Structural, mechanical and electrical properties of alloys in ternary Ag-Bi-Zn system

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ABSTRACT: Structural, mechanical and electrical properties of selected alloys in ternary Ag-Bi-Zn system are presented in this paper. Chosen alloys were investigated using X-Ray Diffraction (XRD), light optical microscopy, Scanning Electron Microscopy combined with Energy Dispersive Spectrometry (SEM-EDS), as well as by electrical conductivity and Brinell hardness measurements. Isolines of electrical conductivity and hardness for the entire Ag-Bi-Zn system were calculated using regression models.

KEYWORDS: Ag-Bi-Zn system; Electrical conductivity; Hardness; SEM-EDS; XRD

RESUMEN: *Propiedades estructurales, mecánicas y eléctricas de aleaciones del sistema ternario Ag-Bi-Zn.* Este trabajo estudia las propiedades estructurales, mecánicas y eléctricas de aleaciones seleccionadas del sistema ternario Ag-Bi-Zn. Las aleaciones elegidas se han caracterizado por medio de difracción de rayos X, microscopía óptica, microscopía electrónica de barrido combinada con espectrometría de dispersión de energía, así como por medio de medidas de conductividad eléctrica y dureza Brinell. Por medio de modelos de regresión se han calculado las líneas de isoconductividad eléctrica y dureza para todo el sistema Ag-Bi-Zn.

PALABRAS CLAVE: Conductividad eléctrica; Dureza; SEM-EDS; Sistema Ag-Bi-Zn; XRD

1. INTRODUCTION

Recent investigations of high temperature lead-free solder systems in the frame of COST MP0602 project (2007), indicated different silver-based systems as possible new more environmentally friendly substitue materials to be applied in electronics. Among mentioned group of systems the Ag-Bi-Zn system presents one of the promising candidates (Ipser, 2007; Abtew and Selvaduray, 2000; Terzieff *et al.*, 2007; Zoro *et al.*, 2005; Hassam *et al.*, 2001).

The first investigations of Ag-Bi-Zn alloys, from thermodynamic and phase diagram point of view, were done in 1988 by Kubaschevski (1988). Miyazawa and Ariga (2001) presented their results

on characterization of Ag-Bi-Zn alloys, while most complete data on that system was given by Ohtani *et al.* (2004). There are also references concerning constitutive binary subsystems (Gandova and Vassilev, 2013; Premović *et al.*, 2013; Jendrzejczyk-Handzlik *et al.*, 2007).

In order to give a contribution to knowledge and better understanding of the Ag-Bi-Zn system, the results of investigation of structural, mechanical and electrical properties of these potential lead-free, ecological aloys are presented in this paper. Knowledge of these properties is very important

for further work with these alloys. Many researchers are examining properties for the different alloys (Soteloa *et al.*, 2014; Vargas-Arista *et al.*, 2014; D'Errico *et al.*, 2013).

2. EXPERIMENTAL

2.1. Samples

The investigated alloy samples were prepared from high purity (99.999%) silver, bismuth, and zinc powders produced by Alfa Aesar (Germany).
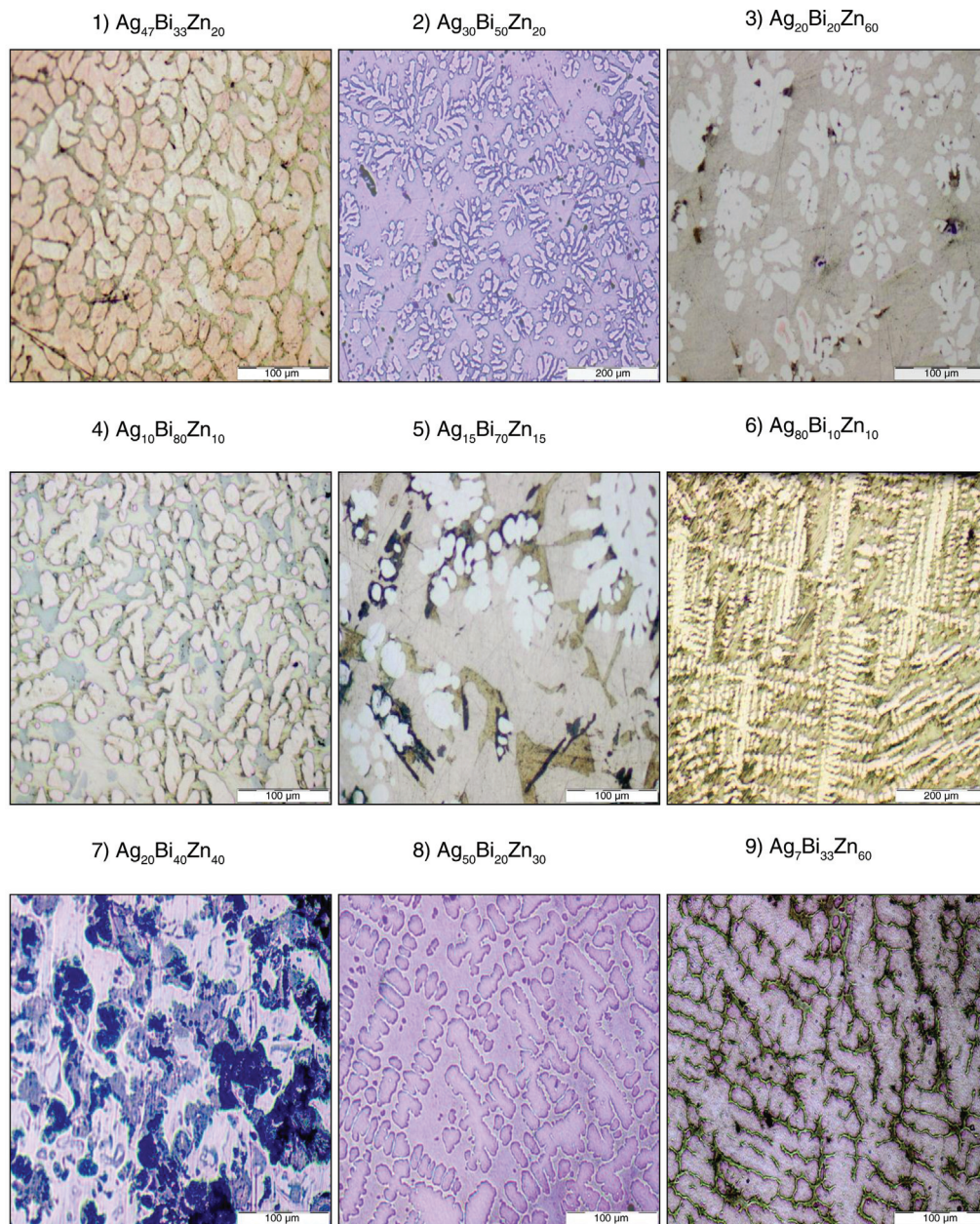

FIGURE 1. Characteristic microphotographs of the alloys in Ag-Bi-Zn system.

The samples of 4 g mass, prepared of mixed powders, were sealed in evacuated quartz tubes, melted in an induction furnace under argon atmosphere and subsequently cooled in air. The mass loss during the melting of the samples were about 1 mass%. The alloys prepared in described way were heated to temperature 50 °C higher than silver melting temperature and then cooled to 473 K by heating rate of 5 °C min^{-1}. Further, the alloys were annealed at mentioned temperature of 473 K for 300 hours, and then rapidly cooled dip into a mixture of ice and water in order to maintain the desired equilibrium. Weight losses during the annealing was less than 0.5 mass%.

2.2. Techniques

Scanning electron microscopy combined with energy dispersive spectrometry, light optical microscopy, x-ray diffraction (XRD), electrical conductivity and Brinell hardness measurements were applied in experimental investigation of examined alloys.

Scanning electron microscopy (SEM) was performed using a JEOL (JSM6460) microscope equipped with Oxford Instruments Energy Dispersive Spectrometer (EDS), while MAC company standards were applied for composition analysis (Brodarac *et al.*, 2014).

TABLE 1.   XRD and SEM-EDS results of the samples annealed at 473 K

Sample	Overall composition (at. %)	Experimentally determined phases	Latice parameters (Å)			
			a=b		*c*	
			Reference from PDF2	Experimental	Reference from PDF2	Experimental
1	47Ag 33 Bi 20 Zn	(Bi) β-AgZn (Ag)	4.536[a] 7.636[b] 4.0773[c]	4.52936(8) 7.64027(1) 4.10037(5)	11.850[a] 2.8197[b] –	11.8263(3) 2.82971(7) –
3	20 Ag 20 Bi 60 Zn	(Bi) AgZn	4.536[a] 2.823[d]	4.49645(7) 2.8245(6)	11.850[a] 4.441[d]	11.7500(2) 4.4443(3)
6	80 Ag 10 Bi 10 Zn	(Ag) (Bi)	4.0773[c] 4.536[a]	4.0773(1) 4.54366(7)	– 11.850[a]	– 11.8461(2)
9	7 Ag 33 Bi 60 Zn	(Bi) (Zn) AgZn	4.536[a] 2.665[e] 2.823[d]	4.5395(7) 2.6650(1) 2.8231(7)	11.850[a] 4.947[e] 4.441[d]	11.8198(2) 4.94688(1) 4.4427(5)

			SEM-EDS		
			Experimentally determined phase composition (at. %)		
			Ag	Bi	Zn
10	70 Ag 10 Bi 20 Zn	(Bi) (Ag)	1.81 78.72	87.39 0.49	10.8 20.79
11	80 Ag 14 Bi 6 Zn	(Bi) (Ag)	1.59 92.43	96.19 0.41	2.25 7.16
12	40 Ag 30 Bi 30 Zn	(Bi) ζ-AgZn	0.36 57.47	98.01 0.36	0.65 42.17
13	60 Ag 14 Bi 26 Zn	(Bi) β-AgZn (Ag)	0.57 62.02 68.89	98.78 0.77 1.03	0.65 37.21 30.08
14	10 Ag 50 Bi 40 Zn	(Bi) AgZn	0.62 16.35	98.22 2.43	1.16 81.22
15	30 Ag 40 Bi 30 Zn	β-AgZn ζ-AgZn (Bi)	53.27 38.54 0.13	0.11 1.09 97.26	46.62 60.37 2.61

[a] Davey (1925); [b] Edmunds and Qurashi (1951); [c] Owen and Williams (1954); [d] Henderson and Wilcox (1964); [e] Swanson and Tatge (1977).

Light optical microscopy (LOM) investigations were carried out using OLYMPUS GX41 inverted metallographic microscope.

Powder XRD data for phase identification of samples were recorded on a D2 PHASER Bruker, powder diffractometer equipped with a dynamic scintillation detector and ceramic X-ray Cu tube (KFL-Cu-2K) in a 2θ range from 5° to 75° with a step size of 0.02°. The patterns were analysed using the Topas 4.2 software, ICDD databases PDF2 2013.

The hardness of the samples was determined using a Brinell hardness tester INNOVATEST, model NEXUS 3001. Electrical conductivity measurements were carried out using the Foerster SIGMATEST 2.069 eddy current instrument. Microhardness of examined alloys was determined using apparatus HV-1000, SINOWON.

The samples used for light microscopy, electric conductivity measurements, and hardness tests were prepared by the classic metallographic procedure without etching. Also, ten samples of alloys investigated by optical microscopy were immersed in resin, while two samples, tested by SEM-EDS method, were not previously immersed.

3. RESULTS

3.1. Structural analysis

Structural analysis of chosen Ag-Bi-Zn alloys was investigated using following experimental techniques: LOM, XRD and SEM-EDS.

Optical microscopy is considered the nine samples of ternary alloys Ag-Bi-Zn system at 473 K. Obtain microstructure are presented in Fig. 1. Samples 1, 3, 6 and 9 are investigated using XRD analysis. Sample from number 10 to 15 are investigated using SEM-EDS analysis.

On characteristic presented microstructure of the considered nine samples from ternary Ag-Bi-Zn system at 473 K it is clearly visible two or three phases. On a larger number of investigated microstructure is basically a solid solution of bismuth (Bi) which composition is different depending on the composition of the considered sample. Besides solid solution of bismuth (Bi) which is basical in microstucture is presented solid solution of silver (Ag) or other compound from silver and zinc which are exist in this ternary system.

In order to confirmation the present phase at the investigated samples were done one more analysis
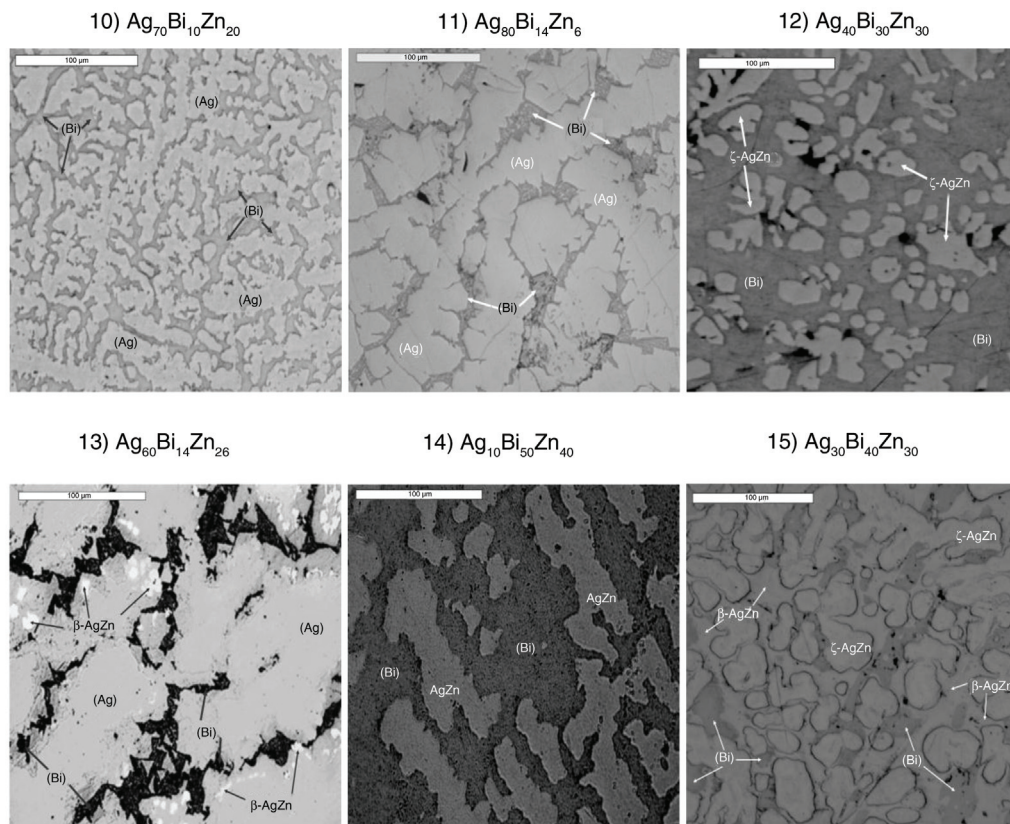

FIGURE 2. SEM microphotographs of chosen Ag-Bi-Zn alloys (compositions given in Table 1).

which is XRD analysis. The results of XRD investigations for four examined Ag-Bi-Zn samples ($1\text{-}Ag_{47}Bi_{33}Zn_{20}$, $3\text{-}Ag_{20}Bi_{20}Zn_{60}$, $6\text{-}Ag_{80}Bi_{10}Zn_{10}$, $9\text{-}Ag_7Bi_{33}Zn_{60}$) are presented in Table 1.

On four tested samples were also determine a lattice parameters. In Table 1 are presented experimentally obtained phases with obtained lattice parameters which are compared with literature data. Observing the experimentally obtained values and literature values it can be clearly see the reasonable agreement between them (Owen and Williams 1954; Davey, 1925; Henderson and Wilcox, 1964; Edmunds and Qurashi, 1951; Swanson and Tatge, 1997).

The results of SEM-EDS, including obtained SEM microphotographs and phases determined by EDS for investigated samples annealed at 473 K are

TABLE 2. Electrical conductivity (σ) values, expressed in MSm^{-1}, of the studied alloys in Bi-AgZn, Zn-AgBi and Ag-BiZn section

Alloy composition			1	2	3	Average value
x(Bi)	x(Ag)	x(Zn)		Bi-AgZn		
0	0.5	0.5	0.282	0.326	0.317	0.308
0.1	0.45	0.45	4.191	4.229	4.237	4.219
0.2	0.4	0.4	0.3615	0.2383	0.3079	0.3025
0.3	0.35	0.35	2.126	2.107	2.261	2.1646
0.4	0.3	0.3	1.182	1.166	1.189	1.179
0.6	0.2	0.2	0.6766	0.702	0.6353	0.6713
0.7	0.15	0.15	1.058	1.059	1.034	1.0503
0.8	0.1	01	0.9108	0.8808	0.8504	0.8806
0.9	0.05	0.05	0.8172	0.8402	0.858	0.8384
1	0	0	0.867	0.867	0.867	0.867
x(Zn)	x(Ag)	x(Bi)		Zn-AgBi		
0	0.5	0.5	11.04	10.62	11.23	10.96
0.1	0.45	0.45	3.237	3.452	3.654	3.4476
0.2	0.4	0.4	1.85	1.891	1.826	1.8556
0.4	0.3	0.3	2.139	2.198	2.191	2.176
0.5	0.25	0.25	0.702	0.823	0.856	0.7936
0.6	0.2	0.2	4.916	4.688	5.238	4.9473
0.8	0.1	0.1	7.806	7.647	7.719	7.724
1	0	0	16.9	16.9	16.9	16.9
x(Ag)	x(Bi)	x(Zn)		Ag-BiZn		
0	0.5	0.5	1.956	1.864	1.902	1.907
0.1	0.45	0.45	1.357	1.657	1.253	1.4223
0.3	0.35	0.35	2.436	2.132	2.415	2.3276
0.5	0.25	0.25	2.762	2.766	2.745	2.7576
0.6	0.2	0.2	4.119	4.117	4.099	4.1116
0.8	0.1	0.1	11.94	11.24	10.94	11.3733
0.9	0.05	0.05	13.79	13.48	14.23	13.8333
1	0	0	62.1	62.1	62.1	62.1

presented in Fig. 2 and Table 1. This study was performed on six alloys, the compositions of the considered alloys are shown below each microstructure in Fig. 2 and in Table 1. On the microstructure are marked defined phases and the compositions of these phases are shown in Table 1.

The microstructure marked with $10\text{-}Ag_{70}Bi_{10}Zn_{20}$ and $11\text{-}Ag_{80}Bi_{14}Zn_6$ are present alloys with high silver content. On this alloys are confirmed two phases ((Ag)+(Bi)), by using SEM-EDS analysis. Also this region is confirmed with XRD method by sample $6\text{-}Ag_{80}Bi_{10}Zn_{10}$. Other two-phases region ((Bi)+(AgZn)) is confirmed by SEM-EDS analysis with sample $14\text{-}Ag_{10}Bi_{50}Zn_{40}$, and by XRD with sample $3\text{-}Ag_{20}Bi_{20}Zn_{60}$. The last investigated region is three-phases ((Ag)+(Bi)+(β-AgZn)) which is investigated by using SEM-EDS (tested sample $13\text{-}Ag_{60}Bi_{14}Zn_{26}$) and XRD (tested sample $1\text{-}Ag_{47}Bi_{33}Zn_{20}$). Based on all experimental tests it can be clearly see that investigated ternary Ag-Bi-Zn system is consist from a larger number of two-phase and three-phase regions In the microstructure are the most presented two solid solution of silver and bismuth and several intermetallic compounds of silver and zinc.

3.2. Electrical conductivity measurements and calculation

The results of electrical conductivity measurements for samples in three vertical sections from bismuth, silver and zinc corner (Bi-AgZn, Ag-BiZn and Zn-AgBi) are presented in Table 2.

Based on experimentally measured values, electrical conductivity of the samples in Ag-Bi-Zn system was calculated according to the regression
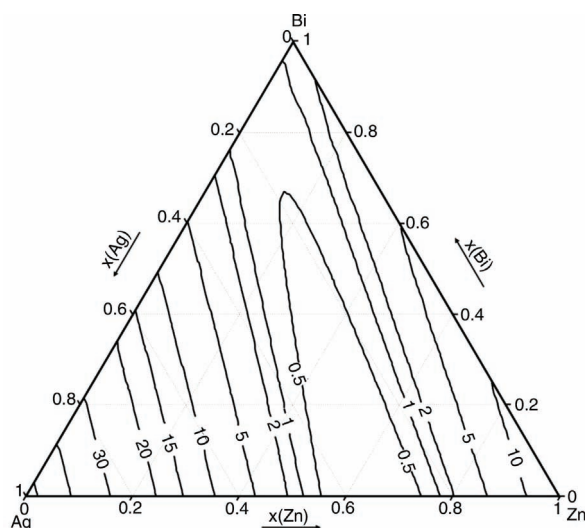

FIGURE 3. Calculated electric conductivities for the samples in ternary Ag-Bi-Zn system.

models of polynomial type, defined by following canonical polynomials (Kolarević, 2004; Cornell, 1990; Lazić, 2004):

- Linear

$$\hat{y} = \sum_{i=1}^{q} \beta_i x_i \qquad (1)$$

- Quadratic

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

- Special cubic

$$\hat{y} = \sum_{i=1}^{q} \beta_i x_i + \sum_{i<j}^{q-1} \sum_{j}^{q} \beta_{ij} x_i x_j + \sum_{i<j}^{q-2} \sum_{j<k}^{q-1} \sum_{k}^{q} \beta_{ijk} x_i x_j x_k \qquad (3)$$

- Full cubic

$$\hat{y} = \sum_{i=1}^{q} \beta_i x_i + \sum_{i<j}^{q-1} \sum_{j}^{q} \beta_{ij} x_i x_j$$
$$+ \sum_{i<j}^{q-1} \sum_{j}^{q} \delta_{ij} x_i x_j (x_i - x_j) + \sum_{i<j}^{q-2} \sum_{j<k}^{q-1} \sum_{k}^{q} \beta_{ijk} x_i x_j x_k \qquad (4)$$

For calculation of multiple regression coefficients, evaluation of model adequacy and best model choice, software RA-TeS v.1.1 (*Regression Analysis in Ternary System*) was used. That program calculates Whitcomb Score and automatically suggests the best polynomial model.

For description of electrical conductivity dependence on composition, quadratic model was suggested and selected.

According to presented calculation results (the results of ANOVA analysis (*Analysis of Variance*) for selected model and the evaluation of selected regression analysis and confidence interval of regression coefficients), final equation of selected model can be given as:

$$\sigma(MSm^{-1}) = 53.275x(Ag) + 14.987x(Zn)$$
$$+ 1.449x(Bi) - 129.870x(Ag)x(Zn)$$
$$- 70.730x(Ag)x(Bi) - 7.693\ x(Zn)x(Bi) \qquad (5)$$

TABLE 3. Hardness values (expressed in Mnm^{-2}) according to Brinell of the studied alloys in ternary Ag-Bi-Zn system

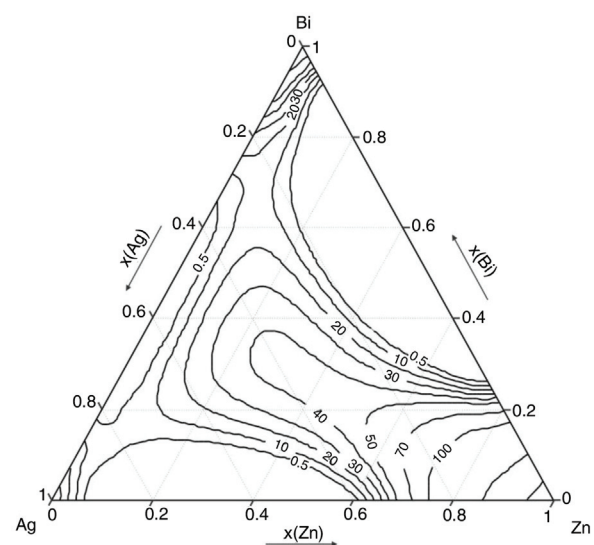| Alloy composition			1	2	3	Average value
x(Ag)	x(Bi)	x(Zn)		Zn-AgBi		
0.5	0.5	0.0	48.2	48.6	47.6	48.1
0.4	0.4	0.2	16.7	17.7	19.9	18.1
0.3	0.3	0.4	34.8	36.2	36.8	35.933
0.2	0.2	0.6	72.3	76.3	80.8	76.467
0.1	0.1	0.8	48.8	53	56.1	52.634
0	0	1	–	–	–	412
x(Ag)	x(Bi)	x(Zn)		Ag-BiZn		
0.0	0.5	0.5	61.6	62.4	61.8	61.9
0.2	0.4	0.4	46.2	45.6	44.8	45.533
0.4	0.3	0.3	33.1	35.6	37.2	35.3
0.6	0.2	0.2	22.3	24.4	25.2	23.967
0.8	0.1	0.1	16.8	20.0	18.6	18.466
1	0	0	–	–	–	24.5
x(Ag)	x(Bi)	x(Zn)		Bi-AgZn		
0.45	0.1	0.45	40.4	43.7	42.6	42.233
0.4	0.2	0.4	20.7	25.5	24.6	23.6
0.35	0.3	0.35	20.8	21.2	20.6	20.866
0.3	0.4	0.3	17.0	20.0	18.2	18.4
0.2	0.6	0.2	15.5	19.2	16.4	17.033
0.15	0.7	0.15	10.6	11.3	10.8	10.9
0.1	0.8	0.1	9.2	9.6	9.6	9.4666
0.05	0.9	0.05	8.6	9.1	9.2	8.9666
0	1	0	–	–	–	94.2
x(Ag)	x(Bi)	x(Zn)		$Bi-Ag_{80}Zn_{20}$		
0.8	0	0.2	42.5	41.8	42.2	42.1
0.64	0.2	0.16	23.3	23.7	23.8	23.6
0.48	0.4	0.12	12.4	16.5	15.4	14.766
0.32	0.6	0.08	18.1	18.8	18.2	18.366
0.16	0.8	0.04	15.7	10.6	12.8	13.033
0	1	0	–	–	–	94.2

FIGURE 4. Calculated hardness by Brinell in ternary Ag-Bi-Zn system.

Two-dimensional conture diagram of electrical conductivity for the alloys in ternary Ag-Bi-Zn system, constructed according to calculation using Eq. (5), is given in Fig. 3.

Observing the obtained experimental values and calculated values it can see that the lowest values of electrical conductivity are in the central part of the ternary system, while in the corners of the ternary system values are in increasing.

3.3. Hardness and microhardness measurements and calculation

The results of hardness measurements for samples chosen in four vertical sections Bi-AgZn, Ag-BiZn, Zn-AgBi and Bi-Ag$_{80}$Zn$_{20}$, done according to Brinell method, are presented in Table 3.

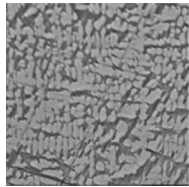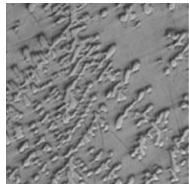In order to determine the iso-lines for hardness of ternary Ag-Bi-Zn system, the calculation based on the experimental data was applied using same mathematical procedure as for the electroconductivity. That procedure was already applied sucesfully in our previous papers (Minić et al., 2011; Minić et al., 2012; Premović et al., 2014; Minić et al., 2014). Special cubic model was selected to describe the dependence of Brinell hardness test on mole fraction of components Ag, Bi, and Zn.
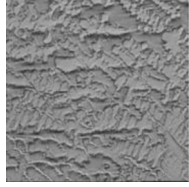
Final equation of the selected model can be written as:

$$\begin{aligned} HB(MNm^{-2}) = {} & 39.804x(Ag) + 368.813x(Zn) \\ & + 93.114x(Bi) - 1009.993x(Ag)x(Zn) \\ & - 333.956x(Ag)x(Bi) \\ & - 1540.501x(Zn)x(Bi) \\ & + 5228.959x(Ag)x(Zn)x(Bi) \end{aligned} \quad (6)$$

Two-dimensional conture diagram for Brinell hardness of alloys in ternary Ag-Bi-Zn system, calculated based on Eq. (6), is shown in Fig. 4.

TABLE 4. Vickers microhardness values, expressed in MNm^{-2}, of the phases in ternary Ag-Bi-Zn system (applying load was 0.245 N and time of loading 20 s)

| Microstructure	HV	1	2	3	4	Average value
		37.24	34.45	39.91	37.64	37.31
		56.94	51.48	50.99	52.48	52.972
Ag-BiZn, x(Ag)=0.4						
		56.38	52.71	62.40	60.58	58.017
		34.86	38.62	42.37	35.62	37.867
Bi-AgZn, x(Bi)=0.6						
		35.97	31.63	24.47	31.68	30.937
		17.17	19.54	20.00	18.46	18.792
Zn-AgBi, x(Zn)=0.2						
		25.54	19.26	19.65	21.26	21.427
		142.92	136.22	102.68	130.23	128.013
Zn-AgBi, x(Zn)=0.6						

In Fig. 4 is clearly visible that the obtained values for hardness by Brinell are increasing with increasing a mole fraction of zinc while in corner rich is silver value are decreasing.

Vickers microhardness was also investigated for four selected alloys in Ag-BiZn, Bi-AgZn and Zn-AgBi sections and presented in Table 4, together with adequate microphotographs. In Table 4 are presented value for Vickers microhardness with microphotographs.

On four investigated samples are obtained value hardness for two phases. In sample rich with zinc it can be seen phase with large hardness 128.013 MNm^{-2}. While other investigated phases have low value for hardness.

4. CONCLUSIONS

- In this paper is examined the isothermal section at 473 K in detail. In the experimental part of the work selected samples were investigated using light microscope, SEM-EDS, XRD, Vickers micro hardness, Brinell hardness and electro conductivity.
- Six samples were investigated with SEM-EDS analysis. Using this type of analysis it is confirmed five regions. Using light microscope on nine samples were obtained characteristic microphotographs of the alloys in Ag-Bi-Zn system. On same samples which are investigated using light microscope, more specifically on the four samples were done additional XRD analysis. With this additional analysis is confirmed four regions. Also for these samples were identified lattice parameters of phases and compared with literature data.
- In the paper are present the Vickers micro hardness of other phases that appear in ternary system. Also in this paper are presented a value for Brinell hardness and electrical conductivity for selected samples. Brinell hardness were experimentally determined for the selected group of alloys from three vertical sections and electrical conductivity were experimentally determined for the alloys from four vertical sections of the ternary Ag-Bi-Zn system. Based on experimentally determined values, using the regression model were calculated iso-lines of Brinell hardness and electrical conductivity of the ternary Ag-Bi-Zn system.

ACKNOWLEDGMENTS

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