Effect of Heat Treatment on Microstructure of Al4Cu-SiC Composites Consolidated by Powder Metallurgy Technique

A. Wąsik, B. Leszczyńska-Madej, M. Madej, and M. Goły

Silicon carbide particles-reinforced Al4Cu composites containing 2.5, 5 and 7.5 wt.% of reinforcement were fabricated using powder metallurgy (PM) technique. The sintered Al4Cu-SiC composites were solution treated for 6 h at two different temperatures (495 and 530 °C) and then aged at 180 °C for various aging periods (4, 12 and 24 h). Effects of heat treatment on the microstructural changes and microhardness were investigated by scanning electron microscope, transmission electron microscope, x-ray diffraction and microhardness tests. The results indicate the ceramic particles-reinforced Al4Cu matrix requires different heat treatment parameters compared to the unreinforced alloy. The applied solution temperature did not allow for thorough dissolution of alloy phases in the matrix material produced by PM route. However, in Al4Cu-SiC composites the main strengthening phase (Al_2Cu) was uniformly distributed in the matrix. The solid-solution temperature increase affects the reduction in time to reach peak hardness. The highest increment in microhardness by aging treatment was observed for composite with the addition of 5 wt.% of SiC solution treated at 530 °C for 12 h (110 HV0.05).

1. Introduction

The wide range of applications of light metal-matrix composites (MMCs) has attracted the attention because of their increased mechanical properties. However, it has to be noted that the obtained properties of composite materials are significantly influenced by the chemical composition and the methodology used to consolidate the materials, which enables formulating permanent matrix–reinforcement bonding, and more importantly by the microstructural evolution associated with applied heat treatment procedure (Ref 1).

One of the methods for improving the mechanical properties of aluminum alloys is heat treatment, which has a strong influence on the microstructure of the composites (Ref 2). Al-Cu alloys with a copper content up to approximately 5% are intended for heat treatment (precipitation hardening), as the copper is one of the most important aluminum alloy elements due to its high solubility and strengthening effect. Precipitation hardening, also known as age hardening, is the heat treatment of aluminum alloys involving solution treatment, quenching

and aging at both room (natural aging) and elevated (artificial aging) temperatures (Ref 3). The strengthening effect of the material subjected to precipitation hardening results from the formation of extremely fine, uniformly dispersed particles of a second phase within the original phase matrix. The different age-hardening behavior has been nevertheless observed in aluminum matrix composites produced by powder metallurgy. Effect of the addition of ceramic reinforcing phase in aluminum alloy matrix on the aging process behavior requires better understanding. Scientific sources report that on the one hand the rapid hardening of composite materials and attaining peak hardness much earlier has been observed compared to the unreinforced alloy, which could indicate the aging characteristic accelerated with the reinforcement (Ref 4, 5). However, at the same time it has been shown that the presence of ceramic reinforcements leads to a significant retardation in the kinetics of hardening (Ref 6, 7). There are a number of factors influencing the nature of change in kinetics and magnitude of hardening during aging the MMCs, such as chemical composition of the matrix material, size, shape and weight fraction of reinforcement and also the method of processing the composite, not to mention the applied heat treatment parameters (Ref 8, 9).

In light of the above, one of the aims of this work was to investigate the influence of the addition of silicon carbide particles to Al4Cu matrix on the heat treatment process and the next one was to research the effect of variable heat treatment parameters on microstructural changes and mechanical properties of Al4Cu-SiC composites.

2. Experimental Procedure

The matrix alloy powders were Al and Cu with weight fraction of Cu of 4%. The average particle size of aluminum powder, produced by argon gas atomization, was below 63 μm.

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(The fractions of the powder are as follows: above 63 μm—5%, in the rage of 32-63 μm—45-70% and below 32 μm—the rest.) The electrolytic copper powder had an average size of < 40 μm. The weight fraction of SiC particles in the composite material was 2.5, 5 and 7.5%, and the average particle size ranged below 2 μm. The morphology of raw material powders used in this study is presented in Fig. 1. Aluminum powder is characterized by an elongated and irregularly shaped particle with a relatively smooth surface. Copper powder has the dendritic shape, which is characteristic for electrolytic manufacturing. Silicon carbide particles are characterized by an angular shape. Al4Cu matrix alloy composites were prepared by blending pure aluminum powder and pure copper powder with silicon carbide particles in appropriate mass proportions of the reinforcing phase to the matrix material for 30 min in a Turbula T2F mixer. Then, the mixed powders were put into a rigid die and pressed at room temperature under a pressure of 300 MPa on a single-action compaction press. The matrix walls were lubricated with glycerin to prevent powder adhesion to the matrix. The obtained green bodies were the cuboid shape (4.5 × 5 × 40 mm). The compacts were sintered under nitrogen atmosphere at 600 °C for 1 h. The not-heat-treated sinters were slowly cooled in the furnace cooler at a rate of about 40 °C.

The sintered Al4Cu-SiC composite samples subjected to heat treatment were solution treated at 495 and 530 °C for 6 h, water quenched and then aged at 180 °C for various aging periodic times: 4, 12 and 24 h. The main purpose of solution treatment is to dissolve the precipitates and obtain a homogeneous solution, and then preserve the solid solution by rapidly cooling to a lower temperature. The heat treatment parameters recommended for the peak hardening (T6 treatment) for alloys of similar compositions mentioned elsewhere (Ref 3, 8, 10, 11) consist of solution treatment in the temperature ranging from 495 to 530 °C and aging treatment at 180 °C. The selected conditions of solution treatment represent two extreme temperatures and thus expected different degrees of dissolution of solute in the matrix. This action is intended to examine the effect of heat treatment procedure on microstructure and mechanical properties of tested materials. Age-hardening behavior of the Al4Cu matrix and Al4Cu-SiC composites was analyzed by measuring the microhardness of the samples at different solution temperatures and aging times using a Vickers hardness testing machine. Vickers microhardness was measured on mirror-shine polished sample surfaces using a Shimadzu HMV-G microhardness tester with a Vickers indenter. A load of 100 g was applied in the microhardness measurement. Microstructure and morphology of precipitants in Al4Cu alloy matrix without and with the addition of SiC particles at each stage of heat treatment were observed with a Schottky electron gun scanning electron microscope with Thermo Scientific NORAN System 7 x-ray microanalysis system (EDS)—Hitachi SU-70. Samples for microstructure examinations were mechanically ground and polished with diamond paste and colloidal suspension of SiO_2. The microstructure was additionally examined by transmission electron microscopy (TEM) using a JEOL JEM 2010 ARP microscope. The thin foils for electron transmission observations were prepared by ion milling. Information about the phase composition was provided by the EDS and x-ray diffraction (XRD) analyses (Bruker, Co Kα = 1. 97 Å radiation).

3. Results and Discussion

3.1 Density of Sinters

Figure 2 presents the density measurements using Archimedes method of not-heat-treated sinters of Al4Cu matrix and composite materials. The highest relative density was characterized by the matrix material (96.99% of theoretical density). The addition of silicon carbide particles resulted in a decrease in the relative density, which remains constant regardless of the weight fraction of reinforcing phase and ranges from 95.87 to 94.99% of the theoretical density.

As it results from the above-presented values of Archimedes density powder metallurgy, processed composites are characterized by a high degree of compaction and the porosity not exceeding 6%. However, it is expected that a further increase in the content of the strengthening phase (above 7.5 wt.% of SiC) will result in a decrease in the degree of compaction of the material. It is associated with porosity at the interface between SiC particles and matrix indicating weak bonding formed during consolidation using conventional powder metallurgy process. Alloyed powders are less prone to compaction than pure metal powders. Thus, the increase in weight fraction of strengthening phase in the form of SiC particles in Al4Cu matrix hinders the achievement a high degree of compaction and resulted in higher porosity. Moreover, the research conducted by the authors (Ref 11) shows that the porosity influences for the aging kinetics, as the porous materials aged slower than the dense materials.

3.2 Microstructural Observation

Figure 3 shows the micrograph of matrix material and composite material with the addition of 5 wt.% of SiC after sintering process. The microstructure consists of regular shape of aluminum grains and locally scare pores. Additionally, in
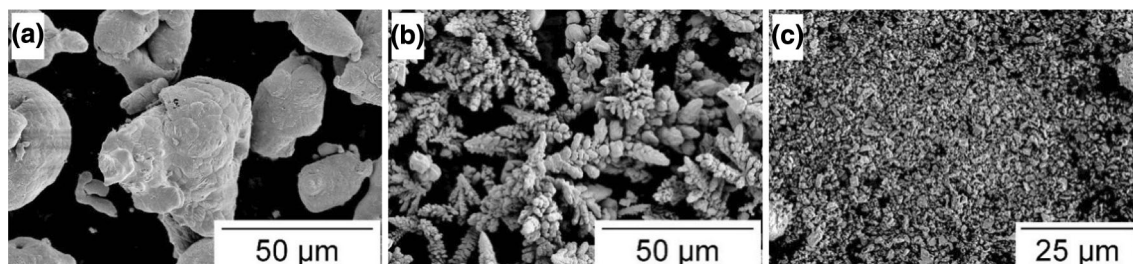
Fig. 1 SEM micrographs of powders: (a) aluminum, (b) copper, (c) SiC

composites with the addition of strengthening phase, the silicon carbide particles are visible on grain boundaries. In the microstructure of non-heat-treated matrix materials as well as composite materials, the primary grain boundaries are revealed by the white continuous network spread along the Al powder particles. The analysis of EDS in Fig. 4 indicated high content of copper in these areas which are alloy phases formed during slow cooling after sintering process. The composition of the precipitates is corresponding to the composition of the Al_2Cu phase (points 1, 2 and 3). The strengthening caused by coarser Al_2Cu precipitates located on the grain boundaries is insignificant. Furthermore, large hard particles on the grain boundaries cause a significant deterioration of fracture toughness (Ref 12). XRD pattern of composite with the addition of 5 wt.% of SiC is shown in Fig. 5. It can be confirmed that the composite

materials are mainly composed of Al matrix, SiC particles and Al_2Cu phase and as the sintering process was carried out under nitrogen atmosphere, aluminum nitride was formed. An advantage of sintering aluminum and its alloy under nitrogen atmosphere is the formation of aluminum nitride which is acting as an additional strengthening factor and in addition penetrating into the pores increases the degree of compaction. The formation of aluminum nitride is an exothermic reaction which may occur if nitrogen gains a direct contact with the freshly exposed aluminum surface (Ref 13).

Copper is one of the most important aluminum alloy elements due to its high solid solubility in aluminum and strengthening effect. The alloy with the content of 4% of copper at thermodynamic equilibrium at ambient temperature is
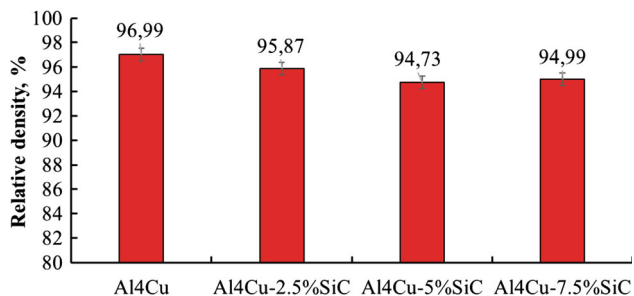

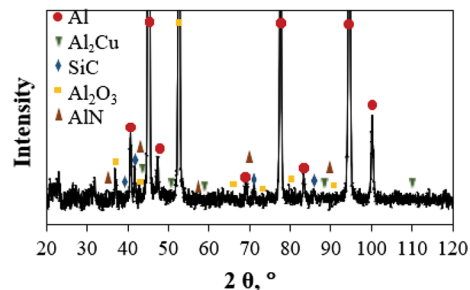**Fig. 2** Variation of Archimedes density of non-heat-treated sinters

Fig. 5 X-ray diffraction pattern of the Al4Cu-5 wt.% of SiC composite material
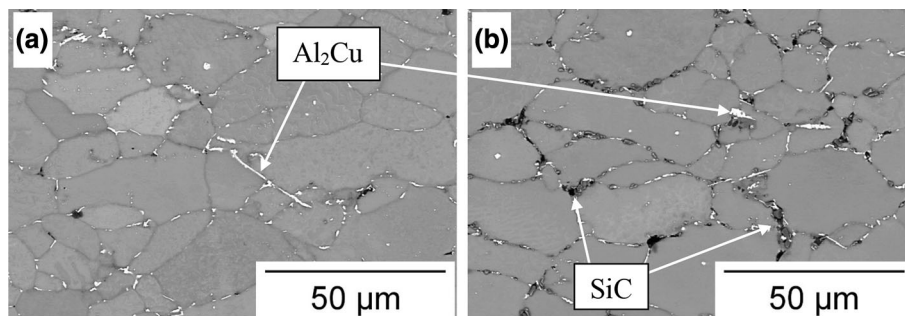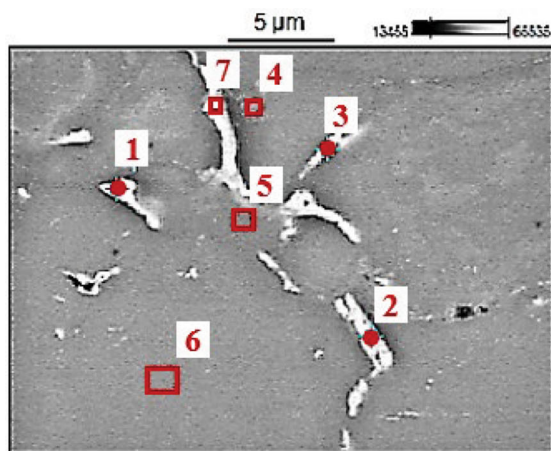

Fig. 3 SEM microstructure of PM: (a) Al4Cu alloy, (b) Al4Cu-5 wt.% of SiC

No	O, wt%	Al, wt%	Fe, wt%	Cu, wt%
pt 1	1	72	–	27
pt 2	3	65	8	24
pt 3	1	76	–	23
pt 4	5	93	–	2
pt 5	1	91	1	7
pt 6	–	95	–	5
pt 7	5	74	5	16

Fig. 4 Chemical analysis carried out in micro-regions of as-sintered Al4Cu matrix, SEM

composed of two phases: a matrix consisting of solid solution α and intermetallic phase θ (Al_2Cu). The purpose of solution heat treatment is to dissolve the θ-phase particles and obtain a uniform solid-solution structure. The maximum solid solubility in aluminum matrix of the copper decreases with temperature dropping. While the solid solubility of copper at 550 °C is 5.65%, at ambient temperature the solid solubility of copper in aluminum drops to 0.2%. Therefore, in solution treatment process the second soluble phase θ gradually dissolves into the aluminum matrix as solution temperature rises (Ref 3).

Figure 6 shows SEM images of matrix material Al4Cu with different heat treatment processes. There is a significant amount of micron coarse intermetallic phases formed inside the grain and on grain boundaries both after solution at 495 and 530 °C. It indicates that the applied solution temperature in the first instance (495 °C) is too low, as in the microstructure there are still a lot of micron particles remaining in the matrix. Precipitate particles of alloy phase are in the form of coagulants with high content of copper. Phase precipitations are accompanied by a reduction in the concentration of dissolved copper atoms in the matrix. As the solution temperature increases, the alloy phase particles would be expected to dissolve, and as a result, the solid solution with the maximum degree of supersaturation would be obtained. However, when the solution temperature reaches 530 °C, alloy phases re-emerge as a result of too high solution temperature. Comparing the x-ray diffraction pattern obtained for the tested solution temperature, it was found that an increase in solution temperature caused the size of alloy phases to become smaller and their number to become greater (Fig. 7). Comparing the x-ray diffraction patterns of the Al4Cu alloy solution treated at different temperatures, it was found the peak intensity corresponding to the Al_2Cu phase is higher in the alloy previously subjected to solution treatment at 530 °C, indicating a higher number of these precipitates, while the peak width corresponding to Al_2Cu phase is higher after solution treatment at 495 °C, indicating a larger size of precipitates. Apart from the growth of the alloy phases previously formed on grain boundaries, new, discoid-shaped precipitate particles of Al_2Cu phase are formed. The microstructure of the matrix after aging, which has been pre-treated at 495 °C, shows a similar character as after solution treatment. An increase in the time of aging favors impoverishment of the precipitate formation in areas adjacent to the grain boundaries (Fig. 6c). The width of these areas is affected by the rate of solution and aging temperature. The increase in the width of zones free from precipitates of Θ phase is caused by applying a lower cooling rate in the solution treatment and a higher aging temperature. The presence of zones free of precipitates on both sides of the grain boundary can be explained by the phenomenon of micron particles of alloy phase growth on grain boundaries, which by increasing their size cause "drawing-in" dissolved atoms from areas adjacent to the grain boundary (Ref 14). An increase in the solution temperature to 530 °C resulted in a decrease in the size of the alloy phases in the matrix subjected to the aging hardening at different time periods. With increasing aging time,
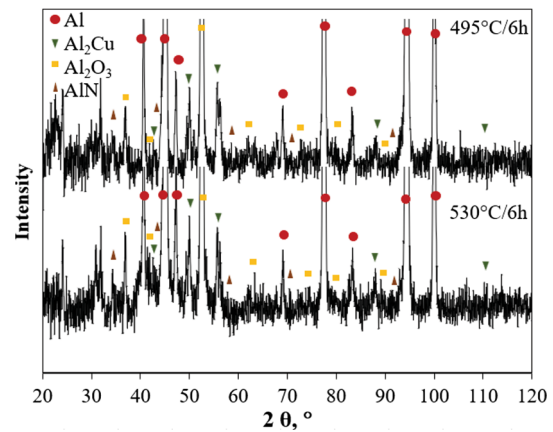

Fig. 7 X-ray diffraction pattern of the Al4Cu solution treated at different temperatures
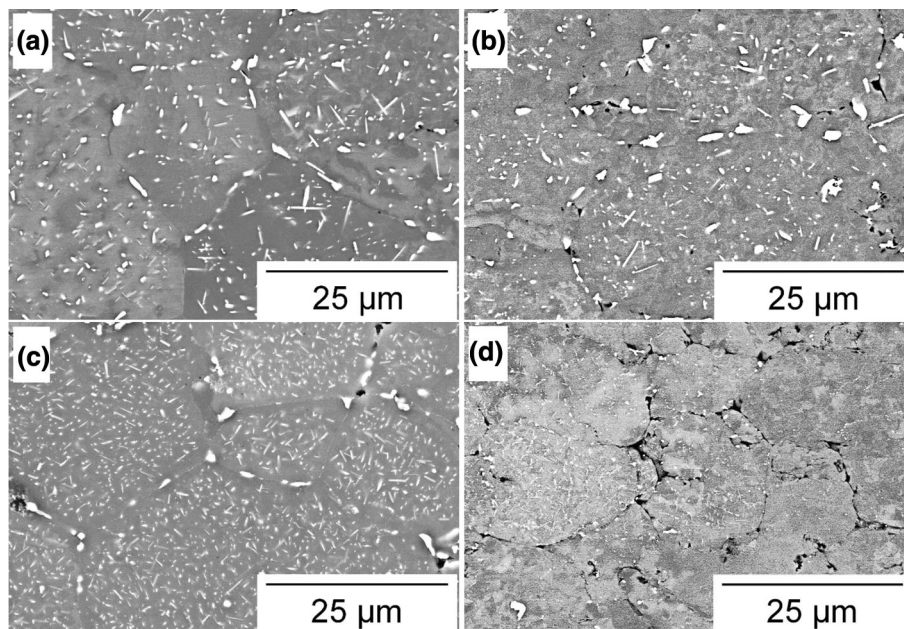

Fig. 6 SEM images of Al4Cu matrix after heat treatment at different temperatures: (a) 495 °C solution; (b) 530 °C solution; (c) 495 °C solution + artificial aging (24 h); (d) 530 °C solution + artificial aging (24 h)

the number of precipitates in grains increases. After 24 h of aging in the microstructure, very fine evenly distributed particles of alloy phase can be observed (Fig. 6d).

There was a difference between the heat-treated microstructure of the matrix material and composite materials reinforced with SiC particles (Fig. 8). The presence of SiC strengthening inhibits the precipitation of Al_2Cu phase which is stopped at the boundaries, mainly on the silicon carbide particles. Therefore, the amount of precipitates of a second phase inside the grain is lower and at the same time their size is finer compared to the matrix material without the addition of ceramic particles. In microstructure of composites, subjected to solution treatment at 495 °C, the precipitations of Al_2Cu phase located on grain boundaries are visible (Fig. 8a). There were no precipitates inside the grains. Increasing the solution temperature up to 530 °C caused the Al_2Cu phase precipitation inside the grain, while the grain boundaries are free of Al_2Cu precipitate particles (Fig. 8b). However, the amount and size of second phase inside the grain are much smaller compared to that of the Al4Cu matrix subjected to the solution at the same conditions. It indicates that applying solution temperature equal to 530 °C in solution treatment of composite materials allows for almost complete dissolution of intermetallic phase.

An increase in the aging time to 24 h of composites previously solution treated at 495 °C favored precipitation inside the grain fine dispersive precipitates (Fig. 8c). The solution treatment carried out at 530 °C and aging for 12 h resulted in precipitation in composite material of needle-shaped, alternately arranged second phases. Figure 9 shows the TEM image of the composite material with the addition of 5 wt.% of SiC treated through solution at 530 °C and aging for 12 h. It can be seen there are many nanosized precipitated phases dispersively distributed in the matrix.

The hard ceramic SiC particles introduced into the plastic aluminum alloy matrix increase the strain energy in the periphery of the particles in the matrix. These tendencies may be due to the mismatch in the thermal expansion coefficients of ceramic particles and Al matrix during solution treatment and contributes to the formation of high dislocation densities at the reinforcement/matrix phase boundary (Ref 15, 16). Thus, dislocations stimulate the increase in hardness as they act as non-uniform nucleation sites in the interface following the age treatment. The residual stress and high dislocation density can lead to formation of excess free vacancies. Enhanced solute diffusivity in the matrix of composites can accelerate the GP zone formation, as the GP zone requires vacancy clusters. The main strengthening phase of Al-Cu alloy is θ (Al_2Cu). High dislocation density in the composite matrix provides more nucleation sites for θ precipitation. The mechanism of strengthening in precipitation hardening consists in impeding the movement of dislocations, which are stopped by incoherent precipitates from a supersaturated solid solution. In TEM images, the phenomenon of dislocation movement inhibition on the precipitated Al_2Cu phases can be observed (Fig. 9).

Additionally, the aging kinetics will be affected by the presence of porosity at the particulates' interface. The aging kinetics of porous materials is slower than that of the dense materials due to the annihilation of dislocation as a result of the presence of higher porosity. It is associated with the location of SiC particles in the free space in-between larger grains of the matrix during quenching and thus with the reduction in the magnitude of misfit strains which affects the reduction in the average dislocation density and reduction in the heterogeneous nucleation volume around the particulates. The presence of porosity in the locally formed agglomerations of SiC particles allows the individual SiC particles to adapt to the compressive forces acting during quenching and hence reduces the precipitation of strengthening phase through reducing the heterogeneous nucleation sites in the form of dislocations.
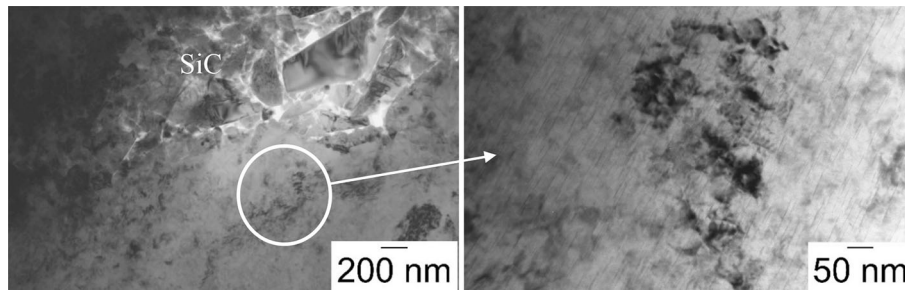
3.3 Age-Hardening Behavior

In the investigated Al-Cu alloy, during age hardening, the precipitation process has the following sequence: GP zone →
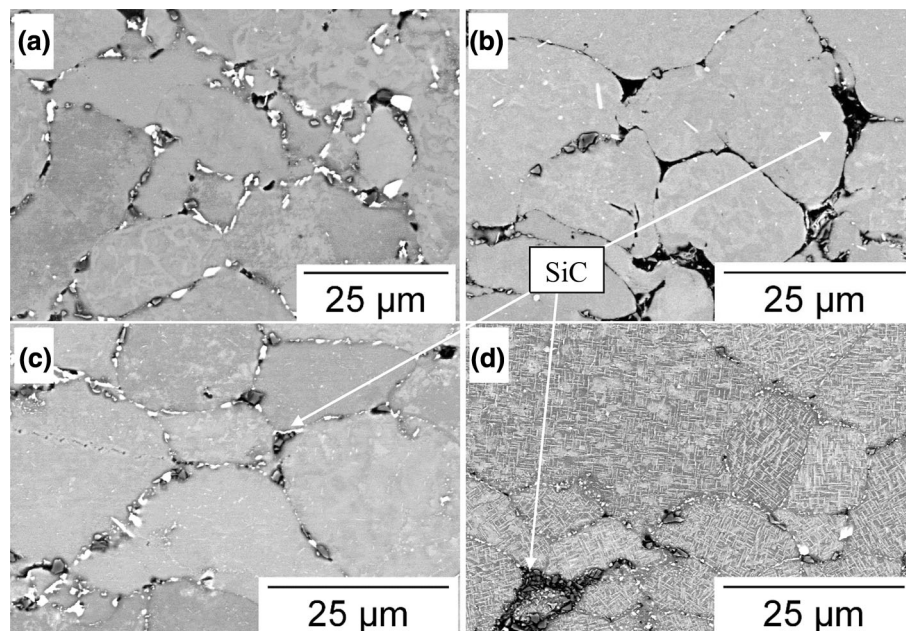
Fig. 8 SEM images of Al4Cu-5 wt.% of SiC composite after heat treatment at different temperatures: (a) 495 °C solution; (b) 530 °C solution; (c) 495 °C solution + artificial aging (24 h); d) 530 °C solution + artificial aging (24 h)

Fig. 9 TEM images of Al4Cu-5 wt.% of SiC composite after solution treatment at 530 °C and artificial aging for 12 h

GPII zone (θ'') $\rightarrow$ θ' intermediate $\rightarrow$ θ (Al$_2$Cu) phase (Ref 17). Therefore, metastable precipitated phases are formed before finally forming the equilibrium phase, such as Al$_2$Cu. The most effective, from the point of view of increasing hardness, are the GPII zone and θ' intermediate (Ref 18, 19). The precipitation of the stable θ phase decreases hardness (over aging) (Ref 3, 20). The addition of SiC reinforcement into aluminum alloy matrix generally accelerates the aging kinetics but does not change its aging precipitation process. Introducing the SiC particles to the aluminum alloy generates additional vacancy sinks due to formation of matrix/reinforcement interfaces. However, at the same time during aging the presence of SiC particles inhibits the precipitation of Al$_2$Cu phase from the solution, as it is pinned at the grain boundaries, mainly on SiC particles. Figure 10(a) and (b) shows the aging plots for the matrix material and composites previously solution treated at 495 and 530 °C, respectively. Additionally, Table 1 presents the values that correspond to aging response of Al-Cu-SiC composites.

The results of microhardness measurement on each stage of heat treatment indicate that the applied solution temperature (495, 530 °C) did not allow for solid-solution strengthening of the Al4Cu alloy produced by the powder metallurgy technique. The coarser Al$_2$Cu phase formed in microstructure after solution treatment did not fulfill the strengthening function. The aging process, regardless of the aging time, also did not result in a significant microhardness change. The heat treatment temperature may be influenced by the presence of pores and higher content of oxygen in the microstructure of sinters, compared to the alloys produced under traditional conditions. The slower kinetics of aging in the matrix material is attributed to the lower concentration of vacancies and dislocation density. Instead, the composite materials seem to respond well to age-hardening process. For the composite materials with the addition of lower content of strengthening phase (2.5 wt.% of SiC), both solution treated at 495 and 500 °C, the incubation period (time for the reaching highest hardness) is found to be much shorter (after 4 h) compared to that of composite materials with the content of SiC particles above 2.5 wt.%. For the composites with the addition of 5 and 7.5 wt.% solution treated at 495 °C the maximum peak microhardness was recorded after 24 h of aging, while after solution treatment at 530 °C after 12 h of aging. Differences in the aging response of Al4Cu alloy and composites can be found in the applied method of manufacturing. Both materials were produced by powder metallurgy route. Due to the strong affinity for oxygen, aluminum particles are covered with a thin oxide layer. Therefore, obtained materials contained an appreciable amount of oxide inclusions. The presence of oxide inclusions and
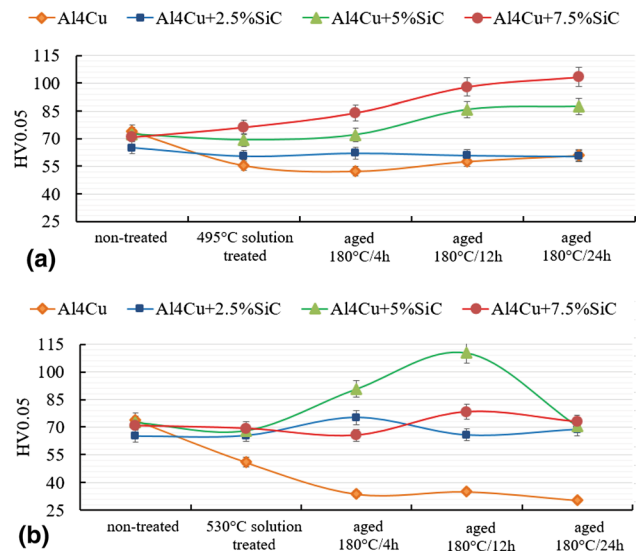
Fig. 10 Age-hardening behaviors of the composites: (a) 495 °C solution; (b) 530 °C solution

oxide-matrix boundaries, but also increased specific surface area, resulting from grain refinement of powder metallurgy-processed materials, is likely to reduce vacancy concentration and consequently suppresses GP zone formation. On the other hand, due to the linear expansion coefficient difference between reinforcement and matrix material, during quenching from the solution temperature, a large amount of plastic strain develops. This strain is accommodated by the high density of dislocation generation in the matrix of composite compared to unreinforced Al4Cu alloy. High density of dislocation also has an influence on the relatively poor vacancy. However, the generated quenching stress might cause the formation of excess free vacancies needed for nucleation of GP zones (Ref 3, 8, 11).

The aging of the composite with the addition of 2.5 wt.% of SiC did not result in major change in microhardness when the aging period increases. In all cases, the microhardness remained at a similar level. A different dependence of microhardness changes was observed in composites with a higher content of reinforcement. It can be seen that microhardness increases as the aging time is extended up to 24 h. Among composites solution treated at 495 °C, the highest microhardness was characterized by the composite with the addition of 7.5 wt.% of SiC after 24 h of aging (103 HV0.05). Increasing the solution temperature up to 530 °C resulted in obtaining peak micro-hardness earlier–after 12 h of aging, and for the composite reinforced by 5 wt.% of SiC, microhardness increased to 110

Table 1 Aging response in Al-Cu-SiC composites

Weight fraction of SiC, wt.%	Aging time, h	Peak hardness, HV0.05	
		Solutionizing temperature, 495 °C	Solutionizing temperature, 530 °C
0	0	55	51
	4	52	34
	12	58	35
	24	61	30
2.5	0	61	65
	4	62	75
	12	61	66
	24	60	69
5	0	69	68
	4	72	91
	12	86	110
	24	88	70
7.5	0	76	69
	4	84	66
	12	98	78
	24	103	73

HV0.05. Extending the aging time to 24 h resulted in its decreasing to 70 HV0.05 due to over aging.

Composite materials require different temperature parameters in solid-solution hardening than unreinforced Al4Cu matrix material. While the solution temperature equal to 495 °C is too low to further successful aging hardening of the pure Al4Cu matrix, in the case of composites, thermal mismatch between the reinforcing phase and matrix leads to high dislocation density and consequently faster aging kinetics. In addition, the presence of a higher dislocation density in composites with the addition of reinforcement provides new nucleation sites for θ' precipitation leading to a finer size of precipitates compared to those in unreinforced matrix alloy.

The aging plots correlate with the performed microstructural investigation. The number of Al_2Cu precipitates increases with increasing the aging time. After 4-h aging, the number of Al_2Cu precipitates is smaller, which corresponds to the lower microhardness of the composites. Instead, the large amount of Al_2Cu precipitant with fine size accounts for the peak microhardness after 12-h aging of composites previously solution treated at 530 °C. After 24 h of aging, the size of Al_2Cu precipitates significantly increases, leading to a decrease in microhardness.

4. Conclusions

In this research work, powder metallurgy-processed Al4Cu alloy composites reinforced with SiC particles were subjected to age hardening. The primary conclusions that can be drawn from the study are as follows:

- In the microstructure of non-heat-treated both matrix and composite materials, the grain boundaries are revealed by precipitations of coarser Al_2Cu phase.
- There was a difference between the heat-treated microstructure of the matrix material and composite materials reinforced with SiC particles, which indicates that composite materials require different temperature parameters in solid-solution hardening than unreinforced Al4Cu

matrix material produced by powder metallurgy technique.
- The solution temperature equal to 495 °C is too low to successful solution treatment of the pure Al4Cu matrix. There are still a lot of micron-sized particles remaining in the matrix that was not fully dissolved into the matrix.
- Applying solution temperature equal to 530 °C in solution treatment of composite materials allows for almost complete dissolution of intermetallic phase.
- The Al4Cu-5 wt.% of SiC composite solution treated at 530 °C shows the highest increment in microhardness by aging treatment. After 12-h aging treatment, numerous nanosized Al_2Cu phases uniformly distributed in the matrix inhibit the dislocation movement and account for the peak microhardness (110 HV0.05).

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