

THE SUPRAMOLECULAR STRUCTURES ON THE BASE OF $A^{IV} B^{VI} - Sb$ (NiSb) EUTECTIC SYSTEMS

K.Sh. KAGRAMANOV, S.Sh. KAGRAMANOV

Scientific-industrial community "Selen" of National Academy of Sciences of Azerbaijan Republic, Baku, Azerbaijan

E.D. MOROUDOR, M.G. PISHKIN

Stambul Technical University "Yildiz", technical department, Stambul, Turkey

The processes of crystallization and morphology peculiarities of microstructure $A^{IV} B^{VI} - Sb$ (NiSb) eutectic are evidence in favor of supramolecular conception with taking into account of substructure interactions.

The anomalous structures are revealed on the obtained direct-oriented (PbTe - Sb) and (PbS - Sb) eutectics, and the regular plane-fibrous structures are revealed on the (PbS - NiSb) and (PbTe - NiSb) eutectics.

The principle of the orientational and dimensional correspondence keeps in (PbS - NiSb) eutectic also as in the normal binary eutectic systems, the plane (111) PbS accretes with plane (0001) NiSb.

The morphology analysis of (PbS - NiSb) eutectic showed the presence of the different defects of types: plate shifting, surfaces of convergence, block (plate) inclinations and excess extra-plate. These defects are analogous to atom defects, obtaining in real one-phase crystals.

The investigations showed that eutectics can be considered as the supramolecular layered ensembles.

Introduction

The eutectic is the special melt (solution), the components of which restrictedly mix in solid and liquid (in some temperature interval) states and spontaneously form the thermodynamic equilibrium liophilic disperse system in this interval. The given disperse system is characterized by significant interphase interaction and salvation.

The main statements of eutectic theory have been analyzed in the ref [1] and the view of the more significant results, obtained at the investigation of eutectic melts of inorganic substances, is given. The evidences in the favor of supramolecular eutectic conception, taking into account the interaction ("non-autonomous phases") are presented. The many direct experimental provements of "interacting phases" conception exist.

The ability of the self-organizing supramolecular objects to the molecular recognition is their main property. The component recognition proposes the "complementarity", i.e., the compatibility of ensemble participants (substratum and receptor) – as geometrical one, so the level of the formation of intermolecular connections [2].

The theoretic analysis proves the possibility of suprastructure self-organizing from the structures, disproportionate in the one crystallographic direction. The consideration of eutectic compositions, in the limit case disproportionate on all three crystallographic directions, in terms of supramolecular chemistry allows us to obtain the answers on many questions and to escape the vaguenesses, mentioned at the discussion of existing eutectic conceptions [1]. The proposed supramolecular conception in eutectic theory [1] doesn't reject the classic conceptions. It differs from them only by the fact, that abstract thermodynamic criterias, such as the surface energy or surface tension are changed by real parameters of interacting substructures: disproportion, hardness, interaction potential.

The given experimental data can be evidence in the proof of supramolecular eutectic conception. The statement about the fact, that interaction of these phases is need to consider in the eutectic melt, consisting the autonomous phases A and B is the base of this conception. The forming of the

nonautonomous phases, similar to the supramolecular ensembles is the result of such interaction. The conditions of the formation of defect composite-nonautonomous phases with changed lattice parameters can be created in the dependence on the interaction potential, disproportion of crystal lattice of suprastructures A and B, their harshness and extension, external mechanic influences. The special properties of eutectic melts, the dependence of their structures on the synthesis conditions (temperature, cooling velocity, mechanical interactions) are defined by the quantity of nonautonomous phase and the degree of it organization [1].

Many works are dedicated to the investigation of the morphology and crystallization peculiarities, physicochemical properties of eutectics [1,3-9]. They reveal the peculiarities in their microstructure. It was the base for the eutectic classification. The classification, on which all eutectics are divided on normal and anomalous ones, was separated.

Normal eutectics have mainly plate or fibrous (rod) microstructure. The definite crystallographic ratios, character for the given melt, should exist in normal eutectic between the phases. The anomalous eutectics form in only that case, when eutectic phases can't increase with the similar velocities.

Eutectic crystallization "is carried out by the way of the creation" and increase of colonies of two-phase formation, the each of them forms on the base of one center [10-11].

On authors' opinion the oriented crystallization, as in eutectic, so at the artificial crystallization of the one crystal on the substrate of another one, is possible only at the condition, that both materials already naturally have some geometric similar crystallographic atom complexes, well mating with each other [5-6]. Authors of the refs [5-6, 12-13], showing on the observance dimensional and oriented correspondence between the phases in eutectic, draw an analogy between atom interaction on the interphase boundary in eutectic melt and this interaction, which is realized at the artificial epitaxial growing of the one material on the substrate of the another one. However, the results of X-ray

and electron-microscopic investigations of eutectic set showed that such analogy can't be drawn. The electron-microscopic investigations of directed-oriented eutectics reveal the presence dislocation variance on the phase boundaries, decreasing the coherent deformations, caused by the convergence of phase lattices on the coupling boundary. As the investigations showed, the dislocation density of the variance on the interphase boundaries in eutectics is defined by the value of the convergence of lattice parameters of mating phases [3]. The dislocation forming in double-layer film structures, obtained by epitaxial growing, is caused not only by variance of crystal lattices, but mainly by interdiffusion [14].

The results of X-ray investigation of the thin structure of eutectic system Si - ZrSi₂ [14], obtained by the directed crystallization, showed that in eutectic the dislocation density of variance decreases exponentially into grain depth, and it is maximal on the interphase surface in the difference from the double-layer film structures, in which the dislocation density, caused by interdiffusion, is maximal in the grain center and decreases to the boundary side. As it was established in this paper, the lattice of Si matrix is pressed, and lattice of ZrSi₂ fibres is extended in the result of the variance of crystal lattices in the direction, which is parallel to the increase axis of Si - ZrSi₂ eutectic system. The given deformations are elastic as shows the selective etching. The lattice inclinations in the transverse direction are absent, that is the interest fact. The above mentioned fact is the important step in the statement of the fact that eutectic structures radically differ from the structures of another crystals.

It can be supposed, that eutectic is the system, which is characterized by the unite energy electron spectrum. The results of the refs [13-15] are the some statements of this supposition. So the authors of the ref [15], studying the possible interphase interaction in SnTe - NiSb by the method of nucleus-gamma resonance, have revealed, that Lorenz curve of tin atoms in this eutectic is asymmetric in comparison of one in SnTe, that is revealed in line broadening to the side of the low velocities. This asymmetry was explained by the authors [15] by the fact, that eutectic spectrum presented itself the superposition of two spectrums of different intensity. The spectrum of bigger intensity corresponds to the tin atoms in SnTe, and the spectrum of low intensity corresponds to tin atoms, situated on the boundaries with NiSb phase.

The analysis of literature data allows us to make the conclusion, that the some chemical interaction takes place in eutectics on the interphase boundaries. However, the electron structure of eutectics and reasons of its creation haven't clarified yet.

The given paper is dedicated to the study of the peculiarities of morphology microstructure, physicochemical properties of eutectic melts A^{IV} B^{VI} – Sb (NiSb) with the aim of the revealing of interphase interaction nature, proving in the proof of the supramolecular conception.

Investigation technique

The eutectic melts of the following systems: PbS(PbTe)-Sb, PbS(PbTe)-NiSb have been grown by Bridgman method at the velocity $v=3$ mm/h and temperature gradient $\Delta T=100^\circ\text{C}$ between the heaters. The purity of the initial materials: Sb, Pb, S, Te was not less, than 99,999%.

The part of the microstructures on MIM-7 microscope, on raster electron microscope (REM) JSM-50A with the prefix for the local X-ray analysis and on electron microscope JSM-2000 in the beams of the secondary electron mission and in the beams of the transmission electrons, X-ray images had been investigated in Te, S, Ni, Sb beams.

The diffractograms of PbS-NiSb eutectic are obtained on DRON-2 installation. The photos are obtained at the beam directions, parallel to the direction of the axes of crystal growth and perpendicular to the surface plane of PbS and NiSb in eutectic. The diffractograms of eutectic powder PbS-NiSb were investigated for the comparison.

The lines of the characteristic X-ray radiation for the one beforehand chosen element (permission method 1000-2000 nm) are registered; white elements on the photos are more hard. For example, regions PbTe on NiSb phone are looked like white. The microstructures of the anisotropic eutectic PbTe-NiSb, obtained on REM give the possibility to see the image of the investigated surface simultaneously as in electron, so in X-ray beams. The images in electron and X-ray beams are turned out interest not only by the fact, that they have best permission in principle, than the ones, obtained with the help of the metallographic microscope, but by only the fact, that they give the possibility to observe the boundary between the phases and to judge about the element distribution along sample surface.

The result discussion

The regular ordered microstructures are character to the eutectic melts (fig. 1-2). The high strength and superplasticity of the eutectics say about the special structure of interphase boundaries. All these peculiarities and many other properties differ eutectics from the homogeneous melts and from the mechanical ones and are evidence in the proof of the supramolecular eutectic conception.

The previous investigations showed that the structure in PbTe-NiSb systems is the one of the same type. Let's consider the crystallization in PbTe-NiSb and PbS-NiSb systems for the distinctness. The PbTe-NiSb eutectic (fig.1 (b)) has the fiber structure and PbS-NiSb has the plate structure (fig.2 (1-4)). The eutectics of (PbS-Sb) PbTe-Sb systems can be related to the anomalous, limited bar ones with the petalled structure of the rods and planes. The investigated eutectic PbS-NiSb (fig.2) can be related to the normal, i.e. to the "regular" ones on the form and phase distribution. Each substructure grows with the one strictly drawn crystallization front, clearly revealing the contact surface of subblocks. The defined crystallographic ratios, character for the given systems exist in the normal microstructures, investigated by us, between the phases which aren't observed in anomalous eutectics.

The element distribution in PbTe-NiSb eutectic, obtained by the local; X-ray analysis between two grains is given on the fig.3. As it is seen from the fig.3 the distribution of Pb, Te, Ni and Sb elements between the phases is clear; in the inclusion region the signal falling from Pb and Te and the increase of the signals from Ni and Sb take place. The insignificant distribution of Ni and Sb in NiSb region is paid attention. The results of the local X-ray analysis of the directed-oriented eutectic of PbTe-NiSb system are given here. According to them Ni and Sb, happen to the needles of the dispersed phase, coincide with minimal content of Pb and Te.

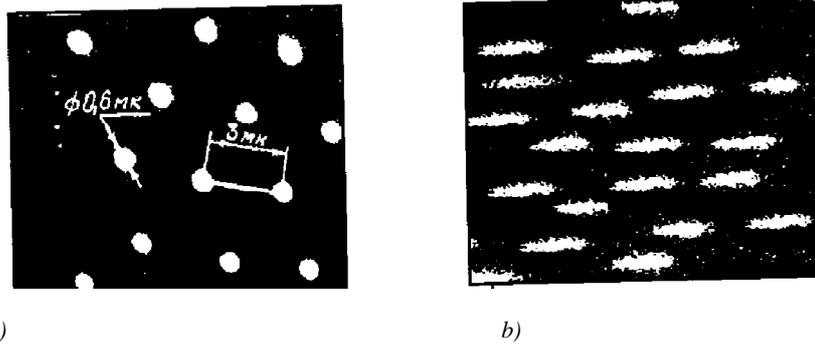


Fig.1. Microstructure of transversal (a) and longitudinal (b) section of the directed-oriented PbTe-NiSb eutectic.

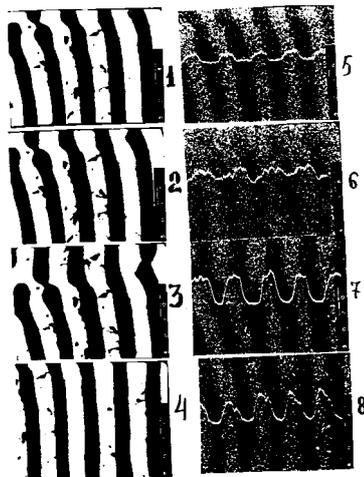


Fig.2. The photos of microstructure and local X-ray analysis of longitudinal section of the oriented eutectic PbS-NiSb ($\times 300$), obtained on JSM-50A: 1,2,3,4 – in Sotro mode; 5-8 are X-ray images in the characteristic beams (5 –Ni, 6-Pb, 7-Sb, 8-S) towards with linear distribution of Pb, S, Sb and Ni.

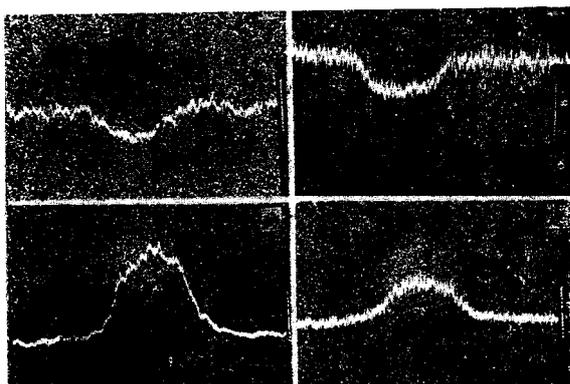


Fig.3. The figures of the local X-ray analysis of the transversal section of the oriented eutectic PbTe - NiSb: a) – X-ray images in characteristic Sb beams with linear distribution ($\times 14000$); b) - the same in Ni beams towards with Ni linear distribution ($\times 14000$); c) - the same in Pb beams ($\times 15000$); d) - the same in Te beams towards with Te linear distribution ($\times 15000$).

The images, obtained in characteristic beams of all four atoms, show that as the matrix, so the inclusions present themselves the suprastructures, consisting from the solid

solutions. The data of local X-ray analysis is the total agreement with the state diagram of PbTe-NiSb system [16]. The same pictures of Pb, S, Ni, Sb element distribution in PbS-NiSb eutectic are presented on the fig.2, (5-8). The alternations of plates of the solid solutions on the base of the lead sulphide and planes of solid solutions on the base of nickel antimonide are clearly seen on the figure 2. Each phase in the system, being the independent subblock, belongs to the united eutectic supramolecular ensemble.

Let's consider the mechanism of the establishment of the oriented ratios in these systems (PbS-NiSb, PbTe-NiSb).

The length of the fibers in eutectic colony of PbTe-NiSb system varies in the interval from 200 nm till 7000 nm at the diameter 160-180 nm. The fibers have cigar-shaped form with the thickening on the one end and needle-shaped bevel on another one (fig. 1 (b)).

It can be said, that the obtaining of such eutectic supramolecular ensembles with regular structure (fig.1 and fig.2) is the enough complex technological task, which not always can be realized even on the one and the same composition on these or that reasons. Thus, the high concentration of convergence defects (mentioned by circle (A) fig.4) and terminations (mentioned by pointer (B), caused by the fact, that phase orientation in this case doesn't coincide with more profit one, is visually seen on transversal section of PbS-NiSb eutectic. The plates turn relatively each other round growth axis on small angles, mating moreover between each other by more successful form for the realization of minimum of interphase energy. The PbS and NiSb mate more successful in bend phase region; the plate shifting relatively each other increases as far as the region widening. The further widening in bend region leads to the plate break with the formation of convergence surfaces (fig.4). The convergence surfaces in energy meaning are comfortable places for termination creations. Though all details of creation and growth of plates of PbS and NiSb are hard to imagine, the profitable orientation relatively to neighbor plates, which endure the bend by the way of formation of subboundaries with off-orientation, causes to quick establishment of favorable crystallographic connections in eutectic.

The PbS-NiSb plates are continuous and parallel in the limits of small regions in eutectics. The excess plane of the one phase is present on the one of the sides. This extra-plate is analogous to atom extra-plane, connected with edge dislocation like the edge of atom half-plane forms the line

defect in space, which is called end substructure defect or termination [10].



Fig.4. The longitudinal section of PbS-NiSb eutectic.

The convergence surfaces are connected with terminations in plate eutectics. These convergence surfaces are situated perpendicular to the plates. Besides the regions with defects and the perfect regions, where the termination is absent, are present.

Thus, the defect density is defined not by crystallization conditions, but by crystallographic factor. The orientation, favorable for the growth of perfect phases, appears in creation process. The subblocks (PbS and NiSb plates) grow perpendicular to crystallization front in conditions of stationary growth, so that contact plane of plates necessarily has the growth direction. As PbS-NiSb has the plate structure, then the condition of regular accretion of phases is the more obvious, and between accreting planes of lead sulphide and nickel antimonide the orientation ratios should exist. It is naturally, that the disparities of mating lattices on the period of crystal lattice and their mutual shifting appear at the mating of eutectic phases. The morphology analysis of subblocks of PbS-NiSb eutectic shows the presence of defect set of termination type, plate shifts, convergence surfaces and their contortions. The studied morphology and carried out analogies show that the big defect number of crystal structure can participate in the forming of interphase boundaries of suprastructure eutectic melts.

The order distribution of crystal directions in sample space is well demonstrated with diffractograms, given on the fig. 5 (a,b,c). The diffractograms are taken at the beam directions, parallel (a) to direction of crystal growth and perpendicular (b) to PbS and NiSb plates, and also in the case of use of eutectic powder PbS-NiSb (c). The obtained photos show on the fact, that the orientation ratio between blocks of eutectic melt, that can be caused by atom distribution along planes (0001) in NiSb and (111) in PbS is present.

It is obvious, that especially this correspondence between the structures of concerning verges is the reason of the creation of layered eutectic suprastructure.

The atom interaction of eutectic superstructures is direct physical reason, which causes the crystal accretions of these

phases on the defined directions, more energetically profitable ones and supplies the high stability of interphase boundary.

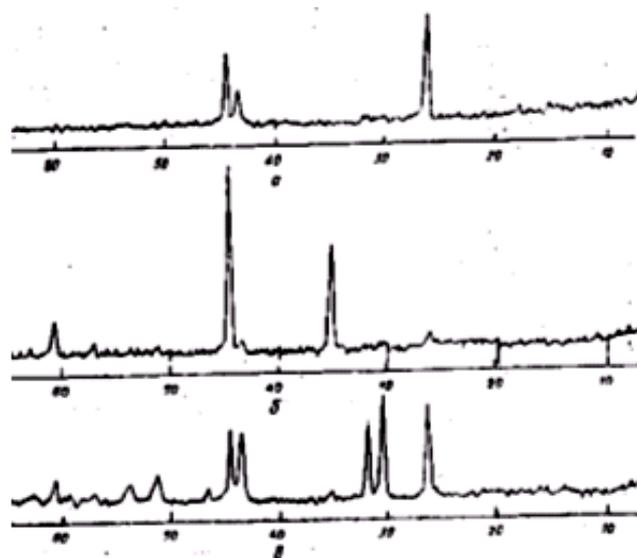


Fig.5. The diffractograms, taken on the beam directions, parallel (a) to direction of crystal growth and perpendicular (b) to plane of plate surfaces PbS and NiSb, and also diffractogram of eutectic powder (PbS-NiSb) – (c).

The investigations of possible orientation ratios in system eutectics by $A^{IV}B^{VI}$ -NiSb type, carried out on the samples, directly grown eutectic of PbS-NiSb system showed, that plane (111) PbS accretes with plane (0001) NiSb in such way, that monatomic stibium layer is situated under the monatomic sulfur layer on the interphase boundary (fig.6). The lead chalcogenides and also SnTe have cubic lattices by NaCl type, differing from each other by the dimensions of elementary cell that is given in table. That's why the analogy to PbS-NiSb eutectic system it can be supposed, that such deorientation correspondence should take place between the phases in eutectics of PbSe(Te)-NiSb and SnTe-NiSb systems, i.e. the monatomic Sb layer should be situated under chalcogen monatomic layer in all eutectics of systems by $A^{IV}B^{VI}$ -NiSb type.

The distances between the chalcogen atoms in direction (111) (a'), (table) had been calculated on the base of such supposition for semiconductor phases of all eutectics. The distance between Sb and NiSb atoms coincides with a parameter of elementary cell.

The circuit of interphase boundary of blocks eutectic of PbS-NiSb system in plane, perpendicular to plane of phase accreting, is given on the fig.6. The sulfur and stibium atoms are striving for the interaction and development in eutectic of high voltages. The crystal lattice of lead sulphide on boundary is situated under the influence of compression stress and crystal lattice of NiSb is situated under the influence of tensile one. The difference of parameters of phases of crystal lattices on coupling boundary (a' and a) at transfer from PbS to PbTe increases. As more this difference, the more voltage should develop on interphase boundary and the more influence should provide the creation of near boundary interaction on the connection weakening between atoms inside each eutectic phases. This supposition is well

evidenced by corresponding decrease of melting point of eutectics in the set of PbS(Se,Te) – NiSb system (table).

Table
The phase parameters, constituting the eutectics of systems by $A^{IV} B^{VI} - NiSb$ type

The eutectic composition, mol %	T°K	α , Å	α' , Å	$\Delta\alpha$, %
46 PbS	1420	5,93	4,19	6,35
54 NiSb		3,94	3,94	
68 PbSe	1170	6,147	4,44	12,7
32 NiSb		3,94	3,94	
92 PbTe	1143	6,45	4,49	13,9
8 NiSb		3,94	3,94	

Notion: $\Delta\alpha$ is difference between α' in lead chalcogenide and α in nickel antimonide.

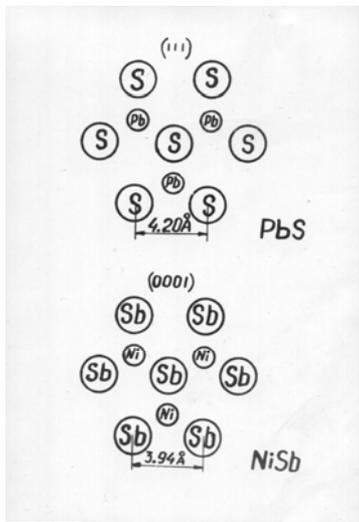


Fig.6. The circuit of atom situation on interphase boundary (111) of PbS and (0001) of NiSb planes.

The microstructure regularity should reflect on the course of periodic potential of all structure eutectic elements in total, in particular: periodic potential of one lattice should be changed by periodic potential of another lattice with some constant period for given eutectic and it is bigger than constant of lattice of each phase. The width of PbS plate is ~1800 nm, and NiSb is ~2500 nm have been considered on results of microstructure investigation, in the result of which the structure period of this eutectic is ~4300 nm (fig.1-4).

The supposition, that disparity compensation of elementary cell parameters on the boundary of phase couplings should be carried out not only by the way of the formation of dislocation grids on this boundary, but by some lattice stretching of one phase and pressing of another one, that is proved experimentally in the ref [14]. The elastic deformation of lattices is maximal one on the boundary and exponentially decreases in phase depth. Along with it, the connection weakening between atoms in near boundary region inside the phases should cause the decrease of order in the given region. That's why potential periodicity inside

supramolecular formations in eutectic in the relation to that, which has the place in crystal of initial component, will significantly change.

According to above mentioned data, at eutectic crystallization of PbS-NiSb system, the (111) plane of one of its phase (97 mol.% PbS+3 mol.% NiSb) accretes with (001) of another phase (99 mol.% NiSb+1 mol.% PbS) in such way, that monatomic stibium layer is situated under monatomic sulfur layer (fig.6). In this connection the distance between Pb and S atoms is 4,2Å, between Sb atoms the distance is 3,94Å in direction, perpendicular to interphase boundary without taking consideration of elastic lattice deformation. These values of interatomic distances will be saved only in crystal center of each phase at taking under the consideration of the field of elastic deformation, about character of which has been mentioned above. The crystal lattice of solid solution on NiSb base will treat the tensile stress, and crystal lattice of solid solution on PbS base will treat compression stress (fig.7).

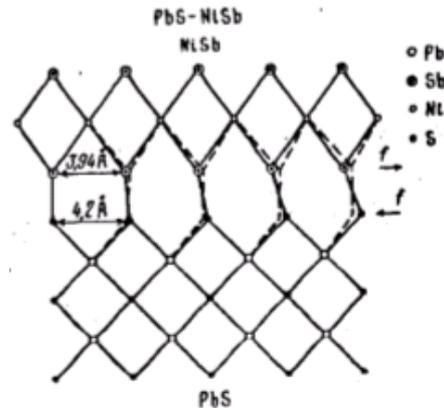


Fig.7. The circuit of possible strained PbS-NiSb eutectic in plane, perpendicular to plane of phase accretion.

The supposed change of periodic potential in eutectic of PbS-NiSb system in direction, perpendicular to plane of phase accretion, where the parameters of elementary cells of component phases with structure periods is shown on the fig.8.

The morphology analysis of plate eutectic of PbS-NiSb system allows to discuss the directed-oriented compositions as the especial class of crystal layered supramolecular systems.

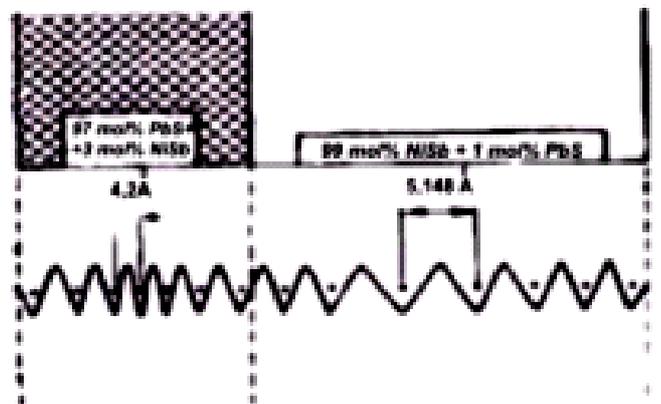


Fig.8. The schematic sketch of supposed change of periodic potential in oriented eutectic suprastructure (PbS-NiSb) in direction, perpendicular to accretion plane of suprastructures (phases).

The microstructure regularity, as we already described, should be reflected in the course of periodic potential of whole eutectic crystal in whole, and in particular: periodic potential of one lattice should be changed by periodic potential of another lattice with some period, constant for given eutectic and more bigger constant lattice of each phase.

The crystallization peculiarity and electron structure of eutectic causes the firm interphase connection, which reveal in such properties of eutectic melts as the high values of mechanical strength, plasticity, and also the microstructure stability to long heat influences.

Conclusion

The glassed eutectic is the supramolecular system, in which the redistribution of electron density, in the result of

which the connection appears between the atoms of different blocks on the boundary, leading to the electron community, and connection becomes weak inside the each subblock in frontier region, takes place in the comparison with initial components.

The oriented crystal eutectic melts can be considered as solid suprastructures, in which the periodic potential of phase alternation is marked on periodic potentials of crystal lattices of each phase, that causes the unite energy structure for the electrons of whole crystal.

The totality of investigated of supramolecular eutectic compositions on A^{IV} B^{VI} base gives the possibility of their use as contact materials at the production of different sublayers for thermoelements in systems: A^{IV} B^{VI} eutectic-metal [17].

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К.Ş. Qəhrəmanov, S.Ş. Qəhrəmanov, E.D. Moroydor, M.Q. Pişkin

A^{IV}B^{VI}-Sb (NiSb) EFTEKTİK SİSTEMLƏRDƏ SUPRAMOLEKULYAR ANSAMLLAR

İşdə A^{IV}B^{VI}-Sb (NiSb) eftektik nümunələrdə kristallizasiya proseslərini və mikrostrukturların morfoloji xüsusiyyətləri öyrənilib. Belə nəticəyə gəlmək olur ki, eftektikaların faza strukturu və onların fiziki əlaqələri molekulyar suprastruktur konsepsiyası ilə izah etmək olar.

(PbTe-Sb) və (PbS-Sb) eftektikalarda anomal, (PbS-NiSb) və (PbTe-NiSb) normal laylı suprastruktur almaq olur.

(PbS-NiSb) eftektik sistemində (111) PbS müstəvisi (0001) NiSb müstəvisinə paralel oriyentasiya və ölçü prinsiplərinə malik olurlar. Bu eftektikada yaranan atom defektləri real bircinsli kristallarda olan defektlərə bənzəyirlər.

К.Ш. Кахраманов, С.Ш. Кахраманов, Е.Д. Моройдор, М.Г. Пишкин

СУПРАМОЛЕКУЛЯРНЫЕ СТРУКТУРЫ НА ОСНОВЕ ЭВТЕКТИК СИСТЕМ A^{IV}B^{VI}- Sb (Ni Sb)

Процессы кристаллизации и особенности морфологии микроструктуры эвтектик A^{IV} B^{VI} - S b (NiSb) свидетельствуют в пользу супрамолекулярной концепции с учетом взаимодействия субструктур.

На полученных направленно – ориентированных эвтектиках – (PbTe - Sb) и (PbS - Sb) выявлены аномальные структуры, а в эвтектиках (PbS - NiSb) и (PbTe - NiSb) - регулярные пластинчато-волокнистые.

В эвтектике (PbS - NiSb), также как и в нормальных бинарных эвтектических системах соблюдается принцип ориентационного и размерного соответствия: плоскость (111) PbS срастается с плоскостью (0001) NiSb;

Анализ морфологии эвтектики (PbS - NiSb) показал наличие различных дефектов типа сдвигов пластин, поверхностей несоответствия, искривлений блоков (пластин) и избыточной экстра - пластины. Эти дефекты аналогичны атомным дефектам, образующимся в реальных однофазных кристаллах.

Исследования показали, что эвтектики можно рассматривать как супрамолекулярные слоистые ансамбли.

Received: 20.06.06