

**EFFECT OF CHEMICAL COMPOSITION ON MICROSTRUCTURE,
HARDNESS AND ELECTRICAL CONDUCTIVITY PROFILES OF
THE Bi-Cu-Ga ALLOYS AT 100 °C**

Dejan Gurešić¹, Nadežda Talijan², Vladan Čosović^{2}, Dušan Milisavljević¹,
Aleksandar Đorđević¹, Milica Tomović¹*

*¹ University in Priština, Faculty of Technical Science, K.M. 7,
4000 Kos. Mitrovica, Serbia*

*² University of Belgrade, Institute of Chemistry, Technology and Metallurgy,
Belgrade, Serbia*

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Abstract

Theoretical calculation and experimental investigation of the isothermal section of a ternary Bi-Cu-Ga system at 100 °C are presented in this paper. Thermodynamic binary-based calculation of the isothermal section was performed using Pandat software. Experimental investigation included microstructural analysis carried out using light optical microscopy (LOM) and scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS), phase composition analysis using X-ray diffraction (XRD), Brinell and Vickers hardness testing and electrical conductivity measurements. In total, thirty alloy samples with compositions along three vertical sections Bi-CuGa, Cu-BiGa and Ga-BiCu were studied. The obtained experimental results support the calculated phase regions of the isothermal section at 100 °C. Hardness of individual phases as well as hardness and electrical conductivity of the studied alloys were measured. Based on the experimentally obtained results iso-lines of Brinell hardness and electrical conductivity along the whole compositional range were calculated by using appropriate mathematical models.

Keywords: ternary Bi-Cu-Ga system, isothermal section at 100 °C, microstructural investigation, hardness test, electrical conductivity

Introduction

It is well known that Cu and Cu based alloys are very important for electronic industry. In addition to good electrical and thermal conductivity and corrosion

* Corresponding author: Vladan Čosović, vlada@tmf.bg.ac.rs

resistance [1-7] it is significant that all used copper is recyclable without any loss of quality. Also copper can easily make alloys with a variety of elements [8-10]. The most important copper alloys are brass [11] and bronze [12] while the largest percentage of copper is used for production of wires for power transfer.

To the best of our knowledge there are no studies on ternary Bi-Cu-Ga system in literature. Hence, the selection of temperature for the isothermal section i.e. for the study of alloy samples was based on analysis of the predicted phase diagrams of three binary subsystems Bi-Ga [13], Bi-Cu [14] and Cu-Ga [15], that are presented on Fig. 1. From the calculated Bi-Ga phase diagram presented on Fig. 1a it can be seen that in temperature range ≈ 30 to ≈ 222 °C L+(Bi) phase is stable. The second binary subsystem Bi-Cu [14] (Fig. 1b) is eutectic system with eutectic reaction at 270.5 °C while the (Bi)+(Cu) phase region is stable from 0 up to eutectic temperature. The third binary system Cu-Ga [15] presented at Fig. 1c is more complex than the other two and along all compositional ranges in temperature interval ≈ 30 to ≈ 260 °C the same phase regions are present. Although, the ternary Bi-Cu-Ga system itself has not been thoroughly investigated the three binary subsystems Bi-Ga [16-18], Bi-Cu [19-21] and Cu-Ga [22-24] have been investigated over the years by a number of different research groups.

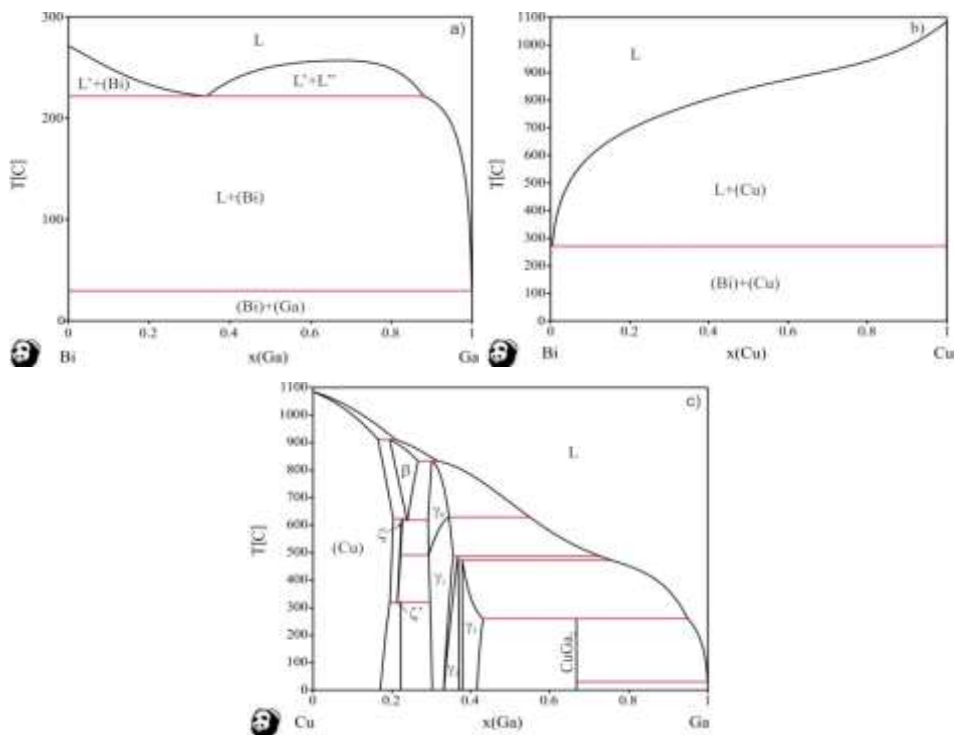


Fig. 1. Calculated phase diagrams of the binary subsystems: a) Bi-Ga [13], b) Bi-Cu [14] and c) Cu-Ga [15]

Taking into account that the working temperature of Cu wires is from 70 to 90 °C, the isothermal section at 100 °C was selected for investigation. The prepared alloys from the selected isothermal section were characterized in terms of microstructure, chemical and phase composition, Brinell and Vickers hardness and electrical conductivity.

Experimental

The studied alloy samples were prepared from high purity elements Bi, Cu and Ga (99.999 at. %). Weight of the alloy samples was 3 g. The samples were melted in an induction furnace under inert atmosphere (Ar) and the average loss of mass during melting was about 1.5 at. %. Subsequently, all samples were placed in evacuated quartz tubes and sealed, then annealed at 100 °C for 2 months and at the end quenched into an ice/water mixture in order to preserve the equilibriums. Microstructure of the samples was observed by inverted light optical microscopy (LOM) using OLYMPUS GX41 metallographic microscope. Additional microstructural analysis was carried out on JEOL (JSM6460) scanning electron microscope with energy dispersive spectroscopy (EDS) (Oxford Instruments X-act). Homogeneity and composition of the prepared alloy samples were checked by EDS elemental mapping. Overall composition and compositions of coexisting phases was determined using EDS. X-ray diffraction analysis (XRD) was performed on D2 PHASER powder diffractometer equipped with a dynamic scintillation detector and ceramic X-ray Cu tube (KFLCu-2K) in a 2θ range of 5 to 75 deg with a step size of 0.02 deg. The patterns were analyzed using Topas 4.2 software and ICDD databases PDF2 (2013). Electrical conductivity measurements were carried out using Foerster SIGMATEST 2.069 eddy current instrument. Hardness of the samples was determined using Brinell hardness tester INNOVATEST Nexus 3001 by applying force of 294.2 N and time of loading 20 seconds. Hardness of the observed phases was determined using Digital micro Vickers hardness tester DHV-1000, applied force was 0.245 N and time of loading 20 seconds. All investigated alloy samples except for those investigated by XRD were prepared by a classic metallographic procedure without etching.

Thermodynamic data from literature were used for calculation of isothermal section of Bi-Cu-Ga ternary system at 100 °C. Data for the Bi-Ga system were taken from Girard [13], for the Bi-Cu system from Teppo et al. [14], and for the Cu-Ga system from Li et al. [15]. Gibbs energy functions for the unary phases were taken from the SGTE compilation by Dinsdale [25]. Calculations were carried out using PANDAT software [26]. The phases from the constitutive binary subsystems considered for thermodynamic binary-based prediction together with their corresponding Pearson symbols are given in Table 1.

Table 1. Considered phases and their crystal structures

Thermodynamic database name	Phase	Pearson symbol	Space
LIQUID	L	-	-
FCC_A1	(Cu)	cF4	Fm3m
RHOMBO_A7	(Bi)	$hR2$	$R\bar{3}m$
ORTHOMBIC	(Ga)	$oC8$	$Cmca$
BCC_A2	β	cI2	$Im\bar{3}m$
HCP_A3	ζ	hP2	$P6_3/mmc$
CU8GA2_ZETA	ζ'
CU9GA4_GAMMA0	γ_0	oP52	$P\bar{4}3m$
CU9GA4_GAMMA1	γ_1	oP52	$P\bar{4}3m$
CU9GA4_GAMMA2	γ_2	cP?	$P\bar{4}3m$
CU9GA4_GAMMA3	γ_3	cP?	$P\bar{4}3m$
CUGA2	$CuGa_2$	tP3	$P4/mmm$

Results and discussion

Thirty alloy samples from the isothermal section at 100 °C were prepared for investigation, from which 27 are ternary alloys and three are binary alloys. Compositions of the all thirty samples lay along three vertical sections (Bi-CuGa, Cu-BiGa nad Ga-CuBi), that originate from all three corners of pure elements while the another two elements were added in the same ratio. Calculated isothermal section at 100 °C is presented on Fig. 2 together with experimentally determined compositions (EDS elemental mapping) of the investigated samples.

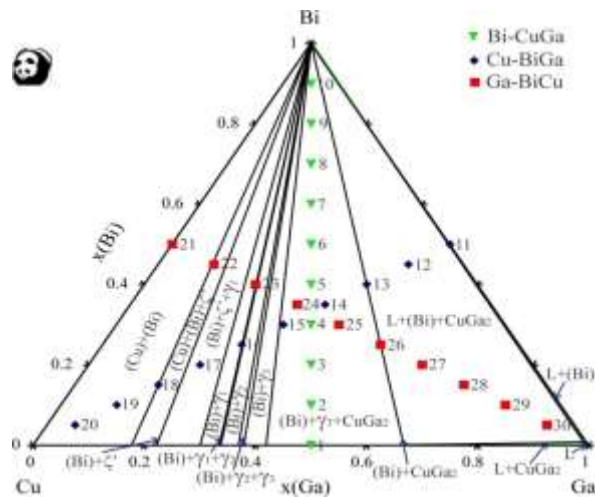


Fig. 2. Isothermal section of the ternary Bi-Cu-Ga system at 100 °C with marked overall compositions of the investigated samples

As can be seen from Fig. 2, the calculated isothermal section at 100 °C consists of fifteen different regions. The investigated alloy samples belong to seven different regions. Majority of the samples are from two largest three-phase regions L+(Bi)+CuGa₂ and (Bi)+CuGa₂+ γ_3 while the rest are from three three-phase regions (Bi)+ γ_1 + γ_2 , (Bi)+ γ_1 + ζ' and (Bi)+(Cu)+ ζ' and two two-phase regions (Bi)+(Cu) and (Bi)+CuGa₂. The obtained results of the combined SEM-EDS analysis of the nine alloy samples are presented in Table 2, together with theoretically predicted phase compositions.

Table 2. Calculated and experimentally determined phase compositions of the studied alloys

Sample	Overall composition (at.%)	Theoretic. Predicted phases	Experiment. Determined phases	Compositions of phases (at.%)					
				Bi		Cu		Ga	
				exp.	calc.	exp.	calc.	exp.	calc.
2	10.03 Bi	(Bi)	(Bi)	98.17±0.1	100.00	0.80±0.3	-	1.03±0.1	-
	45.17 Cu	γ_3	γ_3	0.17±0.3	-	57.01±0.3	58.10	42.82±0.4	41.90
	44.80 Ga	CuGa ₂	CuGa ₂	0.34±0.2	-	32.19±0.2	33.33	67.47±0.1	66.67
6	51.03 Bi	(Bi)	(Bi)	99.07±0.1	100.00	0.45±0.6	-	0.48±0.1	-
	25.03 Cu	γ_3	γ_3	0.08±0.1	-	57.01±0.2	58.10	42.91±0.3	41.90
	23.94 Ga	CuGa ₂	CuGa ₂	0.28±0.4	-	32.01±0.2	33.33	67.71±0.2	66.67
8	69.76 Bi	(Bi)	(Bi)	98.54±0.7	100.00	0.87±0.4	-	0.59±0.3	-
	15.65 Cu	γ_3	γ_3	0.54±0.2	-	57.29±0.2	58.10	42.17±0.6	41.90
	14.59 Ga	CuGa ₂	CuGa ₂	0.23±0.5	-	33.89±0.2	33.33	65.88±0.2	66.67
10	90.89 Bi	(Bi)	(Bi)	99.70±0.4	100.00	0.25±0.3	-	0.05±0.4	-
	4.43 Cu	γ_3	γ_3	0.14±0.1	-	59.78±0.3	58.10	40.08±0.3	41.90
	4.68 Ga	CuGa ₂	CuGa ₂	0.11±0.6	-	34.25±0.5	33.33	65.64±0.3	66.67
17	19.45 Bi	(Bi)	(Bi)	98.20±0.1	100	0.78±0.2	-	1.02±0.6	-
	60.87 Cu	γ_1	γ_1	0.09±0.1	-	70.76±0.5	69.98	29.15±0.1	30.02
	19.68 Ga	ζ'	ζ'	0.15±0.5	-	76.34±0.5	77.80	23.51±0.2	22.20
18	15.54 Bi	(Bi)	(Bi)	99.12±0.3	100	0.67±0.1	-	0.21±0.5	-
	69.84 Cu	(Cu)	(Cu)	0.43±0.3	-	81.75±0.3	82.34	17.82±0.3	17.66
	14.62 Ga	ζ'	ζ'	0.33±0.2	-	78.55±0.2	77.80	21.12±0.3	22.20
19	9.98 Bi	(Bi)	(Bi)	98.87±0.1	100	0.65±0.1	-	0.48±0.5	-
	80.10 Cu	(Cu)	(Cu)	0.33±0.1	-	81.82±0.3	82.34	17.85±0.2	17.66
	9.92 Ga								
22	44.98 Bi	(Bi)	(Bi)	98.93±0.1	100	0.25±0.5	-	0.82±0.5	-
	45.15 Cu	(Cu)	(Cu)	0.27±0.5	-	82.66±0.3	82.34	17.07±0.3	17.66
	9.87 Ga	ζ'	ζ'	0.47±0.3	-	77.34±0.3	77.80	22.19±0.3	22.20
28	15.67 Bi	L	L	0.13±0.1	0.76	0.63±0.4	0.57	99.24±0.5	98.67
	13.56 Cu	(Bi)	(Bi)	98.93±0.8	100.00	0.34±0.5	-	0.73±0.6	-
	70.77 Ga	CuGa ₂	CuGa ₂	0.45±0.2	-	32.17±0.1	33.33	67.38±0.1	66.67

According to the carried out thermodynamic calculations the samples 2, 6, 8 and 10 belong to three-phase region (Bi)+CuGa₂+ γ_3 . The obtained results of EDS analysis confirm existence of these three phases in the samples 2, 6, 8 and 10. Moreover, the results show that solubility of Ga and Cu in solid solution (Bi) are around 1 at. %. The other two phases CuGa₂ and γ_3 can dissolve Bi in small amounts, maximum up to 0.54 at. %. The experimental results show presence of three phases (Bi)+ γ_1 + ζ' in the sample 17, which is in agreement with the thermodynamic predictions. The samples 18 and 22

belong to three phase region (Bi)+(Cu)+ ζ' which is also confirmed by EDS whereas the sample 19 belongs to two phase region (Bi)+(Cu). The detected solid solution (Bi) dissolves only small amount of Cu and Ga which is negligible while solid solution (Cu) can dissolve 17.66 at. % of Ga which is allowed by thermodynamic calculations. Also, three phases, L, (Bi) and CuGa₂ were identified in the microstructure of the sample 28. Some of the analyzed microstructures are illustrated by the obtained SEM images presented on Fig. 3. The detected phases are marked with the corresponding symbols of the individual phases given in Table 1.

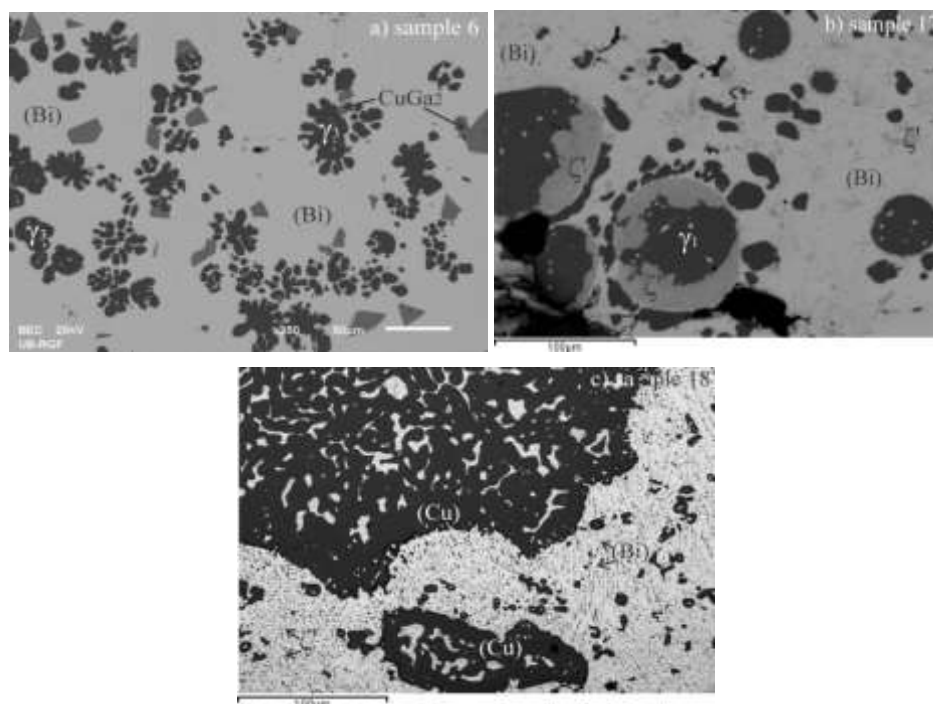


Fig. 3. Selected microstructures of the alloys analyzed by the SEM-EDS technique

In order to additionally confirm phase compositions of the studied alloy samples determined by SEM-EDS technique and predicted by thermodynamic calculations ten samples were selected for further investigation by XRD. The obtained results of XRD analysis are presented in Table 3.

Generally speaking, the obtained results of XRD analysis support the results of SEM-EDS and the theoretical predictions. For the ten alloy samples 3, 5, 7, 9, 14, 15, 25, 26, 27 and 29 the same phase composition was determined both by XRD and SEM-EDS analysis. In addition to confirmation of phase composition, lattice parameters for the detected phases were calculated and compared with literature data. In total, three different phases (Bi), γ_3 and CuGa₂ were identified in the ten studied alloy samples. Lattice parameters from literature [27-29] are presented in Table 3 next to the calculated ones. Similarity between calculated and literature values is clearly visible.

Table 3. Results of XRD analysis: identified phases and calculated lattice parameters compared with literature data

S.	Overall composition (at.%)	Coexisting phases		Lattice parameters (Å)			
		SEM-EDS	XRD	a=b		c	
				Exp.	Ref.	Exp.	Ref.
3	20.19 Bi	(Bi)	(Bi)	4.5487(1)	4.548(5) [27]	11.8589(2)	11.852(12) [27]
	39.75 Cu	γ_3	γ_3	8.7456(5)	8.747(2) [28]		
	40.06 Ga	CuGa ₂	CuGa ₂	2.8365(3)	2.830 [29]	5.8378(1)	5.839 [29]
5	40.17 Bi	(Bi)	(Bi)	4.5478(3)	4.548(5) [27]	11.8527(6)	11.852(12) [27]
	29.87 Cu	γ_3	γ_3	8.7456(2)	8.747(2) [28]		
	29.96 Ga	CuGa ₂	CuGa ₂	2.8309(2)	2.830 [29]	5.8309(2)	5.839 [29]
7	59.75 Bi	(Bi)	(Bi)	4.5425(1)	4.548(5) [27]	11.8534(1)	11.852(12) [27]
	20.87 Cu	γ_3	γ_3	8.7498(1)	8.747(2) [28]		
	19.38 Ga	CuGa ₂	CuGa ₂	2.8345(3)	2.830 [29]	5.8368(1)	5.839 [29]
9	80.58 Bi	(Bi)	(Bi)	4.5493(2)	4.548(5) [27]	11.8536(3)	11.852(12) [27]
	9.92 Cu	γ_3	γ_3	8.7467(5)	8.747(2) [28]		
	9.50 Ga	CuGa ₂	CuGa ₂	2.8347(6)	2.830 [29]	5.8306(4)	5.839 [29]
14	35.03 Bi	(Bi)	(Bi)	4.5467(7)	4.548(5) [27]	11.8578(2)	11.852(12) [27]
	30.15 Cu	γ_3	γ_3	8.7496(6)	8.747(2) [28]		
	34.82 Ga	CuGa ₂	CuGa ₂	2.8320(5)	2.830 [29]	5.8387(9)	5.839 [29]
15	30.19 Bi	(Bi)	(Bi)	4.5498(1)	4.548(5) [27]	11.8567(5)	11.852(12) [27]
	39.78 Cu	γ_3	γ_3	8.7498(9)	8.747(2) [28]		
	30.03 Ga	CuGa ₂	CuGa ₂	2.8376(6)	2.830 [29]	5.8327(3)	5.839 [29]
25	30.17 Bi	(Bi)	(Bi)	4.5479(1)	4.548(5) [27]	11.8576(3)	11.852(12) [27]
	30.02 Cu	γ_3	γ_3	8.7453(3)	8.747(2) [28]		
	39.81 Ga	CuGa ₂	CuGa ₂	2.8390(2)	2.830 [29]	5.8365(5)	5.839 [29]
26	24.87 Bi	(Bi)	(Bi)	4.5492(3)	4.548(5) [27]	11.8587(9)	11.852(12) [27]
	25.39 Cu			2.8356(1)	2.830 [29]	5.8345(1)	5.839 [29]
	49.74 Ga	CuGa ₂	CuGa ₂				
27	19.76 Bi	L	-		-		-
	20.05 Cu	(Bi)	(Bi)	4.5487(9)	4.548(5) [27]	11.8512(3)	11.852(12) [27]
	60.19 Ga	CuGa ₂	CuGa ₂	2.8339(1)	2.830 [29]	5.8354(4)	5.839 [29]
29	9.78 Bi	L	-				
	9.69 Cu	(Bi)	(Bi)	4.5476(6)	4.548(5) [27]	11.8554(9)	11.852(12) [27]
	80.53 Ga	CuGa ₂	CuGa ₂	2.8358(5)	2.830 [29]	5.8367(1)	5.839 [29]

The prepared alloy samples were further investigated by Vickers micro-hardness test. Determined values of hardness for the individual phases are given in Table 4. The presented values represent an average for individual phase hardness measured in all studied samples in which the particular phase is present. For example, intermetallic compound CuGa₂ is detected within sixteen alloy samples. For one sample, hardness of the individual phases was measured approximately five times at different parts of the sample. This means that the reported value for hardness of CuGa₂ phase in Table 4 represents average of eighty measurements. The same procedure was applied for each phase.

Table 4. Vickers microhardness of the detected phases in the ternary Bi-Cu-Ga system

micro-Vickers	Detected phase with EDS and XRD						
	(Cu)	(Bi)	ζ'	γ_1	γ_2	γ_3	CuGa ₂
Mean value MN/m ²	908.13	80.17	308.91	245.35	222.61	214.89	157.46

LOM images of microstructures of the samples 9 and 19 after micro-Vickers measurements are presented on Fig. 4.

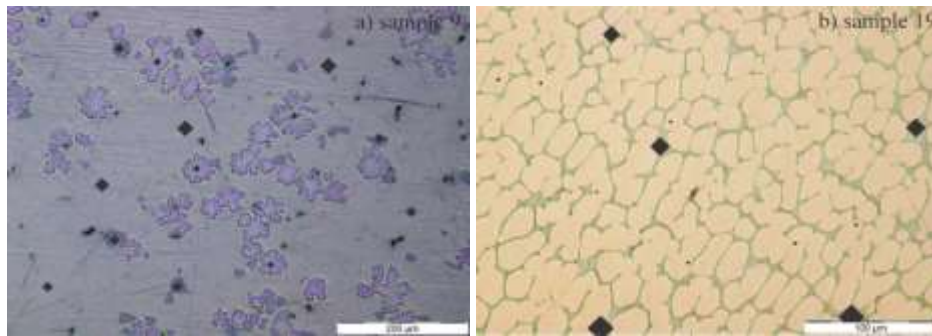
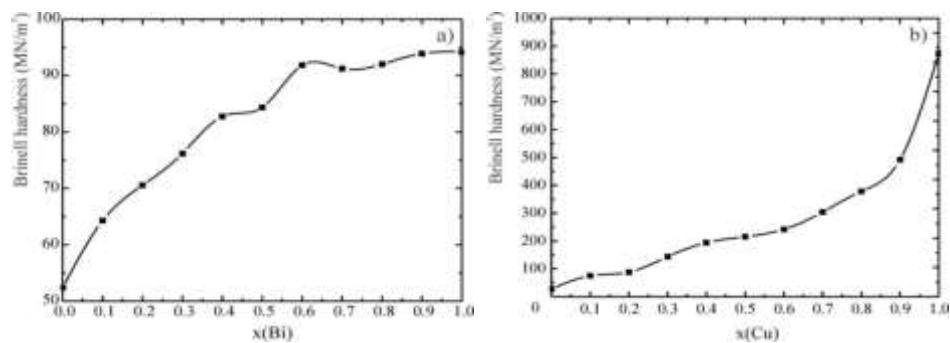


Fig. 4. Microstructures of the alloys after micro-Vickers test: a) sample 9 and b) sample 19

Brinell hardness tests were performed on all prepared alloy samples from the studied ternary system as well as on three binary alloys. The obtained average values after three measurements are presented on Fig. 5 in relation to alloy composition. Considering values of Brinell hardness for the pure elements Bi (94.2 MN/m²), Cu (874 MN/m²) and Ga (60 MN/m²) [30] the observed increase of alloy hardness with an increase of copper content (Fig. 5b) is expected. Similarly, the Cu rich alloy (sample 20) has the highest hardness 492.47 MN/m².



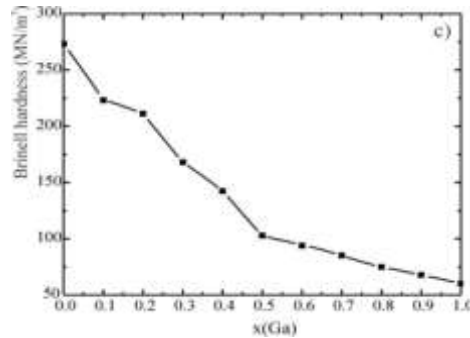


Fig. 5. Graphical presentation of the measured Brinell hardness of the investigated samples: a) Bi-CuGa, b) Cu-BiGa and c) Ga-BiCu

From Fig. 5 it can be seen that the hardness of CuGa alloys increases with addition of Bi. Likewise, BiGa alloys behave in the similar manner as an increase of hardness can be observed with the addition of Cu. However, in this case, the increase of hardness is much more significant. In contrast, the alloys from Ga-BiCu vertical section exhibit quite opposite behavior as the hardness of the alloys BiCu decreases with addition of Ga.

By using experimental results and appropriate mathematical model it is possible to obtain values of hardness along the entire compositional range. To define a mathematical model of dependence of Brinell hardness vs composition of the studied alloys the software package Desig Expert v.9.0.3.1 was used. Out of a possible canonical or Scheffe models [31-33] that meet the requirements of adequacy, the Special Quartic Mixture Model is recommended. Adequacy of the model was confirmed by the Analysis of variance (ANOVA) and the final equation of the predictive model in terms of actual components is:

$$\begin{aligned}
 HB(MN/m^2) = & 9.36810 \cdot x(Bi) + 27.39840 \cdot x(Cu) + 9.57134 \cdot x(Ga) - \\
 & 3.47309 \cdot x(Bi) \cdot x(Cu) - 15.23695 \cdot x(Bi) \cdot x(Ga) - 46.61845 \cdot x(Cu) \cdot x(Ga) + 252.66687 \cdot x(Bi) \\
 & \cdot x(Cu) \cdot x(Ga)^2
 \end{aligned} \tag{1}$$

Iso-lines contour plot of Brinell hardness of the alloys from the Bi-Cu-Ga ternary system at 100°C defined by equation 1 is shown on Fig. 6.

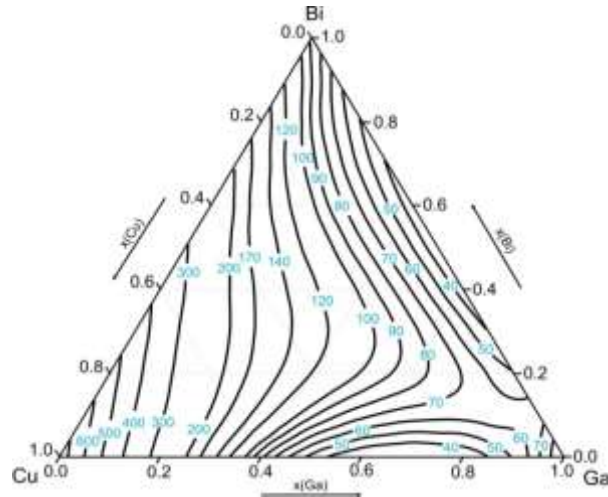
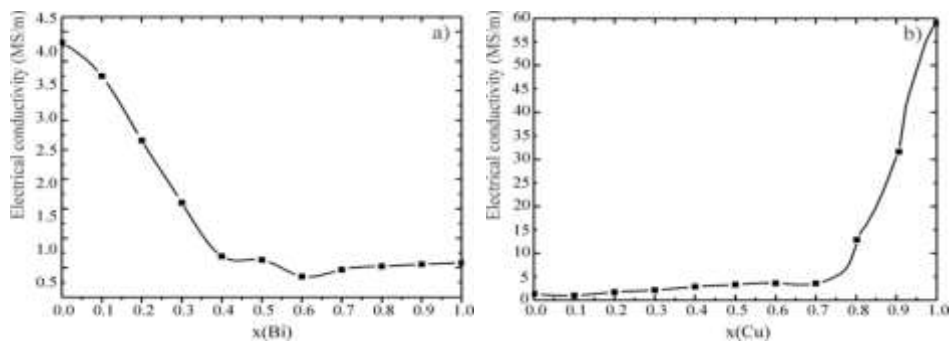


Fig. 6. Iso-lines of Brinell hardness of the ternary Bi-Cu-Ga system at 100 °C

The same ternary alloy samples as well as three binary alloys were used for electrical conductivity measurements. Graphical presentation of the relation between electrical conductivity and mole fraction of components for all investigated samples is shown on Fig. 7. As in case of Brinell hardness, when electrical conductivity of pure elements is considered Bi (0.77 MS/m), Cu (59 MS/m) and Ga (7.1 MS/m) [34], it can be expected that the electrical conductivity of alloys will increase with an increase of copper content.

From Fig. 7 it can be seen that the electrical conductivity of alloys from Bi-CuGa vertical section is decreasing with addition of Bi, on the other hand quite opposite is true for BiGa alloys. As expected, the alloys from the Cu-BiGa vertical section exhibit increase of electrical conductivity with an increase of Cu content and the Cu rich \approx Cu90Bi5Ga5 alloy (sample 20) has the highest value of electrical conductivity (31.18 MS/m).



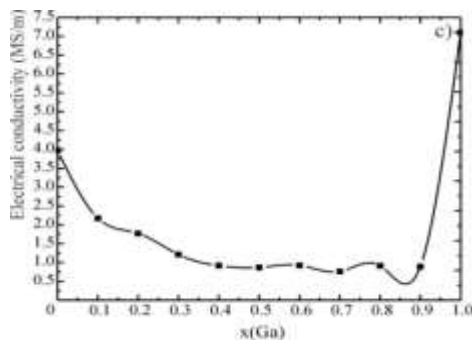


Fig. 7. Graphical presentation of the measured values of electrical conductivity of the investigated alloy samples: a) Bi-CuGa, b) Cu-BiGa and c) Ga-BiCu

Calculation of electrical conductivity for the alloys from the Bi-Cu-Ga ternary system was carried out in the same manner as the aforementioned Brinell hardness calculation. Model summary statistics is suggested Quadratic Mixture Model. The final equation of the predictive model in terms of actual components is:

$$\sigma(\text{MS/m}) = -0.3077 \cdot x(\text{Bi}) + 4.09219 \cdot x(\text{Cu}) + 0.94371 \cdot x(\text{Ga}) - 3.34898 \cdot x(\text{Bi}) \cdot x(\text{Cu}) - 2.38152 \cdot x(\text{Bi}) \cdot x(\text{Ga}) - 5.54438 \cdot x(\text{Cu}) \cdot x(\text{Ga}) \quad (2)$$

Iso-lines contour plot of electric conductivity of the Bi-Cu-Ga alloys defined by equation 2 is shown on Fig. 8.

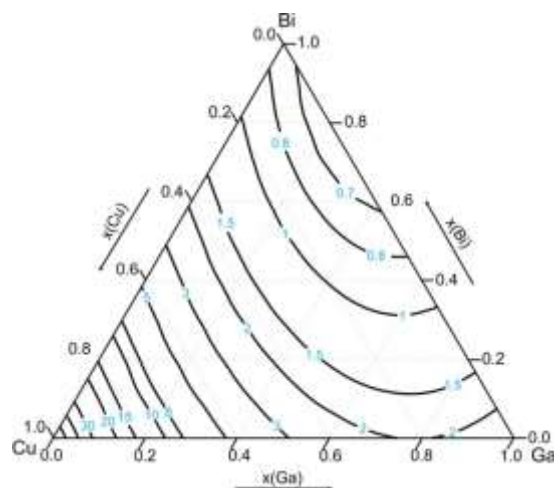


Fig. 8. Iso-lines of electrical conductivity of the ternary Bi-Cu-Ga system at 100 °C

Based on the obtained experimental results and the appropriate mathematical model calculation of electrical conductivity for all alloys from the ternary Bi-Cu-Ga system at 100 °C was possible. The presented plot on Fig. 8 as such may give information on electrical conductivity for all possible ternary Bi-Cu-Ga alloys.

Conclusion

Isothermal section of the ternary Bi-Cu-Ga system at 100 °C was experimentally and theoretically investigated. The presented theoretical prediction of the isothermal section at 100 °C was obtained using thermodynamic calculations and literature data for binary subsystems. Seven of fifteen identified regions from the isothermal section at 100 °C were experimentally tested and in total thirty alloys with compositions along three vertical sections (Bi-CuGa, Cu-BiGa and Ga-BiCu) were prepared and studied. The obtained results of microstructure analysis using optical microscopy and SEM support the theoretical predictions rather well. The results of EDS analysis of the alloy composition and composition of present phases confirm existence of the predicted phases in seven tested regions. In the identified (Bi) solid solution Ga and Cu solubility was ≈ 1 at.% which is negligible. Detected intermetallic compound from binary Cu-Ga system (CuGa_2 , γ_1 , γ_3 , ζ') shows small Ga solubility of maximum 0.54 at. %. Further phase composition analysis of eighteen alloy samples by XRD has also confirmed predicted phase regions. Apart from confirmation of the phase regions, lattice parameters for the three identified phases were calculated and the results show close agreement with literature data (the difference is on third decimal place). With additional micro-Vickers hardness tests hardness of seven phases was measured.

All thirty alloy samples were characterized in terms of Brinell hardness and electrical conductivity. As expected, it was found that the alloy with the highest Cu content has the highest hardness and electrical conductivity. Based on obtained experimental results and mathematical calculation iso-lines for Brinell hardness and electrical conductivity were calculated which can provide information on hardness and conductivity for all possible ternary Bi-Cu-Ga alloys.

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