Surface Properties of Melts Based on Tin

Yu N Kasumov¹, R A Kutuev², A R Manukyants¹ and V A Sozaev¹

¹ North Caucasus Mining and Metallurgical Institute (State Technological University), 44 Nikolayev street, Vladikavkaz, 362021, Russia
² Chechen State University, 17a Dudayev Boulevard, Grozny, 364060,Russia,

E-mail: kasumov60@mail.ru, kra-07@mail.ru, artmanukyants@mail.ru, sozaeff@mail.ru

Abstract. This paper presents the results of the surface properties of the tin-based melts. It has been shown that the best wetting of Al-4 at. % Li grade aluminum, silicon, and piezoceramics are achieved by the eutectics and terectics of tin-aluminum, tin-zinc, and aluminum alloys, respectively. Preliminary photon annealing of the substrates for 3–4 sec improves the wetting of silicon and ceramics. Solder wetting is also improved by the addition of copper and nickel micro powders, alkaline, and alkaline earth elements.

1. Introduction

Reliable data on the surface properties of tin-based alloys are needed to design lead-free solders for aluminum brazing. Knowledge of the surface properties of alloys, such as Sn-Bi and Sn-Sb, is also of interest in connection with the fact that these alloys are promising as anode materials for lithium-ion batteries [1].

Solders based on the Sn-Zn system are widely used for soldering laptop nodes [2]. Similar studies, using Sn-Ag based solders, were carried out in [3-6]. However, surface properties (surface tension, wetting angles, spreading kinetics), as well as patterns of formation of intermetallic compounds using such solders, have not been studied enough.

Particular attention in the study of tin-based surface properties is given to eutectic or near-eutectic solders of the Sn-Ag system: Sn-Ag-Cu, Sn-Ag-Bi, Sn-Ag-Sb, Sn-Cu, Sn-Ag-Cu-Ni; and also Sn-Bi systems: Sn-Bi-Cu. The use of small additives, for example, Cu and Bi, allows lowering the surface tension and reduce the rate of formation of Cu₆Sn₅ intermetallides during brazing (recommended solder is Sn-3.5Ag-0.5Cu) [7], as well as Ni₃Sn₄ intermetallides during brazing of Ni and Ni / Au metallization system [4]. Additives Bi, Sb, Ni improve mechanical properties, fluidity, regulate the temperature of solder and KTP of the soldered joint. Solders based on Sn-3.5 mass% Ag eutectic (melting point Tm ~ 221 °C) with rare-earth metals additives provide high shear strength when soldering semiconductors: Si (> 11 MPa), GaAs (> 8 MPa), GaN (> 14 MPa), diamond (> 14.5 MPa), SiC (> 7.8 MPa), dielectrics: Si₃N₄ (> 12 MPa), Ta₂O₅ (> 5.6 MPa), Al₂O₃ (> 11.5 MPa), SiO₂ (> 7 MPa) [8]. These semiconductors are used in many optical and electronic devices.

High adhesion during brazing of sapphire is achieved by Sn-0.5 wt.% Al melt [9]. Melts based on the Sn – Al system well-wet moisten aluminum nitride [10].
2. Research results

The surface tension of tin-based alloys has been studied in many works, and until 1980, these data were generalized in a reference [11]. A study of the surface tension of Sn-based alloys in recent years has been carried out in the following works: alloys based on the Sn-Ag system [12–14], alloys of the Sn-Sb system [15], alloys: Sn-Ni [16], Sn-Si [17], Sn-Al [18.19], Sn-Tl [20] and other alloys.

Theoretical estimates of the surface tension of tin-based binary and ternary alloys are usually carried out based on the Butler-Zhukhovitsky equation [21, 22]. The analysis of theoretical and experimental works on tin-based surface properties shows that they do not take into account the presence of micro and nanoparticles in alloys. At the same time, it is precisely micro- and nanostructured melts that may turn out to be the best soldiers. These solders can be widely used for soldering aluminum and its alloys.

Aluminum and its alloys are increasingly used in electronic technology. In particular, aluminum is used as bases to which ceramic substrates of integrated circuits are soldered [23, 24].

In known methods of soldering integrated circuits, fluxes are usually used, the pairs of which disable active IP elements. In this regard, it is of interest to develop solders for flux-free tinning and to solder integrated circuits with aluminum substrates. These solders should have a sufficiently high melting point, such that heating the integrated circuits during its operation does not lead to the melting of the solder. At the same time, there are restrictions from above in temperature - no higher than 300–350 °C. Otherwise, when soldering above this temperature, diffusion zones of active elements of integrated circuits can be violated.

It is also desirable that the solder be corrosion-resistant, resistant to thermal cycles, wet aluminum, and metalized ceramics well and provide a sufficiently high strength of the solder joint.

So far, not a single solder has simultaneously satisfied all these conditions. Known solders used for flux-free brazing and tinning of aluminum [25–30] contain components that either worsen the mechanical properties of the solder and aluminum product, either containing expensive metals (silver, gold) or containing volatile metals with toxic properties (lead, cadmium). These solders also have insufficient oxidation resistance.

Of great interest for flux-free brazing of aluminum are solders containing tin and aluminum. A solder (wt. %) Was proposed in [31]: Al 0–17.2, Cu 0–4.6, Zn 12.0–41.6, (Fe, Pb, Cd, Na, Si, Ca, Mg) <2.1, Sn – the rest. The solder is interesting in that it covers the composition and practices of the Sn-Zn-Al system. Using a solid-liquid peritectic state in [32], we proposed modes and methods of tinning and brazing of aluminum.

The use of the solid-liquid state of alloys [33] may turn out to be promising in the development of solders and methods for brazing aluminum and its alloys. The high strength of the solder joint can be ensured by the mechanism of creating a dispersed medium at the solder-aluminum interface.

In the case of eutectics and peritectic, the particles of the solid phase of small sizes contained in them, interacting with the surface of aluminum, are deposited on it. As a result, a dispersed medium is formed, which, due to the side effects of dissolution [34] and capillary forces, enhances the dissolution of the oxide film and accelerates mass transfer between the liquid and solid phases.

In [43], we attempted to study the polytherms of the contact angles of tin and eutectic Sn-Al melt of aluminum alloys Al + 4 at.% Li (Fig. 1), which are widely used in the aircraft industry. At a temperature of 810–820 K, a threshold for wetting the substrates with a pure tin melt is observed (Fig. 1). For curves 1 and 2, the experiment is repeated twice. When a drop melts, intense interaction with the substrate (reactive wetting) occurs with the formation of craters. After these temperatures, tin completely spreads over the substrate, \( \theta \to 0 \).

When Sn-Al is wetted with an eutectic melt, the wetting angles decrease (curve 3 in Fig. 1), and the wetting threshold is observed at a lower temperature: \( T = 765 \text{ K} \). Then again \( \theta \to 0 \).

Such a liquid-solid dispersed medium can be created artificially. To this end, powders of refractory metals with a size of 20–100 \( \mu \text{m} \) were introduced into the solder of the Sn-Al system, which would be wetted by melts of the Sn-Al system. For example, Ni was used. We proposed one such solder in the study [35].
Solders compared with the known ones provide higher strength properties of soldered joints but have low fluidity. For solving this problem, reliable data on the surface properties of solders in the region of low Al concentrations are needed. The surface tension of the Sn – Al system was first studied in [18]. However, in the region of low Al concentrations in Sn (covering the eutectic of the system), the surface tension has not been studied sufficiently. Due to the dimensional effects of physicochemical properties, the addition of micro- and nanopowders to Sn-based melts leads to the intensification of mass transfer at the interface between solids and melts, enhancement of wetting, solubility, and other processes.

An urgent problem is the development of solders and metallization systems of the piezoceramics of the TsTS brand [19].

The most significant application in metallization of ceramics was found in silvering, based on the burning of a coating 0.02–0.03 mm thick into piezoceramics. However, such a coating is not sufficiently resistant to thermocycling loads, and at high burning temperatures, the functional properties of ceramics are lost.

Therefore, this coating is underway for other metallization systems and solders for soldering PZZ piezoceramics with a lower temperature. In this regard, solders based on the Sn – Ag – Cu – Me (Me: Co, Ni, Zn, Zr, Ti) systems [36] may be of interest. But more research is needed, and especially of capillary properties (polytherm, surface tension isotherms, wetting angles).

Currently, to improve the wetting by solders of substrates, attempts are made to change the surface morphology of the substrates [37].

It was shown in [37] that pretreatment of films by photon annealing (ultraviolet radiation) in an argon atmosphere improves wetting. When tin – strontium melts spread on the surface of aluminum films on silicon, preliminarily treated with photon annealing for 4 s, wetting thresholds are detected at temperatures > 850 K. Tin – barium melts and pure tin only partially wet aluminum films on silicon [45].

Data on the polytherms of the wetting angle helps to calculate the adhesion energy, \( W_a \), of the melt to silicon, according to the Dupre formula [1–6, 37–42]. For example, at \( T = 550 \) K, estimates show that the adhesion energy of pure tin to silicon is \( W_a = 70.3 \) MJ/m\(^2\), which is consistent with theoretical calculations [45–47].

Fig. 2 presents the results of experiments on the wetting of aluminum films on silicon. The results show that pure tin does not wet the substrate in any of the studied cases. With increasing temperature, the contact angles decrease. In experiments with an unannealed aluminum film, the most considerable wetting angles were observed. Perhaps this value is associated with a massive disorder of the surface.
structure, a higher roughness. The smallest contact angles were found on substrates on which aluminum films were subjected to photon annealing for three seconds.

\[ \theta_{\text{eff}} \text{, degree} \]

![Figure 2. Polytherms of the contact angle of aluminum films on silicon with pure tin, depending on the duration of photon annealing](image)

The connection of the contact angle on a rough surface \( \theta_{\text{eff}} \) with the contact angle on a smooth surface gives the Wenzel – Deryagin equation. If the roughness coefficient \( k > 1 \), \( | \cos (\theta_{\text{eff}}) | > | \cos (\theta) | \). Thus, the surface roughness improves wetting: \( \theta_{\text{eff}} < \theta \) for \( \theta < \pi / 2 \) and worsens it for \( \theta > \pi / 2 \). In our case, \( \theta > \pi / 2 \). Therefore, a decrease in surface roughness under the action of photon annealing should improve wetting, which is observed in experiments [48].

The results can be explained by the fact that, at an annealing time of 4 s, the structure is ordered on the surface of the samples, and the aluminum film becomes smoother [49]. And at high temperatures, oxides are destroyed on aluminum films, which is confirmed by a sharp drop in contact angles. In the case of a melt, Sn-0.106 at. Sr this occurs at temperatures above 850 K, and in the case of a melt Sn-1.928 at. % Sr at \( T > 865 \) K.

In Fig. Figure 3 shows the polytherms of the contact angles of the surfaces of the substrates with tin-strontium melts. Slight wetting of aluminum films on silicon by Sn melts – 0.106 at. Sr and Sn – 1.928 at. was found at annealing times \( \tau = 0, 2, 6, \) and \( 8 \) \( \tau = 2 \) and 6 s, respectively. With a photon annealing time of aluminum films on silicon of 4 s, the wetting angles are the smallest; moreover, at temperatures \( T > 850 \) K in the case of Sn – 0.106 at. and \( T > 856 \) K in the case of Sn – 1.928 at. % Sr, the wetting angle drops sharply (to 24 ° and 20 °, respectively (shown by arrows in Fig. 3 A and 3 B) [50].

\[ \theta_{\text{degree}} \]

![Figure 3. Polytherms of the contact angles of aluminum films on silicon with tin-strontium melts, depending on the duration of photon annealing of the substrates](image)
Thus, it was found that photon annealing of aluminum films on silicon leads to a decrease in the wetting angle by pure tin and Sn – Sr melts. The maximum decrease in contact angles is achieved at annealing times corresponding to the minimum surface resistance of the films, i.e., 3..4 c.

It is interesting to note that when a droplet spreads over a substrate with an aluminum film on silicon, dendritic structures arise [50], which apparently indicates the formation of a complex eutectic in the tin-strontium-aluminum-silicon system.

Micro and nanopowders can be used in the manufacture of porous tapes, which, by impregnating with tin-based melts, can create new lead-free composite solders [44].

Thin ultrafine metal plates impregnated with solders are used as spacers for dispersion (composite) welding and brazing of metals with metals, semiconductors, and ceramics [51–59].

The data on polytherms of the contact angles of metallic melts of porous metals in the literature is insufficient.

Known results of studies of wetting angles are associated with the design of composite solders and, as a rule, contained a lead-containing low-melting component.

It should also be noted that in the literature, there are no data on the wetting of porous nickel and copper with melts containing additives of alkaline and alkaline earth elements. The presence of such additives opens the way to the creation of new composite cathodes.

We studied the polytherms of the contact angles of porous nickel (porosity of 30 %) and copper by Sn-Ba and In-Na melts.

It has been established that Sn-0.152 wets porous nickel at. % Ba, and Sn-0.396 at. % Ba at temperatures above 475 °C, while Sn-0.061 wets copper at. % Ba and Sn-0.396 at. % Ba melts already at 275 °C.

Additional thresholds for wetting nickel and copper with In-0.3 at. % Na and In-0.5 at. % Na are detected at 250 and 225 °C, respectively. Research morphology of samples after crystallization. The results showed the presence of various structures of small sizes, including intermetallic formations.

In all cases, at the first moment, the melts do not wet the substrates, which is explained, first of all, by the high oxidizability of In-Na, Sn-Ba melts, the oxide films of which are destroyed with increasing temperature. But, possibly, the gas presence in the pores and the high degree of roughness of the substrates led to the partial wetting of the Cassi-Baxter [38]. With increasing temperature and destruction of oxide films, the melts come into closer contact with a rough surface, and the Wenzel mechanism is realized [38]. As the porous substrate is saturated with melts, the heterophase substrate is wetted, and the contact angle takes the form [40]: $\cos \theta = (1-\Pi) \cos \theta + \Pi$, where $\Pi$ is the porosity, $\theta$ is the contact angle according to the Dupre formula [40]. However, this formula is valid in the absence of chemical interaction of the melts with a capillary-porous substrate.

There is no theoretical description of the porous wetting and heterophase systems during the formation of intermetallic compounds.

3. Conclusion

The surface properties of tin-based melts are analyzed to construct solders for brazing aluminum, silicon, and piezoceramics.

It is shown that the smallest contact angles are achieved using tin-aluminum eutectics and tin-zinc-aluminum peritectic with the addition of copper and nickel micro powders. A decrease in wetting angles is also achieved by preliminary photon annealing of the substrates.

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