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Research Article

Investigation of the Effect of High-Frequency Induction Sintering on Phase Structure and Microstructure of SiC Reinforced Aluminum Matrix Composites

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ABSTRACT

In this study, SiC-reinforced aluminum matrix composites were powder metallurgically (PM) prepared and sintered using high-frequency induction system (HFIS). The samples with different ratios of SiC (wt.%10, 20 and 40) added to the aluminum matrix were sintered at 660, 800, and 1000 °C. In addition, Al/SiC composites were compared by sintering with the conventional sintering (CS) method under similar sintering conditions. The heating rate for the sintering process using HFIS was 500 °C/min, while the CS method used a heating rate of 10 °C/min. The effect of the temperature and SiC ratio on the density, hardness, phase structure, and microstructure of composites was investigated. The optimum sintering temperature was determined according to the SiC additive amount. When 10%, 20%, and 40% SiC by weight were added to the aluminum matrix in the sintering process with HFIS, the required sintering temperatures were determined as 660, 800, and 1000 °C, respectively. While new phases were not formed as a result of short-term HFIS sintering, a high-temperature Al_4C_3 phase was detected in CS sintering. HFIS sintered Al/SiC composite samples were obtained in Al and SiC phases with high density and hardness ranging from 43-118 HV. In the high-temperature sintering process with HFIS, the formation of Al_4C_3 was prevented and its physical and mechanical properties were improved.

1. Introduction

Aluminum matrix composite materials are extensively utilized in aerospace, automotive, and electronic industries as they form the primary constituent of engineering products that possess favorable properties such as high strength, low density, high elastic modulus, hardness, and wear resistance. Silicon Carbide (SiC), Titanium Carbide (TiC), Boron Carbide (B_4C) and Alumina (Al_2O_3) are the most widely used inter-metallic compounds in the aluminum matrix composites. SiC is the most widely used reinforcement phase in aluminum matrix composites due to its superior properties such as high elastic modulus, strength, wear resistance and hardness [1, 2]. Evaluation of SiC particles as a reinforcement element in the aluminum matrix composites has some restrictions. High production cost, non-homogenous distribution of reinforcement phase, grain growth in the matrix, and undesired phases occurrence such as Al_4C_3 in the matrix are the most popular restrictive factors of the Al/SiC composites [3, 4].

Previous studies have shown that liquid-phase or solid-phase production is effective in determining the properties of aluminum matrix composites. Pressure infiltration, laser melting, and mold casting methods are the techniques used in the liquid-phase production [5-7]. In production processes involving liquids, production inherently occurs within a liquid medium, and obtaining the proper distribution of

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the reinforcement phase becomes a great difficulty. In addition, agglomeration of the grains leads to a decrease in the mechanical properties of the composite materials. Hot press sintering, hot isostatic press, spark-plasma, and sintering in the argon medium are alternative solid phase methods for production of the net shaped materials. This powder metallurgy method generally needs long operation periods and temperatures, so some of the restricts and obstacles must be controlled and even eliminated during the process [8-10].

High temperature sintering causes to the occurrence of inter metallic compounds which significantly decrease the performances of composites and their ductility properties. In the production of Al-SiC composites, aluminum does not react with SiC particles until the melting temperature of 660 °C, but different products can be formed depending on the temperature by reacting with SiC above the Al melting temperature. Needle shaped, very brittle and hydrophobic Al_4C_3 compound formation occurs in between 650 °C and 920 °C temperature interval. However, under the 650 °C operation conditions, the occurrence of Al_4C_3 phase has a very low possibility in the composite structure. When the composites are obtained at short operation periods and used low amount of SiC compositions, there is not much considerable formation of Al_4C_3 phase. However, when the sintering temperature is high, the Al_4C_3 phase is rapidly formed [11, 12]. For that reason, when Al/SiC system was considered, there is a potential of the development of Al_4C_3 phase as a reaction product because of the dissolution of SiC in the liquid aluminum on the interface of Al/SiC depending on the reaction at the equation 1 [13].

$$3SiC + 4Al \rightarrow Al_4C_3 + 3Si \quad (1)$$

Long term sintering process is required to produce Al/SiC composites in the conventional methods which causes some unfavorable effects such as particle growth and occurrence of undesired phases, so new manufacturing techniques are needed. Moreover, the conventional sintering methods causes disadvantages for commercial productions because of long operation periods needed.

To eliminate these problems, HFIS is a promising sintering technique that provides rapid heating at the desired sintering temperature. HFIS reaches high temperatures with a heating rate of over 500 °C, providing the opportunity to reach the desired temperature in a short time. Heating is obtained from the induction coil by using alternating current. To induce the metal object itself, heating is derived by creating an electromagnetic field around the coil. HFIS is very effective in sintering the composite materials in a few minutes to prevent thermal deformation and undesired grain growth and as a result, it was shown that various properties of composites were improved [14].

In this study, sintering conditions of Al/SiC composites in HFIS were investigated according to the SiC ratio. Densities, microstructures, and phase formations of Al/SiC composites were investigated. Similar conditions were used in the conventional sintering methods and compared with high frequency induction system.

2. Materials and Methods

In this work, aluminum (99%) and SiC (99.5%) were used in the production of aluminum composites reinforced with different ratios SiC. In Figure 1, when the SEM analysis of aluminum and SiC powders is observed, aluminum grains are elliptical sphere shape and SiC particles have sharp corners. It was seen that powders particle size was under the 100 μm. The average grain size of Al and SiC powders was determined to be 58 and 64 μm, respectively (Malvern/ Mastersizer 3000). Aluminum powders to which 10, 20, and 40 wt.% SiC were added were mixed for one hour using a V-type mixer. Prepared compositions were shaped in a cylindrical metallic mold under 40 MPa pressure by means of a uniaxial hydraulic press.

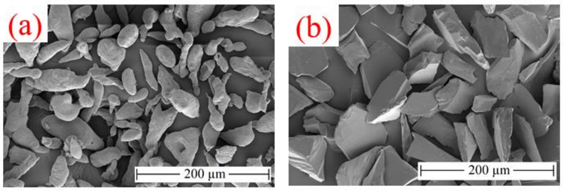

Figure 1. Aluminum (a) and SiC (b) powders microstructure analysis

Samples were sintered in argon atmosphere using HFIS and CS methods. Tube furnace was used in

CS method. The sintering operation was performed at 660, 800, and 1000°C with the heating rates of 500 °C/min and 10°C/min in the HFIS and the CS method, respectively. In Figure 2, a time and temperature graph were given for HFIS and CS methods. The sintering process performed with HFIS at a temperature of 1000 °C was completed in 3 minutes, and with CS sintering in approximately 141 minutes.

Sintered sample characterization was made by using density, phase analysis, hardness, and microstructure. Experimental density was measured with the Archimedes method. Theoretical calculation of the densities of Al/SiC composites was made using the mixture rules. Phase components were determined with XRD (PANalytical Empyrean High-resolution X-ray diffractometer) analysis. XRD analyses were performed by 0.026° and the interval was of 10°–70° (2θ). Microstructure analyses of the composites were measured using scanning electron microscopy (SEM, SUPRA 40VP). Before the microstructure analysis, the samples were sanded with 600, 1000, and 2000 sanding and finally, a smooth surface was obtained using diamond solution. Sintered Al/SiC composite hardness was measured using a Vickers hardness testing machine (HBRV-187.5 Digital Universal Hardness Tester) 30 gf load applied for 10 seconds). Five measurements were taken from each composite and their average was determined.

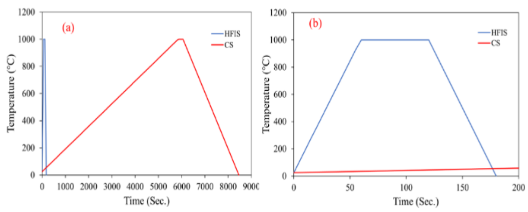

Figure 2. The time and temperature graph for HFIS and CS methods: (a) general graphic, (b) detail graphic.

3. Result and Discussion

In this study, HFIS was used in the sintering of aluminum composites prepared by adding SiC at different rates (10, 20 and 40 wt.%). The optimum sintering temperature was determined according to the amount of SiC additive. The Experimental density values of samples prepared with different SiC ratios are given in Table 1. The experimental densities of the samples increased with sintering temperature. As the SiC ratio increased, the sintering temperature required to increase the experimental density also increased. When 10 wt. % SiC was added to aluminum, a similar density value was obtained at all sintering temperatures. Therefore, the sintering temperature of the composition with 10% SiC was determined to be 660 °C. When the SiC ratio was 20% and 40%, the sintering temperatures were 800 and 1000 °C, respectively. Because the sintering process took place quickly, higher temperatures were needed to achieve the desired density. Liu et al. determined the sintering temperature as 770 °C for the addition of 15% SiC in the microwave sintering process [15].

Table 1. Density analysis of Al/SiC composites produced with HFIS

SiC ratio (wt.%)	Theoretical density (gr/cm^3)	Experimental density (gr/cm^3)		
		660 °C	800 °C	1000 °C
10	2.75	2.70	2.72	2.72
20	2.80	2.67	2.74	2.76
40	2.90	2.71	2.75	2.79

SEM images of the sintered samples are given in Figure 3a-i. The interface formed between aluminum particles and SiC particles is effective on the properties of Al-SiC composites. The SiC ratio and sintering temperature are the determining parameters in forming a good interface. The melting temperature of aluminum metal is known to be 660 °C. In conventional sintering processes, it has been observed that dense materials are obtained with long sintering time, such as 1-2 hours, below the melting temperature. [16]. At high SiC ratios the sintering process was carried out above the melting temperature of aluminum [4, 17]. In this study, the required sintering temperature was determined according to the SiC ratio in the sintering process with HFIS. SEM analysis in Figure 3 showed that a homogeneous microstructure was obtained at all SiC ratios.

Additionally, in Figures 3d, g and j, no fracture occurred in the SiC grains despite the high sintering temperature. Partial aggregation is observed when the SiC ratio is 40%, but it is at a low level. In Figure 3g, it was determined that partially pores were more in the sintering made at 660 °C at 40% SiC ratio. It was observed that these pores decreased as the sintering temperature increased. When the microstructure and density values of the samples sintered by HFIS were examined, it was determined that the sintering temperature increased depending on the SiC ratio. In the sintering process with HFIS, 10, 20 and 40 wt. % SiC was added and the required sintering temperatures were determined to be 660, 800 and 1000 °C, respectively.

Photographs of composites produced under optimum conditions are given in Figure 4. The samples were produced at different temperatures without deformation. It was observed that the samples became darker as the sintering temperature increased. This darkening was caused by a small amount of oxide layer formed due to the sintering process at high temperatures.

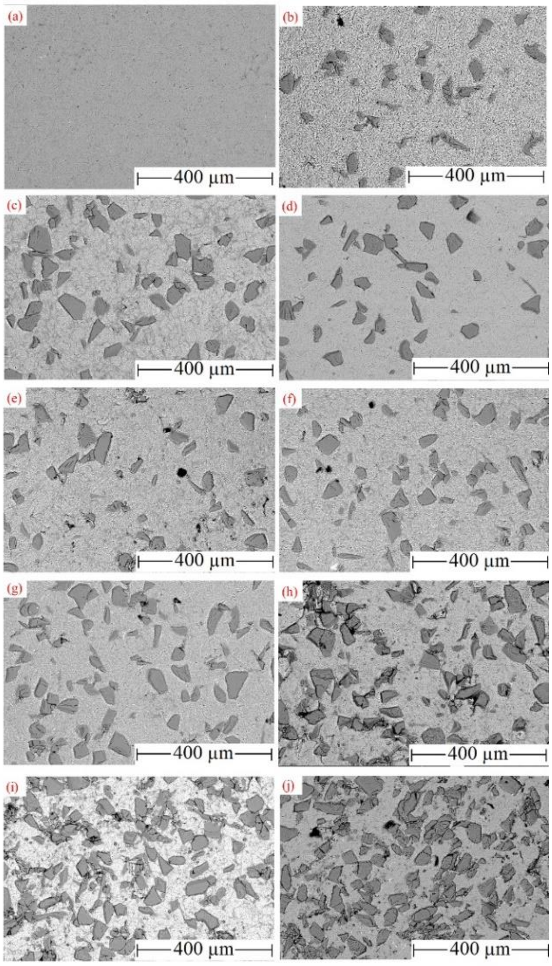

Figure 3. Microstructure images of samples sintered with HFIS at different SiC ratios and different temperatures; (a)Al-660 °C (b) Al10-660 °C, (c) Al10-800 °C, (d) Al10-1000 °C, (e) Al20-660 °C, (f) Al20-800 °C, (g) Al20-1000 °C, (h) Al40-660 °C, (i) Al40-800 °C, (j) Al40-1000 °C

Figure 4. Photograph of Al/SiC composites produced with HFIS at optimum temperatures (from left to right; Al composites with 10, 20, 40% SiC addition)

The microstructure images obtained as a result of sintering the optimum compositions with the CS method were given in Figure 5. It was determined that the Al/SiC composite produced with the addition of 10 wt% SiC at low temperature (660 °C) had a homogeneous microstructure. As the sintering temperature and SiC ratio increased, fractures began to occur in the SiC grains. In addition, short dimensional lath Al_4C_3 structures in white color were formed (Figure 5b). It was stated that Al_4C_3 structures were formed in sintering above the melting temperature.

The formation of a harmful aluminum carbide (Al_4C_3) phase has been detected in long-term contact of SiC grains with the liquid aluminum outer surface [18, 19]. It was observed that when sintering time and SiC amount increased, more Al_4C_3 structures were formed. When the microstructure formed as a result of sintering the Al40 composition at 1000°C was examined, it was observed that the SiC grains were more broken. Al_4C_3 structures also began to form longer. Figure 5d, lath Al_4C_3 length was determined to be around 50 microns. When samples containing Al_4C_3 phase are kept under atmospheric conditions, they slowly hydrolyze and form an Al-hydroxide structure [18]. Moisture-induced decomposition of the Al_4C_3 phase occurs in the reactions given in equations 2 and 3.

$$Al_4C_{3(s)} + 12H_2O_{(g)} \rightarrow 4Al(OH)_{3(s)} + 3CH_{4(g)} \tag{2}$$

$$Al_4C_{3(s)} + 18H_2O_{(l)} \rightarrow 4Al(OH)_{3(s)} + 3CO_{2(g)} + 12H_{2(g)} \tag{3}$$

Al_4C_3 phase is an undesirable phase because it causes deterioration of the mechanical and physical properties of the material. Figure 6 shows the microstructure of a sample containing Al_4C_3 phase, which is formed as a result of keeping it under room conditions for 1 year. As a result of the reaction of the unstable Al_4C_3 phase with water, cracks occurred in the sample due to the formation of $Al(OH)_3$ structure. However, Al_4C_3 structure is not formed in samples exposed to high temperatures for a short time during sintering with HFIS (Figure 3e-j).

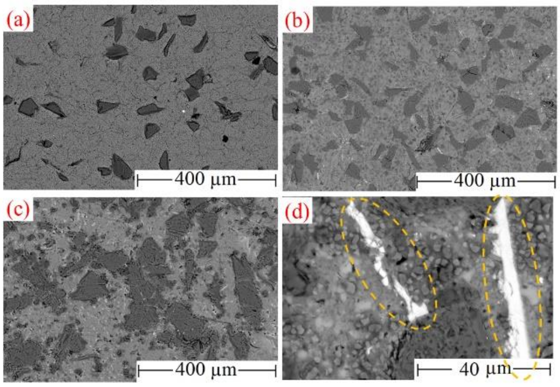

Figure 5. Microstructure images of samples sintered with CS at different SiC ratio and temperatures: (a) Al10-660 °C, (b) Al20-800 °C, (c) Al40-1000 °C, (d) Al40-1000 °C (high resolution)

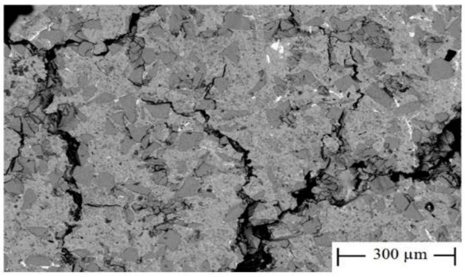

Figure 6. Microstructure image of 40% SiC doped aluminum composite, which is sintered by CS method at 1000 °C and kept for one year at room conditions

In Figure 7, XRD analyzes of samples sintered with HFIS at different temperatures and SiC ratios are given. Al (PDF# 98-007-7363) and SiC (PDF# 00-027-1402) peaks were detected in the samples produced under different conditions. In the XRD analysis, it is clearly seen that the Al_4C_3 phase is not formed. According to the diffraction peaks of Al_4C_3 between 30° and 33° as shown in Figure 8a, the presence of an Al_4C_3 phase could not be detected after the HFIS sintering process at all temperatures. The characteristic peaks of Al_4C_3 were observed when the composite of Al/SiC (wt.%40) was sintered in the convectional system at 1000 °C (Figure 8b). It is stated that the Al_4C_3 phase, which generally occurs at high temperatures, is formed even below 660 °C [18, 20]. Therefore, short-term sintering with HFIS is an advantageous process in preventing the Al_4C_3 phase.

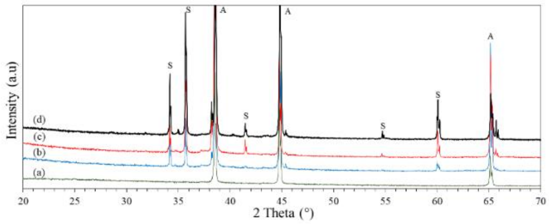

Figure 7. X-ray diffraction patterns of Al/SiC composites sintered with HFIS; (a) Al-660 °C, (b) Al10-660 °C (c) Al20-800 °C, (d) Al40-1000 °C (A: Aluminium, S: SiC)

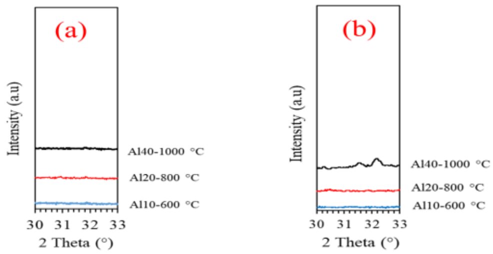

Figure 8. X-ray diffraction patterns of Al/SiC composites after sintering; (a) HFIS, (b) CS

Figure 9 shows the relationship between the hardness of the composites and the SiC content for both sintering methods. Hardness increases with increasing SiC addition [21]. When the SiC ratio was wt.%10, 20 and 40, the hardness values were found to be 43, 69 and 118 HV, respectively. In the literature, the hardness values of Al/SiC composites vary between 25 and 90 HV depending on the SiC ratio [16, 22-25].

It was determined that the hardness values obtained in this study were compatible with the literature. When the SiC ratio was wt.% 40, a very high hardness value was obtained (118 HV). In the sintering process with CS, the hardness value of the composite with wt% 40 SiC addition was determined as 91 HV. This difference in hardness value may result from the breaking of SiC grains during conventional sintering (Figure 5c). HFIS is an advantageous process for sintering Al/SiC composites without degradation.

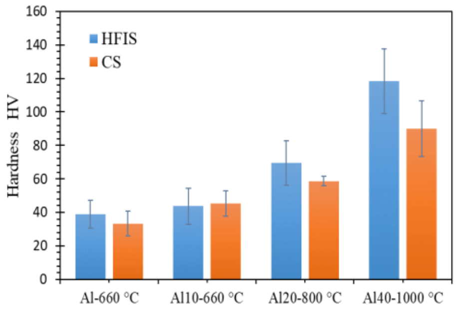

Figure 9. Hardness value of Al/SiC composites

4. Conclusion

In this study, aluminum matrix composites containing different ratios of SiC were successfully produced with HFIS. The required sintering temperature was determined according to the SiC ratio used. When the SiC ratio is 10, 20 and 40%, the required sintering temperature was determined to be 660, 800 and 1000 °C, respectively. Al/SiC composites were produced successfully without the formation of harmful Al_4C_3 phase in sintering with HFIS. Composite samples were obtained in Al and SiC phases, with hardness values of 43-118 HV, at high experimental density above the melting temperature of aluminum.

When the composites were sintered with the CS method with a heating rate of 10 °/min and compared with HFIS, it was determined that the Al_4C_3 phase was formed (wt.%40 SiC added composition). This composition decomposes under long-term atmospheric conditions. Al/SiC composites were successfully produced by HFIS sintering process without new phases forming above the melting temperature.

Article Information Form

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Authors' Contribution
The authors contributed equally to the study.

The Declaration of Conflict of Interest/ Common Interest
No conflict of interest or common interest has been declared by the authors.

The Declaration of Ethics Committee Approval
This study does not require ethics committee permission or any special permission.

The Declaration of Research and Publication Ethics
The authors of the paper declare that they comply with the scientific, ethical and quotation rules of SAUJS in all processes of the paper and that they do not make any falsification on the data collected. In addition, they declare that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered, and that this study has not been evaluated in any academic publication environment other than Sakarya University Journal of Science.

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