DATA BASE OF Pb - FREE SOLDERING MATERIALS, SURFACE TENSION AND DENSITY, EXPERIMENT VS. MODELING

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ABSTRACT

Experimental studies of surface tension and density by the maximum bubble pressure method and dilatometric technique were undertaken and the accumulated data for liquid pure components, binary, ternary and multicomponent alloys were used to create the SURDAT data base for Pb-free soldering materials. The data base enabled, also to compare the experimental results with those obtained by the Butler's model and with the existing literature data. This comparison has been extended by including the experimental data of Sn-Ag-Cu-Sb alloys.

Keywords: Pb-free solders. surface tension, density, Butler's modelling

1 INTRODUCTION

During the Codata conference in Berlin 2004 (Moser, Gąsior & Dębski, 2004a) and consequently in the proceedings of this conference in electronic form (Moser, Gąsior & Dębski, 2005c) the organization of the SURDAT data base for Pb-free soldering materials was reported. SURDAT contains experimental results of surface tension, density and molar volumes as well as a comparison with thermodynamic modeling of the surface tension by Butler's method (Butler, 1932). In Table 1 there are summarized the updated investigated systems with references: (Gąsior, Moser & Pstruś, 2001); (Moser, Gąsior & Pstruś, 2001a); (Moser, Gąsior, Pstruś, Zakulski, Ohnuma, Liu et al, 2001c); (Moser, Gąsior & Pstruś, 2001b); (Gąsior, Moser, & Pstruś, 2003a); (Moser, Gąsior, Ishida, Ohnuma, Liu, Bukat et al, 2005b); (Pamies, Garcia-Cordovilla & Louis, 1984) (Nogi, Oishi & Ogino, 1989); (Gąsior, Moser & Dębski, 2004c); (Liu, Inohana, Ohnuma, Kainuma, Ishida, Moseret al, 2002); (Gąsior, Moser, Pstruś, Krzyżak & Fitzner, 2003b); (Pstruś, Moser & Gąsior, 2004); (Moser, Gąsior & Pstruś, 2005a); (Gąsior, Moser, Pstruś, Bukat, Kisiel & Sitek, 2004e); (Moser, Gąsior, Pstruś & Księżarek, 2002); (Moser, Gąsior, Pstruś, Ishihara, Liu, Ohnuma et al, 2004d); (Gąsior, Moser & Pstruś, 2004b); (Moser, Gąsior, Ishida, Ohnuma, Bukat, Pstruś et al, 2004c); (Gąsior, Moser, Bukat, Pstrus, Sitek, & Kisiel, 2004d); (Kisiel, Gąsior, Moser, Pstruś, Bukat & Sitek, 2005).

Table 1. The investigated liquid metals and alloys

Metals	References	Binary Alloys	References	Multicomponent Alloys	References
Pb	(Gasior et al, 2001)	Pb – Sn	(Gasior et al, 2001)	(Sn-Ag) _{eut} +Zn	*
Sn	(Moser et al., 2001a)	Ag – Sn	(Moser et al., 2001a)	(Sn-Ag) _{eut} +In	(Liu et al, 2002)
In	(Moser et al, 2001c)	Ag – In	(Moser et al, 2001c)	(Sn-Ag) _{eut} +Bi	(Moser et al, 2001b)
Ag	(Moser et al, 2001c)	Bi – Sn	(Gasior et al, 2004c) (Moser et al, 2001b)	(Sn-Ag) _{eut} +Cu	(Gasior et al, 2004e) (Moser et al, 2002)
Bi	(Moser et al, 2001b)	In – Sn	(Liu et al, 2002)	(Sn-Ag) _{eut} +Sb	(Moser et al, 2004d)
Sb	(Gasior et al, 2003a)	Ag – Bi	(Gasior et al, 2003b)	(Sn-Ag) _{eut} +Cu+Sb	(Moser et al, 2005a) (Gasior et al, 2004b)
Cu	(Moser et al., 2005b)	Sb – Sn	(Gąsior et al, 2003a)	(Sn-Ag) _{eut} +Cu+Bi	(Moser et al., 2005b) (Moser et al, 2004c)
Zn	*	In – Zn	(Pstruś et al, 2004)		
		Sn – Zn	(1 strus et al, 2004)		(Gasior et al, 2004d)
Al	(Pamies et al,1984)	Ag – Sb	(Moser et al, 2005a)	(Sn-Ag-Cu) _{eut} +Bi+Sb	
		Cu – Sn			(Kisiel et al, 2005)
Au	(Nogi et al, 1989)	Cu – Sb			

* not published

Butler's method requires excess Gibbs energies of the components of the investigated alloys usually available in the literature for the binary and ternary systems, and the data of the surface tension of pure components. Comparison of Butler's modeling with experimental surface tension from the Institute of Metallurgy and Materials was performed also for higher order systems with co-operation with the Tohoku University where the thermodynamic data base ADAMIS for micro-soldering alloys has been developed (Ohnuma, Liu, Ohtani & Ishida, 1999; Ohtani & Ishida, 1994). This co-operation was next extended on phase diagram calculation (Moser et al, 2001c; Liu et al, 2002) and on simulation of solidification. The enclosed two schemes dealing with the ADAMIS and SURDAT data bases show the structures and the main functioning of both data bases (Moser & Gasior, 2004b; Gasior, Moser & Dębski, 2004a).

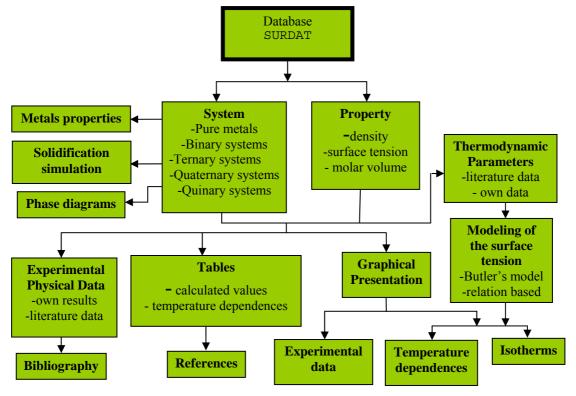


Figure 1. Scheme of the functioning of SURDAT data base

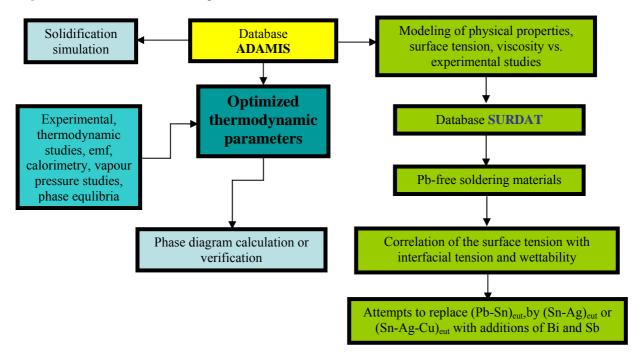


Figure 2. Presentation of the use of the ADAMIS and SURDAT data bases

More details on the functioning of the SURDAT data base have been shown in the proceedings of the Codata conference in Berlin 2004 on the example of Sn-Ag eutectic with Cu additions. It is due to the fact that both eutectics: the binary Sn-Ag and the ternary Sn-Ag-Cu are recommended as the main substitutes for traditional Sn-Pb solders. In further searching for new Pb-free soldering materials various amounts of additions to these two eutectics are tested in parallel to studies on new solders like Sn-In-Zn. Since the Roman Times we have relied on Sn-Pb solders, and as a consequence, the properties of each new Pb-free solder including also wettability are compared with Sn-Pb (eutectic temperature 183°C). Both eutectics Sn-Ag and Sn-Ag-Cu have the temperature above 200°C, and therefore also the melting temperature of new materials is an important factor, as each 10°C above 183°C introduces additional problems to adjust the industry to the new Pb-free solders.

It should be noted, that at the beginning of our co-operation with the Tohoku University we have compared experimentally measured surface tension in the protective atmosphere with the calculated values from Butler's modeling as the lowering of the surface tension by the added component is the qualitative indication of improved wettability. For practical application, usually the meniscographic studies are performed to measure the wetting time, the wetting force and to the calculate contact angles in air with the use of various fluxes. The wetting balance test can be used to observe the dynamic process of wetting by measuring the force that acts between the immersed specimen and the molten solder. In a recent publication on addition of Cu to binary eutectic Sn-Ag (Gasior et al. 2004e), we reported the joint results with industrial institutes dealing with the production and testing of Pb-free solders to combine data of the surface tension from measurements in a protective atmosphere with the results from meniscographic studies. This was possible using two different meniscographs and performing measurements on wetted and not wetted specimens in air. Tests on non - wetted specimens by the method proposed by Miyazaki (Miyazaki Mitutani, Takemoto & Matsunawa, 1997) result in interfacial tension when using the chosen flux, while without flux they exhibit surface tension. Comparison of the surface tension in protective atmosphere with surface tension in air and with interfacial tension at 250°C for various Cu additions on the example of Sn-Ag-Cu alloys (Moser et al, 2002; Gasior et al, 2004e), indicated similar trend, however taking into account that fluxes decrease interfacial tension.

The co-operation with Tohoku University has been extended to include the additions of Bi to alloys close to the ternary eutectic Sn-Ag-Cu (Moser et al, 2004c) and additions of Sb to the quaternary alloys Sn-Ag-Cu-Bi (Gasior et al, 2004d; Moser et al, 2005b; Kisiel et al, 2005) including phase diagram calculations of both systems and the simulation of solidification. The joint presentations at the mentioned conferences are now in preparation for publication.

In this updated paper on the recent proceedings of the Codata conference in Berlin 2004 the main aim is to report the data of the parallel studies on the influence of Sb, starting from surface tension and density of Sn-Sb (Gasior et al, 2003a) and next proceeding to ternary Sn-Ag-Sb (Moser et al, 2004d) and to quaternary alloys Sn-Ag-Cu-Sb. These studies undertaken within the frame of COST 531 program were influenced by literature information on the positive influence of Sb on wetting characteristics when searching for new Pb-free solders (Loomans Vaynman, Ghosh & Fine, 1994) and on the improvement of strength, thermal fatigue resistance and creep strength (Lee, Chen, Jao & Hsu, 2004). Part of these results of surface tension, density and molar volumes of quaternary alloys for dilute Sb alloys have been recently published (Gasior et al, 2004b), and together with the data for Sb, Sn-Sb and Sn-Ag-Sb then will be included in the SURDAT data base to illustrate the functioning and to compare with Butler's modeling. The other aim of these studies on Sb additions is to test in parallel to Butler's thermodynamic method, the other modeling based on the dependence of the surface tension on temperature and concentration of quaternary alloys Sn-Ag-Cu-Sb using the surface tension data of constituent systems forming the quaternary system. These results were presented during Calphad XXXIV in Maastricht, Holland this May (Moser et al, 2005a) and later on, after publication will be included in the SURDAT data base.

Also in this updated paper, as a new feature there has been introduced a comparison with surface tension data for Sn-Pb eutectic (Gąsior et al, 2001) to indicate the distance between the given Pb-free solder and the traditional Sn-Pb materials. In addition, in the updated SURDAT data base, when available, the simulation of solidification will be included to confirm the metastable character of solidified solder, evident when comparing the results of equilibrium solidification with Scheil's method.

2 Influence of Sb additions on surface tension and density of Sn-Sb, Sn-Ag-Sb and Sn-Ag-Cu-Sb alloys. Experiment vs. modeling in the SURDAT data base

After setting in motion the SURDAT software the first window (Figure 3) gives the information where the SURDAT was composed, and in the part "System selection" the kind of the systems has to be chosen to go ahead.

There are possible 5 options from pure metals (used in the investigations of lead-free solders) to the quinary systems. In the present version of the SURDAT data base the data for 10 pure metals, 12 binary, 5 ternary, 2 quaternary and 1 quinary systems are available. The surface tension of pure antimony and the binary tinantimony alloys obtained from (Gasior et al, 2003a) as stored in the SURDAT data base are shown on Figures 4 and 5.

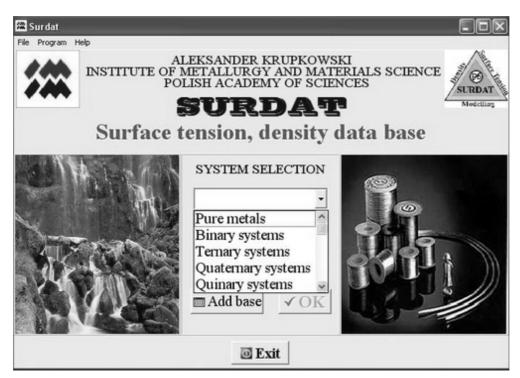


Figure 3. Selection of the kind of the system in SURDAT data base

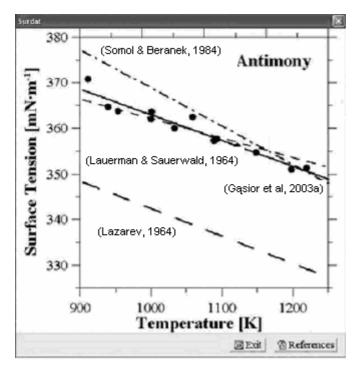


Figure 4. Comparison of the temperature dependences of the liquid antimony accessible in literature (Lazarev, 1964; Lauerman & Sauerwald, 1964; Somol & Beranek, 1984) with experimental points from (Gasior et al, 2003a)

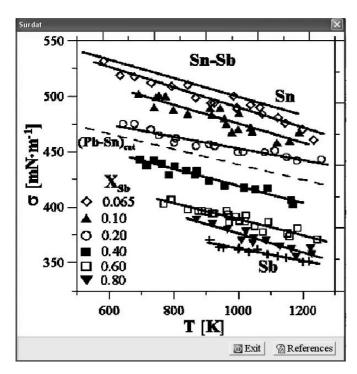


Figure 5. Temperature dependencies of the surface tension for Sb-Sn liquid alloys (Gasior et al, 2003a)

In a similar manner, in the next part of this paper the presentation of the surface tension data of Sn-Ag-Sb alloys from (Moser et al, 2004d) on Figure 6 is reported, together with results for Sn-Pb eutectic.

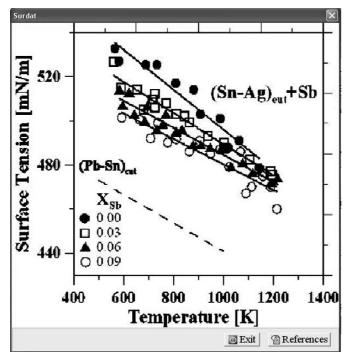


Figure 6. Temperature dependences of the surface tension of the (Ag-Sn)_{eut}+Sb alloys (least squares lines) together with the experimental results (symbols, Moser et al, 2004d). Dotted line from (Gąsior et al, 2001)

Co-operation with the Tohoku University enables to use the optimized thermodynamic parameters from ADAMIS data base not only for modeling of surface tension, but also for phase diagram calculations and simulation of solidification. This simulation of solidification is very helpful when comparing equilibrium

solidification with the Scheil's model not only to confirm the metastable process of solidification, but also to limit excessive third additions to the binary eutectic Sn-Ag. An example is shown in Figure 7 from a recent joint publication (Loomans et al, 1994) using an alloy of the composition Sn-52Sb-8Ag in mass %. In both simulations, solidification starts at 691 K with a primary crystal of (Sb). Under the equilibrium conditions, liquid phase disappears at 643 K, which is consistent with the calculated equilibrium phase diagram. However, tin concentrates into liquid phase with increasing solid phases under the non-equilibrium conditions of the Scheil model. In consequence, the terminating temperature of solidification falls drastically and is completed at 494 K, which is equivalent to Sn-Ag binary eutectic. In the case of the quaternary alloys Sn-Ag-Cu-Bi (Moser et al, 2004c) Bi accumulates in the liquid phase and similarly the decreasing of the terminating temperature corresponding to binary eutectic Sn-Bi is observed. Such behavior of the terminating liquid phase is undesirable as it causes the lifting-off failure of solder from the substrate.

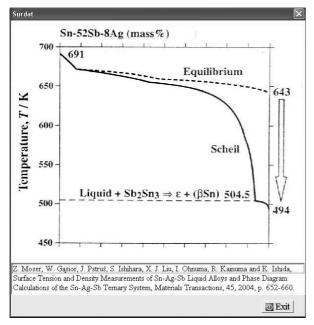


Figure 7. Solidification simulation under equilibrium conditions and the Scheil model of Sn-Ag-Sb alloys

At the next stage of the operation of the SURDAT data base there are three different possibilities of getting information on the surface tension, density or molar volume by choosing one from the three options; 1) temperature dependences, 2) isotherms and 3) Butler's model (only when the surface tension was chosen as the physical property). This will be documented on the example of the quaternary alloy $(Sn-Ag-Cu)_{eut} + Sb$.

The first option offers the possibility to show and compare in one figure the data on the temperature dependences of a selected physical property data from different papers, in the form of experimental points and least squares lines. If only one author's data are in the data base, Figure 8 is shown.

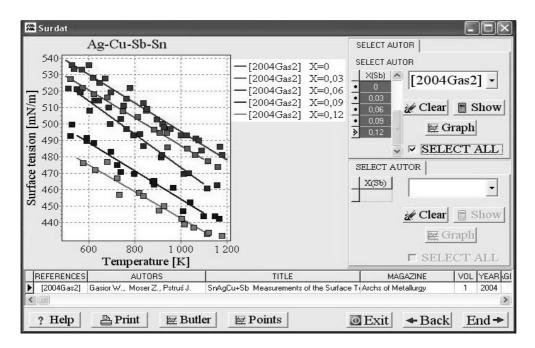


Figure 8. Temperature dependencies of the surface tension for (Sn-Ag-Cu)_{eut}+Sb liquid alloys (Gasior et al, 2004b)

When for the chosen system the results from different papers are given, there is the possibility to see and to compare in one figure all those data.

When the second option is selected, 4 temperatures at most are available and the SURDAT shows the experimental points for each temperature together with the isotherms lines calculated using the equation (worked out by the authors), describing the temperature-concentration dependence of the surface tension (Figure 9).

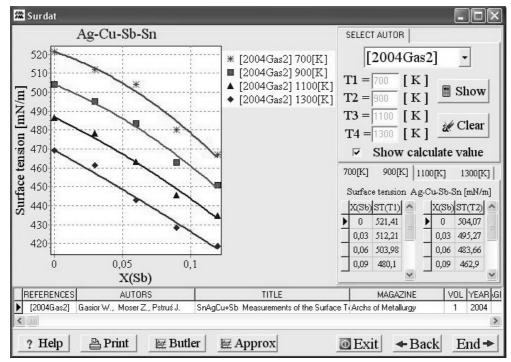


Figure 9. Isotherms of the surface tension of the (Sn-Ag-Cu)_{eut}+Sb calculated at 4 temperatures (700K, 900K, 1100K and 1300K- lines) compared with the experimental data from (Gasior et al, 2004b)

The last option - modeling of the surface tension by Butler's relation is realized after selecting: the "Butler model" button, with the available thermodynamic parameters and the experimental data from various authors (Figure 10) for comparison.

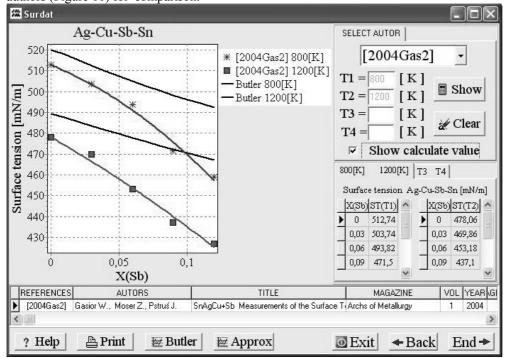


Figure 10. Comparison of the isotherms calculated from Butler's model (black lines) with those calculated from the linear relation worked out by the authors (grey lines) and the experimental data (symbols) at 800K and 1200K

There are possible two additional operations of the data base; calculation of the temperature dependences of the surface tension from thermodynamic parameters (Figure 11) or calculation of the isotherms (Figure 12).

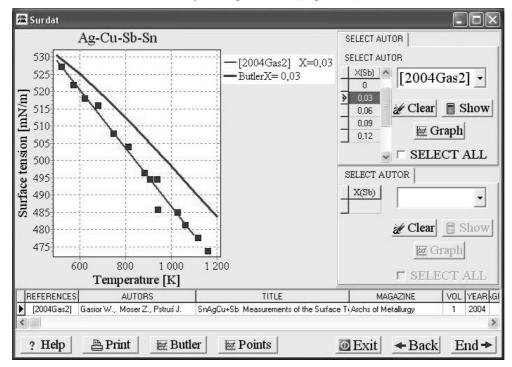


Figure 11. Temperature dependencies of the surface tension, experimental points and Butler's model only for one concentration

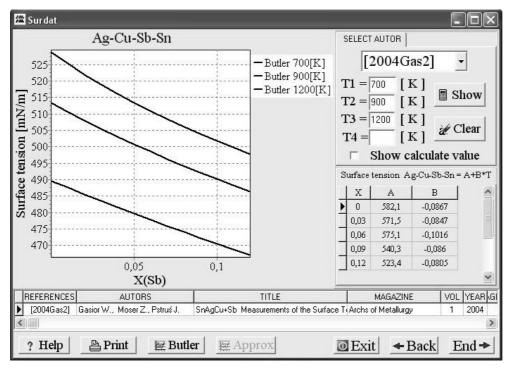


Figure 12. Calculations of surface tension for chosen three temperatures by Butler' method

Modeling of the surface tension using the thermodynamic data from different papers and comparison of the obtained results in one figure is also possible.

The same options as for the surface tension are accessible for the molar volume (Figure 13) and for density, both for temperature dependencies (Figure 14) and for isotherms (Figure 15).

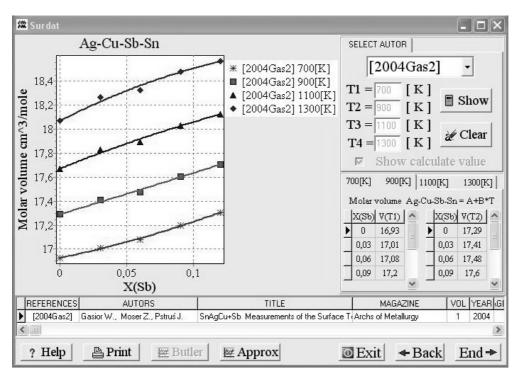


Figure 13. Isotherms of the molar volume calculated at 4 temperatures (700K, 900K, 1100K and 1300K) together with the experimental results (symbols)

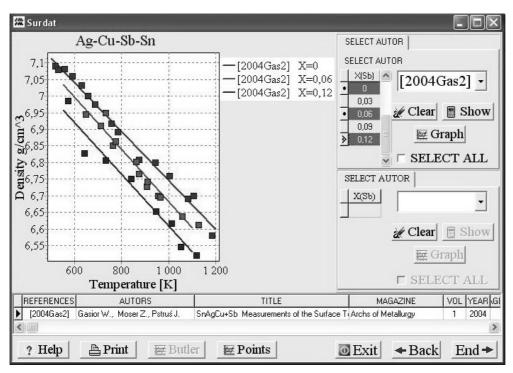


Figure 14. Temperature dependences of density for the (Sn-Ag-Cu)_{eut}+Sb liquid alloys

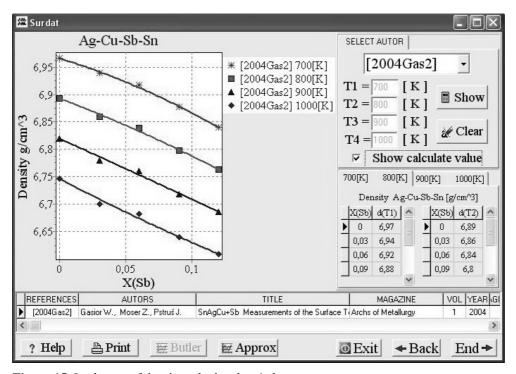


Figure 15. Isotherms of density calculated at 4 chosen temperatures

Additional information concerning the analyzed system (references, temperature, concentrations of alloys) can be obtained at every stage of the SURDAT operation in the side windows.

The co-operation of the ADAMIS with the SURDAT data bases enabled comparison of the surface tension with modeling, using Butler's method for all systems summarized in Table 1. In most cases we have found that the calculated temperature dependence of the surface tension is curvilinear, while the experimental data exhibit linear dependence. One of the possible reasons, discussed in our paper on Ag-Bi alloys (Gasior et al, 2003a), is the assumption of constant parameters in the Butler's method, which probably should be temperature dependent.

4 CONCLUSIONS

- 1. In the recent Calphad XXXIV in Maastricht (Moser et al, 2005b) application of the Butler's modeling to Sn-Sb, Sn-Ag-Sb and Sn-Ag-Cu-Sb alloys was analyzed. It has been found that in all investigated systems, calculations of surface tension by this model result in higher values than the experimental data. For these calculations from the ADAMIS data base there were accepted thermodynamic data of all 6 constituent binaries and two ternaries previously experimentally tested, namely Sn-Ag-Cu (Moser et al, 2002; Gasior et al, 2004e) and Sn-Ag-Sb (Moser et al, 2004d). The SURDAT data base enables calculations of the surface tension in the entire range of Sb additions in a quaternary system, and these results in Figure 16 are compared with experimental data from (Gasior et al, 2004b). The higher values of the surface tension from Butler's modeling were obtained in our attempts to elaborate a relation based only on surface tension. This relation, as mentioned previously, was presented during Calphad XXXIV in Maastricht (Moser et al, 2005a) and based on experimental surface tension data of 6 binaries, the same two ternaries Sn-Ag-Cu and Sn-Ag-Sb like in Butler's method and on one composition of the quaternary alloy. Calculations of the surface tension using this new relation exhibit slightly better agreement in comparison with experimental data, but require additional experimental information. After publication, these new modeling will be included into SURDAT data base. In both modeling, the differences in comparison with experimental surface tension may result also from the fact that we do not use all the required data, as for modeling of the surface tension we need 6 binaries, 4 ternaries in addition to quaternary data.
- 2. Studies on Pb-free soldering materials were particularly accelerated in the last years due to the Directive 2002/95/EC-Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment from 1st July 2006.
- 3. To replace traditional Sn-Pb solders by Pb-free soldering materials different properties of new materials must be studied. In the proceeding of the CODATA conference in Berlin, November 2004 and in this updated contribution, mainly wettability studies are considered. Results of joint studies undertaken at the Institute of Metallurgy and Materials Science in close co-operation with industrial institutes and the Tohoku University enabled organization of the SURDAT data base of surface tension, density and the modeling of the surface tension
- 4. For the accepted two eutectics: binary Sn-Ag and ternary Sn-Ag-Cu as the substitute for traditional Sn-Pb solders, further studies will proceed in the direction of small additions of other components (Ti, Li, Pa), as extensive amounts improving wettability are not accepted due to accumulation of the added component in the liquid phase (for instance Bi in case of Sn-Ag-Cu-Bi alloys) which causes the lifting-off failure of the solder from the substrate.
- 5. The proper choice of the new Pb-free solder is a compromise between wettability, melting temperature, electrical and mechanical properties, and the price.
- 6. The future development of the SURDAT data base will be concentrated on the creation of a software for calculating the viscosity and the molar volume of liquid alloys from their thermodynamic properties together with the physical property of pure metals. The extension of the data on mechanical and electrical properties is also considered.

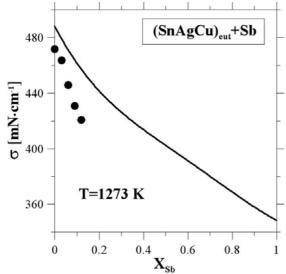


Figure 16 Isotherms of surface tension at 1273 K for quaternary alloys Sn-Ag-Cu-Sb. Experimental points are compared with Butler's model

- 7. An interest is observed for Sn-In-Zn solders, which requires further studies and the protective atmosphere for soldering. We have initiated surface tension and density studies of Sn-In-Zn and the binary systems Zn-Sn and Zn-In are included in SURDAT data base (Pstruś et al, 2004).
- 8. The authors plan to place the SURDAT software at the server of the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences to enable its presentation to a broader audience.

5 ACKNOWLEDGEMENTS

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