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Synthesis and Characterization of $MoSi_2$ Particle Reinforced AlCuMg Composites by Molten Salt Shielded Method

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Molten salt shielded, AlCuMg composite, Powder metallurgy, Microstructure, Sintering, Mechanical properties, $MoSi_2$

Abstract: AlCuMg and its alloys are the most preferred composite materials in areas such as automotive, aerospace and aerospace industries due to their positive properties such as high corrosive properties, heat resistance, high strength and toughness. However, the inadequacy of these alloys in terms of wear and mechanical properties is one of the problems encountered in the industry. As a result of the literature review, it has been determined that there are almost no studies to improve the mechanical properties of these alloys. In this study, $MoSi_2$ particles in different percentages were added to AlCuMg matrix and produced by powder metallurgy method. The aim is to improve the mechanical properties of AlCuMg matrix composite material and to perform characterization processes. Molten salt shielded synthesis/sintering process was used as a protective atmosphere environment for the produced samples to protect them from oxidation during sintering. In salt-protected synthesis, potassium bromide (KBr) was preferred as the salt. After the synthesis process, Scanning Electron Microscope (SEM), Energy Dispersion Spectroscopy (EDS), X-Ray Diffractometry (XRD) analyzes were applied respectively as characterization processes. In addition, microhardness test was performed to determine the mechanical properties of the produced composites. It has been determined that $MoSi_2$ particles reinforced to AlCuMg alloy increase the mechanical properties of AlCuMg composite material because they have high values in terms of hardness and melting temperature. The hardness of the unreinforced sample is approximately 90 $HV_{0.1}$. According to the addition of 5, 10 and 15% $MoSi_2$, the hardness of the samples is 266, 317 and 422 $HV_{0.1}$, respectively. The hardness of the $MoSi_2$ added samples was higher than the non-reinforced sample.

$MoSi_2$ Parçacık Takviyeli AlCuMg Kompozitlerin Erimiş Tuz Korumalı Yöntem ile Sentezi ve Karakterizasyonu

Anahtar Kelimeler
Erimiş tuz korumalı, AlCuMg kompozit, Toz metalurjisi, Mikroyapı, Sinterleme, Mekanik özellikler, $MoSi_2$

Öz: AlCuMg ve alaşımları yüksek korozif özellikleri, ısıl direnci, yüksek mukavemet ve tokluk gibi pozitif özelliklerinden dolayı otomotiv, havacılık ve uzay endüstrisi gibi alanlarda en çok tercih edilen kompozit malzemeler olarak karşımıza çıkmaktadır. Ancak, bu alaşımların aşınma ve mekanik özellik bakımından yetersiz olmaları endüstride karşılaşılan problemlerden biridir. Literatür taraması sonucunda bu alaşımların mekanik özelliklerini iyileştirmeye yönelik neredeyse yok denecek kadar az çalışmaların mevcut olduğu tespit edilmiştir. Bu çalışmada AlCuMg matrisine farklı yüzde oranlarında $MoSi_2$ parçacıkları takviye edilerek toz metalurjisi yöntemi ile üretilmiştir. Amaç, AlCuMg matrisli kompozit malzemenin mekanik özelliklerini iyileştirerek karakterizasyon işlemlerinin yapılmasıdır. Üretilen numuneler için koruyucu atmosfer ortamı olarak, sinterleme esnasında oksidasyondan korumak için erimiş tuz korumalı sentez/sinterleme (molten salt shielded synthesis/sintering process) işlemi uygulanmıştır. Tuz korumalı sentez uygulamasında tuz olarak potasyum bromür (KBr) tercih edilmiştir. Sentezleme işlemi sonrasında karakterizasyon işlemleri olarak sırasıyla, Taramalı Elektron Mikroskobu (SEM), Enerji Dağılım Spektroskopisi (EDS), X-Işını Difraktometresi (XRD) analizleri uygulanmıştır. Buna ek olarak, üretilen kompozitlerin mekanik özelliklerini belirlemek için mikrosertlik testi yapılmıştır. AlCuMg alaşımına takviye edilen $MoSi_2$ parçacıkları, sertlik ve ergime sıcaklığı olarak yüksek değerlere sahip olduğu için AlCuMg kompozit malzemesinin mekanik özelliklerini arttırdığı tespit edilmiştir. Takviyesiz olan numunenin sertliği yaklaşık 90 $HV_{0,1}$'tir. % 5, 10 ve 15 $MoSi_2$ ilavesine göre numunelerin sertliği sırasıyla 266, 317 ve 422 $HV_{0,1}$'tir. $MoSi_2$ katkılı numunelerin sertliği, takviyesiz üretilen numuneden daha yüksek çıkmıştır.

1. INTRODUCTION

Among the recently applied methods, AlCuMg alloys produced by powder metallurgy (PM) were found to be remarkable. Chemical composition control of the alloy can be achieved with the PM method. Powder metallurgy has been developed as an alternative to production methods such as casting, machining, hot and cold pressing. By obtaining composites by powder metallurgy method, materials such as wear resistance, corrosion resistance, surface friction and surface tension can be increased. Composite materials have an indispensable place among the material groups used in engineering applications. As a result of the development of thinner and lighter composites by increasing the strength/weight ratio, the unit cost of production and operating expenses is reduced. Composite materials with very good mechanical properties are produced with AlCuMg alloys, which are successful in terms of lightness and strength. AlCuMg and its alloys are preferred as high engineering alloys in fields such as aerospace, automotive and biomedical due to their excellent heat resistance, corrosion resistance, toughness and strength [1-6]. In addition, these materials are used in industrial and medical applications such as machinery equipment and building materials, medical devices and vehicles, as well as advanced applications such as electronic devices, spacecraft, and many products that facilitate daily life such as super-elastic eyeglass frames, telephone antennas [7,8]. In recent years, it has become widespread in applications in the field of robotics. However, in the industrial use of AlCuMg and its alloys, problems are encountered in terms of mechanical properties and wear properties [9]. Due to these problems, since the mechanical properties and wear properties of AlCuMg alloy are low, it has been produced by adding $MoSi_2$ particles to this alloy by powder metallurgy method. As the abrasion resistance and mechanical properties of the produced samples will increase, a gap needed in the industry will be closed.

Tang et al. In 1997, they carried out the study. In experimental studies, mechanical alloying, SEM, XRD and DSC analyzes were performed. As a result, they successfully produced the Cu-Al-Ni alloy. In their results, they emphasized that the microhardness values increased with the increase of mechanical time [10]. Du et al. In 2017, they carried out the study. The materials used in this experimental study include commercial purity Al, commercial purity crystalline Si, Al-5C master alloys were used. Al-Si based composites reinforced with SiCp can be used in automotive, aerospace and aerospace industries. As a result, in this study, Al-Si composites were made and the effect of SiC on microstructures, mechanical properties and heat treatment procedures were investigated [11]. Karakulak et al. In 2014, they carried out the study. In this experimental study, alloying elements (Si, Ti, Cr, Cu, Mg, Ni) were added to Aluminum. They aimed to improve the mechanical properties of Al alloys and aluminum. As a result, the wear and hardness resistances of aluminum were higher than the matrix composites. Their microstructure and processing details have been studied in the literature [12]. Alizadeh et al. In 2017, they conducted a study. It is aimed to strengthen super high strength nanostructured B4C, Al-2Cu aluminum alloy matrix composites. As a result, they found that increasing the content of B_4C particles increased the matrix grain size [13]. Mandala and Viswanathanb conducted a study. In this experimental study, the effect of heat treatment on microstructure and SiC Aluminum-based 2124 alloy with 10% by weight silicon carbide (SiC) particle reinforced composite was prepared. As a result, they emphasized that the size of SiC particles was reduced after hot rolling [14]. Elkady et al. In 2019, they carried out a study. In this experimental study, Al / Ni-SiC composite was prepared and it was aimed to absorb the Al matrix composite by microwave. As a result, they found that Ni-SiC particles were homogeneously distributed throughout Al [15]. Vrsalović et al. In 2018, they carried out the study. In the experimental study, the effects of heat treatment on the corrosion properties of CuAlNi alloy were analyzed. As a result, the increase in the polarization resistance values of the heat-treated CuAlNi alloy revealed a beneficial effect on the corrosion resistance of the CuAlNi alloy in NaCl solution [16].

The aim of this study is to investigate the effect of the addition of $MoSi_2$ particles on the microstructure and mechanical properties of AlCuMg alloy produced by powder metallurgy method. In this study, the changes in the microstructure and mechanical properties of $MoSi_2$ reinforced AlCuMg composites pressed at constant pressing pressure and sintered at constant temperature were compared.

2. EXPERIMENTAL STUDIES

To be used in the experiments, 4 (four) kinds of alloys were produced by powder metallurgy method. Al, Cu, Mg and $MoSi_2$ powders with approximately 99.9% purity and a grain size of 325 mesh were used in the production of the alloys. In this study, it was produced by powder metallurgy method by adding $MoSi_2$ particles at different rates (5, 10 and 15%) to AlCuMg alloy. For this purpose, firstly, powders for AlCuMg alloy $MoSi_2$ particles were prepared by weighing according to the percentage chemical composition given in Table 1.

Table 1. Chemical compositions of the produced test samples

Sample No	Al (wt %)	Cu (wt %)	Mg (wt %)	$MoSi_2$ (wt %)
1	90	5	5	-
2	85	5	5	5
3	80	5	5	10
4	75	5	5	15

In order to prepare the mixtures, in order to obtain a homogeneous mixture by mixing the pure metal powders thoroughly with each other, an 88 type, closed powder chamber, two kg powder capacity, three-dimensional rotary mixing mixer was used. The powder chamber of this mixer is especially closed type, and after the powder is placed in it, its lid is tightly closed in order to cut off the relationship between the external environment and the powders. After the mixer is started, the powder chamber can rotate 360 degrees in all directions so that the powder can be mixed thoroughly, thus ensuring that the powders are thoroughly mixed with each other. In the pressing application, 600 MPa pressure was applied to the samples as pressing pressure. The sintering process of the produced pellets was carried out with an atmosphere-controlled heat treatment furnace (Protherm) in the Mechanical Engineering Laboratories of the Faculty of Engineering and Architecture of Kastamonu University. The sintering process was carried out in an argon atmosphere at 550 °C for 1 hour.

The sintering process was completed in a total of 180 minutes. In order to discharge the wastes, the sintering temperature was increased to 550 °C in 60 minutes at a rate of 10 °C/minute. Then, the sintering temperature was kept at 550 °C for 60 minutes at a constant temperature. The cooling process of the samples was cooled to room temperature in 60 minutes under atmosphere control.

Metallographic processes were applied to the samples for scanning electron microscopy (SEM) and Energy Dispersive Spectrum (EDS) analyzes of the samples. These applied metallographic processes were performed as sanding, polishing and etching, respectively. The surfaces of the samples were sanded with 200, 360, 600, 1000, 1200 and 2000 mesh abrasives, respectively. Afterwards, the surfaces of the samples were polished with 3 and 1 µ diamond suspensions, respectively. Finally, the etching reagent was prepared by using 95 ml of distilled water, 2.5 ml of nitric acid (HNO3), 1.5 ml of Hydrochloric acid (HCI), 1 ml of Hydrofluoric acid (HF), and they were etched with this reagent by immersion for 20 seconds.

After the samples were etched, SEM images were taken from the "FEI QUANTA 250 FEG" brand device in Kastamonu University Central Research Laboratories. XRD measurements of the samples were made with the Bruker D8 Advance brand device in Kastamonu University Central Research Laboratories. It was investigated as a mechanical property how the hardness values due to the applied pressing pressure and MoSi2 reinforcement and sintering temperature for the samples produced in this study were affected. In order to determine the hardness measurements of the samples in a healthy way, measurements were made from at least 5 points and the average results of the measurements were taken into consideration.

3. RESULTS AND DISCUSSION

3.1. SEM Analysis Results

SEM images of $MoSi_2$ reinforced AlCuMg composites produced by powder metallurgy method were taken and evaluations were made according to the obtained images. When the SEM images of the sample number 1 given in Figure 1 are examined, the AlCuMg matrix structure is clearly seen.

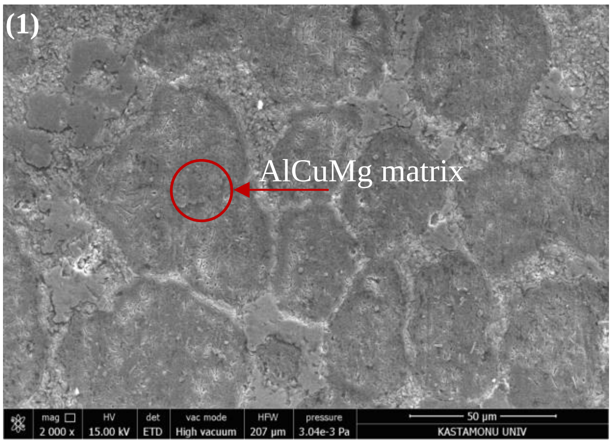

Figure 1. SEM image of sample number 1 produced

SEM images of AlCuMg composites reinforced with MoSi2 in different ratios (5, 10 and 15%) produced by powder metallurgy method were taken and evaluations were made according to the obtained images. When the SEM images of samples 2, 3 and 4 given in Figure 2 are examined, the AlCuMg matrix structure is clearly seen. In addition, it is seen that $MoSi_2$ particles are homogeneously dispersed in the AlCuMg matrix [1,17,18].

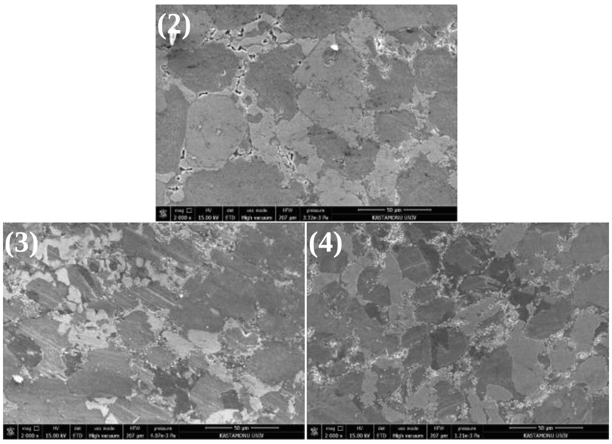

Figure 2. SEM images of samples 2, 3 and 4 produced

Partially cracks and pores are seen in the produced samples. When the SEM images given are examined, it is clearly seen that the amount of pores decreases as the addition of $MoSi_2$ increases. Besides, in the SEM images given, it is seen that MoSi2 particles are homogeneously distributed in the internal structure. It was interpreted that the homogeneous distribution of $MoSi_2$ particles in the internal structure resulted from the mixing and sintering process. This is an expected situation. There are studies in the literature that support this result. In addition, when the SEM images given are examined, the AlCuMg structure used as the matrix is clearly seen. $MoSi_2$ particles used as reinforcements are similar to each other and generally have sharp corners and irregular geometry [19-21]

3.2. SEM-EDS Analysis Results

SEM-EDS analyzes of AlCuMg, AlCuMg-$MoSi_2$ composite samples produced by powder metallurgy method are given in Figures 3, 4 and 5, respectively. When the given SEM-EDS analysis results are examined, it is clearly seen that the results support the chemical composition of the produced samples. In addition, different ratios of Al, Cu, Mg, Mo and Si were found in the internal structures of the produced samples.

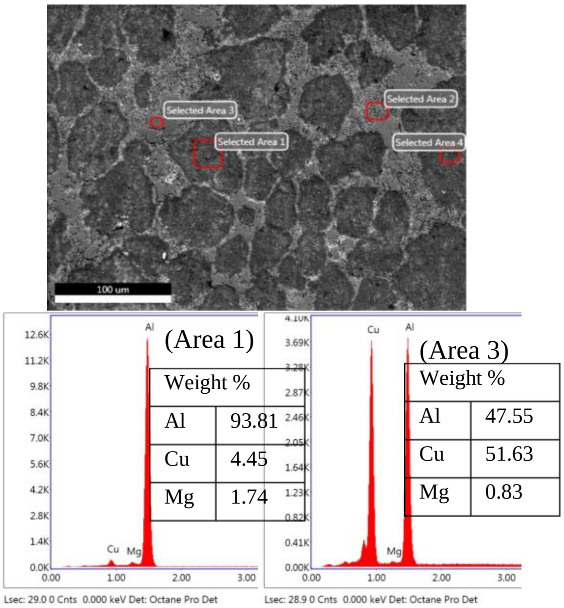

Figure 3. SEM-EDS analysis result of sample number 1 produced

When the SEM-EDS analysis results given in Figure 3 were examined, it was determined that the produced AlCuMg composite sample supported the chemical compositions. According to the results of EDS analysis taken from the selected area 1 in the internal structure of the produced AlCuMg composite sample, it was determined that it was 93.81% Al, 4.45% Cu and 1.74% Mg. In addition, according to the results of the EDS analysis taken from the selected area 3, it was determined that there are 47.55% Al, 51.63% Cu and 0.83% Mg.

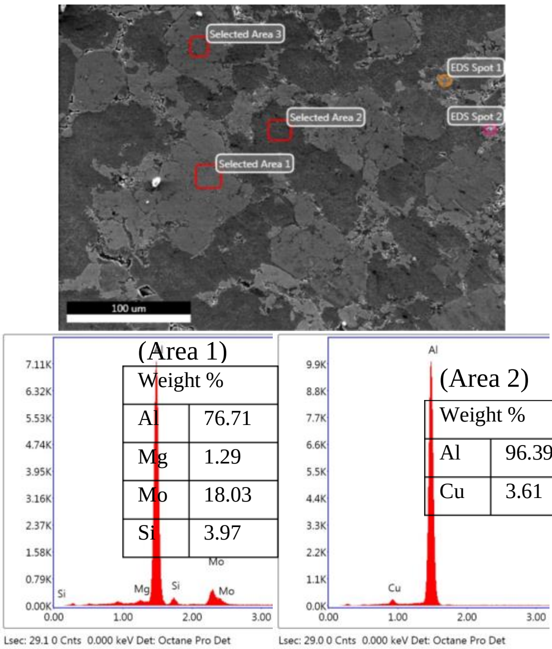

Figure 4. SEM-EDS analysis result of the 2nd sample produced

When the SEM-EDS analysis results given in Figure 4 were examined, it was determined that the produced AlCuMg-$MoSi_2$ composite sample supported the chemical compositions. In the internal structure of the produced AlCuMg-$MoSi_2$ composite sample, it was determined that 76.71% Al, 1.29% Mg, 18.03% Mo and 3.97% Si were determined according to the EDS analysis result taken from the region specified as selected area 1. In addition, it was determined that 96.39% Al and 3.61% Cu were obtained according to the EDS analysis result taken from the region specified as selected area 2.

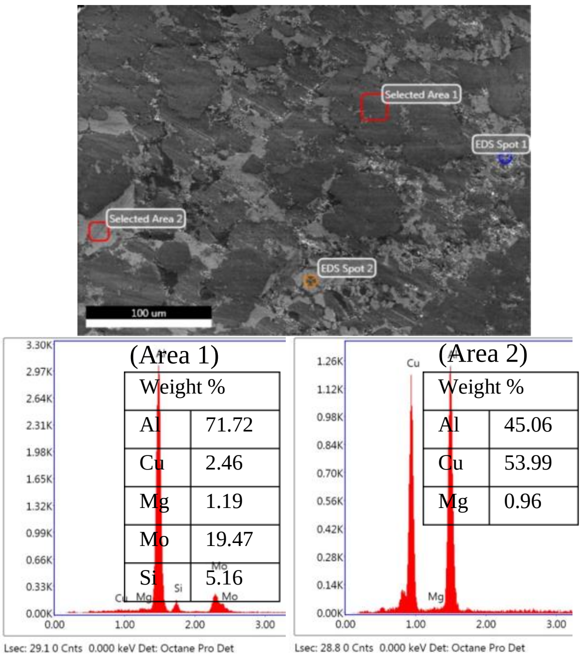

Figure 5. SEM-EDS analysis result of the 3rd sample produced

When the SEM-EDS analysis results given in Figure 5 were examined, it was determined that the produced AlCuMg-$MoSi_2$ composite sample supported the chemical compositions. According to the results of the EDS analysis taken from the selected area 1 in the internal structure of the produced AlCuMg-$MoSi_2$ composite sample, it was determined that there are 71.72% Al, 2.46% Cu, 1.19% Mg, 19.47% Mo and 5.16% Si. In addition, according to the results of the EDS analysis taken from the region specified as selected area 2, 45.06% Al, 53.99% Cu and 0.96% Mg were determined.

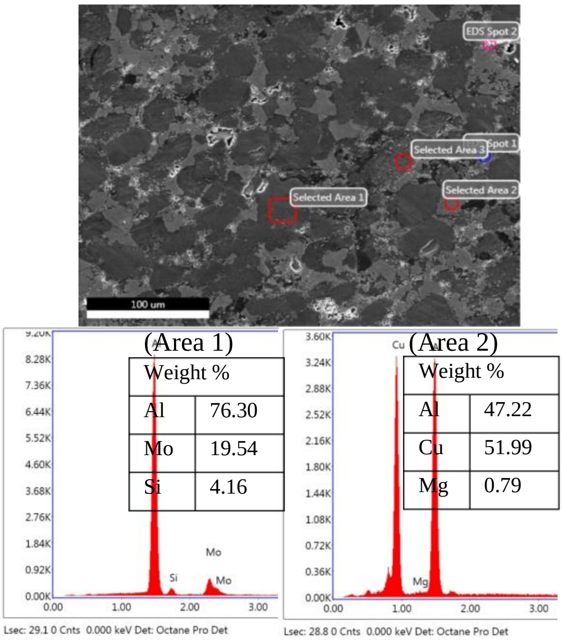

Figure 6. SEM-EDS analysis result of sample number 4 produced

When the SEM-EDS analysis results given in Figure 6 were examined, it was determined that the produced AlCuMg-$MoSi_2$ composite sample supported the chemical compositions. According to the results of EDS analysis taken from the selected area 1 in the internal structure of the produced AlCuMg-$MoSi_2$ composite sample, 76.30% Al, 19.54% Mo and 4.16% Si were determined. In addition, according to the results of the EDS analysis taken from the selected area 2, it was determined that there are 47.22% Al, 51.99% Cu and 0.79% Mg.

3.3. XRD Analysis Results

XRD graphics of AlCuMg and AlCuMg-$MoSi_2$ composites produced by powder metallurgy method are given in Figure 7. When the given XRD graph was examined, it was determined that Al_2CuMg, AlCuMg, Al_2Cu, $Al_{22}Mo_5$, $Mg_{3.78}Al_{4.22}$, Cu_3Mo_2, Cu_6Mo_5, $Si_{1.91}Al_{4.09}$, $Mg_2Al_4Si_5$, MgMo and Al phases were formed. In addition, it is clearly seen that the Cu_3Mo_2, $Al_{22}Mo_5$, Al_2CuMg phases are dominant. Similar peaks are also seen in studies conducted in the literatüre [22-25]

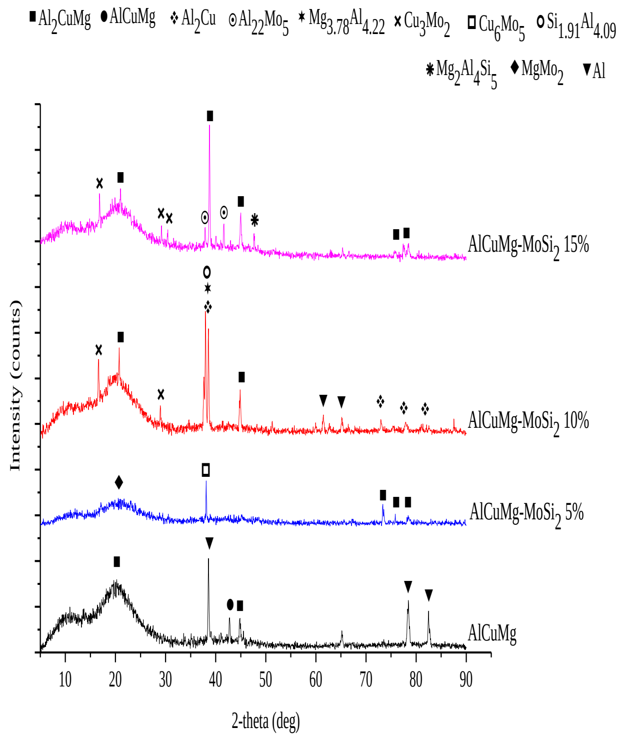

Figure 7. XRD graphs of AlCuMg and AlCuMg-MoSi2 composites

During the sintering process, the above-mentioned phases were formed between Al, Cu, Mg, Mo and Si. In addition, it was determined that the intensity of Cu3Mo2, Al22Mo5, Al2CuMg phases increased as the MoSi2 reinforcement ratios increased in the produced composite samples. The peaks detected in this study were also supported by other studies in the literatüre [22,25]. In addition, whether these precipitates formed in these structures are formed or not is proved by XRD, and the results obtained from the XRD analysis have been found to support the results of point and area EDS analysis made in SEM examinations.

3.4. Microhardness Analysis Results

The microhardness graph of the produced samples is given in Figure 8. Hardness measurements were taken from the sample surface along a line line at 100 μm intervals. The hardness of the unreinforced sample is approximately 90 $HV_{0.1}$. According to the addition of 5, 10 and 15% $MoSi_2$, the hardness of the samples is 266, 317 and 422 $HV_{0.1}$, respectively. The hardness of $MoSi_2$ added samples is higher than the sample produced without reinforcement. This increase was interpreted to be related to the presence of carbide and formed hard phases [26-30]. The use of $MoSi_2$ as reinforcement led to the formation of Cu_3Mo_2, $Al_{22}Mo_5$ and Al_2CuMg phases, and as a result it was found to contribute to the increase in hardness.

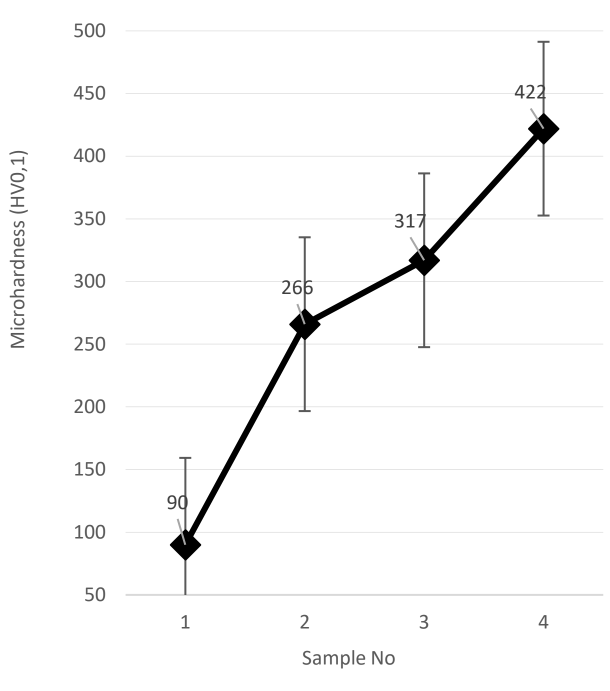

Figure 8. Microhardness graph for the composites

4. GENERAL RESULTS

In this study, $MoSi_2$ reinforced AlCuMg composites were successfully produced by powder metallurgy method. As the production parameter, cold pressing at 600 MPa pressure and sintering process at 550 ℃ in argon atmosphere for 1 hour were performed. SEM, SEM-EDS, XRD and microhardness analysis results of these produced samples were examined. As a result of the studies carried out; $MoSi_2$ reinforced AlCuMg composites were successfully subjected to pressing and sintering by powder metallurgy method. As a result of SEM analysis, it was clearly determined that the amount of pores decreased as the addition of $MoSi_2$ increased. In addition, it was determined that $MoSi_2$ particles were homogeneously distributed in the internal structure in the SEM images taken. It was interpreted that the homogeneous distribution of $MoSi_2$ particles in the internal structure resulted from the mixing and sintering process in the three-dimensional turbula. In the SEM-EDS analysis results of the produced samples, it was clearly determined that the samples supported the chemical compositions. In addition, it has been reported that the produced samples have different ratios of Al, Cu, Mg, Mo and Si in their internal structures. From the XRD analysis results, it was determined that Al_2CuMg, AlCuMg, Al_2Cu, $Al_{22}Mo_5$, $Mg_{3.78}Al_{4.22}$, Cu_3Mo_2, Cu_6Mo_5, $Si_{1.91}Al_{4.09}$, $Mg_2Al_4Si_5$, MgMo and Al phases were formed. In addition, it was determined that Cu_3Mo_2, $Al_{22}Mo_5$, Al_2CuMg phases were dominant. It was determined that the intensity of the Cu_3Mo_2, $Al_{22}Mo_5$, Al_2CuMg phases increased as the $MoSi_2$ reinforcement ratios increased in the produced composite samples. The hardness of the unreinforced sample is approximately 90 $HV_{0.1}$. According to the addition of 5, 10 and 15% $MoSi_2$, the hardness of the samples is 266, 317 and 422 $HV_{0.1}$, respectively. The hardness of the $MoSi_2$ added samples was higher than the non-reinforced sample.

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