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MECHANICAL AND ELECTRICAL PROPERTIES OF TERNARY Ag-Bi-Ga SYSTEM AT 250 °C

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Abstract

This paper presents a comparative review of the experimental and thermodynamic assessment of a ternary Ag-Bi-Ga system. An isothermal section at 250 °C was calculated using optimized thermodynamic data for the constitutive binaries. Microstructures and phase compositions of studied alloys were analyzed by scanning electron microscopy in combination with energy dispersive spectrometry and X-ray powder diffraction technique. The obtained experimental results were found to support the predicted phase equilibria rather well. The hardness of alloys from three vertical sections (Bi-AgGa, Ag-BiGa, and Ga-AgBi) was determined using Brinell hardness test while the hardness of the individual identified phases was determined using Vickers microhardness test. Additional electrical conductivity measurements were carried out on the same alloy samples. Based on the experimentally obtained results iso-lines of Brinell hardness and electrical conductivity for the entire compositional range were calculated.

Keywords: *materials testing; isothermal section at 250 °C; microstructure; hardness; electrical conductivity.*

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Introduction

The ternary Ag-Bi-Ga system was thoroughly investigated in our previous study (*Minic et al.*) [1]. The results of experimental investigations of the liquidus surface; three vertical sections and the isothermal section at 200 °C are presented in the paper [1]. However; in the current study, the focus is on the experimental investigation of mechanical and electrical properties of alloys of the ternary Ag-Bi-Ga system. Besides mentioned properties; the paper also presents results of the SEM-EDS and XRD analysis for alloys from the isothermal section at 250 °C. The selected temperature (250 °C) is rather interesting for investigation since the largest number of different phases is in equilibrium at this temperature and also because at temperatures higher than 250 °C liquidus phases are dominant (melting point of included elements are Ag-961.78 °C; Ga-29.76 °C and Bi-271.3 °C [2]).

The concept of this study and the other similar studies [3 – 5] provides the possibility to follow up how the properties of a base element are changing with the addition of other elements. The significance of the selected ternary system itself reflects in a fact that; as widely known; owing to its outstanding heat and electrical conductivity the silver is practically irreplaceable for various special applications. Also, it is well known that the Ag-Bi [6, 7] or Ag-Ga [8, 9] binaries are extensively used for welding; soldering; in electronics and in the chemical industry.

Experimental procedure

All investigated samples were prepared from Ag; Bi; and Ga shots of 99.99 at. % purity in an induction furnace under high-purity argon atmosphere. In general; the average loss of mass during melting of samples is about 2 mass %. Before annealing; the samples were sealed in evacuated quartz tubes. After that; the prepared samples were heated to the temperature that is 50 °C higher than the melting point of Ag and then cooled to 250 °C at a cooling rate of 5 °C min^{-1}. The samples for all investigation were annealed at 250 °C for 2 months and subsequently quenched into an ice/water mixture in order to preserve the desired equilibrium.

The microstructure of the prepared alloy samples was studied using TESCAN VEGA3 scanning electron microscope in combination with energy dispersive spectroscopy (EDS) (Oxford Instruments X-act). The samples were prepared by the standard metallographic procedure without etching. The polished alloy samples were initially inspected using EDS elemental mapping in order to check compositional homogeneity and possible segregation. Overall compositions of the samples, as well as compositions of coexisting phases, were subsequently determined using EDS point and area analysis.

XRD patterns of the studied samples were obtained using D2 PHASER powder diffractometer with a dynamic scintillation detector and ceramic X-ray Cu tube (KFLCu-2K) in a 5 to 75 ° 2θ range with 0.02 deg step size. The recorded patterns were subsequently analyzed using Topas 4.2 software and ICDD databases PDF2 (2013).

Brinell hardness tester INNOVATEST; model Nexus 3001 was used for testing of the samples while the microhardness of the individual phases was determined using a Vickers microhardness tester Sinowon; model Vexus ZHV-1000V. The electrical conductivity of the studied alloys was measured using Foerster SIGMATEST 2.069 eddy current instrument.

Literature data

The isothermal section at 250 °C of the ternary Ag-Bi-Ga system has been thermodynamically predicted using optimized thermodynamic parameters for the constitutive binaries from literature [10 – 12]. The optimized thermodynamic parameters for the binary Ag-Ga system were taken from *Gierlotka et al.* [10]. Likewise; the parameters for the Ag-Bi system were taken from *Zoro et al.* [11] and for the Bi-Ga binary system from Girard [12]. Thermodynamic calculations were carried out by using PANDAT software package [13].

A list of phases from the binary subsystems considered for thermodynamic binary-based prediction is given in Table 1 together with their corresponding Pearson symbols.

Table 1. Phases considered and their crystal structures [14, 15].

Thermodynamic database name	Phase	Strukturbericht designation	Pearson symbol	Space group
Liquid	Liquid			
Fcc_A1	(Ag)	A1	*cF*4	$Fm\bar{3}m$
Rhombo_A7	(Bi)	A7	*hR*2	$R\bar{3}m$
Ortho_Ga	(Ga)	A11	*oC*8	*Cmca*
Hcp_A3	ζ-Ag_2Ga	A3	*hP*2	$P6_3/mmc$
Hcp_ord	ζ'-Ag_2Ga	C22	*hP*9	$P\bar{6}2m$
Ag_3Ga_2	Ag_3Ga_2	-	-	*Pmmm*

Results and discussion

Fig. 1 presents calculated isothermal section at 250 °C of the Ag-Bi-Ga ternary system. The alloy samples selected for microstructural analysis by SEM-EDS and XRD techniques are marked on Fig. 1 by corresponding symbols. Dashed orange lines are shown on Fig. 1 mark three vertical sections along which lay the compositions of the alloy samples used for hardness and electrical conductivity measurements.

From fourteen regions predicted in the isothermal section 250 °C; eight of them were investigated using SEM-EDS and XRD experimental techniques. The obtained EDS results are given in Table 2 in comparison with the predicted phase compositions.

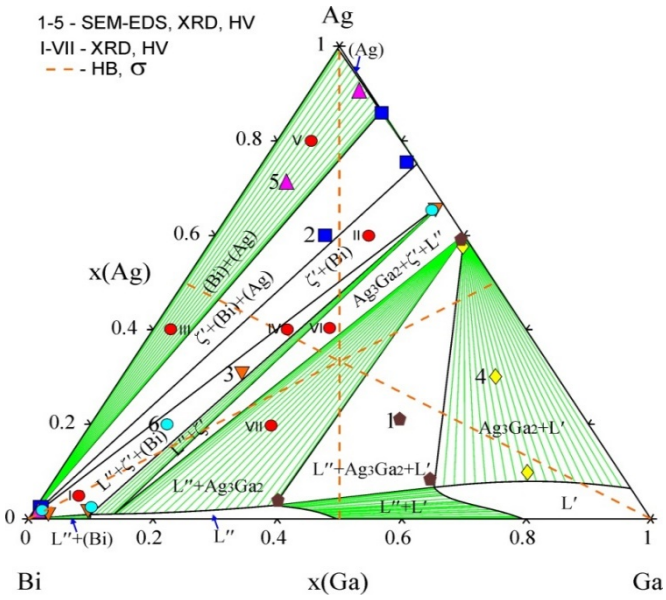
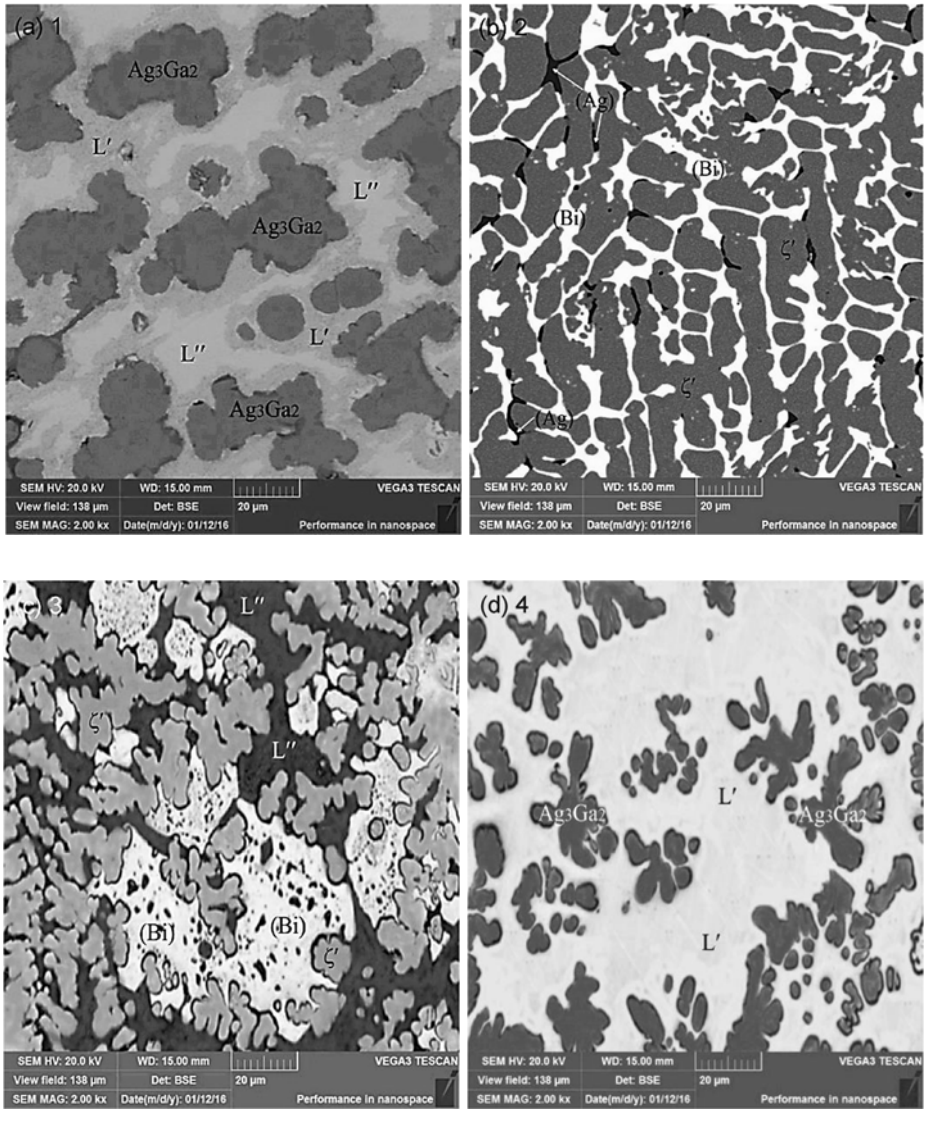


Fig. 1. Isothermal sections of the ternary Ag-Bi-Ga systems at 250 °C with marked overall compositions of investigated samples.

Table 2. Calculated and experimentally determined phase compositions in the ternary Ag-Bi-Ga system at 250 °C.

S.	Overall composition (at.%)	Theor. predicted phases	Exp. determined phases	Compositions of phases (at.%)					
				Ag		Bi		Ga	
				exp.	calc.	exp.	calc.	exp.	calc.
1.	19.32 Ag 30.48 Bi 50.20 Ga	L″ L′ Ag_3Ga_2	L″ L′ Ag_3Ga_2	4.01±0.1 7.55±0.1 58.37±0.3	2.81 6.6 60	57.13±0.5 32.39±0.3 1.46±0.3	59.1 31.5 -	38.86±0.3 60.06±0.5 40.17±0.5	38.09 61.9 40
2.	60.41 Ag 21.87 Bi 17.72 Ga	(Bi) (Ag) ζ′	(Bi) (Ag) ζ′	0.76±0.4 84.97±0.3 75.06±0.6	- 86.03 75.08	98.6±0.2 0.83±0.4 1.44±0.3	100 0.38 -	0.64±0.1 14.2±0.2 23.5±0.2	- 13.59 24.92
3.	31.32 Ag 49.18 Bi 19.50 Ga	L″ ζ′ (Bi)	L″ ζ′ (Bi)	1.53±0.4 65.76±0.2 0.91±0.3	0.89 66.85 -	89.18±0.2 1.41±0.2 97.72±0.3	89.66 - 100	9.29±0.2 32.83±0.4 1.37±0.4	9.45 33.15 -
4.	30.54 Ag 9.84 Bi 59.62 Ga	L′ Ag_3Ga_2	L′ Ag_3Ga_2	8.45±0.3 57.64±0.1	7.86 60	14.46±0.4 1.36±0.2	17.5 -	77.09±0.3 41±0.7	74.64 40
5.	71.08 Ag 23.18 Bi 5.74 Ga	(Bi) (Ag)	(Bi) (Ag)	1.2±0.3 90.58±0.5	- 91.94	98.27±0.6 1.53±0.1	100 0.42	0.53±0.1 7.89±0.3	- 7.64
6.	19.25 Ag 68.42 Bi 12.33 Ga	L″ ζ′ (Bi)	L″ ζ′ (Bi)	1.65±0.3 65.91±0.6 0.91±0.3	0.89 66.85 -	89.5±0.7 1.11±0.3 98.18±0.3	89.66 - 100	8.85±0.3 32.98±0.3 0.91±0.2	9.45 33.15 -

According to the results given in Table 2 the existence of the all predicted phases was confirmed. Phase Ag_3Ga_2 in the samples 1 and 4; has a solubility of Bi of around 1.4 %; the same value is detected for ζ′-Ag_2Ga phase. Also; it can be seen that Bi phases have a very small solubility of Ag and Ga. Phase L′ is rich in Ga and L″ is rich in Bi and this difference was predicted in binary Bi-Ga system. Fig. 2; presents microstructures of the six studied alloy samples. Phases identified using energy dispersive spectrometry (EDS) analysis are marked on the presented microstructures.



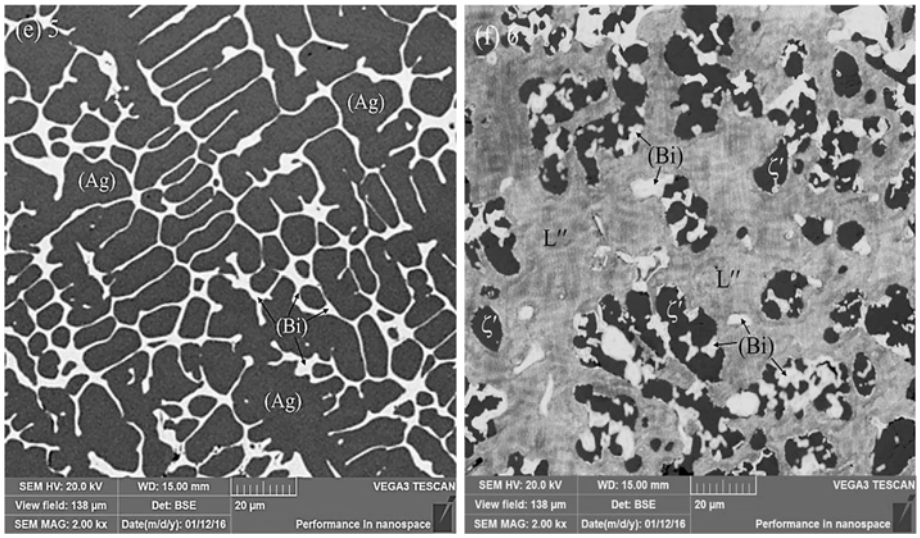


Fig. 2. Microstructures of the alloys analyzed by the SEM-EDS technique.

Additional seven samples from the isothermal section at 250 °C were investigated with XRD technique. Compositions of these samples are marked on Fig. 1 with Roman numerals from I to VII. The samples I and IV belong to the same three-phase region L″+ζ′+(Bi); the samples III and V are from two-phase region (Ag)+(Bi); the sample II is from two-phase region ζ′+(Bi); sample VI is from L″+ζ′+Ag_3Ga_2 region and the sample VII is from two-phase region L″+Ag_3Ga_2. The same alloy samples were further analyzed by XRD technique. The obtained results of XRD analysis are shown in Table 3.

Table 3. Comparative presentation of the calculated and the experimentally obtained XRD data for the studied alloy samples at 250 °C.

S.	Overall composition (at. %)	Phase		Lattice parameters (Å)					
		Pred.	Ident.	*a*		*b*		*c*	
				Exp.	Ref.	Exp.	Ref.	Exp.	Ref.
1	19.32 Ag 30.48 Bi 50.20 Ga	L″ L′ Ag_3Ga_2	L″ L′ Ag_3Ga_2	6.7245(2)	6.7271[1]	3.8753(3)	3.8764[1]	3.1808(5)	3.1802[1]
2.	60.41 Ag 21.87 Bi 17.72 Ga	(Bi) ζ′ (Ag)	(Bi) ζ′ (Ag)	4.5694(3) 7.7482(2) 4.0889(4)	4.5462[16] 7.7460[17] 4.094[18]			11.8576(1) 2.8698(6)	11.8591[16] 2.8704[17]
3.	31.32 Ag 49.18 Bi 19.50 Ga	L″ (Bi) ζ′	L″ (Bi) ζ′	4.5567(1) 7.7457(2)	4.5462[16] 7.7460[17]			11.8451(3) 2.8693(6)	11.8591[16] 2.8704[17]
4.	30.54 Ag 9.84 Bi 59.62 Ga	L′ Ag_3Ga_2	L′ Ag_3Ga_2	6.7287(5)	6.7271[1]	3.8765(2)	3.8764[1]	3.1843(5)	3.1802[1]
5.	71.08 Ag 23.18 Bi 5.74 Ga	(Bi) (Ag)	(Bi) (Ag)	4.5465(3) 4.0872(6)	4.5462[16] 4.094[18]			11.8576(7)	11.8591[16]

6.	19.25 Ag 68.42 Bi 12.33 Ga	L″ (Bi) ζ′	L″ (Bi) ζ′	4.5498(2) 7.7445(6)	4.5462[16] 7.7460[17]			11.8602(3) 2.8756(3)	- 11.8591[16] 2.8704[17]
I	3 Ag 93 Bi 4 Ga	L″ (Bi) ζ′	L″ (Bi) ζ′	4.5476(8) 7.7487(8)	4.5462 [16] 7.7460 [17]			11.8577(5) 2.8698(1)	- 11.8591[16] 2.8704[17]
II	60 Ag 18 Bi 22 Ga	(Bi) ζ′	(Bi) ζ′	4.5561(1) 7.7458(3)	4.5462 [16] 7.7460 [17]			11.8611(1) 2.8732(3)	11.8591[16] 2.8704[17]
III	40 Ag 56 Bi 4 Ga	(Bi) (Ag)	(Bi) (Ag)	4.5531(2) 4.1003(1)	4.5462[16] 4.094[18]			11.8606(5)	11.8591[16]
IV	40 Ag 42 Bi 18 Ga	L″ (Bi) ζ′	L″ (Bi) ζ′	4.5676(1) 7.7458(2)	4.5462 [16] 7.7460 [17]			11.8566(7) 2.8713(5)	11.8591[16] 2.8704[17]
V	80 Ag 13 Bi 7 Ga	(Bi) (Ag)	(Bi) (Ag)	4.5576(1) 4.1001(7)	4.5462[16] 4.094[18]			11.8603(7)	11.8591[16]
VI	40 Ag 33 Bi 27 Ga	L″ ζ′ Ag_3Ga_2	L″ ζ′ Ag_3Ga_2	7.7454(3) 6.7251(3)	7.7460 [17] 6.7271[1]	3.8763(5)	3.8764[1]	2.8712(1) 3.1813(1)	2.8704[17] 3.1802[1]
VII	20 Ag 53 Bi 27 Ga	L″ Ag_3Ga_2	L″ Ag_3Ga_2	6.7251(3)	6.7271[1]	3.8763(5)	3.8764[1]	3.1813(1)	3.1802[1]

Obtained values of lattice parameters for Ag_3Ga_2; (Bi); ζ′ and (Ag) phases are in close agreement with literature data [1, 16 – 18]. The experimentally identified phases by SEM-EDS are same as detected by XRD analysis. XRD pattern of sample I with identified phases is shown on Fig. 3 as an illustration.

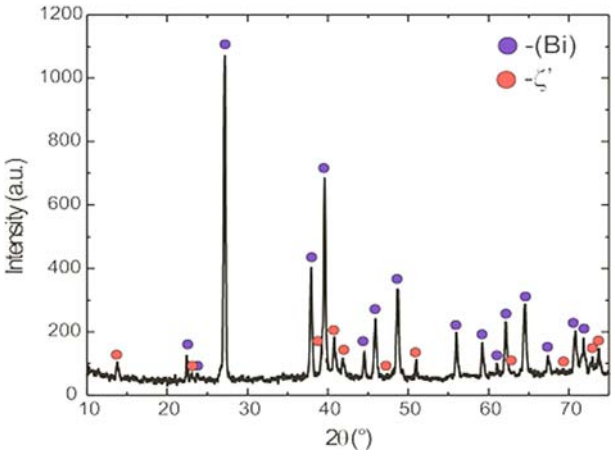


Fig. 3. XRD pattern for the sample alloy I.

Vickers microhardness was investigated using the same alloys samples investigated by XRD. Compositions of these thirteen alloys are marked on Fig. 1. The load of 0.245 N was applied and time of loading was 20 s. The values of Vickers microhardness are given in Table 4. Vickers microhardness for each phase was

determined by five measurements and in Table are given a mean value for investigated phases.

Table 4. Measured Vickers microhardness of the phases in ternary Ag-Ga-Bi system.

Determined phases	Sample												
	1	2	3	4	5	6	I	II	III	IV	V	VI	VII
	Mean value (MN x m^{-2})												
Ag_3Ga_2	130.5			131.8								132.1	131.5
(Bi)		95.5	96.2		93.5	95.4	90.6	91.5	93.8	90.5	94.6		
ζ′		146.4	140.3			141.9	143.4	144.6		143.3		145.2	
(Ag)		55.7			54.7				53.7		56.2		

From all investigated phases; phase rich in silver; solid solution (Ag) has the lowest value of hardness in range 53.7 to 56.2 MN m^{-2}. Mean value of hardness for solid solution (Bi) phase is 93.51 MN m^{-2}. Other two phases ζ′-Ag_2Ga and Ag_3Ga_2 have almost the same hardness. Mean value of hardness for ζ′-Ag_2Ga is 143.59 MN m^{-2}and for Ag_3Ga_2 is slightly lower 131.48 MN m^{-2}.

The hardness of the ternary Ag-Bi-Ga alloys was determined using Brinell test method. As aforementioned; the alloy samples were selected from the three vertical sections: Bi-AgGa; Ag-BiGa; and Ga-AgBi. Summary of the obtained experimental results is given in Table 5; together with literature values of hardness for pure elements [19].

Table 5. Brinell hardness of the alloys of the ternary Ag-Bi-Ga system.

Mole fraction of components			Hardness of Brinell HB (MN x m^{-2}) Values for different measurement			Mean value
x(Bi)	x(Ag)	x(Ga)	1	2	3	
0	0.5	0.5	45.1	41.0	43.05	43.05
0.2	0.4	0.4	39.1	47.4	46.7	44.4
0.4	0.3	0.3	53.68	51.22	52.12	52.34
0.6	0.2	0.2	72.1	74.1	75;7	73.6
0.8	0.1	0.1	82.07	80.23	82.26	81.52
1	0	0				94.2
0.5	0	0.5	27.3	30.5	28.9	28.9
0.4	0.2	0.4	36.46	35.82	37.64	36.64
0.3	0.4	0.3	76.6	78.1	77.5	77.4
0.2	0.6	0.2	52.1	41.5	48.7	47.4
0.1	0.8	0.1	26.06	27.96	27.82	27.28
0	1	0				24.5
0.5	0.5	0	38.2	38.0	38.1	38.1
0.4	0.4	0.2	35.89	33.96	35.87	35.24
0.3	0.3	0.4	45.8	49.0	47.8	47.5
0.2	0.2	0.6	56.2	56.7	53.5	55.4
0.1	0.1	0.8	54.21	54.34	58.49	55.68
0	0	1				60

A graphical presentation of the relationship between the Brinell hardness of the studied alloys from the three vertical sections and the alloy composition is given in Fig. 4.

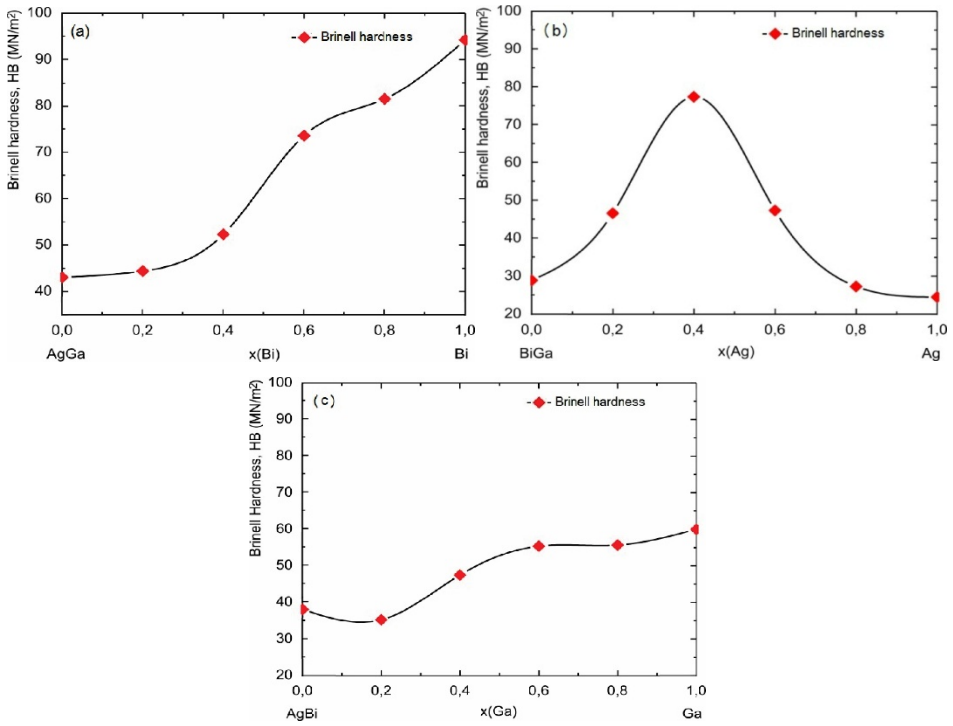


Fig. 4. Brinell hardness for investigated samples from isothermal sections at 250 °C.

Experimentally determined values of hardness given in Table 5; and an appropriate mathematical model were used to calculate values of hardness along the isothermal section at 250 °C of the ternary Ag-Bi-Ga system.

In order to define a mathematical model for the dependence of Brinell hardness vs composition for Ag-Bi-Ga alloys the Design Expert v.9.0.3.1 software package is used. Out of a possible canonical or Scheffe model [20 – 22] that meet the requirements of adequacy a Special Cubic Mixture Model is recommended:

$$\hat{y} = \sum_{i=1}^{q} \beta_i x_i + \sum_{i<j}^{q-1} \sum_{j}^{q} \beta_{ij} x_i x_j + \sum_{i<j}^{q-2} \sum_{j<k}^{q-1} \sum_{k}^{q} \beta_{ijk} x_i x_j x_k \qquad 1$$

The Analysis of variance (ANOVA) confirms the adequacy of the Model. The F-value of the Model is 10.855 and it implies that the model is significant. There is only a 0.01% chance that a ”Model F-Value“ this large could occur due to noise. The final equation of the predictive model in terms of actual components is:

HB = 106.3431·Bi+18.52489·Ag+44.38481·Ga-93.6128·Bi·Ag-170.947·Bi·Ga+

$$60.24555 \cdot Ag \cdot Ga + 552.754 \cdot Bi \cdot Ag \cdot Ga \qquad 2$$

Iso-lines contour plot for Brinell hardness of alloys defined by Equation 2 is shown in Fig. 5.

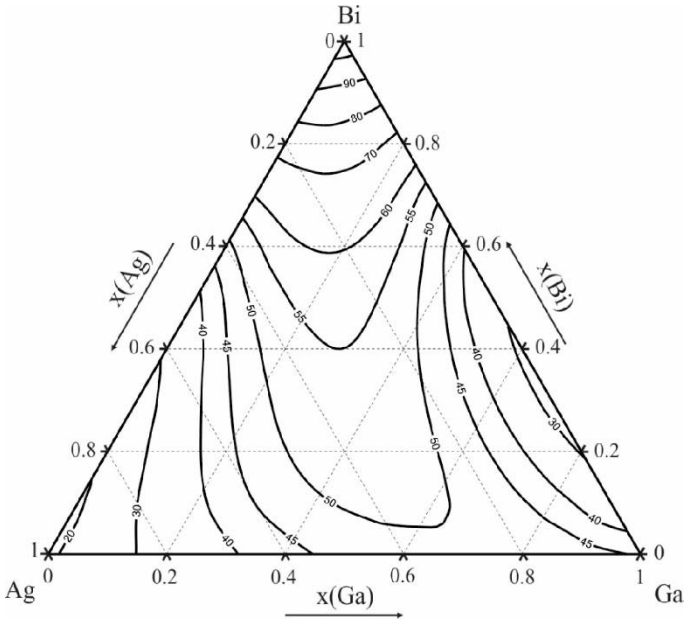


Fig. 5: Iso-lines of Brinell Hardness of the ternary Ag-Bi-Ga system at 250 °C.

Table 6 presents the obtained experimental results of electrical conductivity measurements and literature values of electrical conductivity for pure Ag, Bi, and Ga [23].

Table 6. Electrical conductivity of the alloys from isothermal sections at 250 °C.

Mole fraction of components			Electrical conductivity (MS m^{-1}) Values for different measurement				Mean value
x(Bi)	x(Ag)	x(Ga)	1	2	3	4	
0	0.5	0.5	1.1637	1.2428	1.3058	1.2325	1.2362
0.2	0.4	0.4	1.31	1.315	1.326	1.306	1.3143
0.4	0.3	0.3	1.915	1.863	1.786	1.96	1.8810
0.6	0.2	0.2	0.5518	0.5696	0.5634	0.6054	0.5726
0.8	0.1	0.1	0.5154	0.5065	0.5084	0.4955	0.5065
1	0	0					0.77
0.5	0	0.5	1.3124	1.3482	1.3124	1.3326	1.3264
0.4	0.2	0.4	1.461	1.462	1.518	1.543	1.4960
0.3	0.4	0.3	1.529	1.662	1.727	1.629	1.6368

0.2	0.6	0.2	2.103	2.12	2.109	2.093	2.1063
0.1	0.8	0.1	6.4324	6.4528	6.4486	6.4354	6.4423
0	1	0					62
0.5	0.5	0	1.8826	1.9342	1.9677	1.9227	1.9268
0.4	0.4	0.2	1.52	1.481	1.508	1.507	1.5040
0.3	0.3	0.4	1.32	1.371	1.347	1.348	1.3465
0.2	0.2	0.6	1.146	1.126	1.207	1.135	1.1535
0.1	0.1	0.8	1.773	1.472	1.658	1.734	1.6593
0	0	1					7.1

Graphical presentation of the correlation between electrical conductivity and mole fraction of components for the all investigated samples is shown in Fig. 6.

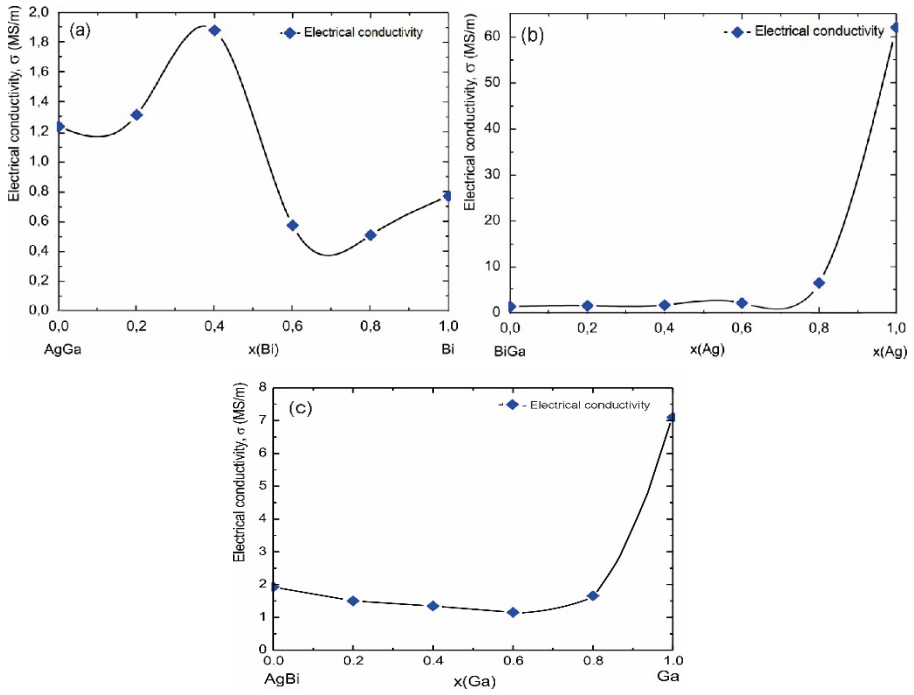


Fig. 6. Electrical conductivity for investigated samples from isothermal sections at 250 °C.

Calculation of electrical conductivity for the alloys from the Ag-Bi-Ga ternary system was carried out in the same manner as the aforementioned Brinell hardness calculation.

Model summary statistics are suggested Special Cubic Mixture Model. The F-value of the Model is 8.89 in the Analysis of variance (ANOVA) and it implies that the

model is significant. The final equation of the predictive model in Terms of Actual Components is:

$$\sigma = 4.818142 \cdot Bi + 27.64718 \cdot Ag + 8.983279 \cdot Ga - 72.3709 \cdot Bi \cdot Ag - 18.314 \cdot Bi \cdot Ga - 84.8721 \cdot Ag \cdot Ga + 173.949 \cdot Bi \cdot Ag \cdot Ga \quad (3)$$

Iso-lines contour plot of Electric Conductivity defined by equation 3 is shown in Fig. 7.

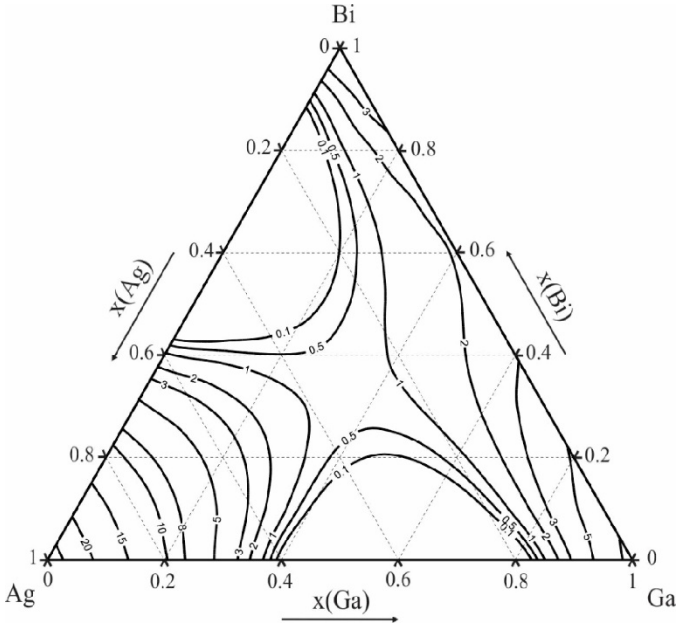


Fig. 7. Iso-lines of electrical conductivity of ternary Ag-Bi-Ga system at 250 °C.

Based on experimental results and used a mathematical model for calculation of electrical conductivity for all alloys in ternary Ag-Bi-Ga system at 250 °C it is clear that all Ag-rich alloys will have better electrical conductivity than Bi-rich or Ga-rich alloys.

Similar studies on other ternary systems were published by the same group of authors in past [24, 25] because temperature-related changes of mechanical and electrical properties of alloys are very important parameters that determine their possible future applications.

Conclusion

In this study; an isothermal section of the ternary Ag-Bi-Ga system at 250 °C was experimentally investigated and thermodynamically extrapolated using optimized thermodynamic unary and binary data taken from literature. Microstructures and phase compositions of the alloys annealed at 250 °C were studied using SEM-EDS and XRD analysis. The experimentally obtained results were found to be in a close agreement with the predicted phase diagram at 250 °C. Vickers microhardness measurements were

carried out for the phases present in the microstructure of the eleven selected alloy samples. Values of Brinell hardness and electrical conductivity were experimentally determined for the selected alloys from the Bi-AgGa; Ag-BiGa; and Ga-AgBi vertical sections of the ternary Ag-Bi-Ga system. Based on the experimentally obtained results iso-lines of Brinell hardness and electrical conductivity for the entire compositional range of the ternary system were calculated.

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