Relationship between temperature dependence of viscosity of Cu – Sn alloys and phase diagram of state

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The relationship between the liquidus line and viscosity isotherms was established for the copper-tin system based on the partial-cluster model within the concept of chaotic particles. The equations of temperature dependences of dynamic viscosity for copper and tin melts, as well as for the partial-cluster model of Cu – Sn alloy at different temperatures are derived. The shapes of the liquidus line curves duplicate the viscosity isotherms from the calculated data, which use smoothed temperature dependences for pure metals Cu and Sn, as well as different thermal chaotization barriers during the transformation of the alloy to the liquid state. The cause of deviation from the regularity of the general features of the liquidus line and viscosity isotherms was found. It consists in the formation of associated complexes of the CuxSny type, which are statistically stable chemical groupings without separation into a separate phase. These complexes (clusters) are in dynamic equilibrium with the solution matrix, characterized by a disorderly distribution of atoms. The formation of such groups is accompanied by the release of heat. This is accounted for in the partial-cluster viscosity model as an additional thermal barrier to chaotization during cluster fracture with further heating of the melt. Consideration of the new component made it possible to obtain an adequate dependence of the viscosity of Cu – Sn alloy on its composition and the thermal barrier on the liquidus line at 1100 °C. Directions for further determination of the model with the purpose of its refinement and wide use in adapting it to experimental data on viscosity at high temperatures of melts from their state diagrams are outlined and verified.

Key words: viscosity, state diagram, liquidus, Cu – Sn alloy, partial-cluster model, liquid state.

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Introduction

The need to study all kinds of properties of substances is determined both by the increasing complexity of technology and by the scientific importance of the results obtained. In this sense, the study of the properties of melts of various systems, which are increasingly used in modern engineering, represents a significant interest. The physical nature of various compounds determines the specifics of their study, both theoretically and experimentally.

Viscosity is one of the most important physical properties of melts. It is a structure-sensitive characteristic, so it can be used in the analysis of various physical and chemical processes in liquid melts to improve the technology of obtaining various alloys [1–11]. In solving such questions, phase diagrams of alloys, which reflect all phase transitions, are considered.

Copper-tin alloys are the basis of tin bronzes, which due to their performance characteristics are widely used in various industries. An important aspect of studying Cu – Sn alloys is also the development of solders based on them that do not contain environmentally harmful lead. The practical importance of copper-tin alloys has led to a large number of studies of the phase diagram of Cu – Sn [12].

In this paper, we study the relationship between the temperature dependence of the dynamic viscosity of the liquid Cu – Sn alloy and their phase diagram of state. These studies are a development of our previously proposed notion of the stochastic nature of clusters as applied to the structure of melts [13, 14].

Methodology and calculation

In the review papers [1, 2], the state diagram of the Cu – Sn system is given on the base of experimental data published before 1990. According to these data, there are two disordered phases with face-centered cubic (FCC) and body-centered cubic (BCC) structures in the region of copper-rich compositions. As tin concentration increases, the equilibrium state diagram reveals a number of intermetallic phases with different structures [2, 15–17].

In the following years, studies of the Cu-Sn system concerned only the phase transition region between the disordered (β) and ordered (γ-Cu₃Sn) BCC-phases [2, 16, 17]. By differential thermal analysis (DTA), energy dispersive *X*-ray spectroscopy (EDMA), and *X*-ray fluorescence (XRF) methods established that the β/γ-Cu₃Sn phase transition belongs to transitions of type II. These

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data were taken into account in the construction of the currently accepted version of the state diagram [12, 18].

The authors [13] developed the partial-cluster viscosity model (PCVM), which is based on the concept that during the melting of pure simple substances as well as their compounds (alloys), their complete phase transformation occurs, but only a partial increase in chaotization, which refers respectively to the melting temperatures of copper and tin and liquidus temperatures on the state diagrams and continues with a further increase in temperature.

The same authors show that the viscosity of the liquid is related to the content of clusters, the fraction of which P_{cl} is equal to the square of the content of particles with energy no greater than the thermal barrier of chaotization RT_{ch}, for simple substances RT_m, and for alloys $RT_{liquidus}$. Since there is no direct relationship between viscosity and the thermal barrier RT_{liq}, it is accounted for by the ratio: how many times at temperature T the fraction of clusters that passed the barrier RT_{liq}, more or less than the fraction of clusters of pure substances that passed the barrier RT_m, as many times the viscosity of the alloy more or less than the known viscosity of pure matter (the effect of the alloy temperature is expressed in the fraction of clusters), taking into account the contribution of alloy components in PCVM:

$$\eta_T = X_A \dot{\eta}_{T,A} \left[\frac{1 - exp(-\Delta_{ch}H/RT)}{1 - exp(-T_{m,A}/T)} \right]^2 +$$

$$+ X_B \dot{\eta}_{T,B} \left[\frac{1 - exp(-\Delta_{ch}H/RT)}{1 - exp(-T_{m,B}/T)} \right]^2 , \tag{1}$$

where η_T — is the alloy viscosity, mPa·s at temperature T, K; $\dot{\eta}_{T,A}$ and $\dot{\eta}_{T,B}$ — viscosity of pure substances A and B, mPa·s; X_A and X_B — atomic fractions (fractions of a unit, u.f.) of substances A and B in the alloy; $\Delta_{ch}H$ — chaotization heat equal a sum of any thermal barriers for the alloy and the melting temperature $T_{m,A}$ and $T_{m,B}$ for pure substances, K. For the system under consideration, A is Cu, B is Sn. Copper melting temperature $T_{m,Cu} = 1083$ °C = 1356 K, tin melting temperature $T_{m,Sn} = 232.08$ °C = 505.08 K [19].

To carry out calculations according to PCVM (1) we need data on the alloy, which would contain a diagram of state in the full range of its composition, the experimental dependence of the viscosity in this range at different temperatures and the physical and chemical features of the structure of the liquid alloy. The necessary data are often grouped by base metal, but for the alloy they are quite rare. One such work is a monograph [12], which presents and systematizes the results of research on the structure and properties of copper melts with aluminum, tin, and lead. The authors [13] previously analyzed an alloy of copper and aluminum and obtained fairly adequate results. For the present study we use data on copper-tin alloy [12].

The system under consideration is quite complex: it has eight phases, which are formed by mixing the pure components [20]. All features of the system are displayed integrally by the liquidus line, which is taken by the authors as a chaotic barrier for the virtual existence of clusters according to the formula (1).

Table shows the data for constructing the liquidus temperature of the Cu − Sn system by recalculating the values of this line from [12]. The step of the atomic fraction of tin was 5% (0.05 u.f.). The obtained values are used to calculate the dynamic viscosity according to PCVM (1). The values taken from [12] were based on real experimental data on the liquidus temperatures of the system, as well as intermediate points obtained from interpolated liquidus lines.

Fig. 1 shows the liquidus line configuration of the Cu − Sn system according to **Table**.

Fig. 1. Liquidus line of Cu − Sn alloy: points – according to Table, line – inter-point interpolation)

Table

Coordinates of the liquidus line of the Cu – Sn system. The points at which phase transformations occur are highlighted

X_{Sn}, % at.	T_{liq}, °C	T_{liq}, K	X_{Sn}, % at.	T_{liq}, °C	T_{liq}, K	X_{Sn}, % at.	T_{liq}, °C	T_{liq}, K
0	1083	1356	35	685	958	75	501	774
5	1030	1303	40	658	931	80	470	743
10	950	1223	43.1	640	913	85	430	703
15	801	1074	45	635	908	86.7	415	688
15.5	798	1071	50	620	893	90	375	648
19.1	755	1028	55	598	871	95	305	578
20	754	1027	60	575	848	98.7	227	500
25	740	1013	65	550	823	100	232.08	505.08
30	710	983	70	528	801			

Results and discussion

A cluster-associated dynamic viscosity model developed earlier by the authors [22] was constructed for copper using experimental data [21], with a correlation coefficient of 0.995:

$$\dot{\eta}_{T,\text{Cu}} = 3.49(1373/T)^{1.778(1473/T)^{0.926}}, \text{ mPa·s.} \qquad (2)$$

Using the reference book [23] we processed the data on dynamic viscosity and obtained a cluster-associated model for tin with a correlation coefficient of 0.99976 [22]:

$$\dot{\eta}_{T,\text{Sn}} = 1.54(573/T)^{0.912(973/T)^{0.479}}, \text{ mPa·s.} \qquad (3)$$

Since the data on dynamic viscosity of Cu − Sn alloys at 700, 800, 900, 1000, 1100, 1200, 1300 °C (973, 1073, 1173, 1273, 1373, 1473, 1573 °K) are available, for the same temperatures we calculated the values of η_T by PCVM (1) using values from equations (2), (3) and by liquidus line from **Table**. The results are shown in **Fig. 2**.

Here it should be noted that for temperatures of 700, 800, 900, 1000 °C the data should be considered starting from X_{Sn} = 60, 50, 25 and 20% respectively, since these compositions are in the liquid state region with $t_{liq} < t_{m,\text{Cu}}$ = 1083 °C and extrapolation of viscosity towards higher copper content is not acceptable due to the fact that the alloy at these temperatures can be homogeneous only in the supercooled state.

As can be seen from **Fig. 2**, the viscosity lines quite clearly repeat the liquidus line (**Fig. 1**) with special features at six points. The minimum viscosity value is at X_{Sn} = 98.7 %, at the corresponding eutectic temperature. In **Fig. 2**, for all of the studied isotherms, there is a noticeable convergence at this temperature.

The authors of [24, 25] show that viscosity isotherms approximately repeat the shape of the liquidus line and may differ in their convergence and leveling as the temperature rises. Such features have been previously confirmed using PCVM (1) for the Cu − Al diagram of state [13, 14].

The authors [26] gave experimental values for the viscosity of the Cu − Sn alloy. The researchers measured the dynamic viscosity of melts containing 10, 20, 25, 32, 38, 50, 60, 65 and 77 wt% Sn at various temperatures. The alloy viscosity isotherms show a maximum in the region of 30−35 wt.% Sn. It is noted that this value corresponds to the existence areas of intermetallic phases γ ($Cu_{31}Sn_8$) and ε (Cu_3Sn) in the state diagram. At concentration of 60 wt.% Sn there is the phase η (Cu_6Sn_5) in solid state.

In [27] the authors obtained data on dynamic viscosity of liquid copper, tin and Cu − Sn alloy with 10, 20, 30, 32, 35, 38, 40, 50, 60 wt.% Sn at temperatures of 800, 900 and 1000 °C. The presence of two maximums in the region of 32−40 wt.% Sn is indicated, which are associated with the formation of Cu_4Sn, Cu_8Sn_2, and Cu_6Sn_2 clusters. However, there are no experimental data after the point corresponding to the 60 wt.% tin content except for the
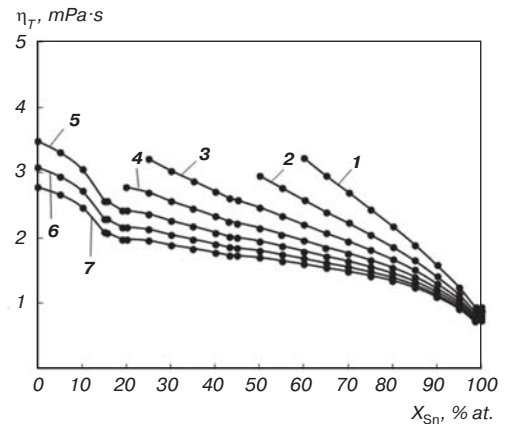

Fig. 2. Temperature dependence of Cu − Sn alloy viscosity according to the partial-cluster model:
1 − 700, *2* − 800, *3* − 900, *4* − 1000, *5* − 1100, *6* − 1200, *7* − 1300 °C

point at 100 wt.% Sn, which is connected by a straight line after 75 wt.% Sn.

Similar results were obtained by the authors of [28] on the dynamic viscosity of Cu − Sn melt at temperatures of 1100, 1200 and 1300 °C. The viscosity peak area is at the concentration at 15−30 at.% Sn. The authors explain this by the weakening of the interaction between Cu and Sn atoms. In the same paper, the authors note that such changes correspond to the extreme values of the enthalpy of mixing, that is, the formation of intermetallic compounds and their destruction when heating the alloy. And here after 65 wt.% Sn there is no data, but the extrapolation to 100% is made curvilinear.

A comparison of all the data in **Fig. 2** and [26−28] reveals quite clear differences:

a) experimental data on Cu − Sn viscosity isotherms form maximums in the 15−40 wt.% and less unambiguously at 60 wt% Sn;

b) values of the maximum exceed the viscosity of the refractory component - copper.

Thus, the repetition of the general features of the liquidus line and viscosity isotherms is supplemented at some Cu: Sn ratios, although the smoothing and convergence of the isotherms with increasing temperature is observed. Therefore, it is necessary to find and consider the reason of the discussed regularity.

In terms of thermodynamics, the reason for the investigated features is the formation of associated unstable complexes of the Cu_xSn_y type, which are statistically defined chemical groupings without the formation of their own phase. These complexes are in dynamic equilibrium with the solution matrix, characterized by a disorderly distribution of atoms. The formation of such groups is accompanied by the release of heat. The papers [29−31] discuss various features of this process.

The thermodynamic properties of the Cu-Sn system phases have been studied in numerous papers and are quite consistent [32−36]. The results of thermodynamic calculations involving all phases occurring in the Cu − Sn system are given in [37−41].
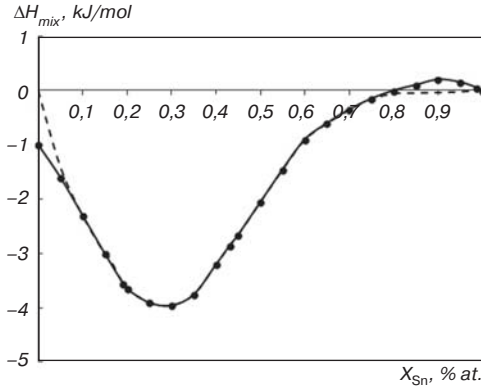
Fig. 3. Enthalpy of mixing of Cu − Sn melts at different temperatures: solid line − enthalpy of mixing for $T = 1373$ K from [12], dashed lines − with extrapolation on the boundaries to $\Delta H_{mix} = 0$ kJ/mol without regard to positive values in the region of $0.75-1$ at.% Sn

The thermodynamic description of the system [41] adopted in this paper was made by the same authors who established the accepted version of the state diagram [12]. This version of the calculation is consistent with experimental data on phase equilibria.

In [12, 18] the results of a detailed theoretical calculation of the enthalpy of mixing from [31] are presented to analyze the scattered data on the thermodynamics of Cu − Sn alloy mixing and further reveal their relationship with viscosity **(Fig. 3)**.

When comparing the dependence of the enthalpy of mixing on the composition and temperature dependences of viscosity, there is a correlation in the region of maximum formation according to the authors [26−28]. In [12] and [26] it is suggested that there is a quantitative relationship between these characteristics (η_T and $\Delta_{mix}H$), especially for systems with intermetallic compounds in the solid state, as in this case Cu_3Sn, Cu_6Sn_5, Cu_2Sn and $CuSn$.

But as for the temperature dependences of the heat of mixing of Cu − Sn alloys, in contrast to the clear separation of viscosity isotherms by [26−28], the heat of mixing isotherms are difficult to distinguish. First, six isotherms are presented by six authors [34−39] at five temperatures, including two at 1400 K, by different authors. Second, there is no consistency in the alternation of isotherms neither in the full range of composition, nor in the region of minimum heat of mixing, and there are intersections of isotherms.

Third, the mixing heat values for each alloy composition should decrease with increasing temperature, especially in the minimum region, but the lowest value of $\Delta_{mix}H$ on these isotherms (-6 kJ/mol) refers to $T = 1393$ K, and the highest (-4 kJ/mol) refers to $T = 1373$ K. In addition, the extrapolation to pure copper in [12] is for some reason shifted to a point with $\Delta_{mix}H = -1$ kJ/mol. As for the area of $60-100$ wt.% Sn, there are no extrapolations. In addition, points with positive $\Delta_{mix}H$ values appeared in it without any explanation.

Our idea of the relationship between the viscosity of pure substances and their alloys can be expressed in terms of the ratio of cluster fractions for any alloy composition above the liquidus line and above the melting point of pure substances, taking into account their partial atomic fractions. In the framework of the concept of chaotized particles, this idea corresponds to the form of the basic partial-cluster model of viscosity:

$$\eta_T = X_A \dot{\eta}_{T,A} \left[\frac{1 - exp\left(\dfrac{-RT_{liq}}{RT}\right)}{1 - exp\left(\dfrac{-RT_{m,A}}{RT}\right)} \right]^2 +$$

$$+ X_B \dot{\eta}_{T,B} \left[\frac{1 - exp\left(\dfrac{-RT_{liq}}{RT}\right)}{1 - exp\left(\dfrac{-RT_{m,B}}{RT}\right)} \right]^2. \tag{4}$$

We found that, in this form, this model serves as an indicator of the various disturbances of the alloy's chaotization due to the formation of unstable chemical compounds and associates in the liquid, the same clusters, the destruction of which requires heat input, similar to what happens with pure substances, and in both cases it is necessary to consider all heat input before complete melting to prevent a break in the system's chaotization, which is displayed integratively by the liquidus lines and the melting point of substances A and B.

Thus, in pure matter, the thermal chaotization barriers to complete melting are $RT_{m,A} + \Delta H_{m,A}$ and $RT_{m,B} + \Delta H_{m,B}$, and the energy cost of overcoming the total current chaotization barrier for pure substances and alloys is the value of RT. For alloys, such barriers are the solidus $\Delta_{sol}H$ and liquidus RT_{liq} points, as well as the heat of mixing of the alloy $\Delta_{mix}H$.

These chaotization parameters are quite accessible except for $\Delta_{sol}H$, which in the first approximation can be identified with the melting heat of the pure component, $\Delta_{sol}H \approx \Delta H_m$. This approximation is necessary to ensure that the structure of the model remains the same, providing at $T = T_{m,A}$ a transition to the viscosity of substance A, and at $T = T_{m,B}$ − to the viscosity $\eta_{T,B}$ at $X_A = 1$ and $X_B = 1$ respectively:

$$\eta_T = X_A \dot{\eta}_{T,A} \left[\frac{1 - exp\left(-\dfrac{\Delta H_{m,A} + RT_{liq} - \Delta_{mix}H}{\Delta H_{m,A} + RT}\right)}{1 - exp\left(-\dfrac{\Delta H_{m,A} + RT_{m,A}}{\Delta H_{m,A} + RT}\right)} \right]^2 +$$

$$+ X_B \dot{\eta}_{T,B} \left[\frac{1 - exp\left(-\dfrac{\Delta H_{m,B} + RT_{liq} - \Delta_{mix}H}{\Delta H_{m,B} + RT}\right)}{1 - exp\left(-\dfrac{\Delta H_{m,B} + RT_{liq} - \Delta_{mix}H}{\Delta H_{m,B} + RT}\right)} \right]. \tag{5}$$
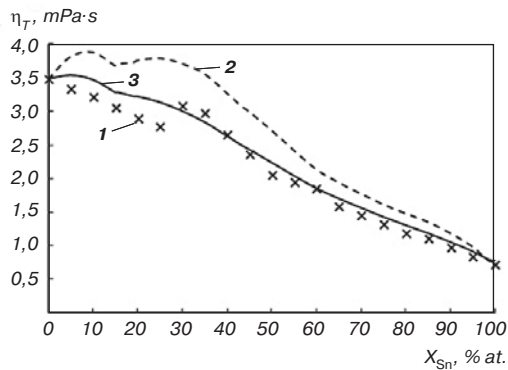
Fig. 4. Dynamic viscosity of Cu − Sn alloy: crosses (*1*) − data [26], dashed line − according to model (*2*), line − according to model (*3*) at *T* = 1373 K

In this case, the basic arguments are the liquidus line RT_{liq} and the fractional accounting of clusters as a material substrate of viscosity, extending the logic of comparison.

To verify the model, we use the reference data on the melting heat of copper $\Delta H_{m,\,Cu}$ = 12930 J/(mole·K), and tin $\Delta H_{m,\,Sn}$ = 7173 J/(mole·K) [19].

The results are shown in **Fig. 4.**

Fig. 4 shows a clear improvement in the correlation between the calculated and experimental data (*R* = 0.982), which is especially important in the area of the viscosity maximum with some displacement and smoothing by the model (2). Exactly in this region (0-0.30) the influence of the factor RT_{liq} (decreasing) and the factor $\Delta_{mix}H$ (increasing) can create their own extremes and deform the position of the maximum $\Delta_{mix}H$. Moreover, even at the maximum point, the contribution of $\Delta_{mix}H$ (as a negative value) from the algebraic sum of ($RT_{liq} - \Delta_{mix}H$) is about 1/3, which emphasizes the dominance of the liquidus factor. In general, we can be confident in the adaptability of PCVM (1) to account for various kinds of interference when identifying the relationship between the melt viscosity of metallic alloys and the liquidus of the phase diagram.

Thus, the proposed modification of the partial-cluster model is the result of an adaptation of the first model, in which the average integral value of the alloy thermal energy at the liquidus temperature RT_{liq} (in the numerator) and in pure components RT_m (in the denominator) was used as the compared chaotization barrier. In the obtained modification the thermochemical quantities RT, ΔH_m, $\Delta_{mix}H$ in some or other combinations for thermal expression of chaotization ΔH_{ch} (in fact as entropy), but always involving the parameter RT_{liq}, and in accordance with the meaning of equilibrium characteristics of phase state diagrams, Boltzmann distribution (energy spectrum) and the concept of chaotic particles.

Conclusion

For Cu − Sn melt, a quantitative relationship between the liquidus line and the fraction of clusters, i.e. thermodynamically unstable virtual formations of the solid phase in the liquid one was established from their phase diagram to express the temperature dependence of dynamic viscosity both for each alloy composition and in their full range (in isotherms).

Within the concept of chaotic particles, clusters are dependent on the size distribution as a function of temperature, and the cluster part of this dependence correlates with the temperature dependence of the dynamic viscosity. The correlation is also known between the liquidus temperature and the viscosity of the liquid alloy, although it is not expressed analytically. Combining these correlations allowed the use of quantitative expression of the first correlation with the second correlation, which received a more stringent general expression to be verified. It should be noted that the concept of the P_{cl} cluster fraction we use for the first time refers to the quantification of the content of clusters, which represent the material substrate of the fluid.

A sufficiently adequate mathematical model containing only the thermodynamic parameters of the pure components and their alloy with the basic characteristic RT_{liq} as the average integral heat of the melt at the liquidus temperature T_{liq}, which initiates melt chaotization upon further increase in temperature, has been obtained. It also serves as a kind of indicator in the simplest modification of the partial-cluster model to identify various disturbances, overcoming which requires the expenditure of thermal energy RT to chaoticize any intermetallic formations and reduce viscosity. In general, obtained results can serve as a confirmation and summarized representation of the influence of a variety of structures on the shape of the liquidus.

The structure of the developed and used model is characterized by additivity of chaotization, which goes through the concept of chaotic particles to the equilibrium distribution (energy spectrum) of Boltzmann. It is capable of predicting extrapolation results into the high temperature region and is open to validation not only on known binary systems, but also on *n*-dimensional.

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