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## AGING BEHAVIOUR OF AA6061/SiC<sub>p</sub> COMPOSITES PRODUCED BY DIRECT EXTRUSION WITH A REVERSIBLY ROTATING DIE METHOD

### OBRÓBKA CIEPLNA KOMPOZYTÓW AA6061/SiC<sub>p</sub> WYTWARZANYCH METODĄ WYCISKANIA Z REWERSYJNIE ROTUJĄCĄ MATRYCĄ

This paper discusses the influence of thermal treatment parameters on mechanical properties of AA6061+x% vol. SiC<sub>p</sub> (x = 0, 2.5, 5, 7.5, 10) composites. The composites were consolidated via powder metallurgy processing using the unconventional method of extrusion (the KoBo method). In order to establish the optimum parameters of the heat treatment two different temperatures of supersaturation (530 and 558°C) were applied. The aging curves were determined at various aging temperatures such as 140, 160, 180 and 200°C. The effects of applied parameters were studied using the microstructure observations and hardness measurements. Obtained results show that the solution treatment at 530°C is sufficient to complete dissolution of the precipitates. Higher temperature of the process resulted in accelerating the aged-hardening. The suitable age treating parameters for the AA6061-5 vol. % SiC<sub>p</sub> composites were 160 - 180°C for 12 - 16 hours.

*Keywords:* Aging treatment; Al/SiC<sub>p</sub> composites; Extrusion; Microstructure; Mechanical Properties

W niniejszym artykule określono wpływ obróbki cieplnej na właściwości kompozytów AA6061+x% obj. SiC<sub>p</sub> (x = 0, 2.5, 5, 7.5, 10). Materiały wytwarzane były przy wykorzystaniu metalurgii proszków. W ostatnim etapie procesu technologicznego, do konsolidacji mieszanin proszków zastosowano metodę wyciskania z rewersyjnie rotującą matrycą (metoda KoBo). W celu określenia optymalnych parametrów obróbki cieplnej zastosowano dwie temperatury przesycań (530 i 558°C) oraz cztery temperatury starzenia (140, 160, 180 and 200°C) kompozytów. Obrabiane materiały zostały poddane obserwacjom mikrostruktury oraz wykonano pomiary twardości w celu określenia optymalnych parametrów procesów przesycań i starzenia. Na podstawie otrzymanych wyników można stwierdzić, że temperatura przesycań 530°C jest wystarczająca do uzyskania przesyconego roztworu stałego. Ponadto określono optymalne warunki starzenia dla kompozytu AA6061-5 obj. % SiC<sub>p</sub>: temperaturę w zakresie 160 - 180°C oraz czas starzenia 12 - 16 godzin.

## 1. Introduction

Aging treatment is one of the most widely used methods for improving the strength of aluminum alloys. This process consists of two stages supersaturation and aging. Additionally the aging process can be divided into: natural (occurring at ambient temperature) and artificial (occurring at elevated temperatures). The main condition for carrying out precipitation hardening is changing solid solubility with temperature. In the case of aluminum alloys, 2xxx and 6xxx alloys are the most commonly used in the process of precipitation hardening. The precipitation sequence of 6xxx alloys, where the major alloying elements are Mg and Si, is as follows [1-4]:

$\alpha(ss) \rightarrow Mg/Si \text{ clusters} \rightarrow GP \text{ zones} \rightarrow \beta'' \rightarrow \beta' \rightarrow \beta (Mg_2Si)$

While the kinetics of the aging process of aluminum alloys is fairly well-known and generally accepted, for the

aluminum matrix composites are still many disparities. Studies of age-hardening kinetics, suggest that the addition of ceramic particles to aluminum alloys has a different effect on precipitation than in the case of unreinforced alloys [5, 6].

Appendino *et al.* [7] examined the 6061 alloy/SiC particles composites. They showed that precipitation sequence in the composites was almost the same, compared to the unreinforced matrix alloys. However, age-hardening kinetics was enhanced by the presence of the reinforcement. The researchers explain acceleration of aging process by increasing dislocation density during heat treatment due to a mismatch in coefficients of thermal expansion (CTE) of matrix and reinforcement. During age-hardening dislocations led to creating heterogeneous nucleation sites. Furthermore, the diffusion rate of solute atoms is enhanced owing to pipe diffusion along dislocation. Other studies confirm the results obtained by Appendino *et*

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*al.* [8-13]. Pal *et al.*, by contrast, seem not to agree with the above mentioned researchers [14]. They examined Al-Cu-Mg matrix composites reinforced with different percentages of silicon carbide particles. They concluded that the presence of SiC particles decelerated aging kinetics compared to unreinforced alloys. They explained this phenomenon by insufficient density of vacancies, dislocation and segregation of alloying elements on matrix-reinforcement interface. Similar observations regarding age-hardening kinetics were made by other authors [15-17]. Furthermore, there are reports in which no discrepancies, in the age-hardening kinetics, between reinforced and unreinforced alloys were noted [18].

Despite the fact that a number of studies on the impact of reinforcement particles on the artificial ageing kinetics have been conducted, their influence has not been thoroughly explained. For this reason, in this study, the influence of supersaturation temperature, time and temperature of aging and the reinforcement particles volume fraction on the age-hardening kinetics were analyzed.

## 2. Experimental procedure

In this work five types of AA6061/SiC<sub>p</sub> composites with different contents of SiC particles (SiC<sub>p</sub> = 0, 2.5, 5, 7.5, 10 % in volume) were fabricated via powder metallurgy technique. The materials used in this work were β-SiC<sub>p</sub> and AA6061 powders with the average particle size of 0.42 μm and 10.6 μm, respectively. The alloy composition of matrix material is shown in Table 1. After blending the AA6061/SiC mixtures were cold isostatic pressed (CIP) to achieve compacts with a diameter of 40 mm and a length of 50 mm. Prepared compacts were then consolidated by direct extrusion with a reversibly rotating die (the KoBo method). The powders mixtures were pressed and consolidated at air atmosphere. The parameters of the extrusion process were as follows: temperature 350°C, ram speed 0.25 mm/s, frequency of the die oscillation was changed within the range of 5-8 Hz, oscillation angle of 4 deg, extrusion ratio 25 which corresponded to true strain of 3.2. More detailed information on the KoBo method is given elsewhere [19-22].

The AA6061/SiC composites rods with the diameter of 8 mm and a length of 1 m were cut perpendicular to the extrusion axis. The AA6061-5 vol. % SiC<sub>p</sub> composite specimens were solution-treated at 530°C for 1.5 hour, water-quenched and then aged at 140, 160, 180, and 200°C for various time, similarly as in [10]. Additionally, to determine the impact of solution temperature on composites properties, the AA6061-5% SiC specimens were solution-treated at 558°C, and aged at 160°C for different amounts of time. Moreover, the influence of the reinforcement volume fraction on the age-hardening kinetics was examined. The age-hardening response of the composites was characterized with the use of a Vicker's microhardness

tester Matsuzawa MMT-X7B. The Young's Modulus was determined utilizing an Optel ultrasonic refractometer. The microstructures of as-extruded and heat-treated specimens were studied with the use of a light microscope (LM) Nikon Eclipse MA200 and transmission electron microscope (TEM) PHILIPS CM 20.

## 3. Results and discussion

### 3.1. Microstructural examination

#### 3.1.1. As-extruded condition

Figure 1 shows the distribution of reinforcement particles on the as-extruded AA6061 – 5 vol. % SiC<sub>p</sub> composite along the transverse directions, relative to the extrusion axis. The small amount of fine SiC<sub>p</sub> agglomerates was observed, indicated by arrows on the micrographs. The average size of agglomerates was determined as 1.6 μm. Hence, it is necessary to improve the uniformity of SiC<sub>p</sub> distribution because the presence of agglomerates negatively affects composite properties [23, 24]. The TEM observations of as-extruded composites showed the occurrence of two types of intermetallic compounds in the aluminum matrix.

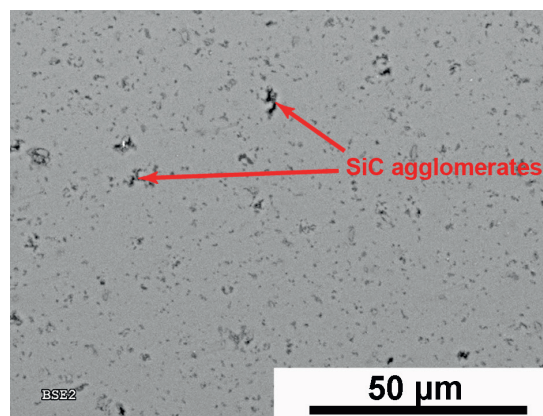


Fig. 1. Electron Microscope observation of AA6061 – 5 vol. % SiC<sub>p</sub> composites

The coarser, marked in Figure 2a as 1, and the finer, marked as 2. They were placed inside and at the grain boundaries of the matrix. In order to determine the chemical composition, an EDS analysis was performed. The intermetallics marked as 1 consisted essentially of Mg and Si (Figure 2b). According to the Al-Mg-Si phase diagram, the compounds could be identified as Mg<sub>2</sub>Si. However, in finer precipitates the Fe, Cu and Cr were detected (Figure 2c). Taking into account the chemical composition of matrix powders, it is believed that these elements are impurities.

TABLE 1

Chemical composition of AA6061 alloy (wt. %)

AA6061	0,272	0,011	1,04	0,111	0,634	0,001	0,179	0,006	0,006	Balance
Alloy	Cu	Mn	Mg	Fe	Si	Zn	Cr	Ni	Ti	Al

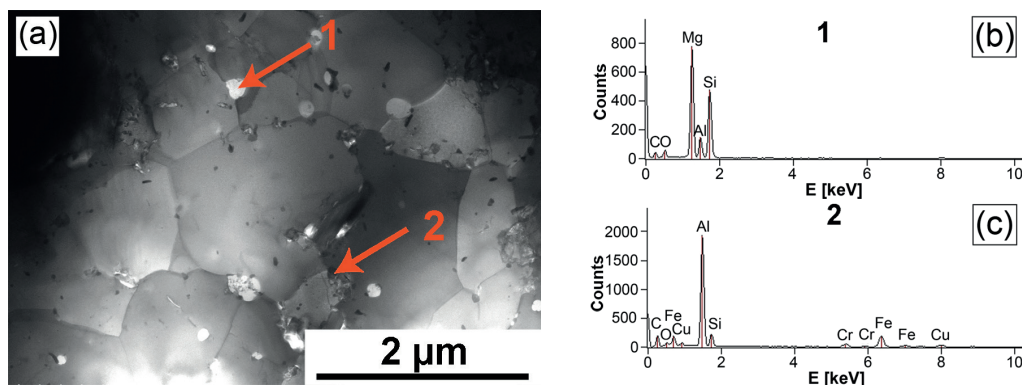


Fig. 2. As-extruded AA6061 – 5 vol. % SiC<sub>p</sub> composites, a) TEM image, b), c) EDS spectrum of a precipitates

### 3.1.2. Aged condition

The microstructures were also analyzed for heat-treated composites. Figure 3 shows the TEM image of the AA6061-5 vol. % composite, aged at 160 °C for 16 h after solution treatment at 530°C for 1.5 h. While comparing the microstructure of the as-extruded and aged-hardened composites coarse precipitants, identified as Mg<sub>2</sub>Si, were not observed. This may indicate that the supersaturation at 530°C was sufficient for complete dissolution of Mg<sub>2</sub>Si precipitates. However, as in the case of as-extruded composites, the finer phases identified as the Al(MnCrFe)Si (marked by arrow at Fig. 3) were noticed. It has already been said, their presence is connected with aluminum alloy impurities. It suggests that Al(MnCrFe)Si phases are stable at solution temperature.

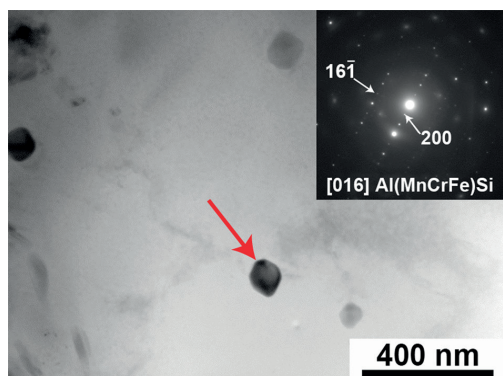


Fig. 3. TEM image showing the Al(MnCrFe)Si precipitants on AA6061-5 vol. % SiC<sub>p</sub> composite solution-treated at 530 °C, aged at 160°C

Additionally, an analysis of the Al-SiC<sub>p</sub> interface was executed and the results are shown in Figure 4. The maps of elements distribution show that during solution treatment there were no precipitations on interface. However, TEM imaging revealed the presence of voids between the reinforcement and matrix. These voids are formed during extrusion and can act as concentrators of local stress, degrading the fracture toughness and ductility of composites [25]. Figure 5a shows bright field TEM micrographs of AA6061 - 5 vol. % SiC<sub>p</sub> composite aged at 160 °C for 16 h. It can be seen that needle-shaped precipitates (marked by arrows) are relatively homogeneous distributed in the matrix.

The diffraction pattern suggests that the precipitates are the β' phases. These phases are the main reinforcing phase in Al - Mg - Si alloys. This confirms that the applied parameters of heat treatment were proper and could lead to achieving optimal properties. In view of the data in the literature on possible presence of precipitates-free zones near the reinforcement [26], the distribution of β'' was analyzed. Figure 5b depicts the interface between the aluminum and SiC particles. It is clearly seen that the distribution of β'' is similar, regardless of the distance from the interface. This may be related to the lack of interface reaction which was considered as a reason of formation precipitation-free zones [27].

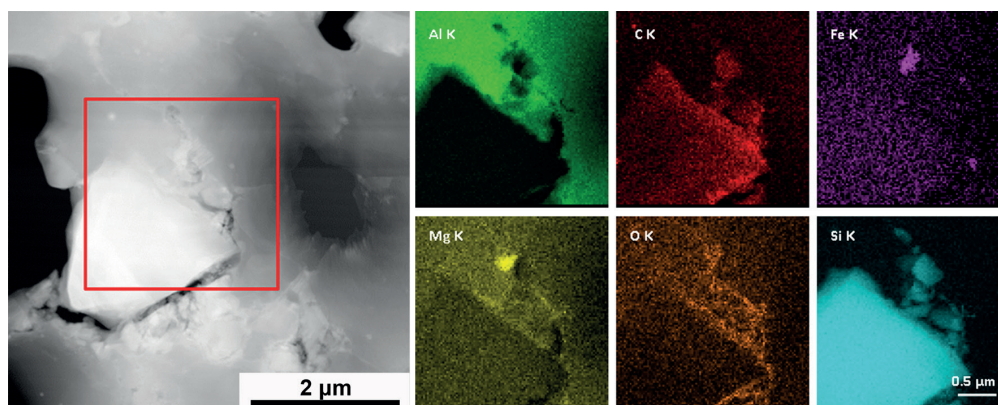


Fig. 4. Maps of elements on the matrix-particle interface for the AA6061-5 vol. % SiC<sub>p</sub> composite after age-treatment

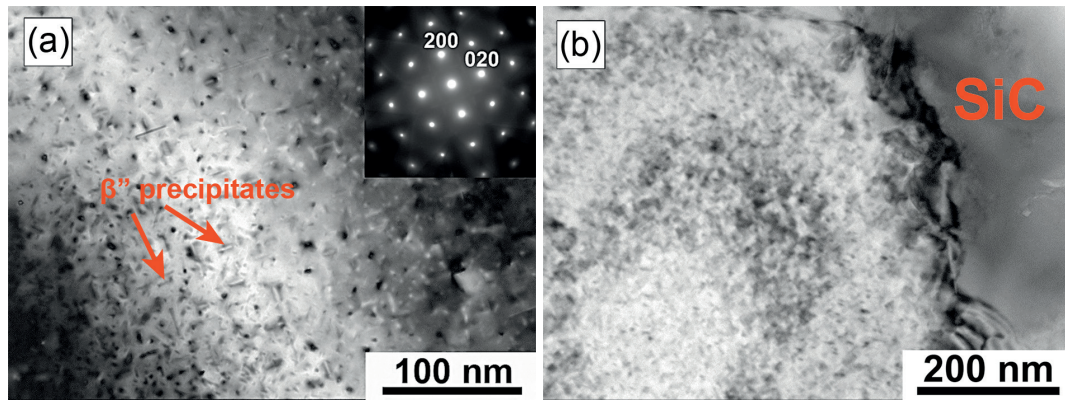


Fig. 5. TEM micrographs of AA6061-5 vol. % SiC<sub>p</sub> composite, a) needle-shape  $\beta''$  precipitates, b) microstructure close to matrix-particle interface

### 3.2. Ageing kinetics

The hardness vs. aging time profiles, at various temperatures, for AA6061-5 vol. % SiC<sub>p</sub> are given in Figures 6 and 7. All profiles demonstrated a rapid increase in hardness at the initial stage of aging. Furthermore, the rate of hardness increase depended on aging temperature. The higher the aging temperature, the faster hardness increased. Moreover, a similar relation was observed for time to reach the peak hardness. At the lowest aging temperature (140°C) the peak hardness occurred after 20 h and decreased to 5 h for aging at 200°C. It is due to the fact that an increase of aging temperature causes a speed up of Mg, Si atoms diffusion. This accelerates formation of clusters and GP zones and consequently improves mechanical properties by developing microstrains in the lattice [11].

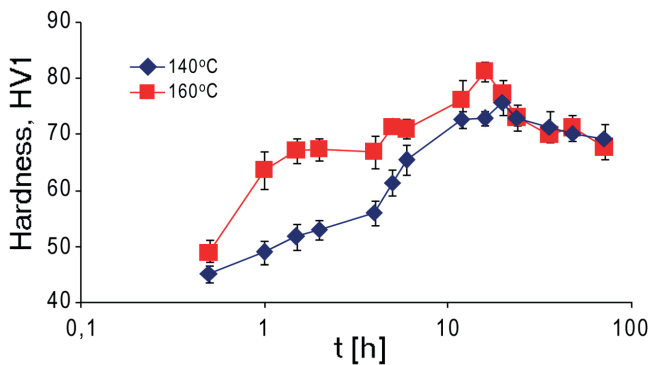


Fig. 6. Variation of hardness of the AA6061-5 vol. % SiC<sub>p</sub> composite solution-treated at 530°C, aged at 140 and 160°C as a function of aging time

The highest values of hardness were achieved for composites aged-hardened at 160 and 180°C. These results entail that the temperatures are proper to heat-treatment of such composites. The peak hardness parameters are listed in Table 2. After achieving peak hardness point, in all cases, hardness gradually decreased which was associated with formation of  $\beta'$  or stable  $\beta$  precipitates [28, 29]. The hardness decrease was more significant for composites aged at the highest temperature.

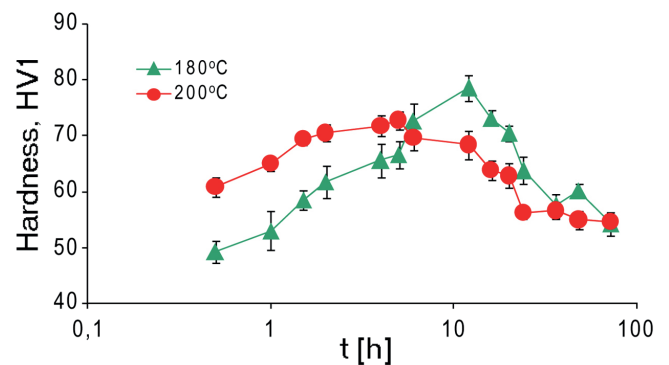


Fig. 7. Variation of hardness of the AA6061-5 vol. % SiC<sub>p</sub> composite solution-treated at 530°C, aged at 180 and 200°C as a function of aging time

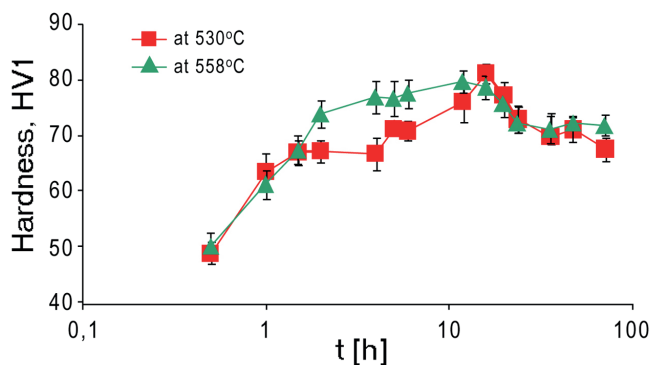
Apart from time and temperature of ageing critical influence, on the mechanical properties has solution heat treatment temperature. According to Al-Mg-Si phase diagram and literature reports, solution treatment at 558°C exhibit better strength properties compared to composites treated at 530°C [11, 14]. This is caused by the fact that the melting of ternary eutectic Mg<sub>2</sub>Si-Al-Mg phase takes place at 558°C and the concentration of alloying elements in the supersaturated solid solution is higher.

In light of the above, the hardness vs. aging time profiles for AA6061-5 vol. % SiC<sub>p</sub> composites solution heat-treated at 530 and 558°C were created. The results are presented in Figure 8. The higher supersaturation temperature resulted in a more rapid growth of hardness compared to composites solution treated at 530°C. This may be due to higher concentration of defects i.e. dislocations or vacancies, after quenching, and accelerates aging kinetics. Also, the time to peak hardness is shorter for higher temperature and reached 12 and 16 hours for composites supersaturated at 558 and 530°C, respectively. Nevertheless, the increase of strength properties was not observed. Similar values of hardness (HV=81±1.8 for 530°C and HV=80±1.6 for 558°C) were noted for these composites. The experiment did not confirm literature reports about the possibility of improving the mechanical properties by increasing the supersaturation temperature.

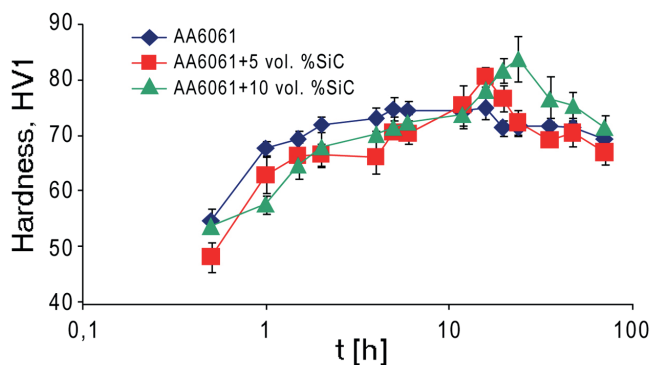
TABLE 2

Peak hardness variations with aging, supersaturation temperature and volume fraction of SiC<sub>p</sub>

SiC <sub>p</sub> volume fraction [%]	Temperature of supersaturation [°C]	Temperature of aging [°C]	Time of aging [hours]	Vickers hardness
5	530	140	20	75±1.3
5	530	160	16	81±1.8
5	530	180	12	79±1.4
5	530	200	5	73±1.6
5	558	160	12	80±1.6
0	530	160	5	75±1.9
10	530	160	24	84±1.8

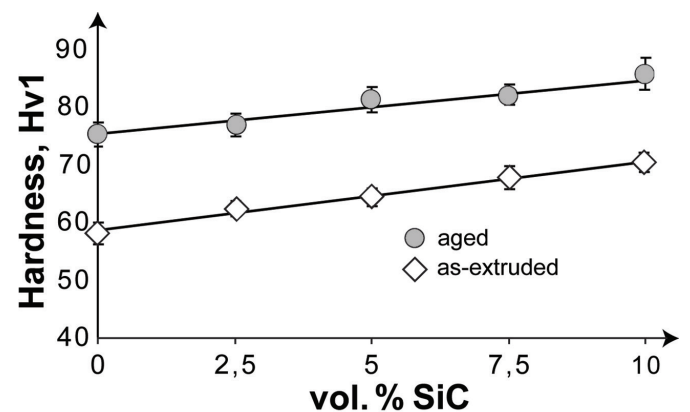
Fig. 8. Variation of hardness of the AA6061-5 vol. % SiC<sub>p</sub> composite solution-treated at 530 and 558 °C (aged at 160 °C) as a function of aging time

In order to determine the influence of the reinforcements volume fraction on aging kinetic the materials containing 0,5 and 10 vol. % SiC<sub>p</sub> were investigated. The composites were aged at 160°C for 16 h after solution treatment at 530°C for 1.5 h. The effect of aging time on hardness increment for the composites as well as unreinforced alloy is shown in Figure 9. It can be seen that for the composites, the peaks aging time are 16 and 24 h for 5 and 10 vol. % SiC<sub>p</sub> respectively, while, for unreinforced alloy occurred after only 5 hours. Detailed parameters are listed in Table 2.

Fig. 9. Variation of hardness of the AA6061, AA6061-5 vol. % SiC<sub>p</sub>, AA6061-10 % vol. SiC<sub>p</sub> composite solution-treated at 530°C, aged at 160°C as a function of aging time

The differences between reinforced and unreinforced alloys are also visible in the final step. For the unreinforced

alloy, after peak aging, the hardness values are stable for a relatively long time, up to 16 h. While, for the composites over aging is reached more rapidly. Similar results are presented in literature [30 - 32]. The deceleration of aging kinetics can be attributed to reducing the volume fraction of the GP zones. The presence of SiC particles causes lower vacancy concentration than in the case of unreinforced alloy, as matrix-particles interface acts as vacancy sinks. Another reason for slower aging kinetics of composites is presence of voids on matrix-reinforcement interface.

Fig. 10. Comparison of hardness of as-extruded and aged composites with different volume fraction of SiC<sub>p</sub>

Coming back to aging kinetics, in other studies, different conclusions were reached [13-18]. The authors observed acceleration of aging kinetics with an increase of reinforcement volume fraction. This effect could be explained by increasing dislocation density, due to the mismatch in coefficients of thermal expansion, which enhances diffusion rate of solute atoms and leads to the creation of heterogeneous nucleation sites.

### 3.3. Comparison of as-extruded and aged properties of composites

Figure 10 shows the hardness vs. SiC<sub>p</sub> volume fraction profiles for peak aged and as-extruded composites. In both cases the increase of hardness values corresponded with increasing of reinforcement volume fraction. The hardness of the aged-treated composites is approximately 25%

higher than that of as-extruded composites. However, the Young's modulus reached lower values compared to as-extruded composites, as can be seen in Figure 11. This can be related to changes in the crystallographic texture of the composites, after heat-treatment. As follows from literature, the Young's modulus for aluminum changes from 77 GPa to 64 GPa respectively for the  $\langle 111 \rangle$  and  $\langle 100 \rangle$  directions [33, 34]. Therefore, on the basis of the literature it is possible to infer that the Young's modulus of composites may change significantly depending on the matrix texture. Another reason for the decrease in Young's modulus after heat-treatment may be creation of microcracks in the matrix after quenching.

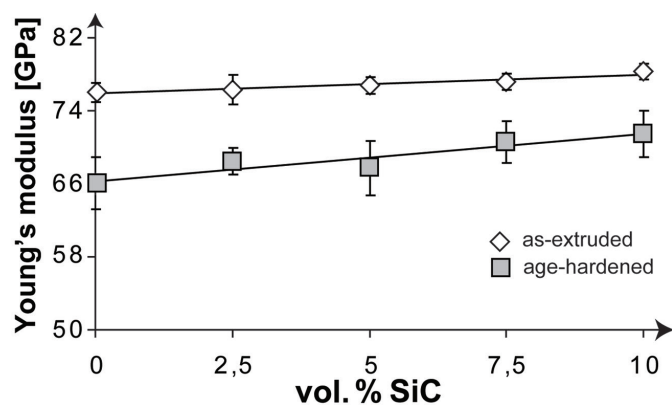


Fig. 11. Comparison of Young's modulus of as-extruded and aged composites with different volume fraction of SiC<sub>p</sub>

#### 4. Conclusions

The conclusions drawn on the basis of this study can be summarized as follows:

- The solution treatment at 530°C for 1.5 h is sufficient to complete dissolution of the precipitates which was confirmed by observation of the composite microstructure. Applying of higher temperature of the process resulted in accelerating the aged-hardening yet increase of strength properties was not observed
- The suitable age treating parameters for the AA6061-5 vol. % SiC<sub>p</sub> composites are 160 - 180°C for 12 - 16 hours. Under these conditions were achieved the highest mechanical properties of heat treated composites.
- The aging kinetics, in terms of time to peak aging, are decelerated with increase of SiC<sub>p</sub>. This can be attributed to increase dislocation density which enhances diffusion rate of solute atoms and leads to the creation of heterogeneous nucleation sites. Moreover, increasing of SiC<sub>p</sub> enhance peak hardness value.

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