



Research Paper / Makale

Investigation of WC Reinforced CuNiSi Composites Produced by Mechanical Alloying Method

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Abstract: In this study, composite samples were produced by adding WC particles in different proportions (2.5, 5, and 10%) by weight to the CuNiSi powder mixture by the powder metallurgy method. The prepared powders were mixed with a mechanical alloying device for 5 and 10 hours. After the mixing process, the powders were pressed with a cold press. In the cold pressing process, 600 MPa pressure was applied as pressing pressure. The sintering process of the pellets produced was carried out with an atmosphere-controlled heat treatment furnace. The sintering process was applied in an argon atmosphere at 900 °C for 1 hour. Scanning electron microscope (SEM-EDS) to determine the microstructure of the samples produced, X-Ray Diffraction method (XRD) analysis to determine the phases formed in the internal structures of the produced samples, and microhardness (HV_{0.05}) to determine the effect of WC particles on hardness. From the microstructure results in the SEM pictures, it was determined that the WC particles were homogeneously distributed in the structure. It was determined that there was an increase in hardness measurement values due to the increasing amount of WC and mechanical alloying time in the produced samples. The highest hardness value of 198 HV_{0.05} was measured on a composite sample reinforced with 10% WC by weight and mechanically alloyed for 10 hours.

Keywords: CuNiSi composite, Mechanical alloying, WC reinforced, Powder metallurgy

Mekanik Alaşım Yöntemi ile Üretilen WC Takviyeli CuNiSi Kompozitlerinin İncelenmesi

Öz: Bu çalışmada CuNiSi toz karışımına ağırlıkça farklı oranlarda (% 2,5, 5 ve % 10) WC parçacıkları toz metalurjisi yöntemi ile ilave edilerek kompozit numuneler üretilmiştir. Hazırlanan tozlar, mekanik alaşım cihazı ile 5 ve 10 saat süre ile karıştırılmıştır. Karıştırma işleminden sonra tozlar bir soğuk pres ile preslenmiştir. Soğuk presleme işleminde presleme basıncı olarak 600 MPa basınç uygulanmıştır. Üretilen peletlerin sinterleme işlemi atmosfer kontrollü ısı işlem fırını ile gerçekleştirilmiştir. Sinterleme işlemi argon atmosferinde 900 °C sıcaklıkta 1 saat uygulanmıştır. Üretilen numunelerin mikroyapısını belirlemek için taramalı elektron mikroskobu (SEM-EDS), üretilen numunelerin iç yapılarında oluşan fazları belirlemek için X-ışını kırınım yöntemi (XRD) analizi ve WC parçacıklarının sertliğe etkisini belirlemek için mikrosertlik (HV_{0.05}) testleri yapılmıştır. Mikroyapı sonuçlarından WC parçacıklarının yapı içinde homojen dağıldığı SEM resimlerinden tespit edilmiştir. Üretilen numunelerde artan WC miktarı ve mekanik alaşım süresine bağlı olarak sertlik ölçüm değerlerinde artış olduğu belirlenmiştir. En yüksek sertlik değeri 198 HV_{0.05} ile ağırlıkça % 10 WC takviyeli ve 10 saat mekanik alaşım yapılmış kompozit numunede ölçülmüştür.

Anahtar Kelimeler: CuNiSi kompozit, Mekanik alaşım, WC takviye, Toz metalurjisi

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1. Introduction

With rapidly developing technology, traditional materials are replaced by new materials with more specific features. For this reason, in the rapidly developing technology, it has still been compulsory to make improvements in the raw materials of the industrial industry. Scientists working in the field of materials generally turned to research new materials that have superior properties to natural and alloy materials. These new materials are produced as composite materials with superior properties, unlike other traditional materials. Composite materials have many advantages over traditional materials. The best features of composite materials are that they are light but have high strength [1,2].

Composites have an indispensable place among the material groups used in engineering applications. As a result of the development of thinner and lighter composites with the increase in strength and weight ratio, the unit cost of production and operating costs were reduced. With Titanium, the most successful metal ever in terms of lightness and strength, parts with both high tensile strength and high fatigue strength can be produced [3].

Powder metallurgy, which is among the metal manufacturing methods, is a method that has a very common usage area. The reason for this widespread use is due to the high quality of manufacturing and the ability to manufacture very complex parts economically. Therefore, the production of various materials with such a method increases over time and replaces traditional metal processing methods [4,5]. Besides increasing the strength of the material, nickel has a healing property against temperature resistance. Besides, nickel increases the hardening depth and decreases the critical cooling rate [6]. There are some studies in the literature on the relationship between microstructure and mechanical properties in copper and nickel-based composites. Tang et al. in 1997, produced CuAlNi composites by mechanically alloying them with powder metallurgy method. In their results, they emphasized that the microhardness values increased with the increase of mechanical time [7]. Yener et al. in 2015, produced the powder metallurgy method by reinforcing graphite powder to Cu composite materials without any atmosphere control. They reported that the hardness values of the produced samples were between 84.5 and 94.6 HB [8]. Du et al. in 2017, produced composite materials by reinforcing SiC to AlSiCu and NiMg piston alloys. As a result, Al-Si composites were made in this study, and the effect of SiC on microstructures, mechanical properties, and heat treatment procedures were examined [9]. Karakulak et al. in 2014, investigated the mechanical and corrosion properties of the samples produced by adding Ni in different proportions to AlCuSiC powders. As a result, aluminum has higher wear and hardness resistance compared to matrix composites [10]. Ni et al. in 2018, added TiC to the Copper material, mixed it with a ball mill for 48, 72, 84, 96, and 120 hours, and then produced the samples using a hot press. The hardness of Cu-matrix composites increased with the increasing duration of the ball mill [11]. Alizadeh et al. in 2017, added Boron Carbide to Al-2Cu aluminum alloy material in different proportions by mechanical alloying with powder metallurgy method, and then examined the microstructure, mechanical and wear characterizations of the samples produced. As a result, they found that increasing the content of B₄C particles increased the matrix particle size [12]. Mandal and Viswanathanb added different proportions of SiC particles to the aluminum alloy 2124 material in 2013 and investigated the effects of heat treatment on the microstructure of the samples produced. As a result, they emphasized that the size of SiC particles is reduced after hot rolling [13]. Salvo et al. in 2019, produced graphene-reinforced copper matrix composites using mechanical alloying and hot pressing [14]. Elkady et al. in 2019, applied the sintering process by adding Ni and SiC particles in different proportions to aluminum matrix composites. As a result, they found that Ni-SiC particles were distributed homogeneously throughout Al [15]. Liu et al. in 2017, mixed Graphene and copper powders in different proportions using a ball mill with powder metallurgy method and then applied cold pressing and sintering to the samples. They applied the hardness, electrical conductivity, corrosion resistance, and relative density tests of the produced samples. As a result of these tests, they reported that the best sample was the sample containing 0.2% graphene [16].

Vrsalović et al. in 2018, investigated the effect of heat treatment on the corrosion properties of CuAlNi alloys. In the experimental study, the effects of heat treatment on the corrosion properties of CuAlNi alloy were analyzed [17]. Wang et al. in 2018, hot-pressed Cu-ZrB₂ composites were produced by adding Copper ZrB₂ particles. They studied microstructural properties, mechanical and electrical properties [18]. Islak et al. in 2014, successfully produced Cu-TiC composite materials using the hot pressing method. They weighed the Cu and TiC powders in different proportions and then applied to mix. As a result of scanning electron microscopy, they determined that TiC particles were homogeneously distributed in the Cu matrix. They determined that the hardness of the samples produced was between 64.5 HV_{0.1} and 85.2 HV_{0.1} as the sintering temperature increased [19]. Şimşek [20] stated in her study that there was an increase in the hardness of the sample mechanically alloyed and sintered for a certain period. Demirtaş determined that with the addition of Ti, the lamellar microstructure of NiAl and Cr-Mo greatly changed and the hardness value increased by approximately 10% [21].

In this study, as a result of literature researches, there is no study about the producibility of WC reinforced CuNiSi composite materials by the mechanical alloying method. For this purpose, WC reinforced CuNiSi composite materials were produced by a mechanical alloying method to fill this gap in the literature.

2. Materials and Methods

This study, it was aimed to produce by powder metallurgy method by adding WC particles in different proportions (2.5, 5, and 10%) to CuNiSi alloy. For this purpose, powders for CuNiSi alloy will be prepared by weighing according to their chemical composition in% given in Table 1.

Table 1. Chemical composition of CuNiSi composite mixture

	Cu (%)	Ni (%)	Si (%)
Preparation of CuNiSi alloy	95	4	1

WC additions to the prepared CuNiSi alloy will be prepared by weighing according to its chemical composition in% given in Table 2.

Table 2. Production parameters of composite materials

No	CuNiSi (%)	WC (%)	Temperature (°C)	Mechanical Alloying Time (hour)
1	100	-		
2	97,5	2,5	900	5
3	95	5		
4	90	10		
5	100	-		
6	97,5	2,5	900	10
7	95	5		
8	90	10		

2.1. Preparation of Samples

2.5, 5, and 10% WC was added to the CuNiSi powder mixture. The prepared mixture powders were mixed with Retsch brand PM100 model traveling ball mill device at 400 rpm for 5 and 10 hours and mechanical alloying was performed.

The prepared powders were mixed for 5 and 10 hours with the mechanical alloying device (Retsch brand PM100 model) in the Metallurgy and Materials Engineering Laboratory of the Faculty of Engineering and Architecture of Kastamonu University. After the mixing process, the powders were pressed with the press in the same laboratory. In the cold pressing process, 600 MPa pressure was applied as pressing pressure. The pressing process was done with the Specac brand GS15011 model hydraulic pellet press. A cylindrical mold with a diameter of 13 mm is used as a mold in the pressing process.

The sintering process of the powders was carried out with the atmosphere-controlled heat treatment furnace (Protherm) in Kastamonu University Metallurgy and Materials Engineering Laboratories. After the pressing process, the samples were sintered in an argon atmosphere at 900 °C for 1 hour.

2.2. Microstructure Analysis of Samples

After the sintering process, metallographic processes were applied in to obtain images from the test samples with the scanning electron microscope (SEM). These applied metallographic processes were applied as sanding, polishing, and etching respectively. The sanding process was applied to the surfaces of the samples with 200, 400, 600, 800, 1000, and 1200 mesh sanders respectively. Then the surfaces of the samples were polished with 3 and 1 μ diamond suspensions respectively. Finally, the reagent below is approximately 10 seconds. They were subjected to the etching process by the dipping method. Etching solution was used 5 ml Fe_3Cl , 20 ml HCl, 100 ml H_2O .

Scanning electron microscope (SEM) images of the samples subjected to the etching process were taken from the "FEI QUANTA 250 FEG" brand device located in the Central Research Laboratories of Kastamonu University. XRD measurements of the produced samples were made with a Bruker D8 Advance brand device. The measurements were made at Kastamonu University Central Research Laboratories.

2.3. Microhardness Measurement of Samples

After sintering, after the metallographic process steps were applied to the test samples in order, the mechanical alloying time applied for the samples produced in this study and how the resulting hardness values were affected by the amount of reinforced WC was investigated as a mechanical property. The microhardness measurement method was used in the hardness measurements of the samples. These measurements were made by using SHIMADZU brand HMV-G21 model microhardness measuring device under $\text{HV}_{0.05}$ kg load in Kastamonu University Metallurgical and Materials Engineering Department Laboratory. Microhardness measurements were made by 50 gr. applying a load for 16 seconds under load.

3. Results and Discussion

3.1. SEM Analysis Results of Samples

SEM images of the samples numbered 1, 2, 3, and 4 produced are given in Figure 1. When the SEM images given in Figure 1 are examined, the CuNiSi structure used as a matrix can be seen clearly. WC powders used as reinforcement elements are similar to each other and generally have sharp angular and irregular geometry.

SEM images of the samples numbered 5, 6, 7, and 8 produced are given in Figure 2. SEM images of samples mechanically alloyed for 5 hours are given in Figure 1, and SEM images of samples mechanically alloyed for 10 hours are given in Figure 2. With the increase of the mechanical alloying time, an improvement in the mechanical properties was detected.

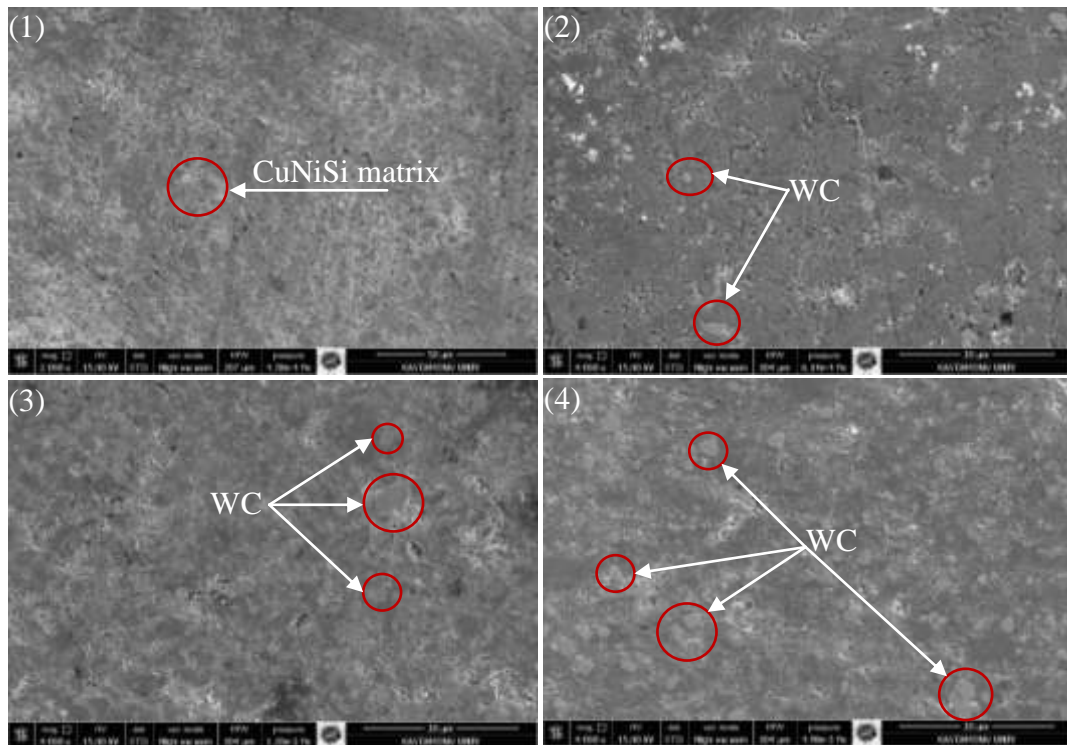


Figure 1. SEM images of produced composites

It is thought that with the increase of mechanical alloying time, the powders are overloaded and the powder becomes extremely brittle, which has a positive effect on mechanical aspects [22]. When Figure 2 is examined, it is seen that the WC particles in the structure have decreased in size with the increase of mechanical alloying time and they are homogeneously distributed in the structure.

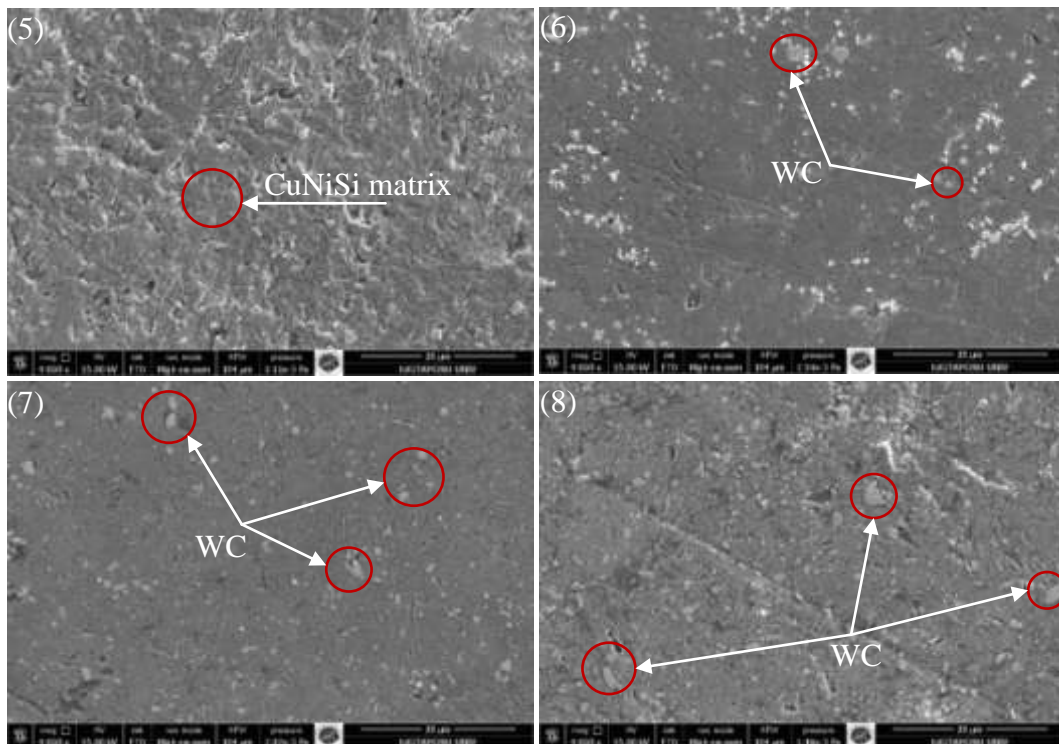


Figure 2. SEM images of produced composites

Homogeneously dispersed WC particles are thought to increase mechanical properties and this idea is supported by the microhardness measurement results. As a result, the sample containing 10% WC and mechanically alloyed by mixing for 10 hours showed the highest microhardness value.

3.2. SEM-EDS Analysis Results of Samples

SEM-EDS analyzes of samples numbered 1, 2, 3, and 4 produced are given in Figure 3.

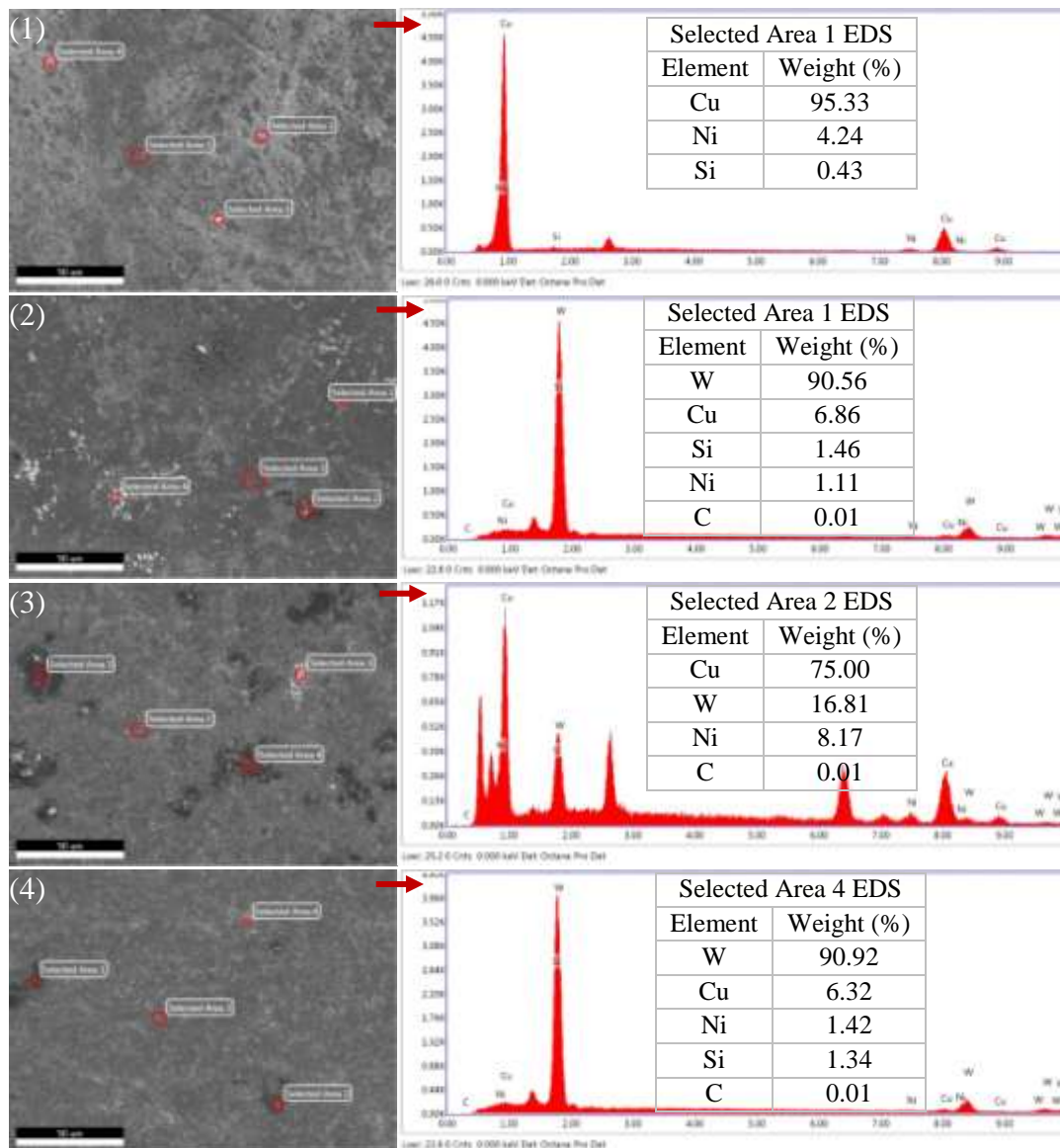


Figure 3. SEM-EDS analysis results of produced composites

The EDS analysis results are given in Figure 3 support the chemical composition of the samples produced. Different proportions of Cu, Ni, Si, W, and C were found in the internal structures of the produced samples. It is seen that the W and C peaks increased with increasing WC percentage (%).

SEM-EDS analysis of the samples numbered 5, 6, 7, and 8 is given in Figure 4.

When the EDS analysis results given in Figure 4 are examined, it supports the chemical composition of the samples produced. Different proportions of Cu, Ni, Si, W, and C were found in the internal structures of the produced samples. It is seen that the W and C peaks increased with increasing WC percentage (%).

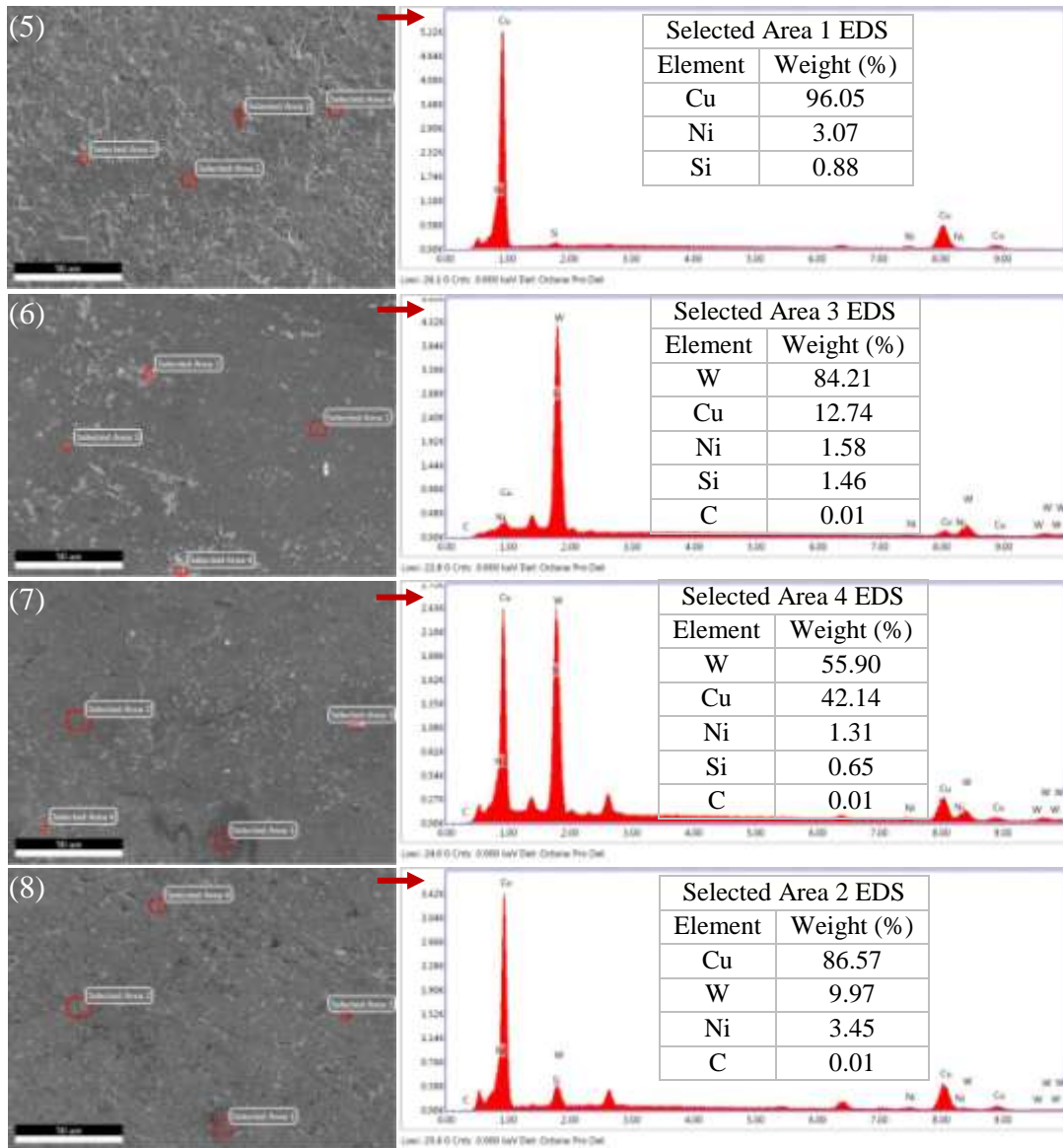


Figure 4. SEM-EDS analysis results of produced composites

Strength-enhancing mechanisms such as carbide particle size reduction, solid melt hardening, and dispersion hardening increase the hardness and wear resistance of materials produced with powder metallurgy. Özdemirler et al., (2017) and Erden et al. (2016) added NbC and AlC to the iron matrix in their studies. They detected NbC and AlC particles in SEM examinations. It was stated that the increase in mechanical properties was achieved by strength-enhancing mechanisms such as particle size reduction of carbides and nitrides detected in the matrix and at the grain boundaries, solid melt hardening, and dispersion hardening. Results Özdemirler et al. and Erden et al. show compatibility with his studies [23,24].

3.3. XRD Analysis Results of Samples

The XRD graphics of the samples numbered 1, 2, 3, and 4 produced by the powder metallurgy method are given in Figure 5.

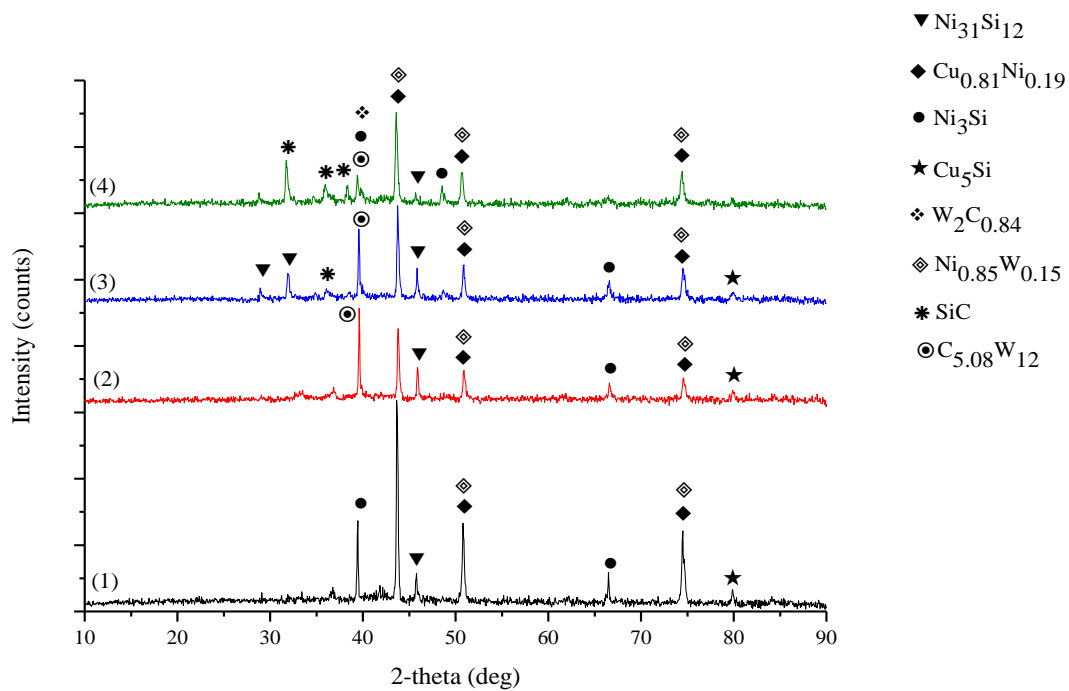


Figure 5. XRD graphs of produced composites

When the XRD graphs given in Figure 5 are examined, it was determined that $\text{Ni}_{31}\text{Si}_{12}$, $\text{Cu}_{0.81}\text{Ni}_{0.19}$, Ni_3Si , Cu_5Si , $\text{W}_2\text{C}_{0.84}$, $\text{Ni}_{0.85}\text{W}_{0.15}$, SiC , and $\text{C}_{5.08}\text{W}_{12}$ phases were formed. It is seen that the $\text{Ni}_{31}\text{Si}_{12}$ phase is dominant. During the sintering process, $\text{W}_2\text{C}_{0.84}$, $\text{Ni}_{0.85}\text{W}_{0.15}$, and SiC phases were formed between the CuNiSi and WC particles. In general, when the given XRD graphs are examined, it was found that the intensity of the $\text{Cu}_{0.81}\text{Ni}_{0.19}$, $\text{Ni}_{0.85}\text{W}_{0.15}$, $\text{C}_{5.08}\text{W}_{12}$, and SiC phases increased as the amount of additional WC increased.

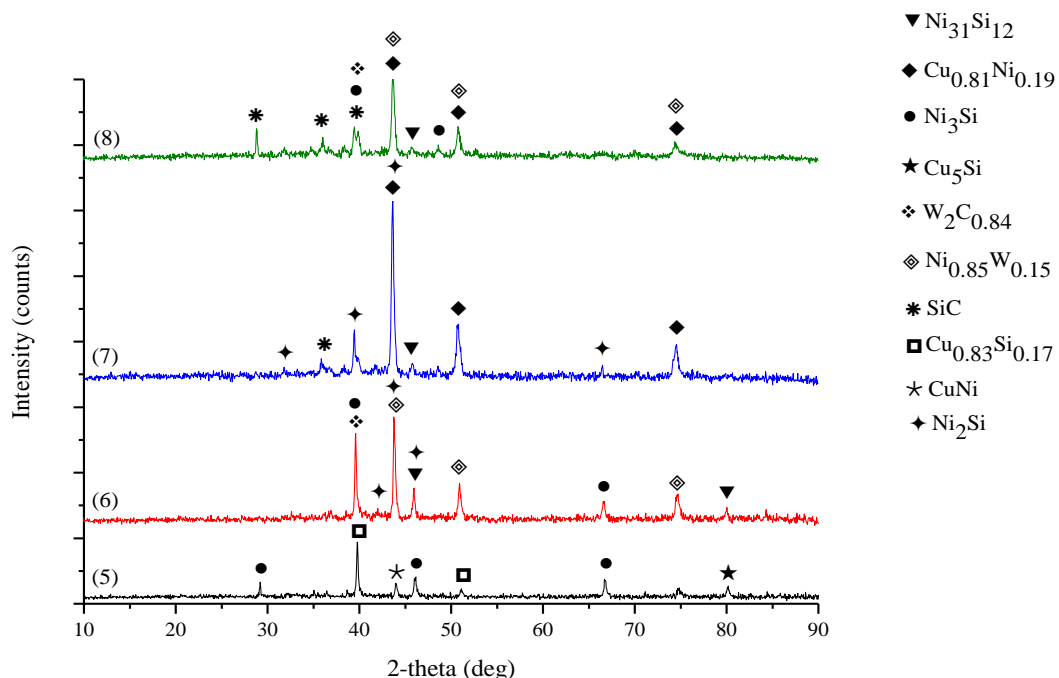


Figure 6. XRD graphs of produced composites

The XRD graphics of the samples numbered 5, 6, 7, and 8 produced by the powder metallurgy method are given in Figure 6.

XRD results show that $\text{Ni}_{31}\text{Si}_{12}$, $\text{Cu}_{0.81}\text{Ni}_{0.19}$, Ni_3Si , Cu_5Si , $\text{W}_2\text{C}_{0.84}$, $\text{Ni}_{0.85}\text{W}_{0.15}$, SiC , $\text{Cu}_{0.83}\text{Si}_{0.17}$, CuNi , Ni_2Si , and WC precipitates as seen in SEM dot and line EDS. It is proved by XRD whether these deposits formed in these structures are formed or not, and the results obtained from XRD analysis support the point and line EDS analysis results made in SEM examinations [25,26].

3.4. Microhardness Analysis Results of Samples

Microhardness graphics of the samples numbered 1, 2, 3, and 4 produced are given in Figure 7.

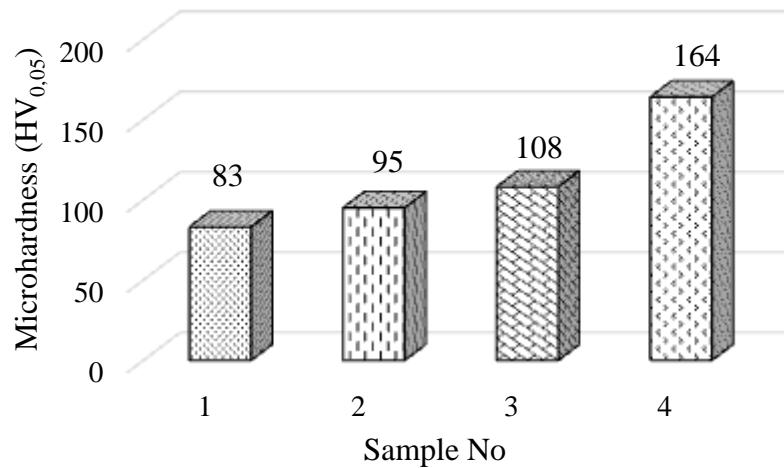


Figure 7. Microhardness graphics of produced composites

Microhardness graphics of the samples numbered 5, 6, 7, and 8 produced are given in Figure 8.

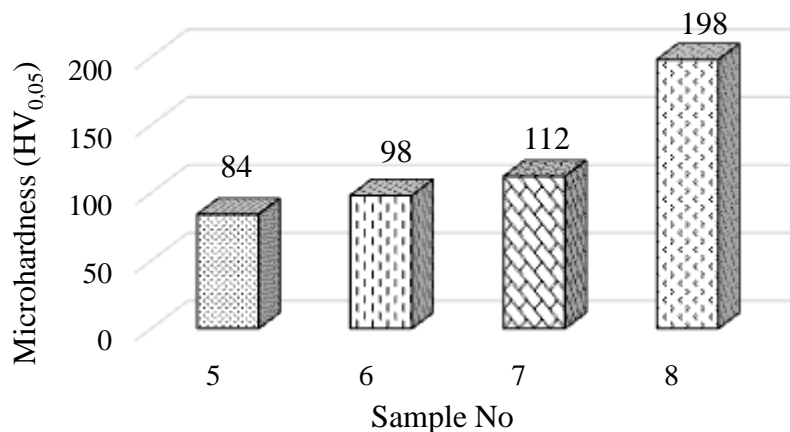


Figure 8. Microhardness graphics of produced composites

The hardness values of the samples produced by mechanical alloying are given in Figures 7 and 8. When the given hardness values are examined, it is seen that the mechanical alloying process increases the hardness values. The increase in the duration of the mechanical alloying process was also increased the hardness values [27-29]. The mechanical alloying process is thought to result from the formation of a homogeneous and fine microstructure with an intensive grinding application, continuous plastic deformation, fracture, cold welding, refracture and welding [30-32].

4. Conclusions

In this study, CuNiSi and CuNiSi-WC composites were successfully produced by applying the mechanical alloying process with the powder metallurgy method. Scanning electron microscopy

(SEM-EDS), X-ray diffractogram (XRD), and microhardness tests were successfully applied to the produced samples. The report of the experimental results can be summarized as follows.

It was reported that the WC particles were homogeneously distributed in the SEM images taken from the samples. In the EDS analysis results, the chemical compositions of the produced samples were supported and it was determined that the samples had different proportions of Cu, Ni, Si, W, and C in their internal structures. Peaks formed between Ni, Si, Cu, W, and C elements were determined in XRD graphs. As a result of the microhardness test, it was determined that the hardness of the samples with WC reinforcement was higher than the sample without reinforcement. The hardness values increased depending on the increasing WC amount and mechanical alloying time. The best microhardness value was measured as 198 HV_{0.05} in the sample to which 10% WC was added and mechanically alloyed for 10 hours.

Authors' Contributions

MA and KFB designed the study, carried out the experiments work and the theoretical calculations. Both authors read and approved the final manuscript.

Competing interests

The authors declare that they have no competing interests.

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