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O.M. Manyk² (<https://orcid.org/0000-0003-2525-5280>),
V.V. Razinkov¹ (<https://orcid.org/0009-0004-2882-5466>),
V.V. Lysko^{1,2} (<https://orcid.org/0000-0001-7994-6795>),
M.M. Krechun^{1,2} (<https://orcid.org/0009-0002-4592-1340>),
D.E. Rybchakov¹ (<https://orcid.org/0009-0002-2891-2258>)

¹Institute of Thermoelectricity of the NAS and MES
of Ukraine, 1 Nauky str., Chernivtsi, 58029, Ukraine;

²Yuriy Fedkovych Chernivtsi National University,
2 Kotsiubynsky str., Chernivtsi, 58012, Ukraine

Corresponding author: M.M. Krechun, e-mail: krechun.mariya@gmail.com

Correlation Models of Phase Transitions in “Thermoelectric Material (Bi-Te) – Solder (Pb-Sn)” Contact Structures of the Bi-Pb-Sn-Te System. Part 1. Chemical Bonding and Phase Diagrams

Theoretical models of phase transitions in contact structures of the Bi-Pb-Sn-Te system are presented. Isothermal sections and phase region distribution schemes for the quaternary system are provided, based on the binary phase diagrams of the constituent elements (Bi-Pb, Bi-Te, Bi-Sn, Pb-Te, Pb-Sn, Te-Sn) and four ternary systems (Bi-Pb-Te, Bi-Pb-Sn, Bi-Te-Sn, Pb-Te-Sn) derived using the inverse triangulation method. Diagrams showing the distribution of equilibrium phase regions at various temperatures have been constructed; these allow for the prediction of chemical reaction kinetics in the contact zone of thermoelements.

Keywords: thermoelectric materials, chalcogenides, solid solutions, contact structures, solders, phase diagrams.

Introduction

The study of phase equilibria in the Bi-Pb-Sn-Te quaternary system is relevant due to the need to establish the patterns of interphase interaction and diffusion degradation processes at the "thermoelectric material (Bi-Te) – solder (Pb-Sn)" interface [1–5].

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The fabrication of stable contact structures for low-temperature thermoelectric materials (Bi-Te) involves joining them with metallic interconnect layers, where lead (Pb) and tin (Sn) serve as the base of the metal solder. However, prolonged thermal loading leads to the formation of significant chemical gradients at the interfaces; these gradients stimulate the interdiffusion of solder elements (Pb, Sn) and thermoelectric material components (Bi, Te), triggering chemical reactions, microstructural changes, and the degradation of contact properties [6].

In this study, the physicochemical analysis of the system under consideration is based on the contrast between the melting points of its pure components. The low-melting metals that form the basis of conventional solders – tin ($T_{\text{melt}} = 232\text{ }^{\circ}\text{C}$) and lead ($T_{\text{melt}} = 327.5\text{ }^{\circ}\text{C}$), as well as bismuth ($T_{\text{melt}} = 271.5\text{ }^{\circ}\text{C}$) – are characterized by low melting points, whereas chalcogenide components exhibit significantly higher thermal stability (for example, $T_{\text{melt}}(\text{Te}) = 449.5\text{ }^{\circ}\text{C}$, while the binary compounds SnTe and PbTe melt at $806\text{ }^{\circ}\text{C}$ and $924\text{ }^{\circ}\text{C}$, respectively) [7].

Analysis of the limiting binary phase diagrams of the system under consideration showed that, in the metallic subsystems at temperatures above $327.5\text{ }^{\circ}\text{C}$, solid-phase regions and intermetallic compounds completely disappear, transforming into a homogeneous liquid state. At the same time, stable semiconducting phases and substitution compounds persist in binary chalcogenide systems under these conditions. Consequently, constructing and analyzing isothermal sections of the Bi-Pb-Sn-Te system over a wide temperature range makes it possible to assess the thermodynamic stability of the phase interface under critical thermal loading conditions; in this state, the metallic solder component (Pb-Sn) exists as a homogeneous liquid, creating a steep chemical concentration gradient and driving intense diffusion of elements into the solid chalcogenide matrix (Bi-Te). For this reason, the subsequent examination of the multicomponent system was based on an analysis of the binary subsystems formed by the constituent elements.

Phase diagrams of binary systems

The bismuth-tellurium system [8] is characterized by complex phase equilibria involving the formation of a homologous series of intermediate compounds and a single base compound that melts congruently. The melting points of the components are $271.4\text{ }^{\circ}\text{C}$ for Bi and $449.5\text{ }^{\circ}\text{C}$ for Te. The key semiconducting phase of the system is bismuth telluride (Bi_2Te_3), which melts congruently at $586\text{ }^{\circ}\text{C}$ and exhibits a liquidus maximum slightly shifted from the stoichiometric composition to 59.8 at.% Te. In the tellurium-rich region, a eutectic reaction $\text{L} (90\text{ at.\% Te}) \rightleftharpoons \text{Bi}_2\text{Te}_3 + (\text{Te})$ occurs at $413\text{ }^{\circ}\text{C}$. In the concentration range of 30–59 at.% Te, crystallization proceeds via a cascade of successive peritectic equilibria forming layered compounds: Bi_4Te_5 at $562\text{ }^{\circ}\text{C}$, Bi_6Te_7 at $540\text{ }^{\circ}\text{C}$, BiTe at $420\text{ }^{\circ}\text{C}$, Bi_4Te_3 , Bi_2Te ($312\text{ }^{\circ}\text{C}$, $375\text{ }^{\circ}\text{C}$), and Bi_7Te_3 at $266\text{ }^{\circ}\text{C}$. On the bismuth-rich side, a degenerate eutectic $\text{L} \rightleftharpoons (\text{Bi}) + \text{Bi}_7\text{Te}_3$ occurs at $266\text{ }^{\circ}\text{C}$ and a concentration of 2.4 at.% Te. The limiting solubility of tellurium in bismuth is approximately 2.4 at.%, whereas the solubility of bismuth in tellurium is negligible.

The lead-tin binary system [9] is a typical eutectic system characterized by limited mutual solid-state solubility of the components. The melting points of the components are $327.5\text{ }^{\circ}\text{C}$ for lead and $231.9\text{ }^{\circ}\text{C}$ for tin. The liquidus lines descend monotonically to a point of ternary

equilibrium at a temperature of 183 °C and a concentration of 73.9 at.% Sn. At this isotherm, a non-variant eutectic reaction $L \rightleftharpoons (Pb) + (Sn)$ occurs. The maximum solid solubility of tin in lead (Pb) reaches 28.1 at.% Sn at the eutectic temperature, but decreases significantly upon cooling to room temperature. On the tin side, the (Sn) solid solution dissolves no more than 1.3 at.% Pb (98.7 at.% Sn). Below the eutectic temperature, across the entire composition range from 28.1 to 98.7 at.% Sn, the alloy structure consists of a two-phase mechanical mixture of (Pb) and (Sn) crystals.

The bismuth-tin binary system [10] is a classic simple eutectic-type diagram characterized by limited solid-state solubility and a complete absence of intermediate chemical compounds. The melting points of the pure components are 231.9 °C for tin and 271.4 °C for bismuth. The liquidus lines slope downward smoothly from the pure components and intersect at the eutectic point at a temperature of 139 °C and a concentration of 43 at.% Bi. At this temperature, the single non-variant eutectic transformation $L \rightleftharpoons (Sn) (13.1 \text{ at.\% Bi}) + (Bi) (99.8 \text{ at.\% Bi})$ occurs. The maximum solubility of bismuth in white tin (Sn) reaches 13.1 at.% at the eutectic temperature but drops sharply to nearly zero at room temperature. The solubility of tin in bismuth (Bi) is very limited and does not exceed 0.2 at.%. At 13 °C, on the pure tin side, the allotropic transformation of white tin into gray tin takes place; this has practically no effect on the overall phase balance of the system.

The bismuth-lead binary system is of the eutectic-peritectic type, characterized by variable mutual solid-state solubility of the components and the formation of an intermediate phase. The melting points of the components are 327.5 °C for lead and 271.4 °C for bismuth. A lead-based solid solution (Pb) is formed, in which the limiting solubility of bismuth reaches a maximum of 22 at.% at the peritectic temperature of 187 °C and decreases gradually to 16 at.% as the temperature drops. At 187 °C, a peritectic reaction occurs – $L (38 \text{ at.\% Bi}) + (Pb) (22 \text{ at.\% Bi}) \rightleftharpoons (29 \text{ at.\% Bi})$ —resulting in the formation of a hexagonal phase. This phase exhibits a significant homogeneity range of 27.5–40 at.% Bi. Upon further cooling of the melt, a eutectic with a composition of 55 at.% Bi crystallizes at 125.5 °C. The solubility of lead in bismuth is extremely low, not exceeding 0.5 at.%. In the solid state at low temperatures (–46 °C), the decomposition of this phase via a eutectoid reaction is observed [11].

The lead-tellurium system [7] forms a single thermodynamically stable chemical compound – lead telluride (PbTe) – with a stoichiometric composition (50 at.% Te), which melts congruently at a high temperature of 924 °C. Due to the high stability of PbTe, the entire system is clearly divided into two independent quasi-binary subsystems of the eutectic type: Pb-PbTe and PbTe-Te. In the Pb-PbTe subsystem, the liquidus line drops sharply from a maximum of 924 °C to a degenerate eutectic equilibrium at 326.8°C, which practically coincides with the composition of pure lead ($L \rightleftharpoons (Pb) + PbTe$). In the PbTe-Te subsystem, the eutectic transformation occurs at a temperature of 410.9 °C and a melt composition of 89.1 at.% Te, according to the scheme $L \rightleftharpoons PbTe + (Te)$. The mutual solid-state solubility of the components – both on the Pb and Te sides – is extremely low, and the PbTe compound itself has a narrow homogeneity range.

The binary tin-tellurium system [12] is characterized by the formation of a single

congruently melting semiconducting compound – tin monotelluride (SnTe) – with a melting point of 806 °C at a composition of approximately 50.4 at.% Te. This compound divides the overall phase diagram into two eutectic regions: Sn-SnTe and SnTe-Te. A distinctive feature of the Sn-SnTe subsystem is the liquid phase's pronounced tendency toward phase separation, manifested by a flat section of the liquidus in the 15–45 at.% Te range and the presence of a liquid-immiscibility region below the liquidus with an upper critical point at 612 °C and a concentration of 28 at.% Te. Crystallization on the tin side concludes with a degenerate eutectic reaction at 231.5 °C, which is only 0.5 °C below the melting point of pure Sn. In the SnTe-Te subsystem, a eutectic equilibrium is observed at 401 °C and a tellurium content of 85 at.%. The solid SnTe phase exhibits a narrow homogeneity range slightly shifted toward a tellurium excess.

Phase equilibria in the ternary systems Bi-Sn-Te, Bi-Pb-Te, Sn-Pb-Bi, and Sn-Pb-Te

The study of phase equilibria in ternary systems is a crucial stage in the analysis of multicomponent materials, as it enables the determination of component interaction patterns and phase coexistence regions, the assessment of intermediate compound stability, and the tracing of potential diffusion pathways. For "thermoelectric material (Bi-Te) – solder (Pb-Sn)" contact heterostructures, the analysis of ternary systems is a necessary prerequisite for developing a physicochemical model of the quaternary Bi-Pb-Sn-Te system.

Each of the ternary systems – Bi-Sn-Te, Bi-Pb-Te, Sn-Pb-Bi, and Sn-Pb-Te – is characterized by specific phase equilibrium features determined by the nature of the interactions between the corresponding binary subsystems. Analysis of their isothermal sections makes it possible to identify the existence regions of solid solutions and intermediate phases, determine three-phase equilibria, and investigate and evaluate potential pathways for the formation of interfacial reaction layers in the contact zone.

Based on the results of the analysis of binary systems, a scheme of phase region distribution for the corresponding ternary systems was constructed using the binary systems as a basis; this serves as an intermediate step in modeling the quaternary Bi-Pb-Sn-Te system.

The obtained results are presented in Figs. 1–4, where the following notations are used:

- α – solid phase based on Bi;
- β – solid phase based on Te;
- γ – solid phase based on Pb;
- λ – solid phase based on Sn;
- δ – solid phases based on Pb-Bi;
- σ – solid phases based on Pb-Te;
- ε – solid phases based on Bi-Te;
- χ – solid phases based on Pb-Sn;
- η – solid phases based on Sn-Bi;
- μ – solid phases based on Sn-Te;
- ξ – solid phases based on Bi-Pb-Te;

θ – solid phases based on Sn-Pb-Bi;
 φ – solid phases based on Bi-Sn-Te;
 ψ – solid phases based on Sn-Pb-Te;
 L – liquid.

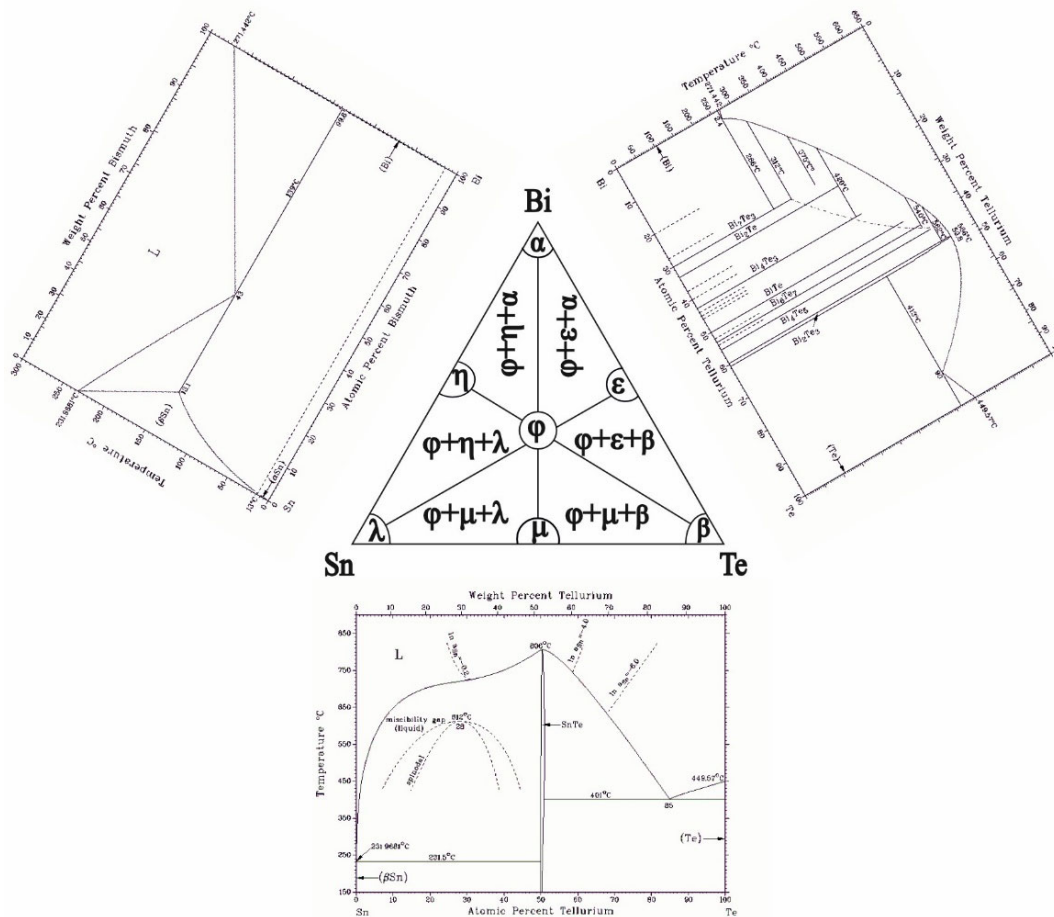


Fig. 1. Diagram of the distribution of Bi-Sn-Te phase regions in the solid state

The phase equilibrium diagram of the Bi-Sn-Te ternary system in the solid state (Fig. 1) is represented by a composition triangle combined with the unfolded binary subsystems Bi-Sn, Bi-Te, and Sn-Te. The vertices of the triangle define the regions of terminal solid solutions of the components – bismuth (α), tin (λ), and tellurium (β) – while binary intermediate phases – η (Bi-Sn), ε (Bi-Te), and μ (Sn-Te) – are located on the corresponding edges. The triangulation is centered on the complex ternary phase φ (Bi-Sn-Te), which is linked by quasibinary sections to corner solid solutions and binary compounds, forming six equilibrium three-phase fields: $\varphi+\eta+\alpha$, $\varphi+\varepsilon+\alpha$, $\varphi+\eta+\lambda$, $\varphi+\varepsilon+\beta$, $\varphi+\mu+\lambda$, and $\varphi+\mu+\beta$. The resulting configuration demonstrates subsolidus phase stability and determines the phase composition of functional thermoelectric materials within this chalcogenide system.

Distribution diagrams for the Bi-Pb-Te (Fig. 2), Sn-Pb-Bi (Fig. 3), and Sn-Pb-Te (Fig. 4) systems are constructed in a similar manner.

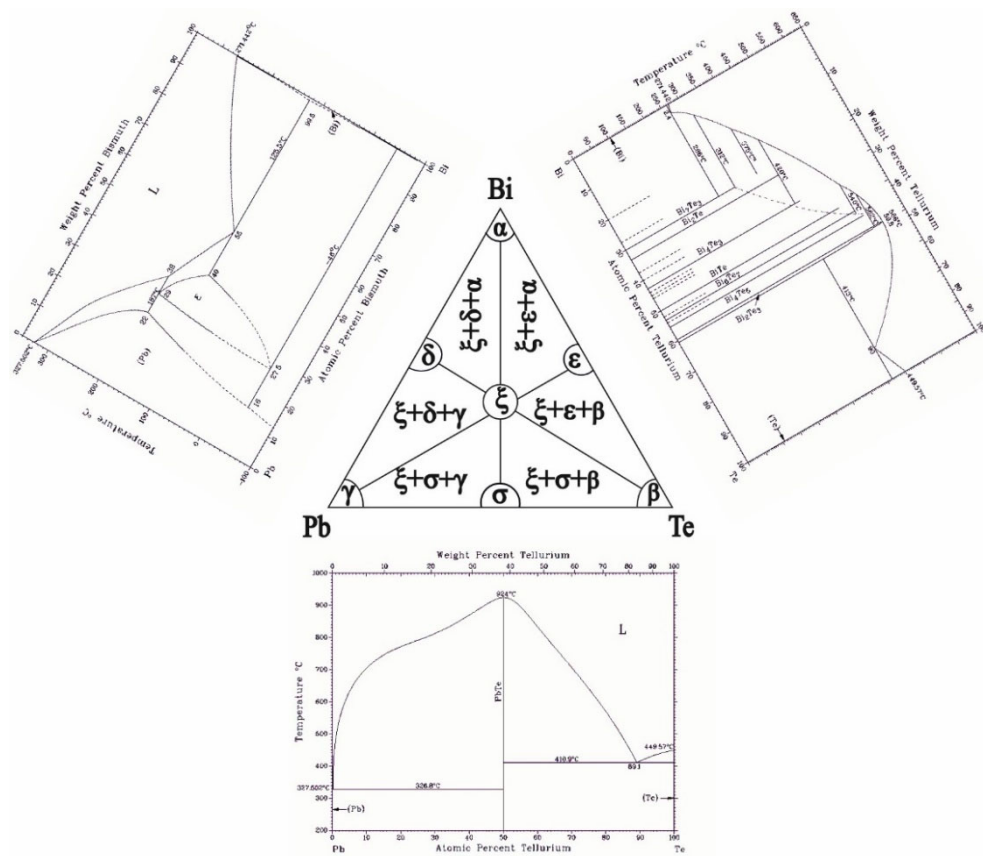


Fig. 2. Diagram of the distribution of Bi-Pb-Te phase regions in the solid state

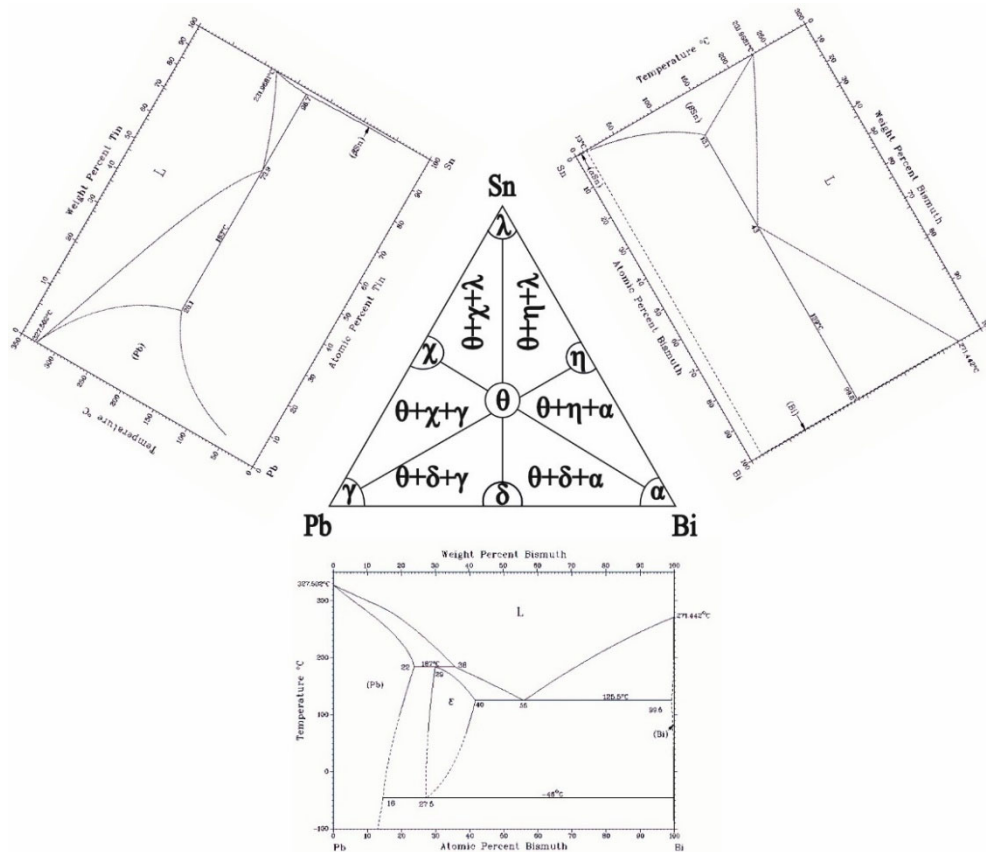


Fig. 3. Diagram of the distribution of Sn-Pb-Bi phase regions in the solid state

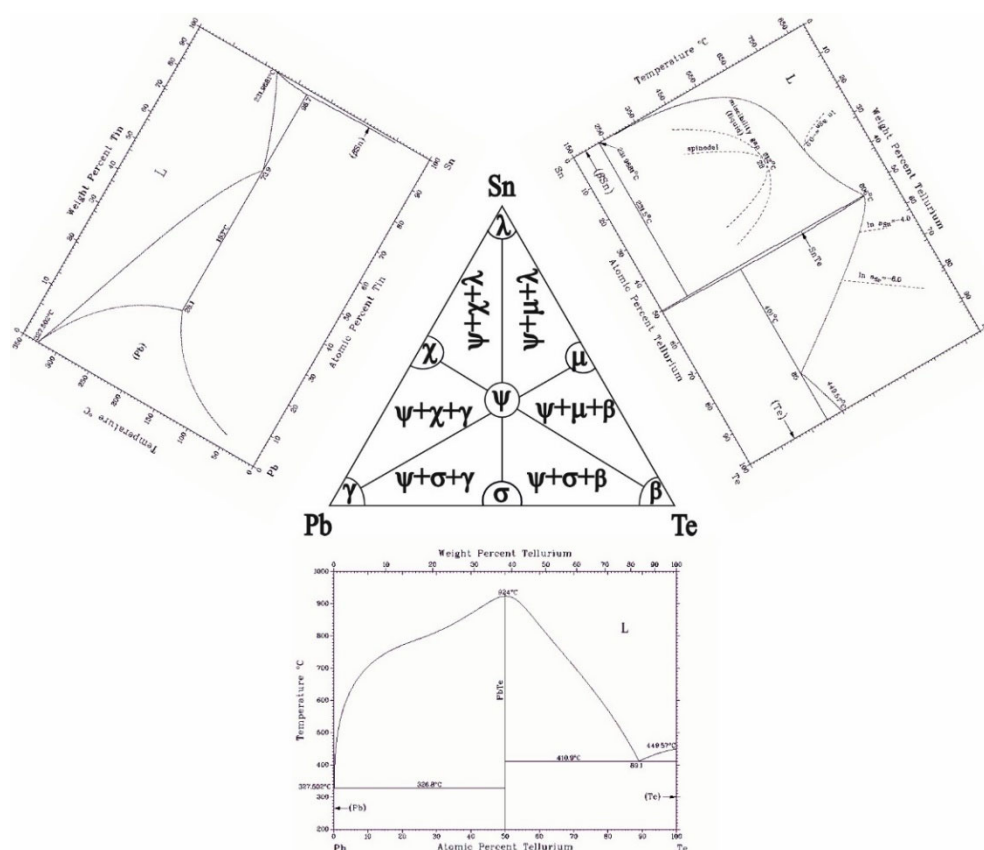


Fig. 4. Diagram of the distribution of Sn-Pb-Te phase regions in the solid state

Analysis of phase distribution reveals a fundamental difference between the systems: while the Bi-Pb-Sn ternary system exhibits a purely metallic interaction character – resulting in the formation of low-melting eutectics and intermetallic compounds – the Bi-Sn-Te, Bi-Pb-Te, and Sn-Pb-Te systems are classified as chalcogenide systems. Phase formation in the latter three is governed by the presence of thermodynamically stable, narrow-gap semiconducting compounds (PbTe , SnTe , Bi_2Te_3), which play a key role in determining the functional properties of thermoelectric materials based on them.

Isothermal sections of Bi-Pb-Sn-Te

Fig. 5 shows the isothermal section of the Bi-Pb-Sn-Te system at 100 °C. The diagram clearly illustrates the division of the quaternary system into four ternary systems, each of which is further subdivided into three intermediate systems. It also clearly demonstrates the conditions for the formation of various phases – compounds, solid solutions, and mechanical mixtures (both ternary and quaternary) – based on these systems.

Figure 6 shows the isothermal section of the quaternary system at 150 °C; this temperature is below the melting points of the components (Bi, Pb, Sn, Te) but above the temperature of the first eutectic in the Bi-Sn and Pb-Bi binary phase diagrams. It is in these systems that the conoid triangles ($L + \lambda + \eta$) and ($L + \alpha + \eta$) begin to appear. This ensures high wettability of the contact surface, results in high thermodynamic activity of the solder components, and promotes their intensive diffusion into the chalcogenide matrix of the thermoelectric material during the long-term operation of the generator module.

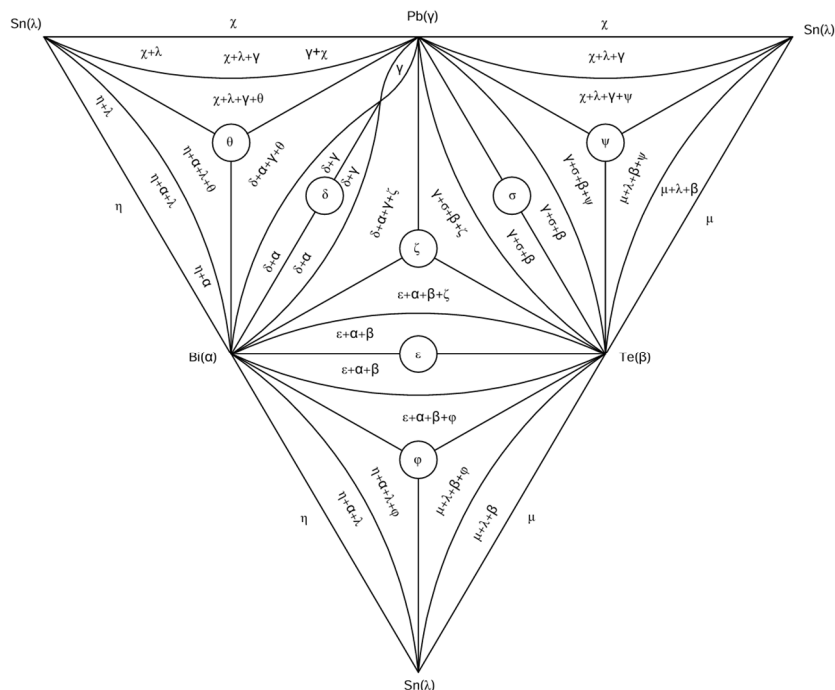


Fig. 5. Isothermal section of Bi-Pb-Sn-Te at 100 °C

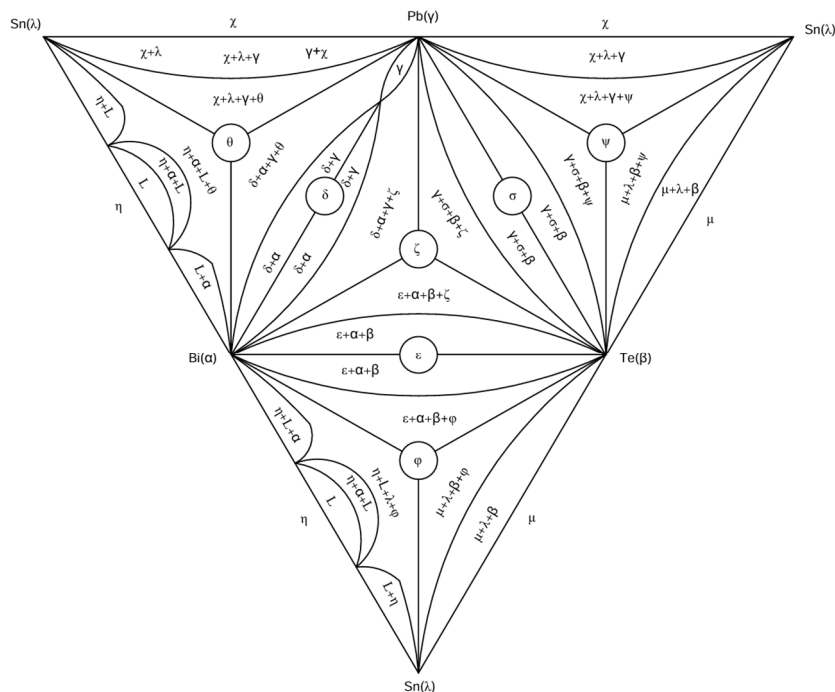


Fig. 6. Isothermal section of Bi-Pb-Sn-Te at 150 °C

Figs. 7–11 show isothermal sections of the quaternary system at temperatures of 200 °C, 300 °C, 400 °C, 500 °C, and 600 °C.

The isothermal section at 200 °C reflects contact interaction under conditions of medium-temperature soldering with tin-lead solders. This temperature exceeds the eutectic equilibrium of the Pb–Sn binary subsystem (183 °C); consequently, significant expansion of phase fields involving the liquid phase (L) is observed along the limiting edges of the metallic subsystems.

The liquid phase equilibrates with solid intermetallic phases and terminal solid solutions of the solder components, whereas the chalcogenide compounds remain stable in the solid state.

The isothermal section at 300 °C (Fig. 8) corresponds to the state of the system following the melting of tin (231.9 °C) and bismuth (271.4 °C). The liquid phase L extends over a large part of the metallic region, forming two- and three-phase fields where the melt coexists with crystalline phases.

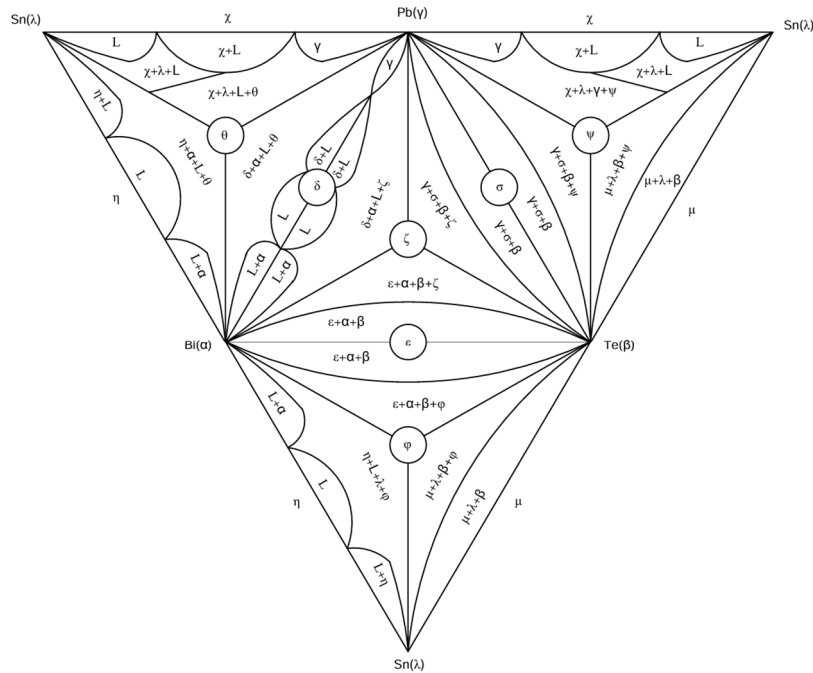


Fig. 7. Isothermal section of Bi-Pb-Sn-Te at 200 °C

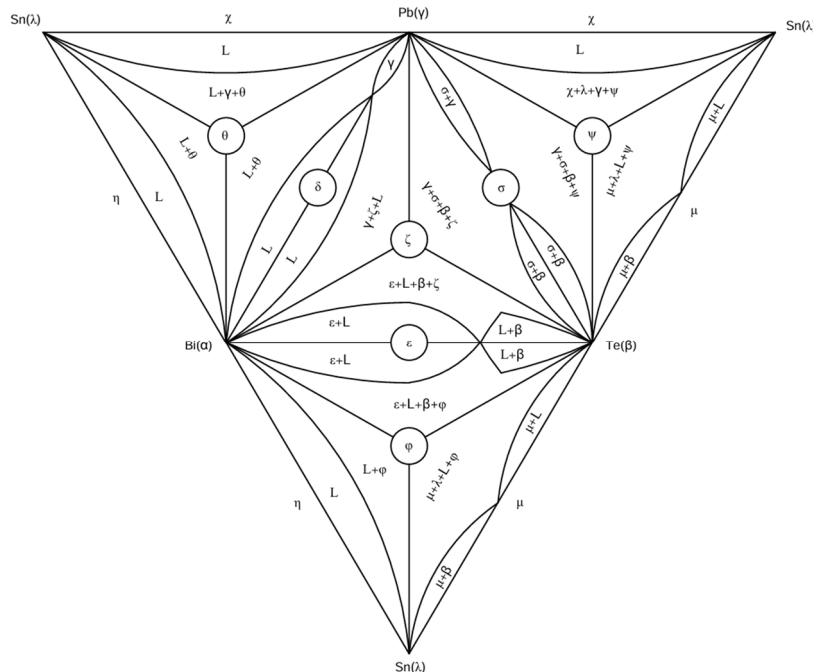


Fig. 8. Isothermal section of Bi-Pb-Sn-Te at 300 °C

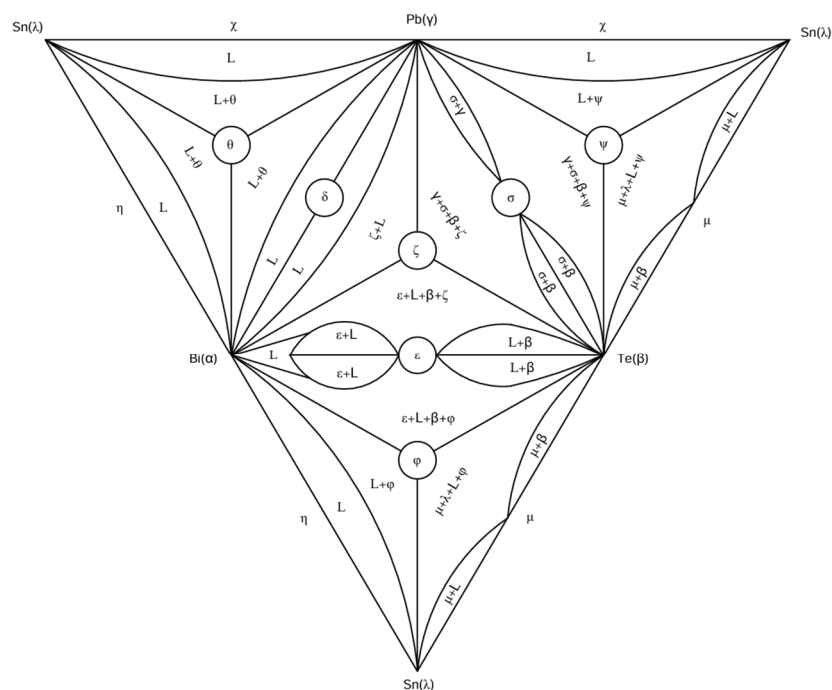


Fig. 9. Isothermal section of Bi-Pb-Sn-Te at 400 °C

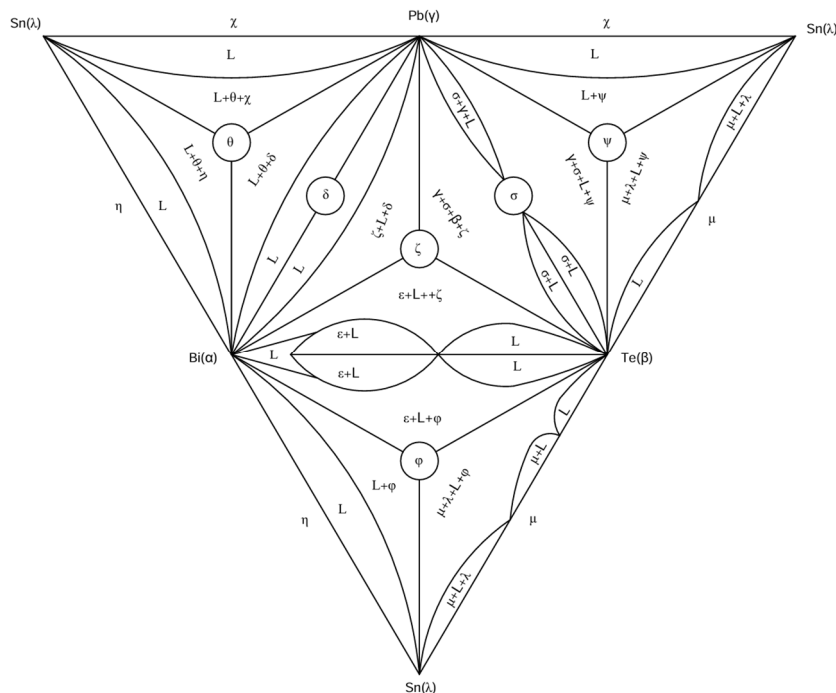


Fig. 10. Isothermal section of the Bi-Pb-Sn-Te system at 500 °C

Further rise in temperature is accompanied by the progressive thermal degradation of contact zones: while at 400 °C (Fig. 9) the melting of lead (above 327.5 °C) transforms the entire Bi-Pb-Sn metallic subsystem into a liquid state (L) with the complete disappearance of metallic intermetallics, at 500 °C (Fig. 10) the melting of tellurium (449.6 °C) extends the melt into the chalcogenide regions while preserving the crystalline compounds Bi_2Te_3 , SnTe , and PbTe ; finally, at 600 °C (Fig. 11), the decomposition of Bi_2Te_3 (586 °C) restricts the solid-

phase region exclusively to the refractory phases of SnTe and PbTe. Thus, the constructed isothermal sections of the Bi-Pb-Sn-Te system clearly illustrate the evolution of interfacial interaction under conditions of high thermal loads, where the molten metallic solder creates a steep chemical gradient and stimulates contact dissolution and the diffusion of elements into the thermoelectric material.

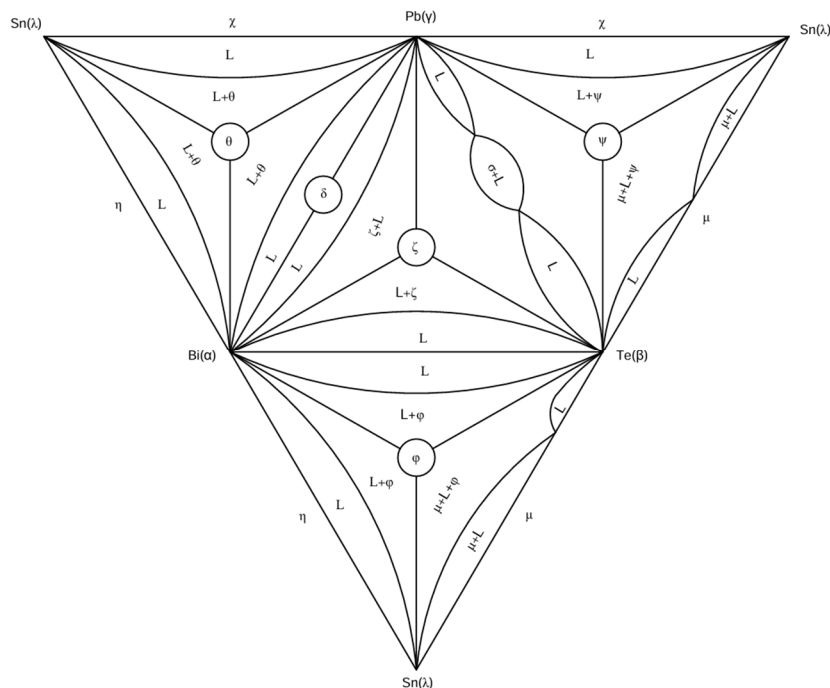


Fig. 11. Isothermal section of Bi-Pb-Sn-Te at 600 °C

Discussion of results and conclusions

The comprehensive approach employed – based on combining binary subsystems into ternary systems and a unified quaternary model – made it possible to describe the processes governing interatomic interaction (at various technological levels) from the standpoint of chemical bonding. This involved, first and foremost, modeling crystal structure formation based on the constituent elements (Bi, Pb, Sn, Te) – incorporating data on their physicochemical properties – as well as the binary compounds derived from them (Bi-Pb, Bi-Sn, Bi-Te, Pb-Sn, Pb-Te, Sn-Te) and the construction of four ternary systems (Bi-Sn-Te, Bi-Pb-Te, Sn-Pb-Bi, and Sn-Pb-Te).

A novel aspect of the study was that complex systems were examined from the perspective of ternary systems, which in turn were constructed by the inverse triangulation method using information on binary compounds and their phase diagrams. This approach enabled the determination of phase equilibrium boundaries in liquid–crystal regions, the differentiation of eutectic and peritectic phase boundaries – facilitating the resolution of technological issues regarding stable and metastable phases – and the identification of congruent and incongruent melting scenarios. The findings can be applied to the development of processing conditions for producing new materials based on the Bi-Pb-Sn-Te system.

Authors' information

O.M. Manyk – Cand.Sc. (Phys.-Math).

V.V. Razinkov – Cand.Sc.(Phys.-Math).

V.V. Lysko – Cand.Sc.(Phys.-Math).

M.M. Krechun – PhD.

D.E. Rybchakov – PhD.

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Маник О.М.² (<https://orcid.org/0000-0003-2525-5280>),
Разіньков В.В.¹ (<https://orcid.org/0009-0004-2882-5466>),
Лисько В.В.^{1,2} (<https://orcid.org/0000-0001-7994-6795>),
Кречун М.М.^{1,2} (<https://orcid.org/0009-0002-4592-1340>),
Рибчаков Д.Є.¹ (<https://orcid.org/0009-0002-2891-2258>)

¹Інститут термоелектрики НАН та МОН України,
вул. Науки, 1, Чернівці, 58029, Україна;

²Чернівецький національний університет імені Юрія Федьковича,
вул. Коцюбинського 2, Чернівці, 58012, Україна

Кореляційні моделі фазових переходів у контактних структурах «термоелектричний матеріал (Bi-Te) – припій (Pb-Sn)» системи Bi-Pb-Sn-Te.

Частина 1. Хімічний зв'язок та діаграми стану

Представлено теоретичні моделі фазових переходів у контактних структурах системи Bi-Pb-Sn-Te. Приведено ізотермічні перерізи та схеми розподілу фазових областей в четверних системах з використанням бінарних діаграм стану вихідних компонентів (Bi-Pb; Bi-Te; Bi-Sn; Pb-Te; Pb-Sn; Te-Sn) та чотирьох потрібних систем (Bi-Pb-Te; Bi-Pb-Sn; Bi-Te-Sn; Pb-Te-Sn), отриманих методом оберненої триангуляції. Побудовані схеми розподілу фазових областей рівноваги при різних температурах, які дозволяють прогнозувати кінетику хімічних реакцій в приконттактній зоні термоелементів.

Ключові слова: термоелектричні матеріали, халькогеніди, тверді розчини, контактні структури, припої, діаграми стану.