Investigation of microstructure and hardness properties of aged AA 7075 matrix B₄C/SiC reinforced composite-hybrid materials

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ABSTRACT

Aluminum hybrid composites are next-generation metal matrix composites having the potential for advanced and new engineering applications. Therefore, in the study, using the powder metallurgy method, microstructure and hardness properties of aluminum (AA7075) composite and hybrid materials were examined in the combination of different ceramic reinforcement phases (5-10-15% B₄C and SiC). Pressed (700MPa) matrix/reinforcement powder mixtures were sintered for 1 hour at a temperature of 560 °C. After the sintering process, density measurements of the test samples were carried out. In order to determine the microstructure and hardness properties of the test samples, optical microscope, scanning electron microscope (SEM), and micro-hardness (HV0.1) measurements were made respectively. At the same time, micro-hardness values were examined by applying the solution-quenching to the test samples at 475 °C temperature for 2 hours and the aging heat treatment at 120 °C temperature for 24 hours. Compared to single ceramic phase composite materials, a decrease in density and hardness value occurred in 5% B₄C-SiC reinforced aluminum hybrid composite materials. Furthermore, it was determined that the micro-hardness values of test samples increased after the aging heat treatment.

ARTICLE INFO

Article history:
Received 16 December 2019
Received in revised form 21 February 2020
Accepted 06 May 2020
Available online 29 June 2020
Research Article
DOI: 10.30728/boron.665080
Keywords:
Composites, AA7075, Powder Metallurgy, Microstructure, Hardness.

1. Introduction

Aluminum and its alloys are widely used in the automotive and aerospace industries due to their properties such as low density and high strength/weight ratio [1-3]. Aluminum can be preferred in industries because of its high corrosion resistance and high damping capacity. However, it is not rigid and strong enough for many specific purposes. Its weakness against abrasion and erosion poses a serious problem for long-term use [4-6]. For this reason, some reinforcements are needed to increase their use. Aluminum composites are the most appropriate one for some applications that require high strength without losing their ductility. Metal-matrix composites have good abrasion and erosion resistance properties, as well as are a new class of materials with higher hardness at lower density [7-10].

Whereas alloying elements added to the aluminum matrix improve their mechanical properties, they can also cause some negative effects. In order to eliminate the negative effects, heat treatment is applied especially to 2XXX, 7XXX and 8XXX series aluminum alloys. In addition to mechanical properties, changes in electrical conductivity and corrosion properties are also observed due to the applied heat treatments. In aluminum alloys, hardness and resistance are generally increased after the aging heat treatment. In order to eliminate non-equilibrium phases and internal structural defects, annealing heat treatment is applied [11-14]. Hybrid composites, on the other hand, attract the attention of researchers all over the world because of their excellent properties such as high weight/weight ratio, better abrasion resistance, and good mechanical properties. In order to make it suitable for use in advanced applications, a large amount of research is being carried out to improve features. Addition of two or more than two reinforcements provides a wider area for optimization of features, and such composites are mainly called as hybrid composites.

The properties of the hybrid composite depend on several factors, of which the type of material used for the matrix and reinforcements is very important. This is because it exhibits good expansion coefficient, abrasion resistance, self-lubrication, impact resistance, and good mechanical behavior at higher temperatures.

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Currently, many aluminum hybrid composites reinforced with hard ceramic particles such as SiC, Al₂O₃, and B₄C are produced in order to improve their mechanical properties [15].

The purpose of this study is to produce a B₄C-SiC reinforced hybrid composite material with aluminum matrix in a combination of double ceramic phase reinforcement, as well as single ceramic phase reinforcement, which is mostly involved in the application. Thus, microstructure and aging-related micro-hardness properties of the obtained non-reinforced AA7075 aluminum alloy, reinforced composite, and hybrid materials were examined.

2. Materials and methods

In the experimental study, AA7075 aluminum alloy powders with an average powder size of 90 µm were used as the starting material in the production of the powder metal composite-hybrid materials. The chemical composition of the used AA7075 (90.66 µm, gas atomizing) aluminum alloy powders is given in Table 1. The physical properties [16] of the ceramic particle reinforcement elements (B₄C, 5 µm and SiC, 40 µm Aldrich) used in the production of composite-hybrid materials are also shown in Table 2.

In the production of the composite-hybrid powder metal test samples, the matrix and reinforcement particles were prepared at different percentage weight ratios (5.10% and 15%). The mixing process was applied to the prepared matrix and reinforcement start powders for 45 min in a 3-axis Turbula T2F device to ensure homogeneous distribution. The powder mixtures obtained after the mixing process applied in the Turbula device were pressed under 700MPa pressure as a result of preliminary trials, as one way, at room temperature in the mold shown in Figure 1. The pressed non-reinforced AA7075 aluminum alloy and composite-hybrid test samples were removed from the mold as having cylindrical 12 mm diameter and 10 mm height. After the pressing process, the test samples were subjected to sintering in the glass tube for 1 hour under argon gas flow by using an atmosphere-controlled heat treatment furnace at a temperature of 560 °C.

Before and after the sintering process, for the density measurements of the test samples, which were produced using the powder metallurgy method, Kern brand precision balance (0.0001) was used and the weights of the samples were measured. Then their heights were determined with the help of micrometers and the density measurements were carried out through the volume calculation. In terms of the microstructural characterization of the sintered samples,

### Table 1. Chemical composition of AA7075 alloy.

| Component | Al  | Zn  | Mg  | Cu  | Cr  | Fe  | Si   | Mn  | Ti |
|-----------|-----|-----|-----|-----|-----|-----|------|-----|----|
| Weight (%)| 87.1-| 5.1-| 2.1-| 1.2-| 0.18-| Max. | Max. | 0.3 | 0.2 |
|           | 91.5| 6.1 | 2.9 | 2   |    |     |      |     |    |

### Table 2. Physical properties of B₄C and SiC powders.

| Reinforcement Material | Density (g/cm³) | Coefficient of Thermal Expansion | Melting Temperature (°C) | Resistance (MPa) | Elasticity Module (GPa) |
|------------------------|----------------|---------------------------------|--------------------------|-----------------|------------------------|
| B₄C                    | 2.52           | 6.08                            | 2420                     | 2759            | 448                    |
| SiC                    | 3.16           | 4.8                             | 1800                     | 700             | 400                    |

![Figure 1. Mold and experiment sample used in pressing.](image-url)
general metallography works including respectively sanding (600-800-1200 sanding), polishing (1 µm diamond paste) and etching (95 ml H2O and 5 ml HCl) operations were carried out.

In order to determine microstructural differences of the produced hybrid/composite samples and distributions of B₄C-SiC ceramic phase particles, optical microscope (Hardway brand) and scanning electron microscope (SEM) (Hitachi SU 1510 model) were used. Hardness measurements of the produced test samples were made using the Hardway brand micro-hardness device (HV0.1) before and after the aging heat treatment. Within the scope of the aging heat treatment, a three-stage process including taking into the solution, quenching in water and aging were applied to the samples. In this context, after 2 hours of the solution process applied to the test samples at 475 °C temperature, the quenching process was carried out. Following the quenching process, the test samples were subjected to an artificial aging process in the oven for 24 hours at 120 °C temperature.

3. Results and discussion

SEM images of the initial matrix and reinforcement elements used for the production of B₄C-SiC reinforced composite/hybrid materials having the powder metal AA7075 matrix are given in Figure 2. When the SEM images are examined, it is understood that the AA7075 aluminum alloy matrix element has a powder shape and morphology called spherical, rod-like, and teardrop. It was determined that the B₄C and SiC ceramic phase structures used as reinforcing elements were irregular and had mostly rod-like and polygonal powder shapes and morphologies.

Following the pressing and sintering processes, the density measurements of the matrix and reinforcing elements were made in terms of powder metal composite and hybrid material production. In particular, the relative density results obtained after sintering are given in Figure 3.

When the relative density values given in Figure 3 were examined, it was seen that the density value of AA7075 Matrix aluminum alloy without reinforcements was

Figure 2. Starting powder material SEM images of matrix and reinforcement; a) AA7075, b) SiC and c) B₄C.

Figure 3. Density changes of samples after sintering; AA: AA7075, B: B₄C, S: SiC, H: Hybrid, 5-10-15: Ratio of reinforce.
measured as 97.45%. When the density values of B₄C and SiC reinforced composite and hybrid test samples were examined, it was identified that there was a decrease in the increased reinforcement rate compared to non-reinforced test samples. Especially in dual ceramic phase reinforced hybrid composite materials, this decrease (96.9, 94.5 and 93.09%, respectively) showed a further increase. Compared to the non-reinforced AA7075 aluminum alloy, for this reduction, especially the grain structure-pore relationship in optical and SEM images (Figure 4-7) can be taken into account. When the microstructure images of the AA7075 aluminum alloy matrix material after sintering are examined in Figure 4, the positive effect of the sintering mechanism can be stated in terms of grain structure-pore interaction. Especially on the SEM image, as occur among many grains, the pore shape and morphology in the triple-grain-contact overlap with the sintering mechanism. In this case, it can be said that the sintering temperature and duration kept going positive. It can be stated that similar cases take place in terms of composite and hybrid materials in Figure 5-7,

Figure 4. Optical and SEM microstructure images of AA 7075 aluminum alloy.

Figure 5. Optical and SEM images of AA7075-B₄C reinforced composite material.
respectively. However, as seen in the decrease in density values, this condition can be observed mostly in hybrid composite microstructures depending on the increased reinforcement rate. This can be considered as the resistance of the ceramic phases, which tend to condensation and flocculation at the grain boundaries, to condensation.

In the B₄C and SiC reinforced composite material microstructures given in Figures 5 and 6, it was determined that based on the increased reinforcement rate, ceramic phases showed distribution mostly on the grain boundary and in the areas close to the grain boundary (Figures 5 and 6-e-f). In a study conducted by Şimsek, it is stated that the B₄C particles added to the matrix get a position at the grain boundaries [17]. It can be stated that depending on the increased amount of reinforcement, B₄C and SiC ceramic phase particles of different sizes and distributions exhibit homogeneous distribution on the matrix structure.

In B₄C and SiC reinforced hybrid composite material microstructures (Figure 7), with the increased double ceramic phase reinforcement, there is considerable flocculation in the microstructure. Compared to the 5% and 10% reinforcement rates (Figure 7-f), at the 15% reinforcement rate, the formation of flocculation and agglomeration occurring at grain boundaries is understood from the matrix grain structure on contact surfaces. It was determined that the amount of pore increased on the surface of the matrix material by the addition of B₄C and SiC. In their study on combining the Al 2024-Based B₄C/SiC Particle-Reinforced Hybrid Composites with TiG welding, Gökmecen mentioned a

Figure 6. Optical and SEM images of AA7075-SiC reinforced composite material.
similar effect [18]. The gaps emerging between the AA 7075 matrix structure and B₄C-SiC particles support the density reduction detected in 15% B₄C-SiC hybrid composites compared to samples at low reinforcement rates (Figure 7 a-d).

Examining Figures 8, 9 and 10, it is seen that SEM, EDS analyses and Element Distribution Mapping of only the 5-10-15% B₄C and SiC reinforced hybrid composite materials having AA7075 matrices are given. In hybrid composite materials produced at different reinforcement rates, the EDS analysis and elemental mapping of Al, Zn, Mg, Cu, Fe elements (in terms of matrix material) and B and Si elements (in terms of reinforcement elements) were studied. According to the obtained elemental mapping results, it is understood that ceramic phases with a pale and grayish structure in hybrid materials define SiC, while ceramic phases with dark-colored distribution define B₄C. Over these elements, the distributions exhibited by the B₄C-SiC reinforcement phases and the microstructural differences can be clearly seen. According to both elemental distribution mapping and EDS analysis results, it can be stated that in the hybrid composite material, the B₄C and SiC ceramic phase reinforcement particles are often clustered at grain boundaries.

As a result of the micro-structure investigation, the obtained micro-hardness results of AA7075 metal-matrix composite-hybrid materials produced by B₄C and SiC ceramic phase reinforcement are given in Figure 11. Micro-hardness results of the composite-hybrid materials are shown compared to the non-reinforced AA7075 alloy and based on the aging heat treatment applied at the same time. Prior to the aging heat treatment, the micro-hardness value of the non-reinforced AA7075 aluminum alloy was measured as 56.52 HV. In B₄C and SiC-reinforced composite samples, the micro-hardness value increased depending on the
increased reinforcement amount, and it was measured at the highest reinforcement rates of 66.82 HV and 64.52 HV, respectively. In their study on conventional and waste-reinforced hybrid composites with Al6061 Matrix, Kamber et al. reported a similar hardness increase [19]. In his study, Gökm en stated that there was an increase in hardness based on the increasing amount of ceramic particles in the Matrix content of Al 2024 [20]. Compared to the non-reinforced aluminum alloy, the most increase was calculated with B\textsubscript{4}C ceramic phase reinforcement. Compared to the samples prepared with both non-reinforced and
single ceramic phase reinforced, the highest hardness value in the hybrid material was measured as 69.59 HV in the 5% B₄C-SiC reinforced sample. In contrast to composite materials produced specifically with single ceramic phase reinforcement, in hybrid material (10% B₄C-SiC and 15% B₄C-SiC), there has been a tendency to decrease in micro-hardness value at the rate of increasing reinforcement. It was
identified that with the aging heat treatment applied in the materials, micro-hardness values increased in all materials. Increase of hardness at the end of aging process can be explained with phases formed in the microstructure, precipitation and change in grain sizes [21]. On the other hand, based on the aging heat treatment, the highest hardness value was determined as 79.4 HV in 15% B₄C reinforced composite material.
4. Conclusions

Using the powder metallurgy method, the microstructure and hardness performance of combinations in the B₄C-SiC ceramic phase reinforced aluminum hybrid composite material with AA7075 metal matrix was studied. In single ceramic phase-reinforced composite materials, the increasing reinforcement phases were observed to exhibit homogeneous distribution in the microstructure. In hybrid composite materials, on the other hand, it was identified that based on the increased amount of reinforcement, ceramic phases tend to cluster at grain boundaries. A tendency to agglomerate and aggregate was determined at the grain boundaries in increasing reinforcement phases especially in hybrid composite materials compared to AA7075 aluminum alloy. Consequently, it was determined that it shows resistance against condensation. The highest micro-hardness value was determined in the single-phase B₄C reinforced composite material. In aluminum hybrid composites, the similar effect of increased reinforcement phases on condensation also exhibited a decrease by being effective in the hardness value. It was determined that with the aging heat treatment, the micro-hardness value of all materials showed an increase.

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