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# Experimental investigation and characterization of selected as-cast alloys in vertical $Cu_{0.5}Ag_{0.5}$–Al section in ternary Cu–Al–Ag system

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**Abstract** Phase transition temperatures of selected as-cast alloys from ternary Cu–Al–Ag system with overall compositions situated alongside vertical section with equal molar ratios of Ag and Cu were experimentally investigated using differential thermal analysis. Microstructures of the as-cast samples were analyzed using scanning electron microscopy with energy-dispersive spectrometry. Experimentally obtained results were compared with the results obtained by thermodynamic calculation of phase equilibria according to CALPHAD approach.

**Keywords** Cu–Al–Ag system · DTA · SEM–EDS · Phase transitions

## Introduction

Cu–Al-based alloys are materials known for their good shape memory characteristics, biocompatibility and low cost of production compared to other shape memory materials [1–5]. These advantages enable wide range of their usage in different areas of industry, engineering, bioengineering, medicine, electronics, aeronautics, etc. [6–8].

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Among other systems in Cu–Al-based group of materials, ternary Cu–Al–Ag system attracts scientific attention in many fields of material science due to occurrence of martensitic transformation ($\beta$ phase) and phenomenon of shape memory effect [29–35], as well as presence of high transformation temperatures, characteristic for mentioned ternary alloys [1].

There are numerous references concerning Cu–Al–Ag system, and its constitutive binary systems Cu–Al, Cu–Ag and Ag–Al, from early twentieth century till now [9–28]. Significant number of thermodynamic studies regarding Cu–Al–Ag system are published [36–39], presenting the base for actual and further investigations in that field. Chang et al. [40] gave a compilation of thermodynamic data for twenty Cu–Ag-based ternary systems including Cu–Al–Ag. Great contribution to the knowledge on examined ternary system and its constitutive binaries Cu–Al, Al–Ag and Cu–Ag has been made by Witusiewicz et al. [28, 37, 41], who investigated thermodynamic properties and phase equilibria in Cu–Al–Ag system. They revised existing thermodynamic descriptions of these systems by modeling Gibbs energy using CALPHAD method and including their experimental results [7]. Phase equilibria regarding this system were studied in a considerable number of researches [40–42, 47, 48], while mechanical properties [43] and microstructure [44–46] of Cu–Al–Ag alloys were investigated too.

According to the presented literature review in the field of research, it can be concluded that—although Cu–Al–Ag system has been studied in detail for a long period of time—there are still some uncertainties regarding thermodynamics, phase equilibria and crystallography, which have to be explored. Therefore, vertical $Cu_{0.5}Ag_{0.5}$–Al section in Cu–Al–Ag system was investigated in the present work from thermodynamic, phase equilibria and structural point

of view. Also, almost all investigations described in the literature up to now are related to the alloys previously subjected to some kind of heat treatment, either only annealed and quenched or additionally aged, while the research results in this work were done with as-cast alloys.

In the frame of examination done in this work, phase transition temperatures of the selected as-cast alloys with constant Cu:Ag molar ratio equal to 1:1 were experimentally determined using differential thermal analysis (DTA) and then compared with the results of thermodynamic calculation by CALPHAD method, while microstructural investigations were done using scanning electron microscopy/energy-dispersive X-ray spectroscopy (SEM–EDS).

## Experimental

Constituent metals Cu, Al and Ag of the 99.99 % purity, respectively, were subjected to the induction melting during alloy samples preparation. All alloys were melted in graphite crucibles under argon atmosphere and cooled repeatedly in order to improve compositional homogeneity. The samples' masses were 3 g with total loss less than 0.5 %. So, the nominal compositions of the samples–prepared alloys were used in further analysis as correct ones. Four samples were investigated with following composition (in at.%): $Al_{20}Cu_{40}Ag_{40}$, $Al_{50}Cu_{25}Ag_{25}$, $Al_{60}Cu_{20}Ag_{20}$ and $Al_{80}Cu_{10}Ag_{10}$.

Phase transformation temperatures of the as-cast alloy samples were determined experimentally by DTA method using NETZSCH STA 449F1 Jupiter instrument, under the following conditions: argon atmosphere, heating rate 10 °C $min^{-1}$, and alumina as reference material.

Scanning electron microscopy was carried out using SEM TESCAN VEGA TS 5136MM instrument and energy-dispersive spectrometry on Bruker spectrometer at 20 kV. Point analysis by EDS was performed in ten or more points depending on the sample for each phase, and average value was calculated.

## Thermodynamic calculation of the Cu–Al–Ag phase diagram

Phase diagram of Ag–Al–Cu ternary system was calculated by the CALPHAD method [49, 50]. CALPHAD method is based on calculation of the Gibbs energy of a phase as a function of its composition, temperature and pressure [7]. Gibbs energy data for all phases appearing in the investigated system should be stored as polynomial functions in the thermodynamic database [7]. Calculation of phase equilibria was done using constrained minimization of system's Gibbs energy method [49, 50]. In this work, calculations were done using PANDAT software [51].

Crystallographic data and thermodynamic models of all phases in the Ag–Al–Cu ternary system, considered for thermodynamic calculation of phase equilibria, are presented in Table 1.

Gibbs energies of pure metals Ag, Al and Cu in their stable and metastable phases from SGTE database [52] were used. Optimized thermodynamic parameters, taken from the literature, used for calculation of the Ag–Al–Cu phase equilibria in this study, are identical to those previously published [7].

Calculated phase diagrams of binary boundary systems Ag–Al, Ag–Cu and Al–Cu are shown in Fig. 1.

## Results and discussion

Phase transition temperatures of four chosen as-cast alloys with equal molar ratios of Cu and Ag and aluminum molar

**Table 1** Considered phases of the Ag–Al–Cu ternary system, their crystallographic data and thermodynamic models [7]

| Phase | TD database name | Pearson symbol | Space group | TD model |
|---|---|---|---|---|
| Liquid | LIQUID | – | – | $(Ag,Al,Cu)_1$ |
| (Ag) (Al) (Cu) | FCC_A1 | *cF*4 | *Fm3m* | $(Ag,Al,Cu)_1(Va)_1$ |
| $\beta$ | BCC_A2 | *cI*2 | *Im3m* | $(Ag,Al,Cu,Va)_1(Va)_3$ |
| $\xi$ | HCP_A3 | *hP*2 | $P6_3/mmc$ | $(Ag,Al,Cu)_1(Va)_{0.5}$ |
| $\mu$ | CUB_A13 | *cP*20 | $P4_132$ | $(Ag,Al,Cu)_1(Va)_1$ |
| $\theta$ | XZ2_C16 | *tI*12 | *I4/mcm* | $(Ag,Al,Cu)_1(Al)_2$ |
| $\eta$ | ALCU_ETA | *mC20* | *C2/m* | $(Al,Cu)_1(Ag,Cu)_1$ |
| $\zeta$ | ALCU_ZETA | – | – | $(Al)_9(Ag,Cu)_{11}$ |
| $\varepsilon$ | ALCU_EPS | *hP4* | $P6_3/mm$ | $(Al,Cu)_1(Cu)_1$ |
| $\delta$ | ALCU_DEL | – | – | $(Al)_2(Cu)_3$ |
| $\gamma D8$ | GAMMA_D83 | *cP52* | *P43m* | $(Al)_4(Al,Cu)_1(Ag,Cu)_8$ |
| $\gamma$ | GAMMA_H | – | – | $(Al)_4(Al,Cu)_1(Ag,Cu)_8$ |

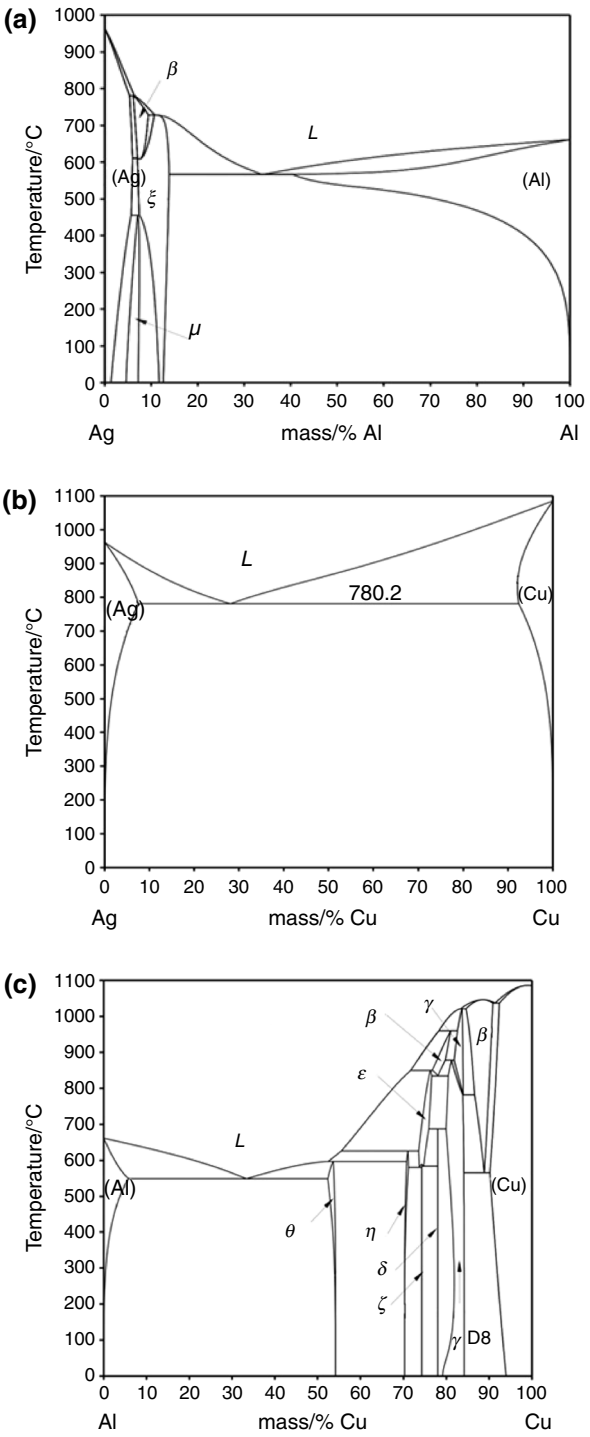


**Fig. 1** Calculated phase diagram of binary system: **a** Ag–Al, **b** Ag–Cu and **c** Al–Cu

content equal to 0.2, 0.5, 0.6 and 0.8 were measured using DTA and compared with the results obtained by thermodynamic calculation of phase equilibria. To insure the reproducibility of the results, DTA measurements included two heating and cooling cycles for each investigated alloy, according to often used procedure [53, 54]. Reading of the phase transition temperatures was enabled by software delivered with the instrument. Phase transition temperatures obtained from the second heating and cooling runs were considered to be more accurate and further analyzed. The experimental error of determined phase transition temperatures has been estimated to ±1 °C. The DTA results are presented in Table 2.

On heating, the extrapolated peak onset temperature was used for the determination of solidus and ternary invariant reaction temperatures, while other phase transition temperatures, including liquidus, were taken from the peak maximum temperature [55]. In cooling curves, only peak onset temperature was used [55].

For investigated samples $Cu_{25}Al_{50}Ag_{25}$ and $Cu_{10}Al_{80}Ag_{10}$ (presented in Table 2), characteristic DTA heating and cooling curves with marked phase transition temperatures are shown in Figs. 2 and 3, respectively.

Comparison between obtained DTA results and thermodynamic calculation is shown in Fig. 4.

For the $Cu_{25}Al_{50}Ag_{25}$ alloy, solidus temperature was detected at 536.6 °C on heating (onset temperature of the first peak on heating—Fig. 2a) and 537.0 °C (onset temperature of the last peak on cooling—Fig. 2b). According to thermodynamic calculation results, this phase transition is related to the appearance of ternary transition-type reaction $L + \eta = \theta + \xi$ at temperature of 562.3 °C (Fig. 4). Experimentally determined temperature of that invariant reaction presented in this study is in better agreement with calculated results (534.7 °C) published by Witusiewicz et al. [41].

Onset of the second sharp peak on heating is identified at temperature of 612 °C. According to calculation results (Fig. 4), thermal effect is connected with the appearance of another invariant transition-type reaction: $L + \varepsilon = \xi + \eta$, at calculated temperature of 602.3 °C.

Maximum of the third and last identified peak on heating, occurring at 710 °C, represents liquidus temperature. From the corresponding DTA cooling curve, the liquidus temperature, determined as onset temperature of the first peak, is 713.7 °C.

According to thermodynamic calculation, solidus of the samples $Cu_{10}Al_{80}Ag_{10}$ and $Cu_{20}Al_{60}Ag_{20}$ represent ternary eutectic reaction $L \leftrightarrow (Al) + \xi + \theta$ at 517.8 °C (horizontal line in Fig. 4).

The existence of this calculated ternary eutectic reaction is confirmed by experimental results from this work.

Figure 3 shows DTA heating and cooling curve for the $Cu_{10}Al_{80}Ag_{10}$ alloy, where onset of the first sharp peak obtained on heating and onset of the last sharp peak obtained on cooling correspond to the temperature of ternary eutectic reaction. Measured temperature of this ternary eutectic reaction obtained on heating is 503.2 °C for the $Cu_{10}Al_{80}Ag_{10}$ sample and 504.8 °C for the

**Table 2** DTA results for the investigated alloys of Cu–Al–Ag ternary system

| Nominal sample composition/at.% | | | Phase transition temperature/°C | | | | | |
|---|---|---|---|---|---|---|---|---|
| Al | Cu | Ag | Solidus | | Other phase transitions | | Liquidus | |
| | | | Heating | Cooling | Heating | Cooling | Heating | Cooling |
| 20 | 40 | 40 | – | – | – | – | 781.6 | 785.0 |
| 50 | 25 | 25 | 536.6 | 537.0 | 612.0 | 632.0 | 710.0 | 713.7 |
| 60 | 20 | 20 | 504.8 | 514.0 | 556.0 | 560.0 | 630.0 | 638.0 |
| 80 | 10 | 10 | 503.2 | 497.4 | – | – | 547.5 | 539.5 |

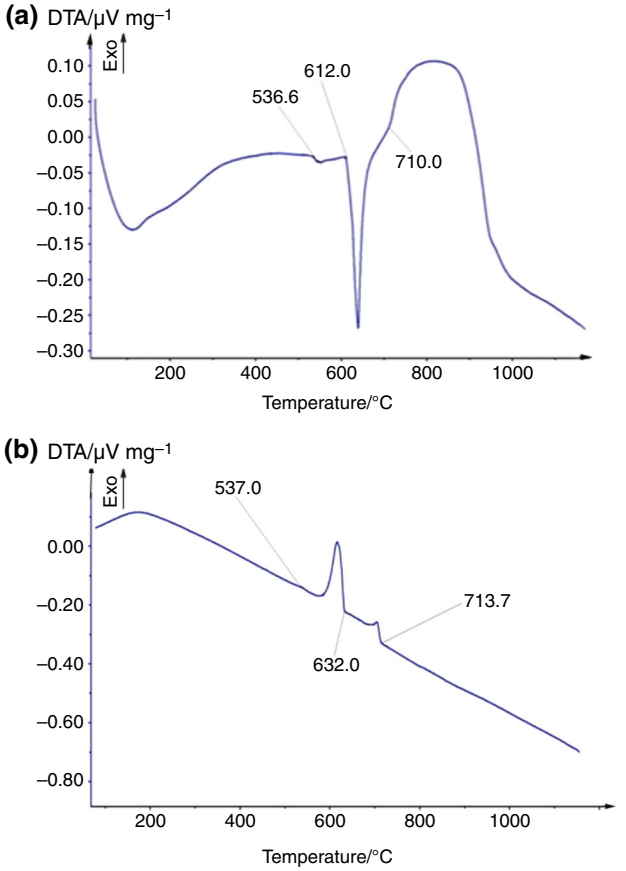


**Fig. 2** DTA heating (**a**) and cooling curve (**b**) of $Cu_{25}Al_{50}Ag_{25}$ alloy

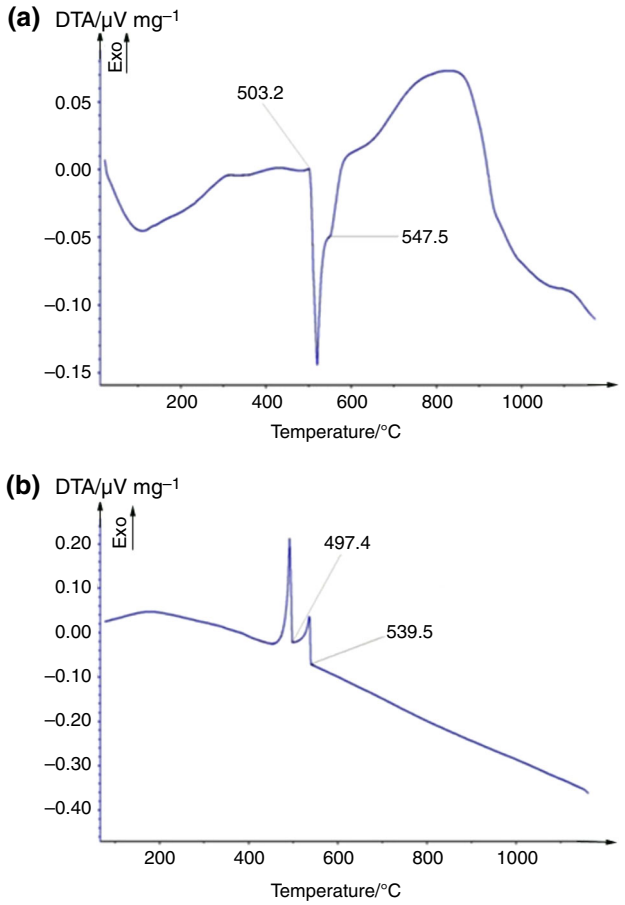


**Fig. 3** DTA heating (**a**) and cooling curve (**b**) of $Cu_{10}Al_{80}Ag_{10}$ alloy

$Cu_{20}Al_{60}Ag_{20}$ sample, which is significantly lower the than calculated eutectic temperature from this work, but it is in reasonable agreement with corresponding temperature of ternary eutectic reaction at 500.5 °C in Ref. [41].

Structural characterization of the investigated sample alloys was carried out using scanning electron microscopy with energy-dispersive X-ray spectroscopy. SEM micrographs of selected samples with marked phases, determined using SEM–EDS, are shown in Fig. 5.

Microstructures of the samples $Cu_{40}Al_{20}Ag_{40}$, $Cu_{20}Al_{60}Ag_{20}$ and $Cu_{10}Al_{80}Ag_{10}$ from $Cu_{0.5}Ag_{0.5}$–Al section with molar ratio Cu:Ag = 1:1 are shown in Fig. 5.

In Fig. 5a, two phases are shown in the as-cast sample alloy $Cu_{40}Al_{20}Ag_{40}$: silver-based solid solution (Ag) as a bright gray phase, and dark gray phase representing γD8 phase. Regarding the micrograph, it is possible that silver precipitates around grains boundaries. Average composition of the (Ag) phase was 17Cu–2Al–81Ag and of the γD8 phase 92.5Cu–3.7Al–3.8Ag.

Figure 5b shows that microstructure of as-cast eutectic alloy $Cu_{20}Al_{60}Ag_{20}$ at room temperature consists of three phases, as it was thermodynamically predicted (Fig. 4). Dark phase corresponds to $\theta$ phase, bright gray grains

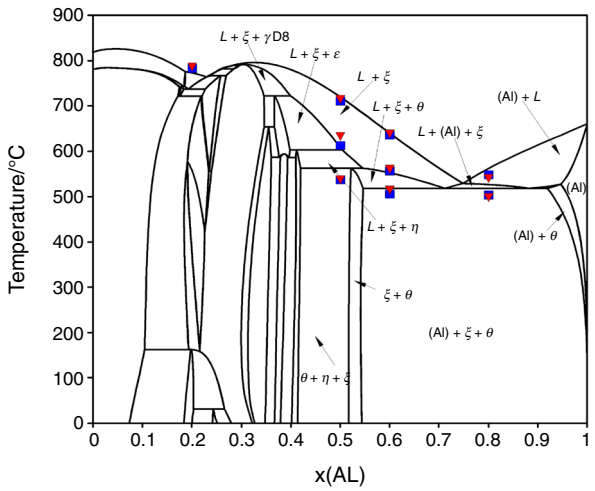


**Fig. 4** Calculated vertical section $Cu_{0.5}Ag_{0.5}$–Al with marked experimentally determined phase transition temperatures for investigated samples (*squares*—heating regime; *inverse triangles*—cooling regime)

represent primary crystallized grains of $\xi$ phase, while light lamellar grains represent aluminum-based solid solution (Al), formed during eutectic solidification. Average phase compositions were: (Al) phase 1.5Cu–65.5Al–33.0Ag; $\xi$ phase 6.0Cu–39.0Al–55.0Ag; and $\theta$ phase 19.0Cu–54.0Al–27.0Ag.

Microstructure of the as-cast sample $Cu_{10}Al_{80}Ag_{10}$, shown in Fig. 5c, has the same three-phase structure as alloy $Cu_{20}Al_{60}Ag_{20}$. In the case of $Cu_{10}Al_{80}Ag_{10}$ alloy, primary crystallized phase is aluminum-based solid solution (Al)—predominantly large gray crystals shown in Fig. 5c. Bright gray phase corresponds to $\theta$ phase, and light areas represent $\xi$ phase visible in ternary eutectic structure. Average compositions of phases presented in micrograph were: (Al) phase 3.3Cu–89.7Al–7.0Ag; $\theta$ phase 30.0Cu–63.0Al–7.0Ag; and $\xi$ phase 7.0Cu–43.8Al–49.2Ag.

As can be seen in Fig. 5, microstructure of the alloy $Cu_{40}Al_{20}Ag_{40}$ differs compared to the other two samples ($Cu_{20}Al_{60}Ag_{20}$ and $Cu_{10}Al_{80}Ag_{10}$). It was expected due to the fact that the microstructure of two samples with higher aluminum content partly consists of eutectic structures.

## Conclusions

Selected as-cast alloys of the ternary Cu–Al–Ag system with variable molar ratio of Al and equal molar ratios of Ag and Cu were experimentally investigated in this work. Phase transition temperatures were determined using DTA heating and cooling regimes. Two ternary transition-type reactions and one ternary eutectic reaction were identified by DTA experiments and microstructure investigations. It

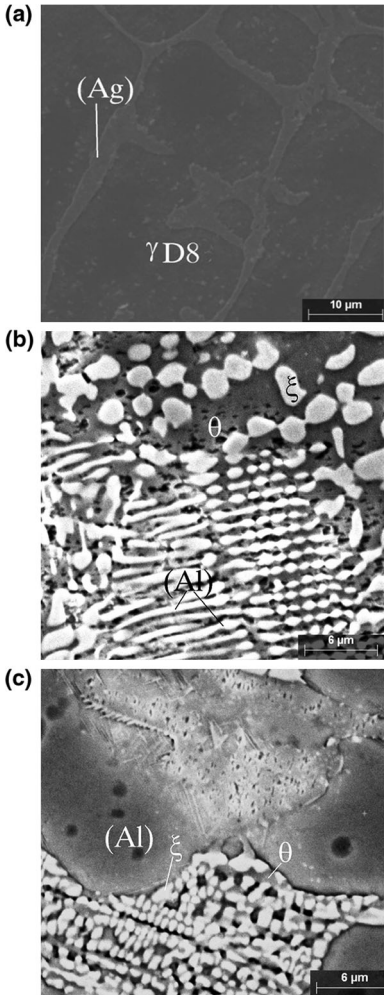


**Fig. 5** SEM micrographs of as-cast alloy samples: **a** $Cu_{40}Al_{20}Ag_{40}$, **b** $Cu_{20}Al_{60}Ag_{20}$ and **c** $Cu_{10}Al_{80}Ag_{10}$ (in at.%)

was determined that Al-rich as-cast alloys (with 60 and 80 at.% of Al) on heating and cooling experience ternary eutectic reaction $L \leftrightarrow (\mathrm{Al}) + \xi + \theta$. Experimentally determined temperature of this invariant reaction obtained as average value from samples DTA heating curves is 504 °C. Eutectic three-phase lamellar regions were observed in the micrographs of investigated alloys with 60 and 80 at.% of Al. Experimentally obtained results were compared with thermodynamically calculated phase diagram of the $Cu_{0.5}Ag_{0.5}$ –Al vertical section and showed reasonable agreement.

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