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RESEARCH ARTICLE

CHARACTERIZATION OF SOLIDUS STRUCTURES OF COMPOSITE MATERIALS.

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Abstract

Growth habits of composite materials from their respective molten states are established by scanning electron microscope and X-ray diffraction studies affirm composite alloys to be a terminal solidus solution of physically distinct and mechanical separable phases. Thermal stability of composite phases is ascertained using DSC. The solidification pattern of the individual constituent phases from the melt is comprehensively viewed and scanned after carefully studying the growth characteristics under scanning electron microscope (SEM). The microscopic observations reveal two types of growth habits of the homogeneous phases; one is edge-wise comprises of the round growth front lamellae distinctively termed nonfaceted lamellae and other is side-wise comprising of sharp growth front lamellae purposefully mentioned faceted lamellae. Evidentially, physical understanding of the single phase solidification predicts that the growth habits of combined constituent phases in binary, ternary and quaternary composite materials are discovered a composite of practically distinct and observable lamellae exploring three possible categories, namely nonfaceted-nonfaceted (nf-nf), faceted-faceted (f-f) and nonfaceted-faceted (nf-f).The classification of composite materials by growth of the lamellae of the homogenous phase in combination, verifies the J. D. Hunt and K. A. Jackson hypothesis.

Introduction:-

A new classification of composites is proposed, based on the entropies of melting of the two composite phases. The classification is used to predict suitable transparent analogs of the metallic systems. Experimental confirmation was obtained consistent with the theoretical shape of the lamellar solidus-liquidus interface, for the fault mechanism of lamellar spacing changes, and for the development of low-energy solidus-solidus boundaries between the lamellae. An explanation is presented to account for the irregular and complex regular structures which are found in some composite systems. From experimental observations, single-phase materials can be divided into two groups according to their solidification characteristics, those that grow as faceted crystals and those that do not. Jackson's showed from thermodynamic reasoning that the type of growth depended on a factor α which is almost the entropy of fusion in dimensionless units. Most non-metals have high entropies of fusion i.e. α greater than 2 and grow with crystalline facets with sharp growth front. Most metals have low entropies of fusion i.e. α less than 2 and grow almost isotropically with non facets implying with round growth front.

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The authors propose that multiphase composites may be classified in a similar manner. In binary phase composites there are three groups of composites, those in which constituent phases have low entropies of fusion ($\alpha < 2$), those in which one of the phases has high entropy of fusion ($\alpha > 2$) and the other phase with low entropy of fusion ($\alpha < 2$) and those in which constituent phases have high entropies of fusion ($\alpha > 2$). Exactly, the same hypothesis is adopted in the present work to classify multiphase composites. Lamellar or rodlike structures are formed in systems in which both phases have low entropies of melting. In these alloys dendrites of phases may be formed or when the composite alloy is rich in the relevant component. Examples are Pb-Sn, Sn-Cd, Pb-Cd, Sn-Zn, Al-Zn. Irregular or complex regular, structures are formed in alloys in which one phase has high entropy of fusion and the other phase has low entropy of fusion. Examples binary phase's composites are Al-Si, Pb-Bi, and Sn-Bi. When the alloys are rich in the low entropy of fusion phases, dendrites are formed; when the alloys are rich in the high entropy of fusion phases, faceted primary crystals are produced. These crystals are sometimes called hoppers or pseudo dendrites. In the present work, the term dendrite will only be used to describe nonfaceted primary crystals. Dendrites are not formed during solidification in high entropy of fusion single-phase materials. The third group of composite includes alloys in which the constituent phases have high entropies of fusion. Each phase grows with a faceted solidus-liquidus interface. Since most metals do not have high entropies of fusion, metallic examples in this composite group are rare. However, they may occur between some intermetallics, semiconductors or semimetals such as silicon, germanium, and bismuth. Metals and metal alloys may be strengthened and hardened by the uniform dispersion of several volume percent of fine particles of a very hard and inert material. The dispersed phase may be metallic or non-metallic and oxide materials are often used. Again, the strengthening mechanism involves interactions between the particles and dislocations within the matrix, as with precipitation hardening. The dispersion strengthening effect is not as pronounced as with precipitation hardening; however, the strengthening is retained at elevated temperature and for extended time periods because the dispersed particles are chosen to be unreactive with the matrix phase. For precipitation-hardened alloys, the increase in strength may disappear upon heat treatment as a consequence of precipitator growth or dissolution of the precipitator phase. The high-temperature strength of nickel alloys may be enhanced significantly by addition of about 3 vol% of thoria (ThO_2) as finely dispersed particles; this material is known as thoria-dispersed (TD) nickel. The same effect is produced in the aluminium-aluminium oxide system. A very thin and adherent alumina coating is caused to form on the surface of extremely small (0.1 to 0.2 μm thick) flakes of aluminium, which are dispersed within an aluminium metal matrix; this material is termed sintered aluminium powder (SAP).

Experimental:-

Materials and their thermal revelation:-

The composite alloys Al-Cd; Al-Sn; Al-Bi; Al-Pb; Al-Cd-Sn; Al-Cd-Bi and Al-Cd-Pb-Bi were prepared in the pyrex tubes by weighing variable amounts of purity 99.999% Al [Alfa Aesar, AR, mp 949 K, $\Delta_f H$ = 10.80 kJmol^{-1}], 99.999% Cd [99.5%, Alfa Aesar, AR, mp 597 K, $\Delta_f H$ = 6.20 kJmol^{-1}], 99.999% Sn [Alfa Aesar, AR, mp 509 K, $\Delta_f H$ = 7.30 kJmol^{-1}], 99.999% Bi (Alfa Aesar, AR, mp 551 K, $\Delta_f H$ = 11.20 kJmol^{-1}and 99.999% Pb[Alfa Aesar, AR, mp 584 K, $\Delta_f H$ = 4.70 kJmol^{-1}) shots with Al-Cd (60 wt % Al & 40 wt % Cd), Al-Sn (60 wt % Al & 40 wt % Sn), Al-Pb (60 wt % Al & 40 wt % Pb), Al-Bi (60 wt % Al & 40 wt % Bi), Al-Cd-Sn (40 wt % Al, 30 wt % Cd & 30 wt % Sn) , Al-Cd-Bi (40 wt % Al, 30 wt % Cd & 30 wt % Bi) and Al-Cd-Bi-Pb (40 wt % Al, 20 wt % Cd , 20 wt % Bi & 20 wt % Pb) . The melting temperatures and enthalpies of fusion of the constituent matels, cited in the parentheses, were obtained by thermal analysis approaching very closely to their literature values. The ampoule tubes were sealed under vacuum to avoid oxidation and subsequently infused in a furnace set at a temperature ~ 900 K for alloying Al, Cd, Sn, Bi and Pb metals. Homogeneity of the alloys were ensured by heat-chill process keeping the temperature of the heater (air oven) ~ 700 K and that of the cooler (water bath) ~300 K. The liquidus temperatures of the composite alloys Al-Cd (597 K), Al-Sn (505 K), Al-Bi (555 K), Al-Pb (602 K), Al-Cd-Sn (457 K), Al-Cd-Bi (429 K) and Al-Cd-Bi-Pb (370 K) were also ascertained by thermal analysis. The corresponding enthalpies of fusion ΔH_f and dimensionless entropies of fusion α, (where $\alpha = \xi\Delta S_f / R$, R is the gas constant which equal to 8.314 Jmol^{-1}K^{-1}, ξ a crystallographic factor which is slightly less than but almost unity and ΔS_f, the entropy of fusion) of the homogeneous materials are provided in the tabular form (Table 1). The computed dimensionless entropy α is found less than 2 ($\alpha > 2$) for Al, Cd, Sn and Pb metals which grow from their melt as non-faceted (nf) crystallites having round growth fronts, whereas α is found exceeding 2 for Bi metal that grows from its melt as faceted (f) crystals implying the crystallite with the sharp growth front.

Table 1:- Computed α values of homogeneous materials

Material	Melting point (T_m)K	ΔH_f (kJ mol^{-1}) Experiment Value	ΔH_f (kJ mol^{-1}) Literature Value	$\Delta S_f = \Delta H_f / T_m$ (Jmol^{-1}K^{-1})	$\alpha = \xi \frac{\Delta S_f}{R}$	Growth front
Al	949	10. 80	10.47	11.38	1.35	nf
Cd	597	6.20	6.19	10.39	1.25	nf
Sn	509	7.30	7.03	14.34	1.72	nf
Bi	551	11.20	11.30	20.33	2.45	f
Pb	584	4.70	4.80	8.05	0.97	nf

Oriented growth:-

Anisotropic solidification composite of the composite alloys and their constituent metals from respective molten state was achieved in the following experimental setup. An experimental sealed pyrex tube containing half-full melt of a freshly prepared composite or metal, was clamped to the centre of an empty graduated beaker (volume capacity ~1dm^3) manipulated midmost in an air oven set at a temperature 30K higher than the melting temperature of the samples. The molten mass in the tube was nucleated by circulating silicone oil at 18 different intervals spanned in the time range 50-60 min from the oil reservoir perforated and plugged with a glass tube carrying valve to control the percolation at ~300K. The melt in the tube started nucleating when the rising level of the oil just touched the bottom of the tube. Several samples of the composites and constituent metals were grown anistropically at different but nearly consistent growth rates determined by circulating approximately the same volume of the oil for the aforementioned intervals.

Instantaneous solidification:-

Isotropic growth was performed by immersing an experimental pyrex tube containing the composite or metal melt in an ice bath maintained at ~ 273 K. The growth being instantaneous in nature is presumed of zero order. Likewise, a good many samples of composite phases were solidified for the isotropic growth for observations.

Non Faceted- Non Faceted (nf-nf, regular) composite materials:-

Composite materials have low entropy of fusion ($\alpha < 2$).

Faceted-Faceted (f-f, irregular) composite materials:-

Composite materials have high entropy of fusion ($\alpha > 2$)

Non Faceted- Faceted (nf-f, complex regular) composite materials:-

Composites materials in which atleast one of the constituent phases with low entropy of fusion ($\alpha < 2$) or high entropy of fusion ($\alpha > 2$) in comparison to other phases

Simple lamellar or rod-type structures:-

Simple lamellar or rod-type structures are only produced in composites in which the constituent phases have low entropy of fusion ($\alpha < 2$).

Complex regular structures:-

Complex regular structures are obtained in composites wherein at least one of the constituent phases has entropy of fusion less than two ($\alpha< 2$) and others may have entropies of fusion greater than two ($\alpha>2$).

Coupled growth:-

Coupled growth occurs in composites phases having entropy of fusion less than two ($\alpha < 2$).

Dendrites:-

When the alloys are rich in the low entropy of fusion phases ($\alpha < 2$), dendrites are formed.

Pseudo dendrites:-

When the alloys are rich in the high entropy of fusion phases ($\alpha>2$), faceted crystal are produced. These crystals are called pseudo dendrites or hopper.

Microscopic studies:-

The specimen grown anisotropically and isotropically were polished at room temperature following a procedure similar to that adopted for analogous problem to reveal the microstructure, a thin layer of the specimen etched in ferric chloride was mounted on stub with gold-coated holder and examined under a scanning electron microscope for micro growth observation. Many samples of each specimen were viewed in this manner and the growth habits of the growing phases during solidification at different growth rates were accordingly photographed.

X-ray diffraction studies:-

The X-ray diffraction patterns exhibited by the experimental composite phases were recorded with Diffraction System-XPERT=PRO using Cuk$_\alpha$ radiation of wavelength 1.5408 Å at room temperature. The powder X-ray was recorded in a 2θ range of 5° to 90° with the step size of 0.05 and step time of 1s.

Results and Discussion:-

Lamellar growth:-

In the present work, the growth of composite phases from the melt strictly follows the hypothesis proposed by J. D. Hunt and K. A. Jackson. An individual material having low entropy of fusion ($\alpha < 2$) grows almost with isotropically. For example, pure aluminium metal has low entropy of fusion, $\alpha = 1.35$ implying less than 2, grows from its melt as nonfaceted crystallites (Fig. 1). On the contrary, bismuth metal has high entropy of fusion, $\alpha = 2.45$ obviously greater than 2, grows form its melt with crystalline facets (Fig. 2). As is evident from the following microstructures, lamellar or rod like structures are formed in composites in which constituent phases have low entropies of fusion ($\alpha < 2$). The composite Al-Cd-Sn is an example of this structure (Fig. 5e), the constituent phases having entropies of fusion, pure Al ($\alpha = 1.35$), pure Cd ($\alpha = 1.25$) and pure Sn ($\alpha = 1.72$). The lamellar structures proves to be less regular compare to rod type growth which does have a perfect ordered structure, particularly interrod spacing, whereas rod like lamellar structure is the intermediate case. Periodic growth of the composite phases tends to provide rod type growth wherein the values of the microstructure parameters, namely, lamella diameter, lamella length, lamella length distribution, volume fraction of lamellae, and the alignment and packing arrangement of lamellae, are repeating themselves exactly after each crystallographic orientation between the lamellae to generate symmetric relation which governs the consistency of the crystallographic orientation relationship in the microstructure entirety.

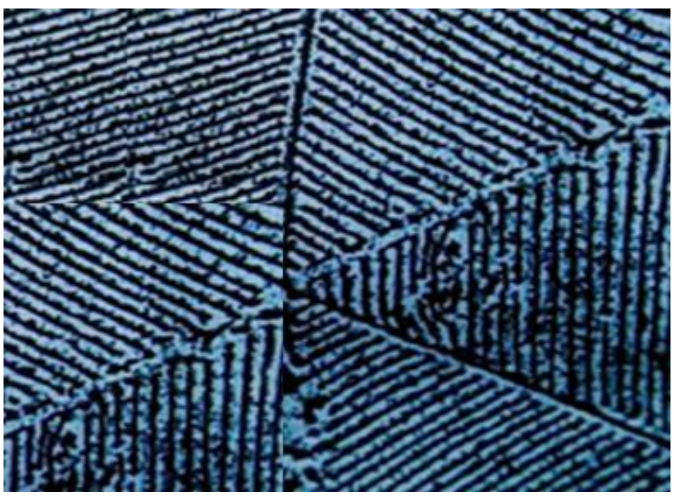


Fig.1:- Microstructure of pure Al metal (1500 x)

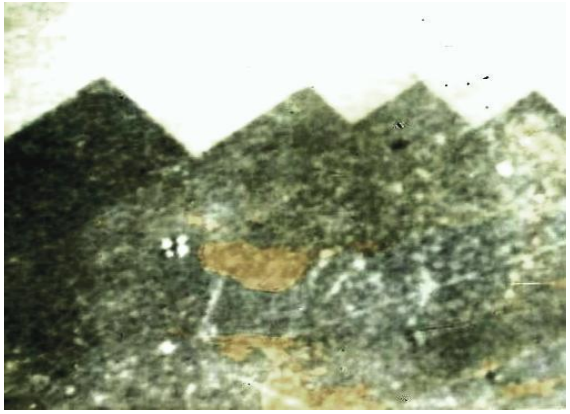
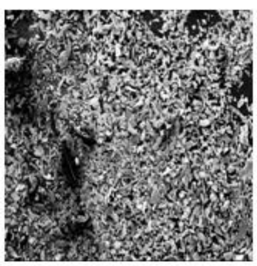


Fig. 2:- Microstructure of pure Bi metal (1500 x).

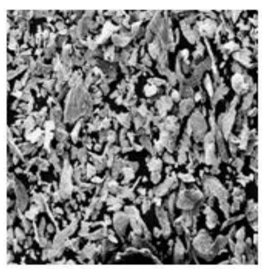
(a) Al-Cd Alloy (1500 x)

(b) Al-Sn Alloy (1500 x)

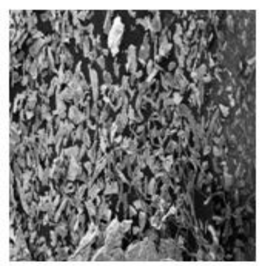
(c) Al-Pb Alloy (1500 x)



(d) Al-Bi Alloy (1500 x)



(e) Al-Cd-Sn Alloy (1500 x)



(f) Al-Cd-Bi Alloy (1500 x)

(g) Al-Cd-Bi-Pb Alloy (1500 x)

Fig. 3:- Distorted microstructure of composite phases in ice bath at (~273) K (a) Microstructure of Al-Cd (b) Microstructure of Al-Sn (c) Microstructure of Al-Pb (d) Microstructure of Al-Bi (e) Microstructure of Al-Cd-Sn (f) Microstructure of Al-Cd-Bi (g) Microstructure of Al-Cd-Bi-Pb.

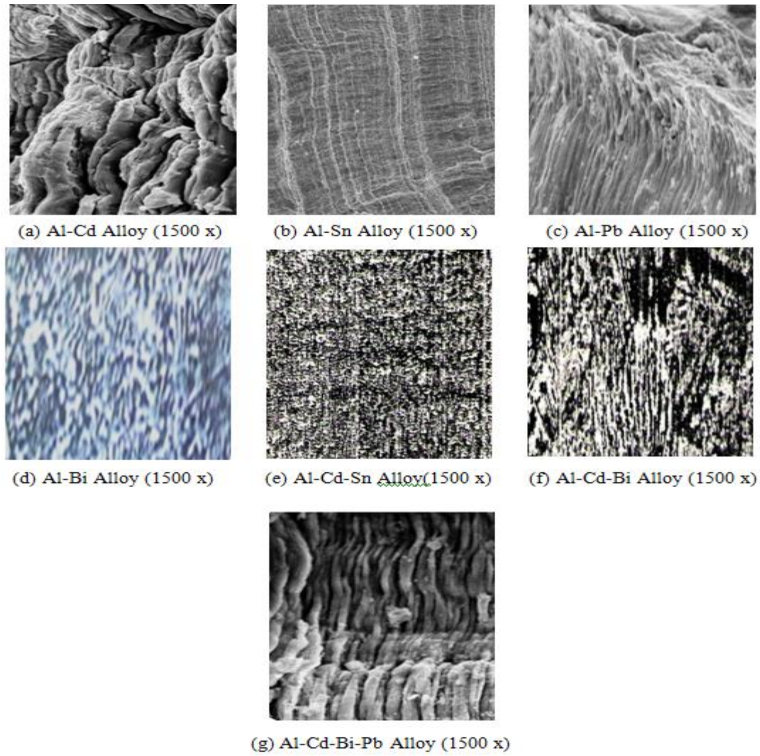


Fig. 4:- Lamellae of composite phases lamellae in growth direction from bottom to top at fast growth velocity 8.10 x $10^{-7}m^3s^{-1}$ (a) Microstructure of Al-Cd (b) Microstructure of Al-Sn (c) Microstructure of Al-Pb (d) Microstructure of Al-Bi (e) Microstructure of Al-Cd-Sn (f) Microstructure of Al-Cd-Bi (g) Microstructure of Al-Cd-Bi-Pb

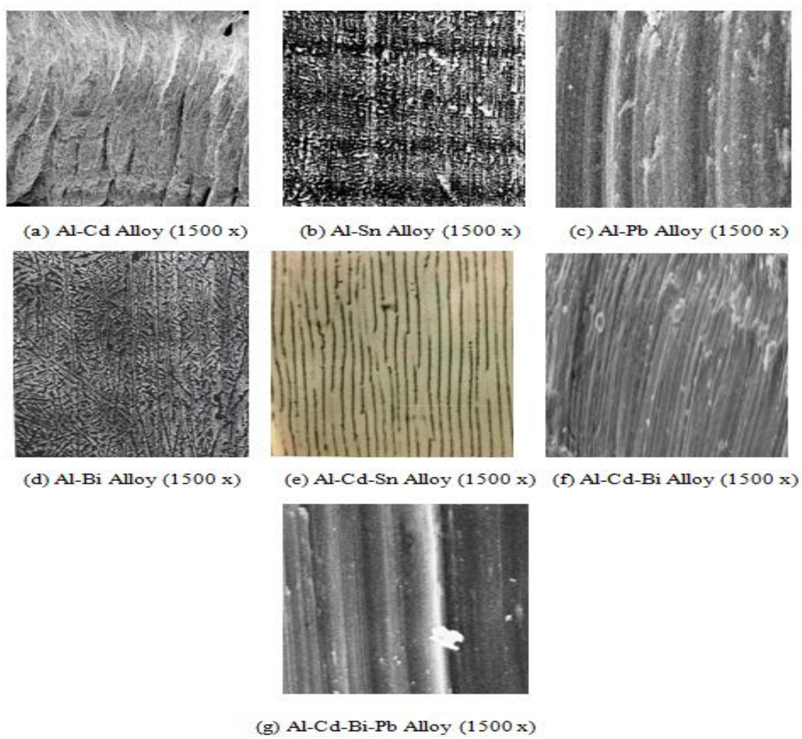


Fig. 5:- Lamellae of composite phases in the growth direction from bottom to top at slow growth velocity 1.71 x $10^{-7}m^3s^{-1}$ (a) Microstructure of Al-Cd (b) Microstructure of Al-Sn (c) Microstructure of Al-Pb (d) Microstructure of Al-Bi (e) Microstructure of Al-Cd-Sn (f) Microstructure of Al-Cd-Bi (g) Microstructure of Al-Cd-Bi-Pb

Complex regular structures are obtained in composites wherein at least one of the constituent phases has entropy of fusion less than two ($\alpha < 2$) and others may have entropies of fusion greater than two ($\alpha > 2$). The composite Al-Cd-Bi is an example of this structure (Fig. 5f). It may be pertinent to mention here that phases with $\alpha < 2$ always grow in contact called coupled growth which does not occur in composite phases having dimensionless entropy of fusion greater than two ($\alpha > 2$). The ingot microstructure of the composite phases, namely Al-Cd, Al-Sn, Al-Pb, Al-Bi, Al-Cd-Sn, Al-Cd-Bi and Al-Cd-Bi-Pb obtained by different modes of growth are presented in Figs. 3-5. The changes in the movement of the lamellae procedure corresponding changes in the mechanical properties which are made comprehensive in term of the approach adopted in the present work. Nevertheless, the orientation of the lamellae offers the possibilities of achieving optimum final products performance by controlling the microstructure parameters which do strengthen the matrix of the resulting lamina to exhibit maximum load bearing capability. Figure 3(a-g) reveals the distorted growth habits of the composite phases, namely, (a) Al-Cd; (b) Al-Sn; (c) Al-Pb; (d) Al-Bi; (e) Al-Cd-Sn; (f) Al-Cd-Bi and (g) Al-Cd-Bi-Pb experienced in an ice bath (~ 273 K) that at a large kinetic undercooling the lamellae formed are of short size, aggressive, disconnected, crossing each other and showing no matrix relationship. These lamella habits, in fact, arise from splitting of the main single lamella into separate single lamellae or group of single lamellae, apparently leading to the distorted morphology. However, the growth habits of the composite phases gradually structure themselves to nonaggressive, attaching and parallel to each other reinforcing the matrix with decreasing kinetic undercooling as is evident from Fig. 4 (a-g). An entirely distinct lamellar microstructures of the composite phases Fig. 5 (a-g) are obtained at the anisotropic growth velocity (~ $1.71 \times 10^{-7} m^3 s^{-1}$) determined by setting the flow-interval of silicone oil at $5 \times 10^{-4} m^3$ for 30 min, which enhances the hardness of the alloys to their optimum value in the present investigation. In the lamellar microstructure, some of the unfavorably oriented lamellae in the composite grains with high configuration energy grew out perpendicularly to the solidus-liquidus interface leaving other lamellae with orientation close to low configuration energy to grow in an aligned preferred crystallographic morphology. The growth habits of the composite phases comprising the rod-like lamellar microstructure from the melt present their relationship with the moderate anisotropic growth velocity, In a binary melt when one of the composite phases, usually the rich one, grows, the vicinal melt region acquires richness in the other phase, the first phase continues its growth as a lamella or a whisker unless and until the other phase nucleates at a certain super saturation. This is another supercrescent lamella growing over that of the first phase, which would also continue its growth till another supercrescent lamella of the first phase appears on it. Thus, the supercrescent lamellae growth of the composite phases from the melt produces a complete lamellar microstructure. The growth front contacting solidus-liquidus interface structure inside the experimental tube, and the rising level of silicone oil outside the tube would move upward nearly with the same pace in a single phase, effectively decreasing the kinetic undercooling which balances the undercooling due to composition, and originating lamella length. Likewise, the growth of composite phases from the ternary and quaternary melts would follow the similar pattern. Accordingly, the growth process produces long lamellae with undamaged surfaces Fig. 5 (a-g) which are embedded parallel to each other in attaching and nonaggressive unidirectional lamiae. The mathematical model for the rod-like growth of the composite systems has been developed elsewhere. In the directional growth, the metals Al, Cd, Sn, Bi and Pb grow as lamellar cells where each cell crystallize either from the bulk of the melt or through secondary nucleation. Since the complete lamella is an attachment of two- , three and four nonaggressive ductile metal lamellae, the efficiency of the lamellae in stiffening and reinforcing the matrix decrease as the lamella length decrease. Lamella ends play an important role in the fracture of short lamella composites (Fig.6) and also in continuous lamella composites (Fig. 7), since the long lamellae may break down into discrete lengths. As mention earlier the lamellae are aggressive, non attaching and irregular thin crystals producing distorted microstructures representing Fig. 3(a-g) which attain fragile matrix of the composite phases. The discontinuous change in spacing in absence of faults is the movement of lamellar-faults, which is an evidence for the fault-mechanism. It is this movement, not the formation of faults, which is an important factor in controlling the spacing among lamellae. The lamellar cells of the pure composite phases would acquire dislocations by virtue of their growth by fault-mechanism from the melt and consequently, exhibit fragile matrix.

Fig.6:- Short Lamellae (1500 x).

Fig. 7:- Long Lamellae (1500 x)

Conclusion:-

The thermal analysis reveals thermal stability and liquidus temperatures of the composites. X-ray diffraction patterns implicitly demonstrate that the composites are terminal solidus solutions comprising of physically distinct and mechanical separable materials because no unique X-ray pattern is observed in the composites other than constituent metals. The growth of composite phases expresses its obedience to the hypothesis proposed by J.D. Hunt and K.A. Jackson that single phase (homogeneous) materials can be divided into two groups according to their solidification characteristics, those that grow as faceted crystals and those that grow as nonfaceted crystals. The homogeneous materials having entropies of fusion less than two ($\alpha < 2$) do grow as nonfaceted crystals marked with regular structures, and those having entropies of fusion greater than two ($\alpha > 2$) do grow with crystalline facets projecting irregular structures. The morphology pattern of the composite alloys may also follow the microstructure classification of the single phase material. The composite alloys that those in which constituent phases have low entropy of fusion ($\alpha < 2$) ; those which comprise constituent phases having high entropies of fusion ($\alpha > 2$) and those in which any constituent phase is inconsistent to the other constituent phases whether low entropy of fusion ($\alpha < 2$) or high entropy of fusion ($\alpha > 2$) . The diversity of the micro morphologies, because of the complex nature of the phenomenon involved in an anisotropically controlled solidification from the melt, is revealed by examining the crystalline specimens in a scanning electron microscope.

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