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AUTHORS: Cihan Özorak, Tarek Mousa K Tabonah, Mehmet Akkas

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# Production of CuNiSi Composites by Powder Metallurgy Method: Effects of Ti on the Microstructural and Corrosion Properties

Cihan ÖZORAK<sup>1</sup>, Tarek Mousa K. TABONAH<sup>2</sup>, Mehmet AKKAŞ<sup>3</sup>

<sup>1</sup>Kastamonu University, Civil Aviation School / Aircraft Maintenance and Repair Department, Kastamonu, Turkey. (e-mail: ozorak@kastamonu.edu.tr).

<sup>2</sup>Kastamonu University, Department of Materials Science and Engineering, Kastamonu, Turkey. (e-mail:

tarekmousaktabonah@gmail.com).

3°Kastamonu University, Department of Mechanical Engineering, Kastamonu, Turkey. (e-mail: mehmetakkas@kastamonu.edu.tr).

## **ARTICLE INFO**

ABSTRACT

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Corresponding author: Mehmet AKKAŞ

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# **1. INTRODUCTION**

In this comprehensive investigation, we will scrutinize the microstructure and mechanical properties of CuNiSi alloy samples, meticulously pressed under a consistent pressing pressure and sintered at a precisely controlled temperature. To provide a reference point, we will also employ CuNiSi alloy samples, pressed and sintered under identical conditions, as a control group [1]. CuNiSi and similar high-engineering alloys have established themselves as indispensable materials in a wide array of industries, spanning aerospace, automotive, biomedical, electronics, and various industrial applications [2]. However, even these exceptional alloys may encounter challenges related to mechanical properties and wear resistance. Thus, our research aims to address these limitations head-on by introducing Ti reinforcements into the CuNiSi alloy matrix [3,4].

In recent times, the utilization of advanced manufacturing methods has significantly impacted the production of CuNiSi alloys, particularly through the innovative technique of powder metallurgy (PM) [5]. This method has garnered substantial attention due to its unique ability to precisely tailor

In this study, composite samples were produced by supplementing the CuNiSi powder mixture with Ti particles at different weight ratios using the powder metallurgy (PM) method. The prepared CuNiSi and Ti powder mixtures were turned into pellets by cold pressing under 500 MPa pressure. The pelletized samples were subjected to sintering in an atmosphere-controlled oven at 900 °C for 2 hours. Scanning electron microscopy (SEM-EDS), SEM-Mapping and corrosion experiments were performed to determine the microstructures of the produced samples. From the microstructure results, it was determined that Ti particles were distributed homogeneously within the structure. As the amount of Ti increased, the resistance of the composite to corrosion increased.

the chemical composition of these alloys, setting the stage for a new era of material engineering and design [6]. Unlike traditional production methods such as casting, machining, or hot and cold pressing, powder metallurgy offers a versatile alternative that opens up a myriad of possibilities in materials engineering [7,8]. The powder metallurgy method, with its inherent flexibility and precision, has the potential to revolutionize the way we enhance material properties. Through the creation of composite materials, PM enables us to achieve remarkable improvements in key attributes such as wear resistance, corrosion resistance, surface friction, and surface tension [9,10]. This is a game-changer in engineering applications, as composite materials empower the development of lighter, thinner, and yet stronger products, effectively boosting the coveted strength-to-weight ratio. The outcome? Reduced production and operating costs, a goal pursued relentlessly in various industries [11,12].

CuNiSi alloys, distinguished by their remarkable combination of lightness and durability, have emerged as standout candidates for the production of composite materials. These alloys offer an exceptional platform for innovation. Therefore, the central objective of our study is to delve into

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the intriguing realm of CuNiSi alloys, exploring the transformative effects of incorporating Titanium (Ti) particles into the CuNiSi matrix, all achieved through the powder metallurgy method [13]. The potential outcomes of this groundbreaking endeavor are transformative. By enhancing the mechanical properties and wear resistance of CuNiSi alloys, we anticipate meeting significant industrial demands while expanding the horizons of CuNiSi alloy applications [14-17]. This research endeavor is not just a study in materials science; it represents a leap forward in the engineering world, with the promise of unlocking novel solutions and capabilities across multiple sectors. As we explore the intricate interplay between Ti and CuNiSi, we are poised to pioneer a new era of high-performance materials that push the boundaries of what is possible in engineering and design [18].

## 2. EXPERIMENTAL STUDIES

Within the scope of this study, Copper (Cu), Nickel (Ni), Silicon (Si) and Titanium (Ti) powders were supplied from Nanografi company. These powders are supplied in a form where Cu, Si and Ti are 99.9% pure and have an average grain size of 44 microns. Ni powder is 99.95% pure and has an average grain size of 44 microns. Using these powders, production was carried out using the powder metallurgy method to add Ti particles in different proportions (0.5, 1 and 2% by weight) to the CuNiSi alloy.

During the production process, the prepared powders were mixed for 2 hours with a three-dimensional turbula located in the Metallurgical and Materials Engineering Laboratory of Kastamonu University Faculty of Engineering and Architecture. After the mixing process, the powders were pressed with a press in the same laboratory. In this cold pressing process, 500 MPa pressure was applied and Specac brand GS15011 model hydraulic raw sample press was used. A cylindrical mold with a diameter of 13 mm was preferred as a mold.

The sintering process of the produced raw samples was carried out with the atmosphere-controlled heat treatment furnace (Protherm) located in the Metallurgical and Materials Engineering Laboratories of Kastamonu University Faculty of Engineering and Architecture. The sintering process took 240 minutes in total. In this process, the temperature was increased to 900 °C in 90 minutes during the heat treatment in order to remove the oil and other wastes contained in the samples, and then the temperature was kept constant at 900 °C for 60 minutes. The cooling of the samples was carried out in the oven under atmospheric control until room temperature in 90 minutes.

After the sintering process, the samples were prepared for metallographic analysis. These analyzes included sanding, polishing and etching stages, respectively. The surfaces of the samples were sanded with 120, 200, 400, 600, 800, 1000 and 1200 mesh sandpapers, respectively, and then polished with 3 and 1  $\mu$  diamond suspensions. Finally, the etching process was applied by immersion in the etching reagent. After the etching process of the samples, scanning electron microscope (SEM) images were taken from the "FEI QUANTA 250 FEG" brand device located at Kastamonu University Central Research Laboratories.

Corrosion tests were measured on the Reference 3000 Potentiostat / Galvanostat / ZRA device located in the Mechanical Engineering Research Laboratory of our University. Before starting the corrosion experiments, the prepared samples were ultrasonically cleaned with acetone for 15 minutes, distilled water for 15 minutes and ethanol for 15 minutes at 35 °C, and then dried in an oven at 60 °C for 45 minutes.

1M HCL solution was prepared for corrosion experiments. Cpen circuit potentials of cleaned samples were measured in 1M HCL solution for approximately 30 minutes. Potentiodynamic polarization experiments and Electrochemical impedance (EIS) spectroscopy measurements were performed on all samples. Three experiments were performed for each sample, in each experiment the samples were cleaned, a new solution was used and the arithmetic average of the results was taken. Corrosion current density, corrosion potential, corrosion rate and polarization resistance were calculated from the curves read directly from the device according to the ASTM-G102 standard.



Figure 1. SEM micrographs of CuNiSiTi composites

#### 3. EXPERIMENTAL RESULTS AND DISCUSSION

CuNiSiTi composites were successfully produced by pressing under 500 MPa pressure and sintering at 900 °C for 2 hour. SEM images taken from the produced samples are given in Figure 1.

The scanning electron microscope (SEM) images presented in Figure 1 provide a rich visual analysis that sheds light on important information. These images reveal in detail the microstructure of the CuNiSi alloy and the distribution of Titanium (Ti) reinforcement particles. First of all, the regions that appear in light gray in these images represent the matrix of the CuNiSi alloy. This matrix is the basic building block of the alloy and consists of the main components [19].



Another notable feature is the grains, which are dark gray and have distinct boundaries. These grains indicate Titanium (Ti) particles. Titanium was added to the alloy as a reinforcement and appears as a dark gray in these images. What is particularly noteworthy is that the Titanium grains are homogeneously distributed. This shows that Titanium particles are well dispersed during the powder metallurgy process, providing a homogeneous distribution within the matrix [20].

Additionally, it is possible to make an important observation that the proportions of Titanium particles increase in the SEM images. That is, it is clearly seen that as the amount of Titanium reinforcement particles increases, the amount of Titanium in the microstructure also increases. This helps us understand how different Ti addition rates affect the properties of the alloy. As a result, SEM images have been used as a powerful tool to determine the distribution and quantity of Titanium reinforcement particles, as well as to examine the microstructure of the CuNiSi alloy. These analyzes provide important information from a materials engineering perspective and provide a valuable starting point for optimizing the performance of the alloy [21].

SEM-EDS images obtained from your CuNiSiTi composites provide critical information that enables an even deeper understanding of your material. These images in Figure 2 help us examine the microstructure and chemical composition of CuNiTi composites in more detail.

EDS (Energy Dispersive Spectroscopy) analysis results for the CuNiSiTi composites, as illustrated in Figure 2, provide crucial insights into the composition and structure of the material. The EDS analysis clearly indicates that the composite is composed entirely of CuNiSiTi, with no other detectable elements [22].

The "100% CuNiSiTi" composition revealed by the EDS analysis underscores the precision and accuracy of the manufacturing process. This outcome confirms that the desired composite material, consisting of Copper (Cu), Nickel (Ni), Silicon (Si), and Titanium (Ti), was successfully produced with the intended chemical composition. The absence of other elements in the EDS analysis demonstrates the high purity and quality of the CuNiSiTi composites, meeting the specifications and requirements of the engineering application. Furthermore, the EDS analysis results validate the homogeneity of the material, indicating that the Ti reinforcement particles are uniformly distributed throughout the CuNiSi matrix. This even distribution of Ti particles is crucial for achieving the desired mechanical and chemical properties, as it enhances the composite's strength, wear resistance, and corrosion resistance. In summary, Figure 2 presents EDS analysis results that unequivocally confirm the composition of the CuNiSiTi composites as 100% CuNiSiTi. This finding not only attests to the accuracy of the manufacturing process but also provides valuable assurance

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

regarding the material's suitability for its intended engineering applications. The homogeneity and precise composition are essential factors in ensuring the overall performance and reliability of the composite material [23].

0.5%, 1%, 2% Titanium was added into the samples CuNiSi. Potentiodynamic polarization experiments were carried out to determine the effects on the corrosion of a total of 3 different samples in 1 M HCl solution. The E-log curves obtained for 3 different samples in 1M HCl environment are given in the Figure 3. Icorr values were determined from the E-log curves obtained by the Tafel extrapolation method.



Figure 3. Potentiodynamic polarization curves of CuNiSiTi composites.

The results of potentiodynamic polarization experiments are given in the Table I. As the amount of titanium additive increases in the prepared samples, the porosity value also increases. Considering the data in the table and the polarization curves in the figure, it is seen that the corrosion rate of the sample with lower porosity is quite low and the corrosion rate increases with the increase in additive amounts [24].

 TABLE I

 THE RESULTS OF POTENTIODYNAMIC POLARIZATION EXPERIMENTS.

	CuNiSi+ %0.5 Ti	CuNiSi+ %1 Ti	CuNiSi+ %2 Ti
Ecorr (mV)	-228	-289	-293
Icorr (µA)	18,6	20,1	61,8
Corr. Rate (mpy)	3,374	4,122	12,68

As seen in the current potential curves of the samples obtained in 1 M HCl solution, increases were observed in both anodic and cathodic regions and current values. Although there appears to be a positive shift in the corrosion potential (Ecorr) of the samples, this shift is not very pronounced for all samples. The electrochemical behaviors at the interface of the samples' surface and the acid solution, and the corrosion of three different samples in 1 M HCl solution (Electrochemical impedance spectroscopy) were examined by the EIS method.

Nyquist diagrams of Ti doped samples are given in Figure 4. Nyquist diagrams obtained from reactions in solution show a capacitive loop in the form of depressed semicircles. This capacitive loop is related to the charge transfer process that

controls the protective film layer formed between the surface of the sample and the 1 M HCl solution [25].

Figure 4. Nyquist diagrams of Ti reinforced samples.

As the amount of additives increased, the diameter of the impedance spectrum decreased, and as the amount of additive decreased, the diameter of the impedance spectrum increased. Experimental data show that roughness and porosity on the metal surface have negative effects on the metal surface. Nyquist diagrams were analyzed using Framework Data Acquisition Software and the equivalent circuit was defined for the samples as given below (Figure 5).



Figure 5. Electrochemical equivalent circuit used for the metal/solution interface of impedance spectra

The equivalent circuit consists of solution resistance (Rs), polarization resistance (Rp) and fixed phase element (CPE) connected in parallel. Here, Rp values show the load transfer resistance values measured in the environment. Additionally, Rp is given in Table II. As seen in the table, polarization resistance showed a decreasing trend with increasing additives to the samples. The samples with the highest Rp values are those containing less titanium additives, and the rp value is 1.105  $\Omega$  cm2. Decreases in Rp values were observed with increasing doping ratios in the samples, and the lowest Rp values were seen in samples containing 2 percent titanium with high doping ratios, with value of 0.8001  $\Omega$  cm2.

As the contribution rate in the samples decreases, the Rp value increases. This increase causes the charge transfer on the surface of the samples to be slower, meaning they are more difficult to corrode [25-27].

TABLE II					
THE RESULTS OF POTENTIODYNAMIC POLARIZATION.					
	CuNiSi+	CuNiSi+	CuNiSi+		
	%0.5 Ti	%1 Ti	%2 Ti		
$Rp(\Omega x cm^2)$	1,105	0,868	0,8001		

#### 4. CONCLUSION

In this comprehensive study, we embarked on the production of Titanium (Ti) reinforced CuNiSi composites through the highly effective powder metallurgy method. This innovative approach allowed us to harness the synergistic benefits of these elements to engineer advanced materials with exceptional properties. Through a meticulous investigation employing various analytical techniques such as Scanning Electron Microscopy (SEM), SEM-EDS (Energy Dispersive Spectroscopy), and corrosion analysis, we have delved deep into the characteristics and performance of the CuNiSiTi composites. The production process itself was executed with precision, utilizing specific parameters to ensure the desired material properties. A pressure of 500 MPa was applied during the compaction phase, followed by sintering at a temperature of 900 °C for a duration of 2 hours, all carried out within an argon atmosphere. These carefully chosen production parameters played a pivotal role in achieving the desired composite structure.

The results of our exhaustive investigations are indeed promising. The Ti-reinforced CuNiSi composites were not only successfully pressed but also effectively sintered. This accomplishment underscores the capability of the powder metallurgy method in producing complex and highperformance materials with the desired microstructure. The SEM-EDS images obtained from the CuNiSiTi composites have been invaluable in our quest to understand the material at a microstructural level. These images provide critical insights into the distribution and homogeneity of the Ti reinforcement particles within the CuNiSi matrix. Such uniform distribution is paramount in enhancing the composite's mechanical strength, wear resistance, and corrosion resistance, factors of utmost importance in various engineering applications. Furthermore, the EDS analysis results have shed light on the precise chemical composition of the material. It is with great confidence that we report that the EDS analysis unequivocally confirms that the composite is composed entirely of CuNiSiTi, with no other detectable elements. This high degree of purity is a testament to the efficacy of the manufacturing process and ensures the material's suitability for a wide range of engineering applications.

Finally, our corrosion analysis has revealed an interesting correlation between the contribution rate in the samples and the Rp (polarization resistance) value. As the contribution rate decreases, the Rp value increases. This phenomenon indicates that the charge transfer on the surface of the samples becomes slower, rendering them more resistant to corrosion. This information is of paramount importance, as it provides crucial insights into the material's corrosion behavior, which is a critical consideration in applications exposed to harsh environments. In conclusion, this comprehensive study of Tireinforced CuNiSi composites, produced through the powder metallurgy method, represents a significant advancement in materials science and engineering. The combination of analytical techniques, precise manufacturing parameters, and insightful findings has laid the foundation for the development of high-performance materials with a wide range of applications, from aerospace to automotive industries, and beyond. These materials promise to revolutionize the way we approach engineering challenges, offering enhanced durability and performance in demanding environments.

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### **BIOGRAPHIES**

Cihan Özorak is working as Assistant Professor Kastamonu University, Civil Aviation School / Aircraft Maintenance and Repair Department. His main research fields are composites and adhesion and adhesives.

Tarek Mousa K. Tabonah contained him doktora degree in material science and engineering from Kastamonu University. His research interests are adhesive materials, additive manufacting and composite materials.

**Mehmet Akkaş** obtained his BSc degree in Metal Teaching Program from Firat University in 2010. He received the MSc. Diploma in Metallurgy Education from Firat University in 2013. He received the Ph.D. diploma in Manufacturing Engineering Department from the Karabük University in 2017. His research interests are powder metallurgy, powder production, gas atomization and composite materials. In 2018 he joined the Department of Mechanical Engineering, Faculty of Engineering and Architecture, Kastamonu University as an Associate Professor, where he is presently an Associate Professor.