslinked polymers may also be classified as a network polymer [3, 10].

Effect of chain configuration on polymer microstructure:

For linear polymers, crystallization is easily accomplished because there are few restrictions to prevent chain alignment. Side branches in branched polymers interfere with crystallization. Network and crosslinked polymers are almost totally amorphous because the crosslinks prevent the polymer chains from rearranging and aligning into a crystalline structure [10].

I.2.1.3. Copolymerization:

Polymers that have only one type of repeating unit (mer) are homopolymers; those with two or more types of mer units are known as copolymers.

There are four different types of copolymers:

- **Random copolymer:**

No sequential order exists among the two or more mer units along the polymer chain.

- **Alternating copolymers:**

The two repeat units alternate chain positions.

- **Block copolymer:**

Identical monomer units occur in relatively long sequences (blocks) along the main polymer chain.

- **Graft or branched copolymer:**

Sequences of one type of mer unit are “grafted” onto a backbone chain of a second type of mer unit to form a branched configuration [2, 3].

Effect of copolymerization on polymer microstructure:

Copolymerization disrupts chain regularity reducing the average length of crystallizable sequences. The more irregular and random the repeat unit arrangements, the greater is the tendency for the development of amorphous structure. Therefore, crystallization occurs more easily in alternating and block copolymers compared to random and graft copolymers [10, 11].

I.2.2. Polymer crystallinity:

Polymer crystallinity occurs by the packing of molecular chains to produce an ordered atomic arrangement. Polymers are often only partially crystalline (or semicrystalline) with crystalline regions dispersed within the remaining amorphous region which is characterized by chain disorder. Sufficient time must be allowed for the chains which are highly random and entangled in the viscous liquid state to move and align themselves in ordered configuration [10].

I.2.2.1. Degree of crystallinity:

Degree of crystallinity ranges from completely amorphous to almost (up to about 95%) crystalline. The degree of crystallinity by weight determined from the following equation:

$$\% \text{ crystallinity} = \frac{(\rho_c (\rho_s - \rho_a))}{(\rho_s (\rho_c - \rho_a))} \times 100$$

ρ_s is the density of a specimen for which the percent crystallinity is to be determined, ρ_a is the density of the totally amorphous polymer, and ρ_c is the density of the perfectly crystalline polymer. Therefore, percent crystallinity of semicrystalline polymer depends on density, and densities of totally crystalline and totally amorphous materials[10].

I.2.2.2. Effect of degree of crystallinity on the physical properties of polymers:

Crystalline polymers are usually stronger and more resistant to dissolution and softening by heat. Moreover, the density of a crystalline polymer is greater than an amorphous one of the same material and molecular weight because the chains are more closely packed together for the crystalline structure[10].

I.2.2.3. Polymer crystals:

Polymer crystals are regularly shaped, thin platelets (or lamellae) approximately 10 to 20 nm thick, and 10 μm long which form a multilayered structure. The molecular chains within each platelet fold back and forth on themselves forming the chain-folded model[10].

I.2.2.4. Spherulite structure:

Bulk polymers that are crystallized from a melt as natural rubber are semicrystalline and form a spherulite structure. Spherulite is roughly spherical in shape and consists of an aggregate of ribbon-like chain-folded lamellae of 10 nm thickness that radiate outward from a single nucleation site in the center (in the 3 dimensions) [10]. At a microscopic scale, semi-crystalline polymers are heterogeneous materials consisting of co-existing amorphous and crystalline phases. The crystalline phase consists mainly of crystal lamellae. The lamellae are joined together by tie-chain. Stress in the polymer is transmitted between the two phases through tie-molecules [12].

I.2.3. Examination of polymer microstructure:

➤ Optical microscope:

Optical microscope could be used to study the structure of spherulite and to measure spherulite radius. Etching of the amorphous phase is performed to increase the contrast between the two phases. The contrast between the two phases makes possible the observation of the crystallite lamellae by optic or electronic microscope. The amorphous phase appears in dark while the light patterns are the crystallites [12].

➤ Raman spectroscopy:

Vibrational spectroscopy provides investigation of chemical composition of amorphous polymers, crystal content and molecular weight. It can perform spatially resolved measurements and it is important in situations that require local analysis. Moreover, there is no need for sample preparation. Raman spectroscopy generates distinctive vibrational signatures associated with polymer chains [11].

I.3. Structure of Ceramics:

Dental ceramics are nonmetallic, inorganic structures containing compounds of oxygen with one or more metallic elements (e.g., aluminum, lithium, magnesium, potassium, sodium, and zirconium). Many dental ceramics contain a crystal phase and a silicate glass matrix phase [2].

I.3.1. Silicate glass (matrix Phase):

Silicates are the basis of many ceramic materials and glasses. The silicon atom is tetravalent and forms bonds with four oxygen atoms in a tetrahedral structure [13]. Glasses are non-crystalline structures that based on silica and silicates; therefore, glasses contain SiO_4 tetrahedra. However, these tetrahedra are not assembled in an ordered and symmetrical manner. Glasses are formed by cooling from the fully molten state at a rate sufficiently rapid that crystallization is prevented [14].

Some oxides can form glass networks together with silica such as alumina, Al_2O_3 that can form a joint glass network with silica. Alkaline oxides such as CaO , Na_2O and K_2O are added to lower the melting temperature. These alkaline oxides are not capable of glass forming on their own as they

cannot form continuous networks. They are referred to as glass or network modifiers. The metal cations enter holes within the network [2, 14].

I.3.2. Crystalline Phases:

Leucite Crystalline Phase (KAlSi_2O_6 , $\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$):

Present in felspathic dental ceramics which contains leucite crystalline phase in amounts of 15- 25 vol % dispersed in glassy matrix. Moreover, it presents in Leucite based heat pressed ceramics in which leucite crystals (1 - 5 μm) present in the amounts changing from 35% - 55% by volume and dispersed in a glassy matrix.

Lithium Disilicate Crystalline Phase:

Lithium disilicate ($\text{Li}_2\text{Si}_2\text{O}_5$) is the major crystalline phase (65% by volume) in Lithium Disilicate based heat pressed ceramics. It is highly interlocking prismatic crystals (5.2 μm in length, 0.8 μm in diameter) dispersed in a glassy matrix with 1 vol% porosity.

Alumina crystalline phase:

Alumina-based core ceramics produced by pressing and sintering contain about 40 - 50 wt.% alumina with a mean grain size of about 4 micrometers. Alumina based ceramics produced by slip cast technique contain 68 vol % alumina crystals (0.5 - 3.5 μm) dispersed in glass phase (27 vol %) and porosity (5 vol %).

Zirconia Toughened Alumina (ZTA):

Zirconia-toughened alumina slip-cast ceramics contain 34 vol % alumina and 33 vol % of 12 mol % ceria-stabilized zirconia (12Ce-TZP). The glass phase represents 23 vol % with about 8 vol % porosity.

Zirconia Crystalline Phase:

Partially stabilized tetragonal zirconia consists of tetragonal zirconia polycrystals partially stabilized by addition of 3 mole percent yttrium (3Y-TZP). The microstructure of polycrystalline 3Y-TZP ceramics involved densely packed tetragonal zirconia grains with a grain size 0.2 - 0.7 μm [15].

II. Phase Equilibria:

II.1. Definitions:

Component: Components are the pure metals of which an alloy is composed. For example, Gold – Copper alloy, the components are Au and Cu.

System: The series of possible alloys consisting of the same components but without regard to alloy composition (e.g., the Gold –Copper system).

Phase: A homogeneous portion of a system that has uniform physical and chemical characteristics (pure material, solid, liquid, and gaseous phases).

Phase equilibria:

Equilibrium occurs when the system's free energy is at a minimum under specific temperature, pressure, and composition.

Free energy is:

Thermodynamic quantity

Function of the internal energy and the randomness or disorder of the atoms or molecules of a system.

If a change in temperature, pressure, and/or composition, an increase in the free energy will occur and a spontaneous change to another state by which the free energy is lowered [10].

II.2. Phase Diagram (equilibrium diagram):

II.2.1. One-component (Unary) phase diagram (Pressure–Temperature diagram):

The simplest and easiest type of phase diagram for a one-component system (phase diagram for a pure substance). The composition is held constant which means that pressure (vertical axis) and temperature (horizontal axis) are the variables.

Example: phase diagram for H₂O contains regions for three different phases; solid, liquid, and vapor. By crossing a boundary when temperature and/or pressure is changed, one phase transforms into another. Triple point is the point at which solid, liquid, and vapor phases are simultaneously in equilibrium with one another. Any deviation from this point by a change of temperature and/or pressure will cause at least one of the phases to disappear.

- Sublimation: is when the substance goes directly from solid to the gas state.
- Deposition: occurs when a substance goes from a gas state to a solid state; it is the reverse process of sublimation.
- Vaporization (or evaporation): is when a substance goes from a liquid to a gaseous state.
- Condensation: occurs when a substance goes from a gaseous to a liquid state, the reverse of vaporization.
- Melting: occurs when a substance goes from a solid to a liquid state.
- Freezing: is when a substance goes from a liquid to a solid state, the reverse of melting [10].

II.2.2. Binary phase diagram:

Binary phase diagram of binary alloys is a phase diagram in which temperature and composition are variable parameters and pressure is held constant at 1 atm. Binary phase diagrams represents the relationships between temperature (vertical axis) and the compositions (horizontal axis) and quantities of phases at equilibrium that influence the microstructure of an alloy. Binary system of known composition and temperature at equilibrium provides information about present phases, their compositions, and the percentages or fractions of the phases. Binary phase diagrams are helpful in predicting phase transformations and the resulting microstructures. Phase transformations occur when the temperature is altered (cooling) resulting in the transition from one phase to another or the appearance or disappearance of a phase [10].

II.2.2.1. Binary Isomorphous Systems:

Isomorphous means complete liquid and solid solubility of the two components. This complete solubility is explained by the fact that both elements have the same crystal structure, nearly identical atomic radii and electronegativities, and similar valences [16].

II.2.2.1.1. Development of Microstructure in Isomorphous Alloys:

- **Equilibrium Solidification:**

Equilibrium solidification occurs because of extremely slow cooling rates. As the temperature changes, readjustments in the compositions of the liquid and solid phases are accomplished by diffusional processes. To maintain equilibrium during cooling, sufficient time must be allowed at each temperature for the appropriate compositional readjustments [16].

- **Nonequilibrium Cooling:**

In practical solidification situations, cooling rates are much too rapid to allow compositional readjustments and maintenance of equilibrium leading to nonequilibrium solidification and formation of cored structure during solidification [16].

II.2.2.2. Binary Eutectic Phase Diagram:

In the phase diagram for the eutectic system, the components are not sufficiently soluble to form a complete series of solid solutions such as copper and silver, which are sufficiently different in atomic size that their atoms are only partially soluble in one another. As the liquid alloy solidifies, the solubility of copper in silver decreases, and three solid phases are formed: silver-rich (α - phase), copper-rich (β -phase) and silver-copper phase ($\alpha + \beta$) [1].

An important feature of the phase diagram of the Ag–Cu system is the depression of the temperature of the liquidus at a composition of 72% Ag: 28% Cu. At a temperature of 780°C, this composition of the alloy can exist as three phases: α , β and liquid. This is called the eutectic point, and the temperature at the intersection of the three phases is the eutectic temperature. The composition is called the eutectic composition of the alloy. If a eutectic liquid is cooled, it changes directly into two solid phases, without an interposing state as a liquid–solid mixture which occurs at all other

compositions. This feature of some alloy systems can be utilized to form low melting temperature materials, such as solders [1]. Alloys in eutectic systems with a composition less than that of the eutectic are called hypoeutectic alloys and those with a composition greater than the eutectic are known as hypereutectic alloys [2].

II.2.2.3. Equilibrium diagram of intermediate compounds:

The intermediate compound is formed between two metals resulting in the formation of crystalline material called an intermetallic compound. The intermetallic compounds should have a distinct chemical formula (stoichiometric) with fixed ratio of involved atoms. Stoichiometric intermetallic compounds appear as a single vertical line. However, in many cases a certain degree of atomic substitution takes place that results in the formation of nonstoichiometric compound. It appears as a range of composition [16].

II.2.3. Ternary phase diagram:

Ternary phase diagrams have been determined for metallic and ceramic systems containing more than two components. Ternary or three-component phase diagram is presented by a three-dimensional model. Compositions on ternary phase diagrams are usually constructed by using an equilateral triangle as a base with the pure component at each end of the triangle [10, 16].

II.2.4. Polymers phase diagram:

Many polymer products are multi-component mixtures of polymers or of polymers and other compounds such as solvents, monomers, and modifiers like plasticizers. In most cases, mixtures of polymers are not miscible, only few pairs of polymers are known to be miscible in a narrow temperature and concentration range.

- **Amorphous equilibrium in polymer systems:**

When mixing two partially miscible polymer liquids, a system decomposes on two liquid coexisting phases – saturated solutions of one component to another. Change of temperature results in change of components solubility and causes variation of coexisting phase compositions. At definite temperature, known as critical, boundaries between phases disappear and a system passes from hetero-phase (two phases) to homo-phase (one phase). The equilibrium values of coexisting phases compositions form unique binodal curve (The binodal line is the separation between the single- and two-phase region)[17].

Convex upwards binodal curve corresponds to the case of increasing miscibility with increase of temperature. Its top reflects the limiting temperature above which a system becomes single-phase at any concentration of components. This temperature is known as the Upper Critical Solubility Temperature (UCST). If miscibility of components increases with decrease of temperature, the binodal curve has a shape of convex downwards curve and critical temperature is named as Low Critical Solubility Temperature (LCST). LCST is the critical temperature below which the components of a mixture are miscible for all compositions. Some systems can have both critical

temperatures and their phase equilibriums are expressed by non-closed (UCST<LCST) diagram, sand-glass shape (UCMT~LCMT) diagram, or closed circle like (UCST>LCST) diagram.

- **Crystalline phase equilibria in polymer systems:**

Crystalline equilibrium occurs when at least one component is capable to crystallize. At cooling from the region of the total compatibility (isotropic solution) at definite temperature the system decomposes on two equilibrium phases: deposition of the crystalline component and its saturated solution into the other component [17].

II.3. The Gibbs phase rule:

A thermodynamics law that controls the conditions for phase equilibria. This rule represents the number of phases that coexist within a system at equilibrium

$$P + F = C + N$$

where P is the number of phases, F is the number of degrees of freedom or the number of externally controlled variables (temperature, pressure, composition), C is the number of components (elements/ compounds and the materials at the two extremes of the horizontal compositional axis in phase diagram), N in Equation is the number of non-compositional variables (e.g., temperature and pressure).

Examples:

In binary temperature–composition phase diagrams where pressure is constant (1 atm), thus, N =1 (temperature) and C= 2. In case of single-phase fields on the phase diagram, P will equal 1.

$$1 + F = 2 + 1$$

$$F = 2$$

This means that to completely describe the characteristics of any alloy that exists within one phase field, we must specify two parameters: composition and temperature. For binary systems, when three phases are present, there are no degrees of freedom because $F = 3 - P = 3 - 3 = 0$ This means that the compositions of all three phases and the temperature are fixed, e.g., eutectic isotherm [10, 16].

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