



Mehmet Akkaş

Kastamonu University, mehmetakkas@kastamonu.edu.tr, Kastamonu-Türkiye

Tarek Mousa K. Tabonah

Kastamonu University, tarekmousaktabonah@gmail.com, Kastamonu-Türkiye

Abdelsalam Mohamed A. Elfghi

Kastamonu University, elfghiabdelsalamhamed@gmail.com,
Kastamonu-Türkiye

Cihan Özorak

Kastamonu University, ozorak@kastamonu.edu.tr, Kastamonu-Türkiye

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ORCID ID	0000-0002-0359-4743	0000-0002-3899-5088
	0000-0003-1932-3072	0000-0003-3052-3024
Corresponding Author	Mehmet Akkaş	

POWDER METALLURGICAL FABRICATION OF CO REINFORCED CUNISI MATRIX COMPOSITES: MICROSTRUCTURAL AND CORROSION CHARACTERIZATION

ABSTRACT

Copper (Cu) and its alloys are used in various composite materials in different industries such as automotive, aviation, biomedical, and space, due to their superior properties such as high corrosion resistance, heat resistance, electrical properties, strength, and toughness. However, some difficulties are encountered in the use of Cu and its alloys in industry in terms of their mechanical properties. For this reason, it is observed that studies aimed at increasing the mechanical properties of CuNiSi alloy are limited in the literature. In this study, cobalt particles were added into the CuNiSi matrix at different rates and mixed homogeneously with a three-dimensional turbula for 3 hours. The results of experimental studies reveal that CuNiSi matrix composite materials can be successfully produced in terms of microstructure and mechanical properties. During the production phase of the samples, the powder metallurgy method was used by mixing Cu, Ni, Si, and Co powders in different chemical composition ratios. In this production process, argon atmosphere was used to prevent oxidation of the samples and for the synthesis process during sintering. After production, Scanning Electron Microscopy (SEM) analyses were applied for the characterization of the samples. Additionally, corrosion tests were carried out for corrosive characterization of the produced samples. As a result of the analyses, it was determined that the high melting temperature, high strength, and high mechanical properties of Co particles increased the microstructure and mechanical properties of the CuNiSi alloy. These findings show that CuNiSi matrix composite materials can be made more effective and durable in industrial applications by improving them with the addition of Co. The results of the potentiodynamic polarization experiments are presented and contain important data on the corrosion behavior of the samples with different cobalt reinforcement amounts. There is an increase in porosity values with the increase in the amount of cobalt reinforcement in the samples.

Keywords: CuNiSi composites, Co reinforced, Powder metallurgy, Microstructure, Corrosion

1. INTRODUCTION

Among the methods applied recently, CuNiSi alloys produced by powder metallurgy have emerged as an important research area. The TM

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method used in the production of these alloys allows precise control of the chemical composition [1, 2 and 3]. Powder metallurgy was developed as an alternative to traditional production methods and offers an alternative to techniques such as casting, machining, and hot and cold pressing. This method offers the potential to increase important properties of materials such as wear resistance, corrosion resistance, surface friction, and surface tension, especially through composites obtained by powder metallurgy. In engineering applications, composite materials play an important role in reducing production and operating costs by optimizing durability and weight ratios [4, 5 and 6].

CuNiSi alloys are very successful in terms of lightness and strength, and using these properties, composite materials with very good mechanical properties are produced [7]. CuNiSi and similar alloys stand out as high-engineering alloys in aerospace, automotive, and biomedical fields because they have superior properties such as high heat resistance, corrosion resistance, toughness, and strength. These features make the material preferred in a wide range of industrial applications [8]. In particular, they are used in many sectors such as machinery equipment, building materials, medical devices, vehicles, electronic devices, and spacecraft, and even find a place in products that make our daily lives easier, such as super-elastic eyeglass frames and telephone antennas. They have also been widely used in robotic applications in recent years. However, some difficulties are encountered in the use of CuNiSi and similar alloys in the industry regarding their mechanical properties and wear resistance [9, 10 and 11]. In this context, due to the low mechanical properties and wear resistance of the CuNiSi alloy, cobalt particles were added to this alloy and produced by the powder metallurgy method. The production of these reinforced alloy samples will open new and advanced areas of use for industry, along with increasing mechanical properties and wear resistance [12]. The results of this research show that reinforcements made to CuNiSi alloy, especially by the powder metallurgy method by adding cobalt particles, can provide a more effective use in industrial applications by increasing mechanical durability and wear resistance. These developments can be considered an example of progress, especially in the field of materials science and engineering [13, 14 and 15].

2. RESEARCH SIGNIFICANCE

This study aims to investigate the effect of the addition of cobalt particles to the CuNiSi alloy produced by the powder metallurgy method on microstructure and mechanical properties. In this context, the changes in the microstructure and mechanical properties of the CuNiSi alloy pressed at constant pressing pressure (300MPa) and sintered at constant temperature (950°C) were examined in detail. The results obtained indicate the potential success of these alloys in the production of composite materials with improved properties.

Highlights:

- Co-reinforced CuNiSi composite samples were successfully produced by the powder metallurgy method.
- As a result of SEM image analysis, it was determined that the decrease in the amount of pores varied depending on the cobalt addition rate.
- As the cobalt reinforcement ratio in the samples decreased, the Rp value increased. This increase causes the load transfer on the surface of the specimens to occur later, i.e. more difficult to corrode.



3. MATERIAL AND METHOD

Four types of mixtures were produced by the powder metallurgy method to be used in the production of composite materials. The properties of the powders used in the production phase of the samples were Cu, Ni, Si, and Co, with 99.9% purity and 1-44µm grain size. First of all, the CuNiSi powder mixture was prepared for experimental studies. The chemical composition of the prepared CuNiSi powder mixture is given in Table 1.

Table 1. Chemical composition of CuNiSi alloy

	Cu (at. %)	Ni (at. %)	Si (at. %)
Preparation of CuNiSi alloy	96.90	2.55	0.55

Later, in experimental studies, 0.5, 1, and 1.5% Co powder was added to the CuNiSi matrix and they were manufactured by the powder metallurgy method. The chemical compositions of the powders used in experimental studies were prepared by weighing them with a precision scale according to the values given in Table 2.

Table 2. Chemical compositions of the produced test samples

Sample No	CuNiSi (at. %)	Co (at. %)
1	99.50	0.50
2	99	1
3	98	2

To homogeneously mix the powders whose chemical composition ratios are given in Table 1 above, the mixing process was carried out for 3 hours in a three-dimensional turbula. After this process, a pressing process was applied to the samples whose chemical composition was adjusted. The pressing pressure was made under 300 MPa. The Sintering process was applied to the pressed samples. The sintering process was carried out in a tube-type furnace under an Argon atmosphere for 180 minutes and at a temperature of 950°C. After the sintering process, sanding, polishing, and etching processes were applied to the samples for characterization processes. Corrosion measurement analyses were performed to determine mechanical properties depending on the increasing Co content in the produced composite samples.

Corrosion tests were carried out using the Reference 3000 Potentiostat/Galvanostat/ZRA device in the Mechanical Engineering Research Laboratory of our university. Before starting the corrosion tests, carefully prepared specimens were cleaned and brought to optimal test conditions. The samples were ultrasonically cleaned with acetone at 35°C for 15 minutes, followed by distilled water for 15 minutes and finally ethanol for 15 minutes, and then dried in an oven at 60°C for 45 minutes. A 1M HCL solution was prepared for corrosion experiments. In this solution, the open circuit potentials of the cleaned samples were measured for about 30 minutes. In this process, all samples showed their corrosion potential by interacting with the solution. Potentiodynamic polarization experiments and Electrochemical Impedance Spectroscopy (EIS) measurements were carried out to analyze the corrosion behavior of the samples in more detail. Three experiments were performed for each sample and the samples were cleaned and a new solution was used in each experiment. The results were obtained by taking the arithmetic mean of the data obtained. According to the ASTM-G102 standard, corrosion current density, corrosion potential, corrosion rate, and polarization resistance were calculated using the curves read from the device. This standard provides an important reference point for the data to provide reliable and reproducible results. These comprehensive analyses and

measurements provide a solid foundation for an in-depth understanding of the corrosion behavior of CuNiSi composite samples.

4. FINDINGS AND DISCUSSIONS

4.1. Scanning Electron Microscopy Analyzes

To examine in detail CuNiSi composite samples reinforced with different amounts of Cobalt (Co), scanning electron microscopy (SEM) analyses of the samples produced using the powder metallurgy method were carried out. These analyses reveal the morphological and structural changes within the matrix of Co dopant added to the CuNiSi matrix (Figure 1).

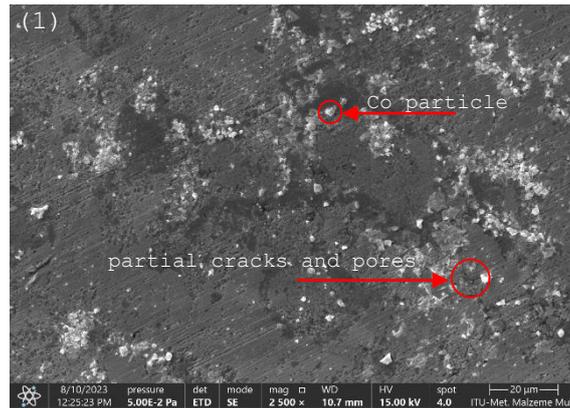


Figure 1. SEM micrograph of 0.5% Co reinforced CuNiSi composite

Figure 1 visually expresses the microstructure of samples with different Co ratios. With Co supplementation, a significant evolution in the microstructure was observed. Co particles were observed to be homogeneously distributed within the matrix, highlighting the diversity in matrix-matrix interactions and phase balance within the matrix. The obtained data show that Co supplementation affects the crystal structure within the matrix and this reinforcement provides a homogeneous distribution. This indicates the potential to increase the mechanical strength and toughness of the material. Moreover, SEM analyses showed that Co reinforcement formed a regular morphology on the matrix surface. This may provide an advantage for long-lasting use by increasing the wear resistance of the material. Evaluations show that Co reinforcement can positively affect the overall performance of the material by affecting the microstructural and morphological properties of CuNiSi matrix composite materials. These findings emphasize that higher performance can be achieved by modifying materials used in industrial applications with special reinforcements [16, 17 and 18].

The main structure of the CuNiSi compound is observed in the SEM images presented in Figure 1. It is observed that the Co particles added as reinforcement particles at different rates into this main structure are distributed homogeneously. This emphasizes the importance of matrix-matrix interactions in the internal structure of the produced samples and the uniform integration of reinforcement particles. Examination of the SEM images in Figure 1 shows that partial cracks and pores were detected in the fabricated samples. These observations suggest that the interactions of the reinforcement particles within the matrix, the details of the sintering process, and the stress distribution within the material are important. This may have a potential impact on the mechanical properties of the material. Furthermore, examination of SEM images reveals that the amount of pores varies depending on the Co addition rate. This suggests that the addition of Co can increase the

density of the material by reducing the voids in the matrix and thus increase the mechanical strength. It has been determined that the Co material distributed homogeneously in the main structure depends on the correct mixing of the powders during the production phase and the proper performance of the sintering process. This shows that a uniform distribution of reinforcement particles within the matrix is a critical factor in achieving the desired properties of the composite material. This determined situation is supported by existing studies in the literature, which emphasize the necessity of a careful production process to control the microstructural properties of the produced CuNiSi composite materials [19].

4.2. SEM Mapping Analysis

Scanning Electron Microscope (SEM) mapping images obtained from CuNiSiCo composites allow us to gain a more comprehensive understanding of the properties of your material. These images, especially those presented in Figure 2, provide important information about the microstructure and chemical composition of CuNiSiCo composites. SEM mapping images visualize details on the material's surface and distribution among components, providing researchers with critical insights into the structural properties of the material. For example, factors such as density, particle size and distribution of components in a particular region can have a direct impact on the performance and functionality of the material. The SEM mapping images in Figure 2 offer the opportunity to examine the internal structure of CuNiSiCo composites in detail. This investigation provides a deeper understanding of important properties of the material, such as its mechanical strength, thermal conductivity, and electrical properties. Additionally, the distribution of specific chemical components can provide guidelines for optimization and control processes in the material's manufacturing process.

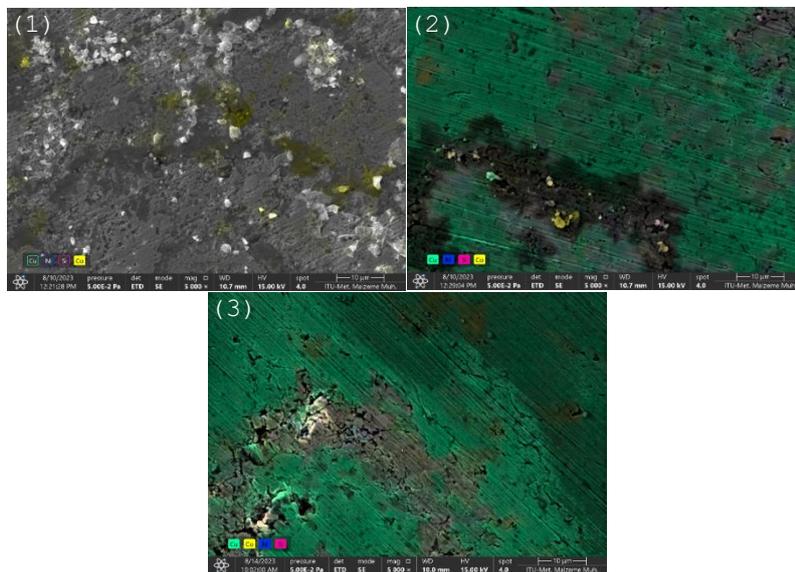


Figure 2. Mapping marked in the SEM micrographs for CuNiSiCo composites

The mapping analysis outcomes derived from the CuNiSiCo composites, as vividly depicted in Figure 2, not only offer essential insights into the composition and structure of the material but also pave the way for a more nuanced understanding of its inherent characteristics. The meticulous examination distinctly points out that

the composite is exclusively comprised of CuNiSiCo, exhibiting a noteworthy absence of any other detectable elements. This revelation carries significant implications for comprehending the purity and homogeneity of the material. The absence of detectable foreign elements underscores the precision and accuracy of the manufacturing process, attesting to the high quality and integrity of the CuNiSiCo composite. This finding is of paramount importance, especially in applications where material purity is a critical factor, such as in electronic components or advanced engineering materials. Furthermore, the mapping analysis goes beyond mere compositional confirmation, delving into the structural intricacies of the CuNiSiCo composite. It unveils the spatial distribution of Cu, Ni, and Si within the material, shedding light on the microstructure and interfacial relationships. This level of detail is invaluable for researchers and engineers seeking to optimize the material's properties for specific applications. In conclusion, the mapping analysis results presented in Figure 2 not only confirm the exclusive presence of CuNiSiCo within the composite but also provide a foundation for a more comprehensive exploration of its compositional and structural attributes. These insights contribute to the broader understanding of the material's properties and open avenues for further research and development, ultimately influencing the optimization and application of CuNiSiCo composites in various technological domains [19].

4.3. Potentiodynamic Polarization Experiments

The samples were prepared by adding different proportions of cobalt (0.5%, 1%, 2%) to the CuNiSi matrix. These different cobalt additions were evaluated by potentiodynamic polarization experiments to determine their effects on the corrosion behavior of the samples. These experiments were carried out to investigate the corrosion resistance of the samples in 1 M HCl solution and to understand the effect of cobalt addition on this resistance. The E-log curves presented in Figure 2 show the potentiodynamic polarization data obtained for three different samples in 1 M HCl. These curves represent the corrosion behavior of samples with various cobalt addition rates. Using the Tafel extrapolation method, i_{corr} values were determined by analyzing the E-log data obtained from these curves. The i_{corr} values obtained quantitatively express the effects of cobalt addition on corrosion resistance. Changes in i_{corr} values were observed with increasing cobalt addition rate. These values provide important information on how cobalt addition affects the corrosion rate of the samples.

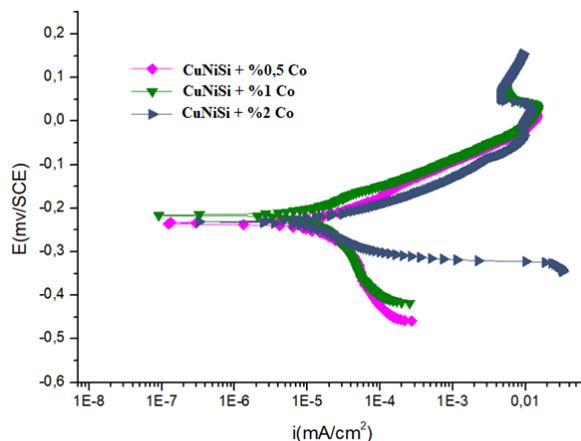


Figure 2. Potentiodynamic polarization curves of Co reinforced CuNiSi composites

The results of the potentiodynamic polarization experiments are presented in detail in Table 3. This table contains important data on the corrosion behavior of the samples with different cobalt reinforcement amounts. With the reinforcement rate increasing to 2%; It has been determined that it increases approximately 30 times according to a 0.5% reinforcement rate and approximately 6 times compared to a 1% reinforcement rate. In addition, the effects on the porosity values of the samples are also observed in this table. There is an increase in porosity values with the increase in the amount of cobalt reinforcement in the samples. This shows that the addition of cobalt affects the structural properties in the matrix and increases the porosity. When the data in Table 3 and the polarization curves in Figure 2 are examined, it is observed that the corrosion rate of the sample with lower porosity is quite low, but the corrosion rate increases with increasing reinforcement amount. These observations emphasize the effects of porosity on corrosion resistance. When the porosity is low, the matrix structure is tighter, resulting in less corrosion damage to the material. However, an increase in porosity with an increase in the amount of reinforcement can reduce corrosion resistance. This suggests that cobalt reinforcement in the matrix is one of the factors that determine the corrosion resistance of the material. These findings help us to understand the complex effects of cobalt reinforcement on corrosion resistance. This information can be used in design processes to optimize CuNiSi composite samples for specific application areas and to improve the durability of the material [20]. When the current potential curves obtained in 1M HCl solution are examined, increases in both anodic and cathodic regions and current values of the samples are observed. Although there is a positive shift in the corrosion potentials (E_{corr}), this shift is not evident for all samples.

Table 3. The results of potentiodynamic polarization experiments of Co reinforced CuNiSi composites

	CuNiSi+%0.5Co	CuNiSi+%1Co	CuNiSi+%2Co
E_{corr} (mV)	-234	-216	-231
I_{corr} (μ A)	5.1	24.7	142
Corr. Rate (mpy)	1.045	5.061	29.06

4.4. Electrochemical Impedance Spectroscopy Measurements

The corrosion electrochemical behavior of three different samples in 1M HCl solution was investigated by EIS. Nyquist diagrams of cobalt-reinforced samples are presented in Figure 3.

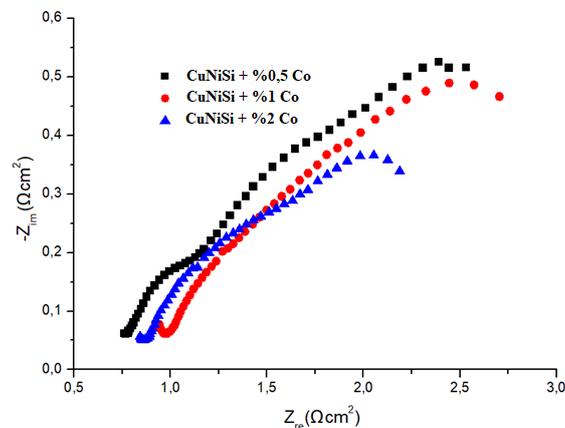


Figure 3. Nyquist diagrams of Co reinforced CuNiSi composites

The Nyquist diagrams obtained show a capacitive loop resulting from reactions in solution. This capacitive loop is associated with the charge transfer process that controls the protective film layer formed between the surface of the sample and the 1 M HCl solution.

A decrease in the diameter of the impedance spectra was observed with an increase in the reinforcement ratio of the samples, while an increase in the diameter of the impedance spectrum was observed with a decrease in the reinforcement amount. According to the experimental data, it appears that roughness and porosity on the composite surface have negative effects on the composite surface. The Nyquist diagrams were analyzed using Framework Data Acquisition Software and the equivalent circuit for the produced specimens was defined as follows (Figure 4).

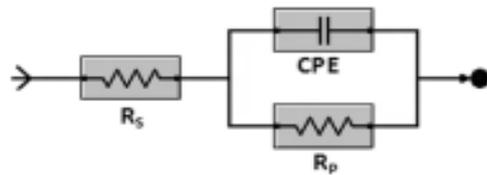


Figure 4. The electrochemical equivalent circuit used for the composite/solution interface of impedance spectra

The equivalent circuit consists of a Solution Resistor (R_s), a Polarization Resistor (R_p), and a Constant Phase Element (CPE) connected in parallel. The R_p values represent the measured load transfer resistance in the medium and these values are given in Table 4.

Table 4. The results of potentiodynamic polarization experiments of Co reinforced CuNiSi composites

	CuNiSi+%0.5Co	CuNiSi+%1Co	CuNiSi+%2Co
R_p ($\Omega \times \text{cm}^2$)	1.119	0.994	0.814

Examination of Table 4 reveals that the fabricated samples show a decreasing trend in polarization resistance with increasing Co reinforcement. The specimens with the highest r_p values are the specimens with lower cobalt reinforcement ratio and the r_p value is $1.119 \Omega \text{cm}^2$. As the cobalt reinforcement ratios in the samples increased, decreases in R_p values were observed and the lowest R_p value was determined as $0.814 \Omega \text{cm}^2$ in the sample containing 2% cobalt. On the contrary, as the cobalt reinforcement ratio in the samples decreased, the R_p value increased. This increase causes the load transfer on the surface of the specimens to occur later, i.e. more difficult to corrode [21].

5. CONCLUSION AND RECOMMENDATIONS

In this study, CuNiSi matrix composites produced by powder metallurgy method by adding Co at different rates were manufactured. As experimental parameters; A pressing pressure of 300MPa, a sintering temperature of 950°C , and a sintering time of 120 minutes were applied. SEM, SEM-Mapping, and corrosion tests were performed on these composite samples. Co-reinforced CuNiSi composite samples were successfully produced by the powder metallurgy method. As a result of SEM image analysis, it was determined that the decrease in the amount of pores varied depending on the cobalt addition rate. As the cobalt reinforcement ratio in the samples decreased, the R_p value increased. This increase causes the load transfer on the surface of the specimens to occur later, i.e. more difficult to corrode.



CONFLICT OF INTEREST

The authors have no conflicts of interest to be disclosed.

FINANCIAL DISCLOSURE

The authors declare that this study has received no financial support.

DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods used in this study do not require an ethical committee.

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