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Letter to Editor

ACTIVITY OF BISMUTH IN LIQUID Ag-Bi-Ni ALLOYS DETERMINED BY EQUILIBRIUM SATURATION METHOD

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Abstract

Activity coefficients of Bi in liquid Ag-Bi-Ni alloys were experimentally determined by means of the equilibrium saturation method at 1173 and 1273 K. The measurements were carried out for alloy compositions along three sections of fixed Ag to Ni mole fraction ratio equal to 4/1, 3/2, and 2/3. The excess Gibbs free energy of Bi was calculated basing on the experimental activity coefficient data. The experimental activity data were compared to the values calculated for two sets of assessed thermodynamic data.

Keywords: activity; Ag-Bi-Ni; equilibrium saturation method; liquid

1. Introduction

The Ag-Bi-Ni system attracts interest due to its importance in high temperature lead-free soldering. Bi-Ag eutectic based alloys with alloying additions and hypereutectic Bi-Ag [1-3] alloys are considered as possible replacement for Pb-Sn solders, whereas Ni is sometimes used for metallization [4]. The knowledge of the Ag-Bi-Ni phase diagram is crucial for better understanding of interfacial chemistry of such joints [1]. The only published assessment of the Ag-Bi-Ni phase diagram [5] is based on the limited amount of experimentally obtained data, namely DTA data for 7 alloy compositions and metallograpic analysis of samples annealed at four different temperatures. Although the authors [5] provided the set of thermodynamic parameters for the liquid phase, it has to be stressed that the ternary interaction parameters were estimated, as no experimental thermodynamic data for the liquid phase were available at a time. In the scope of the COST Actions 531 and MP0602, the thermodynamic databases were developed [6, 7], containing data of limiting binaries of the Ag-Bi-Ni system, so a need for the reassessment of the Ag-Bi-Ni phase diagram occurred in order to provide data for the COST MP0602 database.

The equilibrium saturation method, which was applied in this paper, belongs to a group of methods [8] enabling determination of the activity coefficient of one component in a solution. The basic condition is that (a) this component is significantly more volatile (i.e. its vapor pressure at the test temperature is a few orders of magnitude higher) than the remaining components and (b) its activity coefficient in a reference alloy is known. This method was successfully applied to determine activity of Bi in Bi-Ni alloys [9] at 1773 K. The experimental data [9] agree with the activity calculated from assessed thermodynamic data; in particular, the activity of Bi showed the positive deviation from additivity up to 0.55 mole fraction of Bi and slight negative deviation above that mole fraction [9]. In the case of the Ag-Bi system, the activity of Bi determined by electromotive force measurements [10] shows positive deviation from additivity above 0.2 mole fraction of Bi at 1000 K.

The aim of this work is to determine activity of Bi in liquid Ag-Bi-Ni alloys experimentally and compare it to the values calculated on the basis of data of Gao et al. [5], COST 531 and MP0602 databases [6, 7].

2. Experimental

The experiments were carried out by means of the equilibrium saturation method [11-14] in a set of graphite cells placed in an isothermal zone of the resistance furnace (see Fig. 1 in [13]). Half of the cells contained the studied Ag-Bi-Ni alloys, while the other half contained the reference alloys - Cu-Bi or Sn-Bi. During the experiments, the system had to attain the state of equilibrium, in which bismuth activities will be equal in all alloys. The equilibrium was executed through the gaseous phase. Since the vapor pressure

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of Bi is much higher than vapor pressure of other elements (at T=1173K: p^*_{Ag}= 8.24*10^{-4} Pa, p^*_{Bi}= 6,27*10^{-1} Pa, p^*_{Ni} = 7.41*10^{-9} Pa, at T=1273K: p^*_{Ag}= 7.35*10^{-3} Pa, p^*_{Bi}= 2.56 Pa, p^*_{Ni} = 2.17*10^{-7} Pa [15]), it can be assumed that only pure bismuth vapor was present in the gaseous phase. Due to the fact that only Bi evaporated, the amount of Ag and Ni in alloys did not change during experiments and their ratio remained fixed. Alloys of appropriate compositions were prepared by melting carefully weighed masses of metals (Ag 99.99 wt%, Bi 99.999 wt%, Ni 99.99 wt%). In order to limit evaporation of Bi during melting and ensure good mixing, the alloys were induction melted [16] in quartz crucibles. The compositions of the alloys after saturation were determined by the weighing. It had been established in the preliminary tests, that at 1173 K, the time necessary to reach equilibrium was 180 min and at 1273 K – 150 min. The argon (99.999 %) pressure was 1 hPa at 1173K and 3 hPa at 1273K, respectively. The time, necessary to reach equilibrium was not determined precisely and the saturation time in the tests was longer - 6 hours at 1173K and 5 hours at 1273K, respectively.

The bismuth loss was between 2 and 15 mass% and depended on the time of saturation. The highest bismuth loss (15%) was caused by leaving a small leak in the apparatus in order to check if the bismuth loss influences the attainment of the equilibrium. The equilibrium was reached, so the bismuth loss does not influence the attainment of the equilibrium state.

At the equilibrium state, the activity of Bi is the same in all of the samples inside a closed system. If the activity of Bi in the reference Cu-Bi and Sn-Bi alloys is known, it is possible to calculate the activity of Bi in the Ag-Bi-Ni alloy:

$$a_{Bi(Ag-Bi-Ni)} = a_{Bi(Bi-Cu)} \tag{1}$$

$$\gamma_{Bi(Ag-Bi-Ni)} = \gamma_{Bi(Bi-Cu)} \cdot X_{Bi(Bi-Cu)} / X_{Bi(Ag-Bi-Ni)} \tag{2}$$

or

$$a_{Bi(Ag-Bi-Ni)} = a_{Bi(Bi-Sn)} \tag{3}$$

$$\gamma_{Bi(Ag-Bi-Ni)} = \gamma_{Bi(Bi-Sn)} \cdot X_{Bi(Bi-Sn)} / X_{Bi(Ag-Bi-Ni)} \tag{4}$$

where $X_{Bi(Ag\text{-}Bi\text{-}Ni)}$, $X_{Bi(Bi\text{-}Sn)}$, $X_{Bi(Bi\text{-}Cu)}$ denote equilibrium mole fractions of Bi in Ag-Bi-Ni, Sn-Bi and Cu-Bi respectively, and $\gamma_{Bi(Ag\text{-}Bi\text{-}Ni)}$, $\gamma_{Bi(Bi\text{-}Sn)}$, $\gamma_{Bi(Bi\text{-}Cu)}$, are corresponding activity coefficients of Bi.

3. Results and discussion

The activities of bismuth in liquid Ag-Bi-Ni alloys at 1173 and 1273 K were determined by the equilibrium saturation method. The activity coefficient of bismuth in the alloys, $\gamma_{Bi(Ag\text{-}Bi\text{-}Ni)}$, was determined by Eq. (2) or (4). The values of activity coefficient of Bi (γ_{Bi}) in reference alloys were calculated using relations (5) and (6) and binary interaction parameters for Bi-Cu [17] and Bi-Sn [18] (see Table 1):

$$\Delta G^{ex}_{Bi} = R \cdot T \cdot \ln \gamma_{Bi} \tag{5}$$

$$\Delta G^{ex}_{Bi} = X^2_X \cdot \left[\begin{array}{l} {}^0L_{Bi,X} + {}^1L_{Bi,X} \cdot (3X_{Bi} - X_X) + \\ + {}^2L_{Bi,X} (5X_{Bi} - X_X)^2 (X_{Bi} - X_X) \end{array} \right] \tag{6}$$

where: ΔG^{ex}_{Bi} is excess Gibbs free energy of Bi, R is universal gas constant, T is absolute temperature, ${}^iL_{Bi,X}$ are binary interaction parameters, X_X is mole fraction of the second component in binary Bi-X liquid solution.

Fig. 1 illustrates the activity of Bi determined at 1173 K, whereas Fig. 2 illustrates the activity of Bi in the liquid Ag-Bi-Ni alloys determined at 1273 K. The experimental data (Tables 2-5) are represented by squares in the case of the Bi-Cu liquid reference alloy and by triangles in the case of the Bi-Sn liquid reference alloy. Continuous line corresponds to the activity of Bi calculated with the use of set of binary and ternary interaction parameters from Gao et al. [5] assessment, while the dashed line corresponds to activity calculated based on binary interaction parameters from the COST531 and COST MP0602 databases [6, 7]. There are certain discrepancies between the activities calculated based on both sets of interaction parameters. In the case of the COST dataset, the activity of Bi shows negative deviation from additivity over the whole concentration range and this negative deviation deepens with decreasing Ag to Ni mole fraction ratio. In the case of the Gao et al. [5] dataset, on the other hand, for section of X_{Ag}/X_{Al}= 4/1, the s-shaped activity dependence on Bi concentration shows the negative deviation up to about 0.4 mole fraction of Bi and positive above the mentioned fraction. For section of X_{Ag}/X_{Ni}= 3/2 the range of positive deviation is reduced to approximately 0.5 – 0.7 mole fraction of Bi, whereas for section X_{Ag}/X_{Ni} = 2/3 the activity of Bi shows negative deviation over the whole concentration range despite the s-shape being retained. It should be noticed that the difference between activities calculated from both datasets decreases with decreasing X_{Ag}/X_{Ni} ratio. The comparison between the experimental activity data and those calculated thermodynamically indicates that for sections X_{Ag}/X_{Ni} = 4/1 and 3/2 the experimental data are more similar to data calculated from the COST dataset than those calculated from the Gao et al. [5] dataset. For section X_{Ag}/X_{Ni} = 2/3 the experimental data show smaller deviation from those calculated on the basis of the Gao et al. dataset. Nevertheless, since experimental data show slight negative deviation over the whole concentration range and this deviation becomes stronger with decreasing X_{Ag}/X_{Ni} ratio, it can be concluded that the set of thermodynamic interaction parameters from the COST databases better describes thermodynamic properties of liquid Ag-Bi-Ni alloys.

Table 1. *Thermodynamic parameters for the liquid phase used in calculation*

system	Thermodynamic parameters (J/mol)	Reference
Bi-Cu	${}^{0}L_{Bi,Cu} = 23844.75 - 9.84341 \cdot T$ ${}^{1}L_{Bi,Cu} = -1260.32 - 1.19289 \cdot T$	Teppo et al. [17]
Bi-Sn	${}^{0}L_{Bi,Sn} = 500 + 1.5 \cdot T$ ${}^{1}L_{Bi,Sn} = -100 - 0.135 \cdot T$	Vizdal et al. [18]
Ag-Bi-Ni (Gao et al.)	${}^{0}L_{Ag,Bi} = 4589.8 + 23.73047 \cdot T - 3.93814 \cdot T \cdot \ln(T)$ ${}^{1}L_{Ag,Bi} = -5716.6 - 0.91452 \cdot T$ ${}^{2}L_{Ag,Bi} = -2630.2 + 0.88522 \cdot T$	Kattner et al. [19]
	${}^{0}L_{Ag,Ni} = 54055.6 - 2.2 \cdot T$ ${}^{1}L_{Ag,Ni} = -1910$	Liu et al. [20]
	${}^{0}L_{Bi,Ni} = 15000 - 3.316 \cdot T$ ${}^{1}L_{Bi,Ni} = -17000$	Vassilev et al. [21]
	${}^{0}L_{Ag,Bi,Ni} = 10000 - 190 \cdot T$ ${}^{1}L_{Ag,Bi,Ni} = -5000$ ${}^{2}L_{Ag,Bi,Ni} = 50000 - 130 \cdot T$	Gao et al. [5]
Ag-Bi-Ni (COST)	${}^{0}L_{Ag,Bi} = 3340.81 + 39.1675 \cdot T - 5.96988 \cdot T \cdot \ln(T)$ ${}^{1}L_{Ag,Bi} = -5485.45 - 1.07133 \cdot T$ ${}^{2}L_{Ag,Bi} = -3055.34 + 1.77449 \cdot T$ ${}^{0}L_{Ag,Ni} = 63052.7 - 7.11927 \cdot T$	COST531 [6]
	${}^{1}L_{Ag,Ni} = 4654.55 - 3.63684 \cdot T$ ${}^{0}L_{Bi,Ni} = -6440 + 13.288 \cdot T$	COST531 [6]
	${}^{1}L_{Bi,Ni} = -11315 - 1.457 \cdot T$	Vassilev et al. [9]

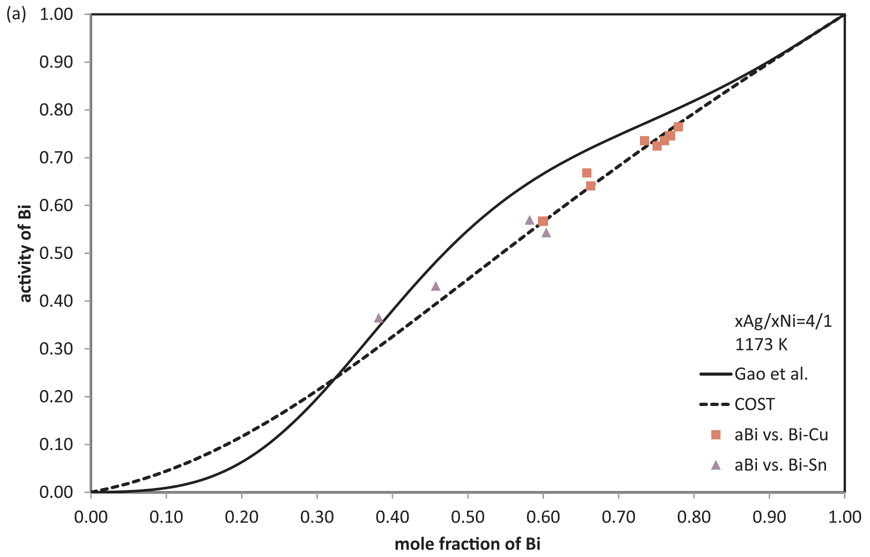

Figure 1. *Activity of Bi in the liquid Ag-Bi-Ni alloys at 1173 K for X_{Ag}/X_{Ni} constant ratio, respectively: (a) 4/1*

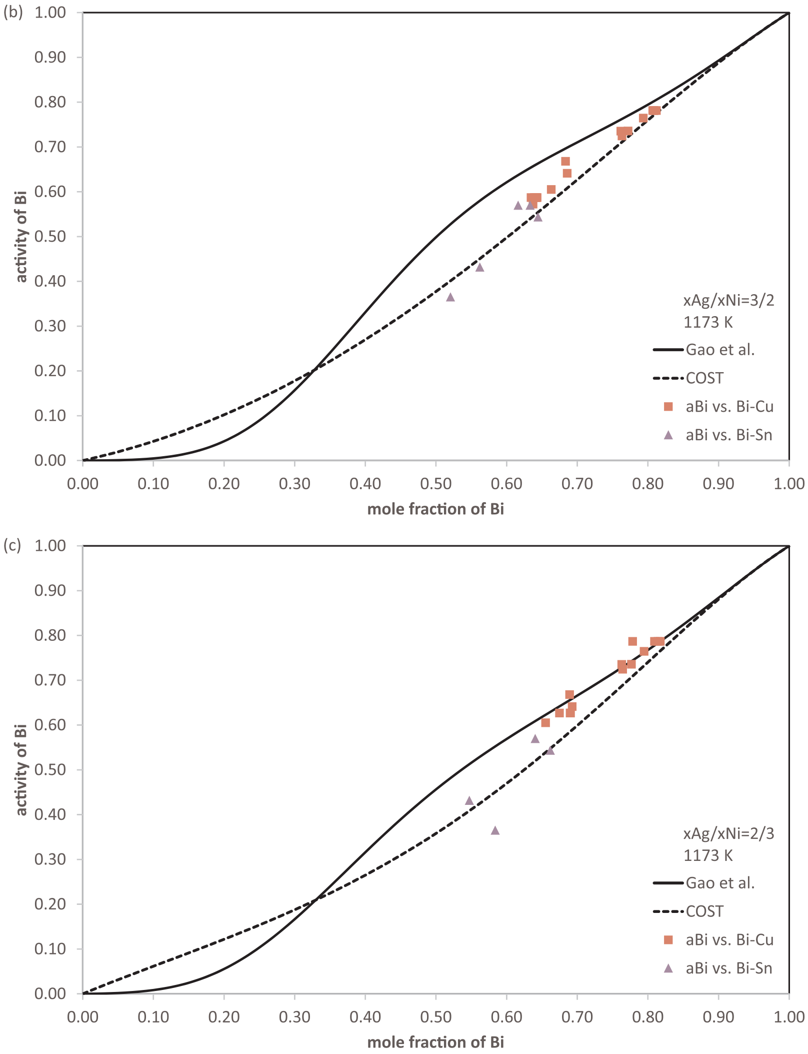

Figure 1. *Activity of Bi in the liquid Ag-Bi-Ni alloys at 1173 K for* X_{Ag}/X_{Ni} *constant ratio, respectively: (b) 3/2, (c) 2/3.*

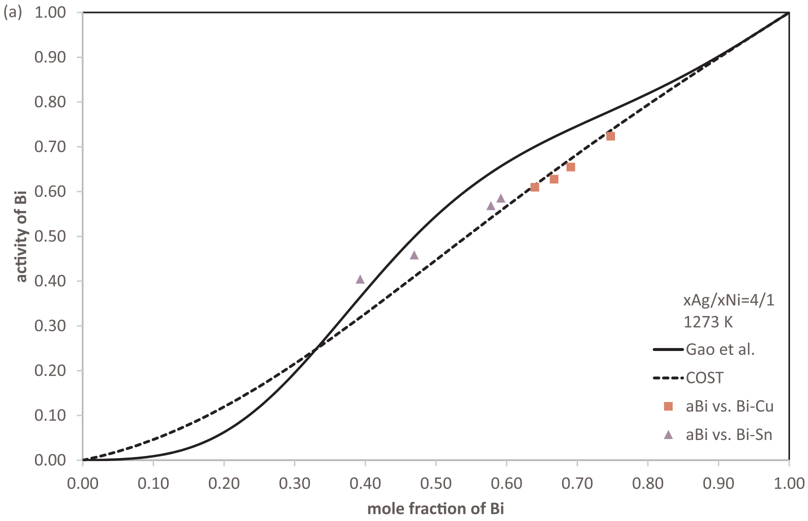

Figure 2. *Activity of Bi in the liquid Ag-Bi-Ni alloys at 1273 K for* X_{Ag}/X_{Ni} *constant ratio, respectively: (a) 4/1*

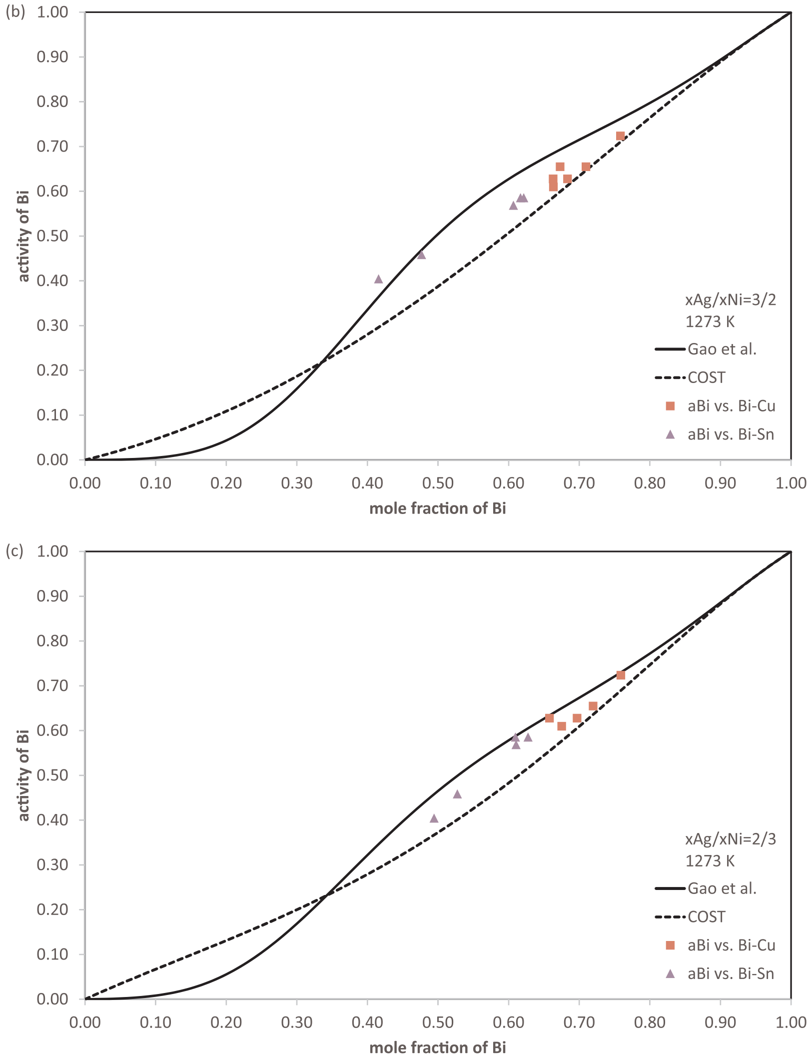

Figure 2. *Activity of Bi in the liquid Ag-Bi-Ni alloys at 1273 K for* X_{Ag}/X_{Ni} *constant ratio, respectively: (b) 3/2, (c) 2/3.*

Table 2. *Activity coefficient of Bi, activity of Bi, excess Gibbs free energy of Bi in the liquid Ag-Bi-Ni alloys determined at 1173 K with Bi-Cu as the reference alloy*

X_{Ag}/X_{Ni}	X_{Ag}	X_{Bi}	X_{Ni}	γ_{Bi}	$X_{Bi(Bi\text{-}Cu)}$	$\gamma_{Bi(\text{Bi-Cu})}$	a_{Bi}	$\Delta G_{Bi}^{ex}\ (J/mol)$
4/1	0.1764	0.7792	0.0444	0.9811	0.7215	1.0597	0.7645	-186
	0.1846	0.769	0.0465	0.97	0.6933	1.0759	0.7459	-297
	0.1851	0.7682	0.0466	0.971	0.6933	1.0759	0.7459	-287
	0.1858	0.7675	0.0467	0.9719	0.6933	1.0759	0.7459	-278
	0.1912	0.7607	0.0481	0.967	0.6771	1.0865	0.7356	-327
	0.1991	0.751	0.0499	0.9648	0.6591	1.0994	0.7246	-350
	0.2122	0.7343	0.0535	1.0016	0.6769	1.0866	0.7355	16
	0.2696	0.6628	0.0676	0.9672	0.5019	1.2774	0.6411	-325
	0.2739	0.6577	0.0685	1.0156	0.5566	1.1999	0.6679	151
	0.3198	0.6001	0.0801	0.9456	0.3459	1.6404	0.5675	-545
	0.3204	0.5995	0.0801	0.9466	0.3459	1.6404	0.5675	-535
	0.3207	0.599	0.0803	0.9474	0.3459	1.6404	0.5675	-527

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3/2	0.1131	0.812	0.075	0.9622	0.7458	1.0477	0.7813	-375
	0.1147	0.8088	0.0765	0.966	0.7458	1.0477	0.7813	-338
	0.1149	0.8083	0.0768	0.9666	0.7458	1.0477	0.7813	-331
	0.1159	0.8067	0.0773	0.9685	0.7458	1.0477	0.7813	-312
	0.1239	0.7934	0.0827	0.9636	0.7215	1.0597	0.7645	-362
	0.1368	0.7718	0.0914	0.9531	0.6771	1.0865	0.7356	-469
	0.142	0.7633	0.0947	0.9493	0.6591	1.0994	0.7246	-507
	0.1433	0.7616	0.095	0.9657	0.6769	1.0866	0.7355	-340
	0.1899	0.6834	0.1267	0.9774	0.5566	1.1999	0.6679	-223
	0.1883	0.6856	0.126	0.9351	0.5019	1.2774	0.6411	-654
	0.2021	0.6632	0.1347	0.9123	0.4239	1.4272	0.605	-895
	0.2138	0.6431	0.1431	0.9129	0.3856	1.5226	0.5871	-889
	0.2151	0.6414	0.1435	0.9154	0.3856	1.5226	0.5871	-862
	0.2165	0.6417	0.1417	0.9149	0.3856	1.5226	0.5871	-868
	0.2166	0.6391	0.1443	0.9186	0.3856	1.5226	0.5871	-828
	0.2175	0.6375	0.145	0.8977	0.3553	1.6106	0.5723	-1052
	0.2192	0.6346	0.1462	0.9251	0.3856	1.5226	0.5871	-759
2/3	0.0732	0.8174	0.1094	0.9627	0.7537	1.0442	0.7869	-370
	0.0743	0.8143	0.1114	0.9663	0.7537	1.0442	0.7869	-334
	0.0762	0.8094	0.1144	0.9723	0.7537	1.0442	0.7869	-274
	0.0821	0.7946	0.1233	0.9621	0.7215	1.0597	0.7645	-377
	0.0886	0.7784	0.1331	1.011	0.7537	1.0442	0.7869	107
	0.0894	0.7767	0.1339	0.9471	0.6771	1.0865	0.7356	-530
	0.0943	0.7642	0.1415	0.9481	0.6591	1.0994	0.7246	-520
	0.095	0.7629	0.1421	0.9642	0.6769	1.0866	0.7355	-356
	0.123	0.6928	0.1842	0.9254	0.5019	1.2774	0.6411	-756
	0.1239	0.6905	0.1856	0.9078	0.4714	1.3299	0.6268	-944
	0.124	0.6899	0.1861	0.9086	0.4714	1.3299	0.6268	-935
	0.1243	0.6891	0.1866	0.9692	0.5566	1.1999	0.6679	-305
	0.1301	0.6748	0.1951	0.9289	0.4714	1.3299	0.6268	-719
	0.1379	0.6553	0.2068	0.9233	0.4239	1.4272	0.605	-778

Table 3. *Activity coefficient of Bi, activity of Bi, excess Gibbs free energy of Bi in the liquid Ag-Bi-Ni alloys determined at 1173 K with Bi-Sn as the reference alloy*

X_{Ag}/X_{Ni}	X_{Ag}	X_{Bi}	X_{Ni}	γ_{Bi}	$X_{Bi(Bi\text{-}Cu)}$	$\gamma_{Bi(\mathrm{Bi\text{-}Cu})}$	a_{Bi}	$\Delta G^{ex}_{Bi}\ (J/mol)$
4/1	0.4939	0.3817	0.1244	0.9564	0.3302	1.1054	0.365	-435
	0.4334	0.4574	0.1092	0.9427	0.3988	1.0812	0.4312	-576
	0.3341	0.5819	0.0841	0.9788	0.5465	1.0421	0.5695	-209
	0.3168	0.6039	0.0793	0.8993	0.518	1.0484	0.5431	-1035
3/2	0.2883	0.5206	0.1912	0.7012	0.3302	1.1054	0.365	-3462
	0.2626	0.5622	0.1752	0.7671	0.3988	1.0812	0.4312	-2586
	0.2199	0.6332	0.1469	0.8994	0.5465	1.0421	0.5695	-1035
	0.2133	0.6445	0.1422	0.8427	0.518	1.0484	0.5431	-1669
	0.2303	0.6163	0.1535	0.9241	0.5465	1.0421	0.5695	-770
2/3	0.1668	0.5838	0.2495	0.6253	0.3302	1.1054	0.365	-4580
	0.1809	0.5472	0.2719	0.7881	0.3988	1.0812	0.4312	-2322
	0.1439	0.6405	0.2156	0.8892	0.5465	1.0421	0.5695	-1145
	0.1352	0.6619	0.2029	0.8205	0.518	1.0484	0.5431	-1929

Table 4. *Activity coefficient of Bi, activity of Bi, excess Gibbs free energy of Bi in the liquid Ag-Bi-Ni alloys determined at 1273 K with Bi-Cu as the reference alloy*

X_{Ag}/X_{Ni}	X_{Ag}	X_{Bi}	X_{Ni}	γ_{Bi}	$X_{Bi(Bi\text{-}Cu)}$	$\gamma_{Bi(\mathrm{Bi\text{-}Cu})}$	a_{Bi}	$\Delta G^{ex}_{Bi}\ (J/mol)$
4/1	0.2877	0.6398	0.0725	0.9528	0.4963	1.2284	0.6096	-512
	0.2469	0.6909	0.0622	0.9479	0.5735	1.142	0.6549	-566
	0.2659	0.6672	0.0669	0.9409	0.5278	1.1893	0.6277	-645
	0.2021	0.7473	0.0506	0.9684	0.6789	1.0659	0.7236	-340
3/2	0.2024	0.6634	0.1342	0.9189	0.4963	1.2284	0.6096	-895
	0.1743	0.7095	0.1163	0.923	0.5735	1.142	0.6549	-848
	0.1898	0.6834	0.1268	0.9186	0.5278	1.1893	0.6277	-899
	0.145	0.7584	0.0967	0.9542	0.6789	1.0659	0.7236	-496
	0.2022	0.663	0.1348	0.9467	0.5278	1.1893	0.6277	-579
	0.1962	0.6729	0.1308	0.9732	0.5735	1.142	0.6549	-288
2/3	0.1301	0.6753	0.1946	0.9027	0.4963	1.2284	0.6096	-1083
	0.112	0.7197	0.1683	0.91	0.5735	1.142	0.6549	-999
	0.1213	0.697	0.1817	0.9006	0.5278	1.1893	0.6277	-1108
	0.0964	0.759	0.1447	0.9535	0.6789	1.0659	0.7236	-504
	0.1369	0.6578	0.2053	0.9543	0.5278	1.1893	0.6277	-495

Table 5. *Activity coefficient of Bi, activity of Bi, excess Gibbs free energy of Bi in the liquid Ag-Bi-Ni alloys determined at 1273 K with Bi-Sn as the reference alloy*

X_{Ag}/X_{Ni}	X_{Ag}	X_{Bi}	X_{Ni}	γ_{Bi}	$X_{Bi(Bi\text{-}Cu)}$	$\gamma_{Bi(\mathrm{Bi\text{-}Cu})}$	a_{Bi}	$\Delta G^{ex}_{Bi}\ (J/mol)$
4/1	0.4851	0.3927	0.1222	1.0288	0.3705	1.0907	0.4041	301
	0.424	0.4692	0.1068	0.9766	0.4273	1.0724	0.4582	-251
	0.3261	0.5918	0.0821	0.9888	0.5634	1.0386	0.5852	-120
	0.3377	0.5777	0.0846	0.9834	0.545	1.0424	0.5681	-177
3/2	0.3513	0.4158	0.2329	0.9719	0.3705	1.0907	0.4041	-302
	0.3139	0.4767	0.2094	0.9613	0.4273	1.0724	0.4582	-418
	0.227	0.6214	0.1516	0.9416	0.5634	1.0386	0.5852	-637
	0.2359	0.6067	0.1574	0.9364	0.545	1.0424	0.5681	-696
	0.2299	0.6168	0.1532	0.9487	0.5634	1.0386	0.5852	-558
2/3	0.2026	0.4944	0.303	0.8172	0.3705	1.0907	0.4041	-2137
	0.1889	0.5274	0.2838	0.8689	0.4273	1.0724	0.4582	-1487
	0.1491	0.6275	0.2234	0.9326	0.5634	1.0386	0.5852	-739
	0.1557	0.6106	0.2337	0.9304	0.545	1.0424	0.5681	-763
	0.1562	0.6096	0.2342	0.9599	0.5634	1.0386	0.5852	-434

4. Conclusion

Activity of Bi in liquid Ag-Bi-Ni alloys was studied by the equilibrium saturation method for three sections of X_{Ag}/X_{Ni} equal to 4/1, 3/2, 2/3, respectively, at 1173 and 1273 K. The experimental data were found to show slight negative deviation from additivity which deepens with decreasing X_{Ag}/X_{Ni} ratio. Presented experimental results agree with activities calculated from the COST dataset of thermodynamic interaction parameters better than with activities calculated on the basis of the set of parameters assessed by Gao et al. [5] Therefore, the COST dataset should be preferred for the future phase diagram reassessment of the Ag-Bi-Ni system.

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