DTA MEASUREMENTS IN THE TERNARY Ag-Au-Ga SYSTEM

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Abstract

In this work, the ternary Ag-Au-Ga system was studied experimentally by differential thermal analysis (DTA). The measurements were carried out along two chosen cross-sections determined by the ratio of mole fractions $X_{Ag}/X_{Ga}=1:1$ and $X_{Au}/X_{Ga}=1:1$ by applying Pegasus 404 apparatus form Netzsch. The experiments were performed at three rates: 1 K min^{-1}, 5 K min^{-1}, and 10 K min^{-1}. Next, the obtained experimental results were used to estimate the temperatures of liquidus by applying extrapolation to a zero rate. Moreover, the temperatures of invariant reactions and other phase transformations were investigated from DTA measurements which were carried out with the rate 1 K min^{-1}. Finally, the experimental results were compared with the isopleths obtained from the prediction and calculation of the phase diagram which were done by using CALPHAD method. The experimental data obtained in this work are in good agreement with the calculation results.

Keywords: Ag-Au-Ga system; DTA measurements; CALPHAD method

1. Introduction

Metals that are liquid at or near room temperature are the only materials that have both metallic and fluidic properties. Mercury is a metal with the lowest melting temperature, but it is toxic. The second one is gallium which provides low-toxicity alternatives and recently its alloys are used in many areas. It was shown that the introduction of gallium into multicomponent alloys can decrease their melting temperature and facilitate the formation of proper interconnections between high melting transition metals [1]. Schmid–Fetzer [1] suggested that Ga-based amalgams with metals like Mo, V and even transition metals like Hf, Nb and Zr can be used for soldering in high temperature applications. Alloys containing gallium may find more interesting applications. Gallium has excellent wetting properties and decreases the melting temperature [2]. Used as a solder alloy, the Cu-Ga system is a part of Cu-In-Ga-Se photovoltaics [3] and Cu-Ga-N semiconductor systems [4]. Moreover, gallium is the component of the following interesting shape memory alloys (Ga-Mn-Ni alloys exhibit the giant magneto-mechanical effect) [5, 6]. The Au-Ga-In system is especially interesting in electronics where it is encountered during the growth of Au-seeded III-V semiconductor nanostructures, so-called nanowires (NWs) containing Ga and In from group

III) [7-10]. Some gallium alloys have a range of properties that make them an attractive option for stretchable and soft electronics [11, 12]. Gallium alloys are used in the production of diodes [13] which are made of very thin layers of semiconductor materials. Gallium arsenide (GaAs), gallium phosphide, and gallium arsenide phosphide are used for LED manufacturing. The semiconductor materials with different impurities result in different colors of light coming from LED. To complete the device, it is necessary to bring electricity to it and from it. Thus wires must be attached onto the substrate. These wires must stick to the semiconductor and be strong enough to withstand subsequent processing such as soldering and heating. Gold and silver compounds are most commonly used for this purpose, because they form a chemical bond with the gallium at the surface of the wafer.

The ternary Ag-Au-Ga system is quite interesting from a practical point of view. The alloys based on noble metals with gallium are used mainly in three areas: medicine (especially in dentistry) [14], electronics (one among most interesting multicomponent possible lead free solder is quaternary Au-Ga-In-Sb system whose examination has just begun) [15], jewelry (special gold colors such as purple, blue, brown and black) [16-18], and they are used as a part of lead-free solders as well [19]. Thus, to gain the necessary knowledge about

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the phase diagram and melting behavior of the Ag-Au-Ga system, new experimental data are needed.

2. Literature review

The information about the ternary Ag-Au-Ga as well as its properties is missing in the monographs of Villars et al. [20], and Petzow and Effenberg [21]. In the literature one can find four experimental works related to this ternary system. The first one of Andronov et al. [2] provides information about the wettability of the Ag-Au-Ga thin films. Consequently, the ternary Ag-Au-Ga system was studied as one which can be applied as a solder. The other one, published by Jendrzejczyk-Handzlik [22], reported the results of calorimetric measurements of the enthalpy of mixing along two cross-sections of this ternary system, with X_{Ag}/X_{Ga}=1:1 and X_{Au}/X_{Ga}=1:1, as well as at two temperatures 1223 and 1323 K. The results obtained in that experimental work showed that the enthalpy of mixing in this ternary system does not have the temperature dependence. Next, Jendrzejczyk-Handzlik [23] reported the results of EMF measurements from which the activity of gallium in liquid Ag-Au-Ga system was determined. The measurements were carried out along three cross-sections, with X_{Ag}/X_{Au}=1:2, 1:1 and 2:1 in the temperature range from 1023 to 1348 K. The activity of gallium shows a negative deviation from Raoult's law in the whole range of gallium concentration in which the measurements were done. Next, the experimental data which were obtained from the calorimetric and EMF measurements [22, 23] were used in the optimization of liquid phase in this ternary Ag-Au-Ga system by using the CALPHAD method [24, 25]. Finally, in the subsequent work by Jendrzejczyk-Handzlik [26], the phase equilibria in the ternary Ag-Au-Ga system were established for two isothermal sections at 523 K and 723 K by applying SEM/EDS and XRD analysis to the equilibrated samples. No ternary compounds were found.

Thermodynamic properties and phase equilibrium data for three binary systems Ag-Au [27], Au-Ga [28] and Ag-Ga [29, 30] have been studied and can be found in the literature. They are described in details in the previous work [26].

3. Experimental

The gold rod of 2 mm in diameter (99.99 mass %) was obtained from Mint of Poland. A metallic gallium piece and indium shots (every 99.999 mass %) were obtained from Alfa Aesar, Germany. The gas atmosphere was pure argon of minimum mass fraction (99.9999 mass%) obtained from Air Products. DTA measurements were carried out on

Ag-Au-Ga alloys along two cross-sections, with X_{Ag}/X_{Ga}= 1:1 and X_{Au}/X_{Ga}= 1:1, and in the temperature range from 473 K to 1300 K. The experimental procedure of DTA measurements is the same as in the case of Au-Ga system [28], Ag-In-Sb system [31], and Ag-Cu-Ga system [32]. The samples of the alloys were prepared from pieces of pure silver, gold, and gallium metals. The weight of samples used for DTA experiments were about 300 mg. These samples were prepared with the following compositions: X_{Ag}/X_{Ga}= 1:1 for X_{Au} from 0.1 to 0.8 and X_{Au}/X_{Ga}= 1:1 for X_{Ag} from 0.1 to 0.8. The samples were placed inside the self-made DTA quartz crucibles, and then they were sealed under vacuum. The melting and annealing processes were performed in the operating furnace VMK22, Linn High Therm. The samples in the quartz-ampoules were placed in the alumina crucibles and they were held at the setting temperature of 1373 K for nearly 6 h to ensure complete melting and homogenization. Next, they were cooled slowly (2 K/min) to the temperature of 473 and annealed for 8 weeks to achieve thermodynamic equilibrium and then quenched in ice water. The overall compositions of all samples were checked by using WD-XRF method and the differences between nominal and measured compositions were negligible. The homogeneity of chosen samples was also checked by using an optical microscope. After these analyses the samples were transferred to DTA apparatus. The measurements were performed on Pegasus 404 apparatus from Netzsch, Selb, Germany. The device was hooked up to a PC in order to control all DTA procedures during the experiment by PROTEUS software, also obtained from Netzsch. The acquired data were recorded on the PC. The calorimeter was calibrated by comparison of the melting temperatures of metallic In, Sn, Pb, Zn, Al, and Au (99.999 mass% purity) with tabulated values with the accuracy of the melting temperature of ± 2°C to establish an internal calibration file. The experiments were carried out at three rates: 1 K min^{-1}, 5 K min^{-1}, and 10 K min^{-1} in two heating and cooling cycles. The working side was a ternary alloy of the chosen composition sealed in the self-made DTA quartz crucibles while the empty self-made DTA quartz crucible was the reference side. Next, the obtained results were used to estimate the temperature of liquidus by applying extrapolation to zero rate for the data obtained from heating and cooling cycles. Additionally, the temperature of phase transformations were estimated from thermal curves obtained from the experiments which were carried out with the rate equal to 1 K min^{-1}. Based on the repeated DTA measurements the overall uncertainty of the determined phase transition temperatures was estimated to be ±2 °C.

4. Results and discussion
4.1 Experimental data

As a result of DTA experiments, phase transition temperatures and the liquidus line along two selected cross-sections, namely X_{Ag}/X_{Ga}=1:1 and X_{Au}/X_{Ga}=1:1 were determined in the Ag-Au-Ga system. The temperatures of the liquidus and other phase transformations were taken from DTA curves for the first heating and cooling cycles for every studied sample (the liquidus temperatures were taken from the peak maximum on heating and on cooling from the corresponding onset). The examples of DTA heating curves recorded for a chosen composition of the ternary Ag40Au20Ga40 alloy with various heating rates: 1 K min^{-1}, 5 K min^{-1}, and 10 K min^{-1} are shown in Fig. 1. All of them showed clearly detected phase transitions. In the inset in Fig. 1 the dependence of the recorded liquidus temperature on the heating rate is shown. It demonstrated that, depending on the applied heating rate, the determined liquidus temperature may vary by more than 20 K. The liquidus temperature is shifted with the increasing heating rate towards higher temperatures. This can be explained by the process dynamics and by the detection abilities of the instrument.

The data of liquidus temperatures obtained from DTA experiments which were carried out with three rates: 1 K min^{-1}, 5 K min^{-1}, and 10 K min^{-1} for heating and cooling cycles were extrapolated to a rate which was equal to zero. Finally, the temperature of the liquidus for every analyzed ternary alloy was determined. The obtained results from DTA experiments carried out with three different rates along the chosen cross-section where X_{Au}/X_{Ga}=1:1 are shown in Figs. 2-4. Similar results were recorded for X_{Ag}/X_{Ga}=1:1 cross-section. All the obtained results from DTA experiments are gathered in Table 1 where sample compositions, the temperatures of liquidus from heating and cooling cycles and other effects from heating are given. According to the DTA results, two invariant reactions were identified, which are given in Table 2.

4.2 Thermodynamic Calculation

The prediction of ternary Ag-Au-Ga diagram by applying the CALPHAD method was done by using Pandat 2017 software [33] and is compared with the experimental data obtained in this work. At first, this prediction was based on the existing information about the assessed binary systems: Ag-Au [27], Au-Ga [28], and Ag-Ga [30]. The binary thermodynamic parameters used for the prediction of phase equilibria in the ternary Ag-Au-Ga system were taken from the previous work [26]. Then, a new set of thermodynamic parameters for the liquid phase of the
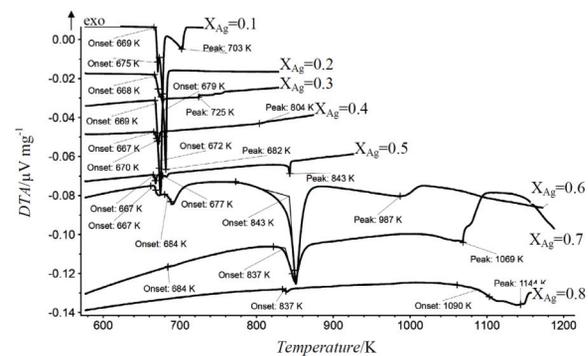
Figure 2. DTA heating curves of Ag-Au-Ga alloys, X_{Au}/X_{Ga}=1:1 with r=1 K min^{-1}
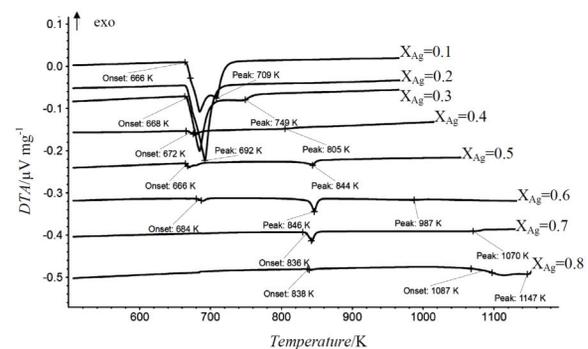
Figure 3. DTA heating curves of Ag-Au-Ga alloys, X_{Au}/X_{Ga}=1:1 with r=5 K min^{-1}

Figure 1. DTA heating curves of Ag40Au20Ga40 alloy with various heating rates
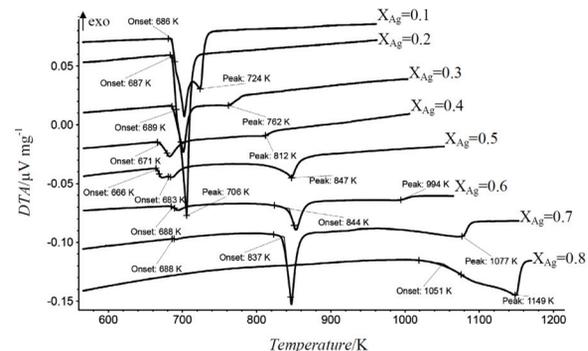
Figure 4. DTA heating curves of Ag-Au-Ga alloys, X_{Au}/X_{Ga}=1:1 with r=10 K min^{-1}

***Table 1.** Summary of measured thermal effects obtained in the Ag-Au-Ga system*

Composition X_i	mass of sample [g]	$T_{Effect\ heating}$ / K $r = 1\ K\ min^{-1}$	$T_{Liquidus\ heating}$ / K				$T_{Liquidus\ cooling}$ / K			
(i = Ag or Au)			$r = 1\ K\ min^{-1}$	$r = 5\ K\ min^{-1}$	$r = 10\ K\ min^{-1}$	$r = 0\ K\ min^{-1}$	$r = 1\ K\ min^{-1}$	$r = 5\ K\ min^{-1}$	$r = 10\ K\ min^{-1}$	$r = 0\ K\ min^{-1}$
X_{Ag} / X_{Ga} = 1:1										
0.1	0.295	588, 661	709	711	712	708	710	710	710	710
0.2	0.298	677, 683, 688	734	744	757	732	694	694	694	694
0.3	0.293	668, 679, 685	736	759	760	738	684	719	726	686
0.4	0.305	670, 678	761	762	763	760	678	695	723	671
0.5	0.294	612, 621	776	778	782	775	696	713	715	697
0.6	0.287	612	908	911	939	900	908	910	912	907
0.7	0.294	681	1069	1101	1125	1064	1072	1075	1075	1075
0.8	0.303	684	1157	1166	1167	1157	1164	1174	1175	1164
X_{Au}/X_{Ga}=1:1										
0.1	0.301	669, 675	703	709	724	699	659	666	683	645
0.2	0.298	668, 679	691	692	706	688	636	644	659	632
0.3	0.301	669, 672	713	749	762	713	680	682	695	677
0.4	0.295	667, 670	804	805	812	802	742	747	766	737
0.5	0.299	667, 677	843	844	847	842	812	857	858	816
0.6	0.298	667, 684	987	987	994	985	986	987	987	986
0.7	0.297	684	1069	1070	1077	1067	1074	1075	1077	1074
0.8	0.302	837	1144	1147	1149	1144	1143	1144	1144	1143

***Table 2.** Temperatures of identified invariant reactions in the Ag-Au-Ga system*

Composition X_{Au}	Type of reaction	Temperatures of reaction /K		
		DTA results	prediction (binary systems)	calculation (optimization of liquid phase)
X_{Ag}/X_{Ga} = 1:1				
0.1	$\zeta'+L = AuGa_2+Ag_3Ga_2$	588	575	580
0.2	-	-	-	-
0.3	-	-	-	-
0.4	-	-	-	-
0.5	$Au_7Ga_2+L=\alpha+Au_7Ga_3$	612	617	614
0.6	$Au_7Ga_2+L=\alpha+Au_7Ga_3$	612	617	614
0.7	-	-	-	-
0.8	-	-	-	-

Ag-Au-Ga system was evaluated by using the following experimental data taken from the literature: the heat of mixing data obtained along two cross-sections where x_{Ag}/x_{Ga}=1:1 and x_{Au}/x_{Ga}=1:1 at two temperatures 1223 K and 1323 K [22], the activities of gallium determined from the EMF method by using solid oxide electrolyte in the temperature range from 1023 to 1348 K [23], as well as the experimental data taken from the present work. Each piece of the selected information was given a certain weight based

on the experimental uncertainty. The optimization was carried out step by step in agreement with Schmid-Fetzer's et al. [34] guideline. Ternary interaction parameters were calculated by using ThermoCalc 2019 software [35].

The description of the thermodynamic parameters of the liquid phase was carried out in the following manner. The substitutional solution model was used to express the Gibbs free energy of the liquid solution in the following form:

$$G^M = x_{Ag} G^0_{Ag} + x_{Au} G^0_{Au} + x_{Ga} G^0_{Ga} +$$
$$RT\left(x_{Ag}\ln x_{Ag} + x_{Au}\ln x_{Au} + x_{Ga}\ln x_{Ga}\right) + G^E \tag{1}$$

where parameters denote free energy of pure component *"i"* taken from the SGTE database [36]. The function denotes the excess free energy and is described by the Redlich-Kister-Muggianu formula:

$$G^E = x_{Ag} x_{Au}\, {}^0 L^{Liquid}_{AgAu} + x_{Ag} x_{Ga} \left(\begin{array}{l} {}^0 L^{Liquid}_{AgGa} + {}^1 L^{Liquid}_{AgGa}\left(x_{Ag}-x_{Ga}\right) \\ + {}^2 L^{Liquid}_{AgGa}\left(x_{Ag}-x_{Ga}\right)^2 \end{array} \right) +$$
$$x_{Au} x_{Ga}\left({}^0 L^{Liquid}_{AuGa} + {}^1 L^{Liquid}_{AuGa}\left(x_{Au}-x_{Ga}\right) + {}^2 L^{Liquid}_{AuGa}\left(x_{Au}-x_{Ga}\right)^2 \right) +$$
$$x_{Ag} x_{Au} x_{Ga}\left(x_{Ag}\, {}^0 L^{Liquid}_{AgAuGa} + x_{Au}\, {}^1 L^{Liquid}_{AgAuGa} + x_{Ga}\, {}^2 L^{Liquid}_{AgAuGa} \right) \tag{2}$$

Parameters ${}^i L^{Liquid}_{AgAuGa}$ are dependent on the temperature and are given in J mol⁻¹. The evaluated thermodynamic parameters are gathered in Table 3. Using these optimized parameters the Ag-Au-Ga system was calculated. Finally, the experimental results obtained from DTA measurements were compared with the results of prediction and optimization, and are shown in Figs 5-6.

Table 3. *Binary and ternary interaction parameters for liquid phase*

Phase	Thermodynamic parameters /J mol⁻¹	Ref
Liquid	${}^0 L^{Liquid}_{AgAu}$=-16402+1.14T	[27]
	${}^0 L^{Liquid}_{AgGa}$=-19643.799+63.659T -8.620Tln(T)	[30]
	${}^1 L^{Liquid}_{AgGa}$=-38747.760+140.716T -16.112Tln(T)	[30]
	${}^2 L^{Liquid}_{AgGa}$=-25745.683+140.455T -17.417Tln(T)	[30]
	${}^0 L^{Liquid}_{AuGa}$=-64836.418-1.669T	[28]
	${}^1 L^{Liquid}_{AuGa}$=-26517.288+8.315T	[28]
	${}^2 L^{Liquid}_{AuGa}$=-12480.212+14.248T	[28]
	${}^0 L^{Liquid}_{AgAuGa}$=-28623-18.65T	this work
	${}^1 L^{Liquid}_{AgAuGa}$=-114876+93.69T	this work
	${}^2 L^{Liquid}_{AgAuGa}$=119584-63.5T	this work

4.3 Discussion

In the case of liquidus temperatures which were determined in the present work by extrapolation of the obtained experimental data to *r*=0 K min⁻¹, the experimental values are in good agreement with the results of the calculations in which the liquid phase was optimized. The biggest difference between liquidus temperatures obtained from the experimental results and this calculation is found for the sample from cross-section X_{Au}/X_{Ga}=1:1, where silver concentration is 0.4 mole fraction. The obtained experimental value for X_{Ag}=0.4 is 802 K while the calculated temperature is 779 K. In the case of the second cross-section the biggest difference between the temperatures of liquidus obtained from the experimental results and this calculation is found for the sample X_{Au}=0.6, where experimental value is 985 K while the calculated temperature is 955 K. However, in the case of other samples the difference between experimental liquidus temperatures and the calculation is less than 20 K. The comparison of
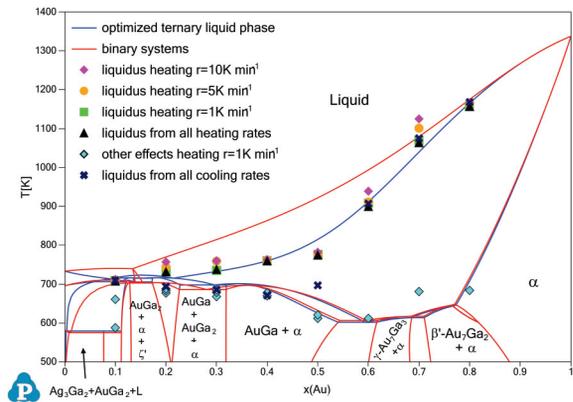
Figure 5. *Experimental data compared with the calculation of the Ag-Au-Ga system given for isoplethal section with X_{Ag}/X_{Ga}=1:1*
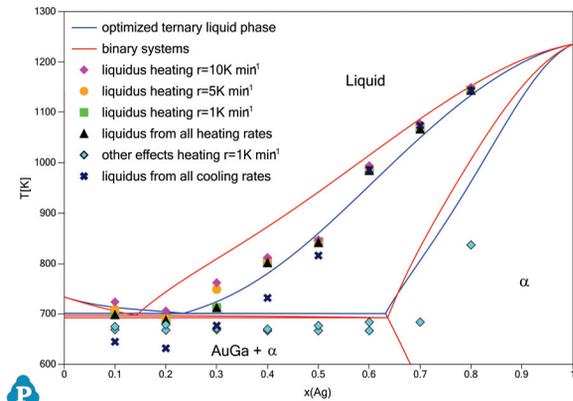
Figure 6. *Experimental data compared with the calculation of the Ag-Au-Ga system given for isoplethal section with X_{Au}/X_{Ga}=1:1*

liquidus temperatures obtained in the present work by extrapolation to r=0 K min^{-1} with the calculation of isopleths from only binary systems data shows that the difference is much larger. It must be remembered that in this prediction the ternary interactions were not taken into account. The comparison of experimental liquidus temperatures which were determined in the present work from cooling cycles with both calculations gives the following result. For alloys with following compositions: along cross-sections X_{Ag}/X_{Ga}=1:1 for X_{Au} which is in the range from 0.1 to 0.5, and along cross-sections X_{Au}/X_{Ga}=1:1 for X_{Ag} which is in the range from 0.1 to 0.5, the supercooling effect is present. For the rest of ternary alloys the experimental liquidus temperatures are in good agreement with the calculation which was done by the optimization of the liquid phase. The determined temperature of the invariant reaction ζ'+L=AuGa$_2$+Ag$_3$Ga$_2$ is based on one sample for X_{Au}=0.1 from the cross-section X_{Ag}/X_{Ga}=1:1. This invariant reaction was found at 588 K. The calculated temperature of this invariant reaction from the information taken only from binary systems is 575 K,while the calculated temperature of this invariant reaction was determined with the optimized liquid phase and it is equal to 580 K. The analysis of the experimentally determined values given in Table 3 shows that in case of this alloy the obtained temperature of this invariant reaction is higher than that obtained from both calculations. Next, the temperature of the invariant reaction Au$_7$Ga$_2$+L=α+Au$_7$Ga$_3$ was determined from the DTA analysis of two samples: X_{Au}=0.5 and 0.6, while X_{Ag}/X_{Ga}=1:1. It is equal to 612 K. In turn, the predicted temperature of this invariant reaction from only binary systems is 617 K, while from the calculation with optimized liquid phase is 614 K. It can be seen from Figs. 5-6 that in the case of the above listed invariant reactions the obtained experimental results are in good agreement with the results of both thermodynamic calculations of this ternary system.

5. Conclusions

The phase equilibria in the Ag-Au-Ga system were experimentally investigated by using differential thermal analysis. The analyses of sixteen samples along two cross-sections (X_{Au}/X_{Ga}=1:1 and X_{Ag}/X_{Ga}=1:1) gave information about liquidus temperatures, invariant reactions, as well as about other phase transitions. The comparison of these experimental results with the calculations of the Ag-Au-Ga system leads to the following conclusion: it seems that the real liquidus surface is located in good agreement with the one obtained from the calculation in which the thermodynamic properties of the liquid phase as well as the data from the present work are

taken into account. Experimentally determined temperatures of two invariant reactions are close to those obtained from the calculations. The comparison of temperatures of other phase transitions obtained from the experiment with the results of calculations showed some discrepancy.

Experimental DTA results obtained in the present work together with the other existing experimental results [22, 23, 26] will be used in the near future in the final optimization of the phase diagram of the ternary Ag-Au-Ga system by applying the CALPHAD method.

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DTA MERENJA U TROJNOM Ag-Au-Ga SISTEMU

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Apstrakt

U ovom radu trojni Ag-Au-Ga sistem je eksperimentalno ispitivan pomoću diferencijalne termijske analize (DTA). Ispitivanja su vršena duž dva izabrana preseka koji su određeni odnosom molarne koncentracije X_{Ag}/X_{Ga}=1:1 i X_{Au}/X_{Ga}=1:1 primenom Pegasus 404 aparata kompanije Netzsch. Eksperimenti su izvedeni pri vrednostima brzine zagrevanja od 1 K min-1, 5 K min⁻¹, i 10 K min⁻¹. Nakon toga su dobijeni eksperimentalni rezultati korišćeni za procenu likvidus temperature primenom ekstrapolacije do nulte brzine zagrevanja. Pored toga, temperature invarijantnih reakcija i drugih faznih transformacija ispitivane su na osnovu DTA merenja koja su rađena pri brzini od 1 K min⁻¹. Konačno, eksperimentalni rezultati su upoređeni sa vertikalnim presecima dobijenim iz predviđanja i proračuna faznog dijagrama koji su urađeni primenom CALPHAD metode. Eksperimentalni podaci u ovom radu su u saglasnosti sa rezultatima proračuna.

Ključne reči: Ag-Au-Ga sistem; DTA merenja; CALPHAD metoda