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DEVELOPMENT of Cu MATRIX COMPOSITE CONTACTORS REINFORCED by NICKEL COATED SiC

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Highlights

- 1 In this study, Cu-Ni SiC (Copper-Nickel coated SiC) composite samples were produced by electric current assisted sintering (ECAS) method
- 2 The matrix component Copper powder was successfully produced by cementation method.
- 3 SiC powders were coated with Ni by electroless plating method.

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ABSTRACT: In this study, Cu-Ni coated SiC composite samples were produced by electric current assisted sintering (ECAS) method by adding electroless nickel coated SiC powders to copper powders produced by cementation method at the ratios of 0.5, 1 and 1.5 wt%. The relative densities of the produced samples were measured by Archimedes' principle, their microstructures were examined by SEM-EDS, dominant phases were determined by XRD technique; microhardness and electrical conductivity measurements were made. The relative density of undoped copper was determined as 99.42% and the relative density value of Cu-Ni SiC composite samples decreased to 98.35% at most with increasing SiC ratio. The hardness values of Cu-Ni SiC composite samples increased from 120HV to 145HV with the addition of SiC; electrical conductivity values decreased from 90.41 IACS (International annealed copper standard) to 58.56 IACS.

Keywords: *Cu-Ni SiC, Copper Matrix Composite, Cementation, Relative Density, IACS*

1. INTRODUCTION

Copper is the 26th most abundant element on earth. The electrical conductivity of copper ranks 2nd after silver among all metals. It has an important place in the industry and has a wide range of uses because it makes alloys such as brass and bronze [1]–[3]. There are different copper production methods depending on the properties of the ore used as raw material. These methods are grouped as hydrometallurgy, pyrometallurgy and electrometallurgy.

In this study, the matrix material was produced by precipitating copper powder from copper sulphate solution by cementation method. Reduction and precipitation of copper from dilute sulfuric acid leach solutions with the help of iron is the oldest hydrometallurgical method. The process of reducing and precipitating a metal in its aqueous solution with the help of another metal without the aid of any electric current is called cementation. In the cementation method, the more noble metal ion in the solution is precipitated by adding a more basic metal to the solution in a neutral state. In this method, the metal reduced from the solution is called "cement" and the more basic metal added to the solution is called "cementator". The general reaction equation for this precipitation process is as follows :

$$mN^{n+} + nM = nM^{m+} + mN \quad (1)$$

In this equation, "N" indicates the more noble metal and "M" indicates the reducing metal. In copper cementation, Cu^{2+} ions are reduced by more basic metals such as iron, zinc, aluminum as a result of the electrochemical process. The reactions that take place in the cementation of copper with iron are as follows;

Semicellular reactions;

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$Cu^{2+} + 2e^- = Cu$ (E_o = +0.34 V) (2)

$Fe = Fe^{2+} + 2e^-$ (E_o = +0.44 V) (3)

Full reaction;

$Cu^{2+} + Fe = Fe^{2+} + Cu$ (E_o = +0.78 V) (4)

The reaction for copper sulphate solution is as follows;

$CuSO_4+Fe=FeSO_4+Cu$(E_o=+0.78V) (5)

In addition to these, there are also side reactions that can occur in the solution, which can be shown as the main factors that reduce the efficiency of the cementation process. Solutions used on an industrial scale may contain Fe^{2+} and Fe^{3+} ions. Another side reaction that will occur in this case;

$Fe^{3+} + e^- = Fe^{2+}$ (E_o = +0.771 V) (6)

The other reaction that may occur is as follows;

$Fe + Fe_2 (SO_4)_3 = 3FeSO_4$ (E_o = +1.211 V) (7)

The reverse reaction that can occur with reduced copper is as follows;

$Cu + Fe_2 (SO_4)_3 = 2FeSO_4 + CuSO_4$ (E_o = +1.211 V) (8)

Since waste copper solutions usually contain sulfuric acid, another reaction that can occur during the cementation of copper with iron is as follows;

$Fe + H_2 SO_4 = FeSO_4 + H_2$ (9)

Copper and its alloys are very important engineering materials to date due to their high corrosion resistance, excellent electrical and thermal conductivity, attractive appearance, high ductility and ease of forming. They have the best electrical conductivity after silver and very high thermal conductivity between silver and gold [4]. However, due to their high cost, silver and gold are rarely used in conductive applications. In terms of bulk applications, the most commonly used conductive materials are aluminium and copper. Copper is preferred mainly because of its higher conductivity and higher strength compared to aluminium [5], [6]. A significant disadvantage of copper is its strength of 390 MPa. This is because pure copper, even if hardened by cold forming, recrystallizes at temperatures close to 100°C and therefore loses its strength quickly. Adequate electrical conductivity and high strength and hardness compared to pure copper can be obtained by alloying pure copper. However, since it is not desirable to reduce the electrical conductivity too much, the alloying elements to be added to pure copper

are only 2%, and in many applications in smaller amounts. The disadvantage of low- alloyed copper used in conductivity applications is the rapid loss of properties due to the coarsening of precipitate particles at high temperature. In order to solve this problem, copper matrix metal matrix composites reinforced with ceramics such as oxide, boride and carbide have been developed due to the stability of ceramic particles at high temperatures [3]. In this study, in order to increase the strength of the copper matrix, it is aimed to reinforce it with SiC due to its high elastic modulus and high thermal conductivity. In order to avoid wetting problems at the Cu and SiC interface, SiC particles were coated with Nickel by electroless plating method.

Electroless nickel plating is a pretreatment technique especially for materials with high surface resistance [7]. Electroless nickel plating is the more popular variation of electroless plating with some different properties [8]. Electroless nickel plating technique is based on the conversion of nickel ions into nickel metal by reducing the effect of nickel ions on the catalytic material surface. It was first developed by Brenner and Riddel in 1946. A reductant, sodium hypophosphite, added by the researchers to remove unwanted oxidation products from electrolytic nickel plating baths, led to nickel deposition and thus a very important industrial metal plating method was permanently discovered. The major advantage of electrolytic nickel plating is that it provides a uniform coating thickness so that it can be easily coated on uneven surfaces and in any contact with the solution [9]. It is used in almost every field. It has a wide range of applications from simple knitting needles to aerospace applications. The most important feature of electroless nickel coatings is wear resistance and corrosion resistance [8], [9].

Cu-SiC composites combine both the superior ductility and toughness of copper and the high strength and high modulus of SiC reinforcements. They can be used as electrical contact materials in relays, contactors, switches, circuit breakers and other switchgear components [10].

In this study, Cu-Ni SiC composite samples were obtained by ECAS method by using the powder mixtures of cemented copper and Ni coated SiC. The microstructure and morphologies of these samples were examined by SEM, phase structures were dedected by SEM-EDS and XRD analysis and their relative densities, hardnesses and electrical conductivities were measured and compared with each other.

2. MATERIAL AND METHODS

2.1. 1. Copper Powder Production

Anhydrous $CuSO_4$ (99% purity, Merck), H_2SO_4 (Sulfuric Acid) (99% purity), Pure Fe Powder (99% purity, 10 μm Merck) and Distilled Water were used to produce Cu powders by cementation. Copper powder was obtained by precipitation from 0.1 M $CuSO_4$ solution by cementation method. The equation for the cementation method is as in Equation 10.

$$CuSO_4 + Fe = FeSO_4 + Cu \quad (10)$$

The production flow chart of copper powder is given in Figure 1.

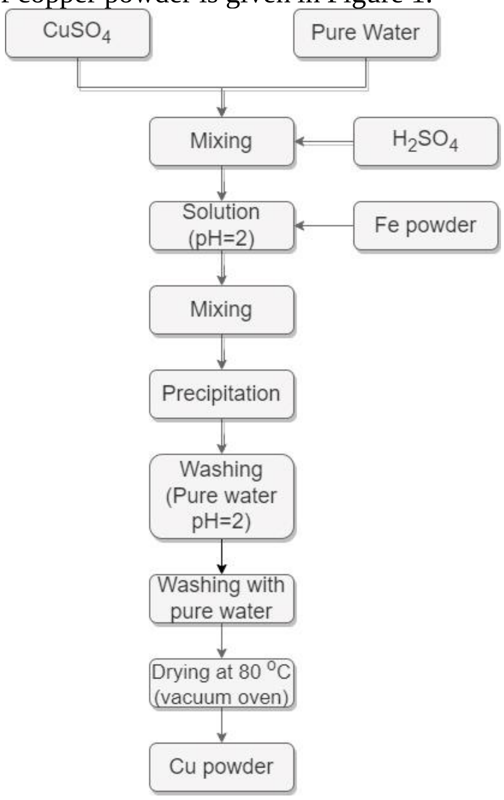


Figure 1. Schematic representation of the production of cemented copper powder

2.2. Nickel Plating of SiC

For electroless Ni plating of SiC powders; SiC powder (Struers 80-99.9% purity), $SnCl_2$ solution (Sigma-Aldrich, 99.9% purity), purified water, HCl Acid, Ammonia (NH3) (Sigma-Aldrich, 99% purity), Sodium stearate (Sigma-Aldrich, 99% purity), and nickel powder (Sigma-Aldrich 1μm) were used. The main purpose of electroless Ni coating on SiC is to increase the wetting between SiC and Cu. In order to perform this process, the solution prepared from the mixture of SiC, $SnCl_2$, H_2O, and HCl weighed in cytometric ratios was stirred with a magnetic stirrer at 1500 rpm for 1 hour. After filtration, activation, coating, washing and drying, Ni coating on SiC powders were obtained. The main objective of electroless nickel plating technique is to keep the coating thickness at minimum level and to maximize the wettability with the matrix material. The electroless Ni coating process is shown schematically in Figure 2.

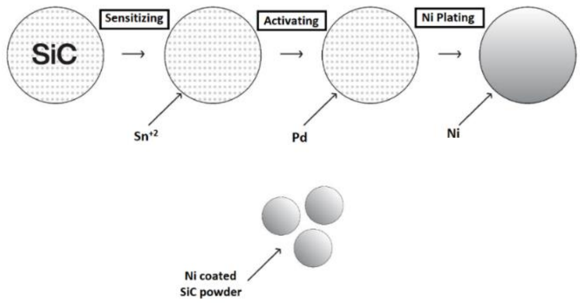


Figure 2. Schematic representation of Electroless Ni coating on SiC.

After sintering, the compacts were hot pressed at 400°C under 200 MPa pressure with a uniaxial chydraulic press. After pressing samples were grinded and metallographically prepared. The schematic representation of sintering by ECAS method is given in Fig. 3. The produced composite samples were grinded to 1200 grit sandpaper level and polished with 0.3 μm alumina in accordance with metallographic sample preparation methods. The polished samples were characterized by SEM-EDS, hardness and electrical conductivity methods.

2.3. Fabrication of Copper-Ni coated SiC Composites

Cemented copper powders and Ni coated SiC powders were homogeneously mixed as Cu-0,5-1-1,5wt.% Ni-SiC and pressed under 150 MPa pressure in a uniaxial hydraulic press to obtain compact samples. The compacts were embedded in graphite powder and placed in the mould in an ECAS process .

Using ECAS for sample consolidation results in shorter process and faster heating times, as well as lower sintering temperatures. To consolidate or synthesis the sample for required configuration and density, this technology applies an electric current while also applying mechanical pressure [11]–[14]. The compacts were sintered with an electric current in the range of 2200-2500 amperes for 2 minutes.

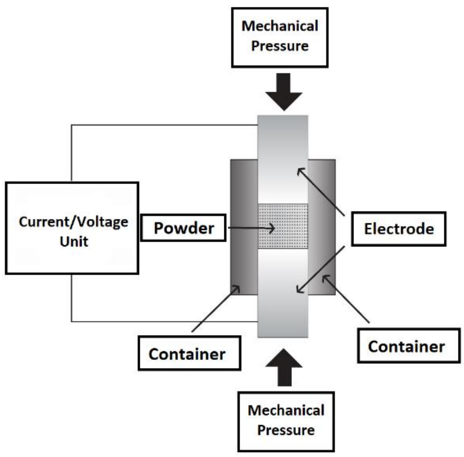


Figure 3. Schematic representation of sintering by ECAS Method [11], [15].

2.4. Characterization

Microstructural characterization and elemental examination of the phases of as-prepared composites were carried out by using SEM-EDS (JEOL LV6060, in HV with 20 kV)

Phase characterization and verification of EDS were realized by XRD analysis using Cu-K_α radiation with wavelength of 1.54 nm in a range of 20-90°.

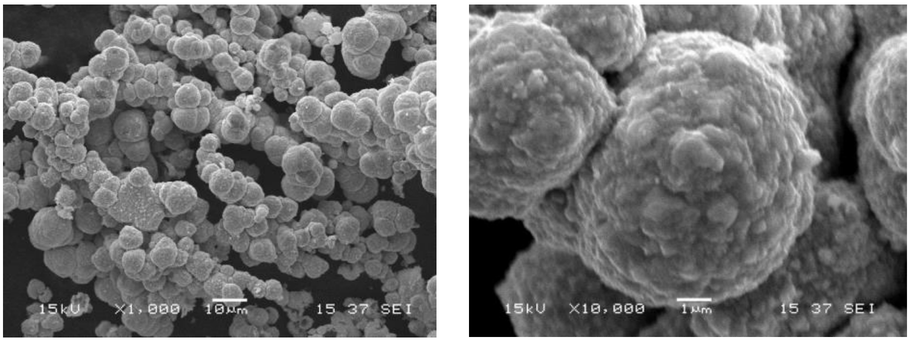
Hardness measurements of the samples are important in terms of providing information about the properties of the material such as strength and elastic modulus. Hardness values were determined by taking the arithmetic mean of 6 different measurements under 100 g load from metallographically prepared samples with an Emcotest DuroScan 70G5 model microhardness indenter.

Electrical conductivities of composites as well as cemented copper sample were measured by using Sigmatest D 2.068 model instrument.

Relative densities of composite samples were measured according to Archimedes method by taking of both dry and wet weights of each samples.

3. RESULTS AND DISCUSSION

SEM images of copper powders produced by cementation method at different magnifications are given in Figure 4.

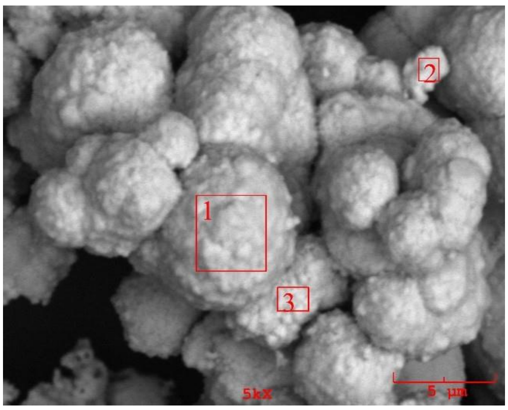


(a) (b)

Figure 4. SEM image of copper powder produced by cementation at different magnifications

SEM images show that the powders are about 5-10µm in size (Fig. 4a), but at higher magnification it is seen that real size of Cu powders are in submicron size and they agglomerated (Fig. 4b). It is thought that the first nucleated powders were submicron sized and then coalesced and agglomerated. The formed powders have a spherical morphology. G. Granata et al. reported that both temperature and surfactants have an important role on particle size [16]. R. Jhajharia et al. stated that the powders obtained in cemented copper production have polygonal morphology [17].

Figure 5 shows the SEM-EDS analyses of cemented copper powders. It is seen from the SEM image in the analysis that the powders are submicron in size. In addition, it was determined from the SEM-EDS analysis result that the powder grains are completely copper and contain trace amounts of oxygen.



Wt. %	1	2	3
O	0.142	1.561	0.416
Cu	99.858	98.439	99.584

Figure 5. SEM-EDS analysis of copper powders produced by cementation

XRD analysis of copper powders produced by cementation method is shown in Fig. 6. Characteristic peaks of copper and a very small peak of Cu_2O were obtained. It is thought that the trace amount of Cu_2O formation occurs during the drying stage of the cemented copper powder after production.

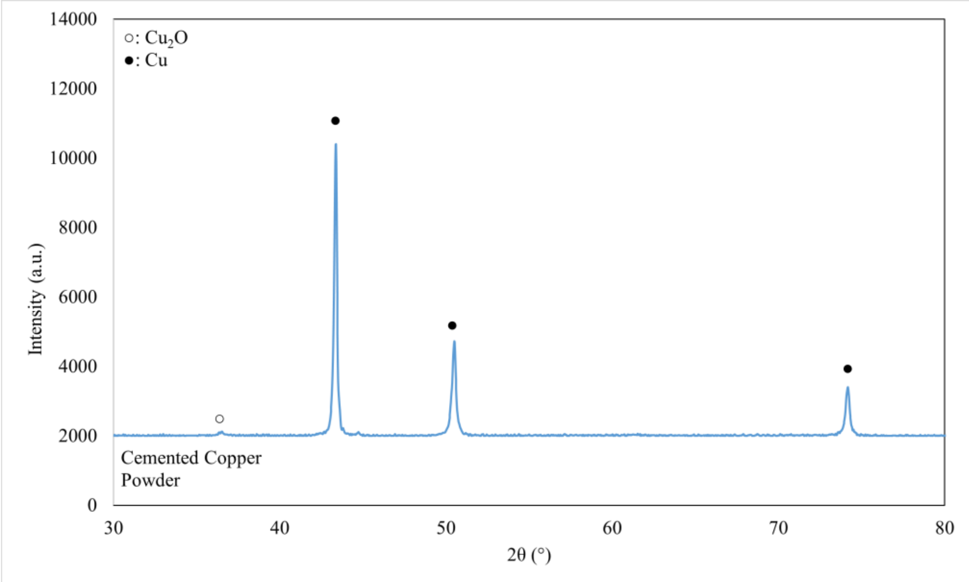
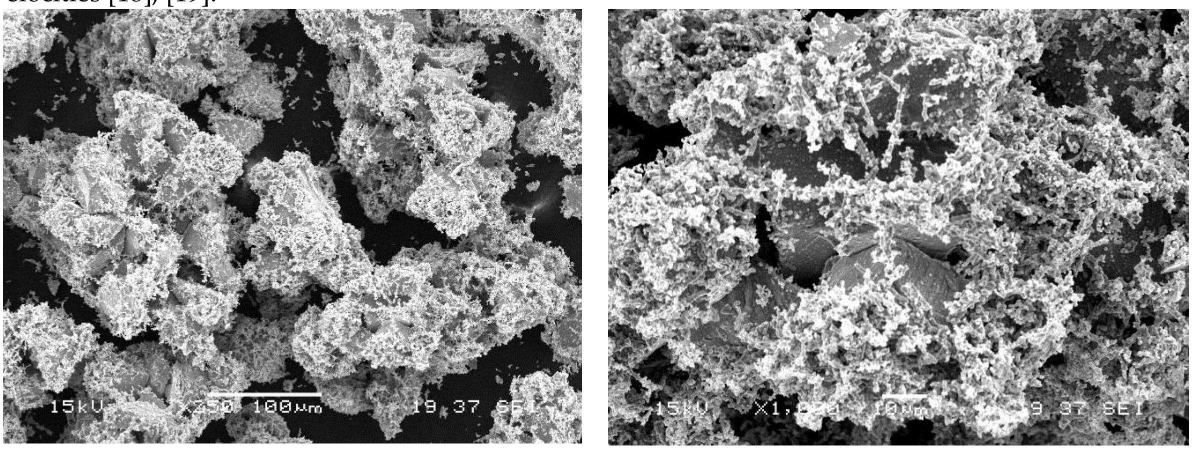


Figure 6. XRD graph of cemented copper powders.

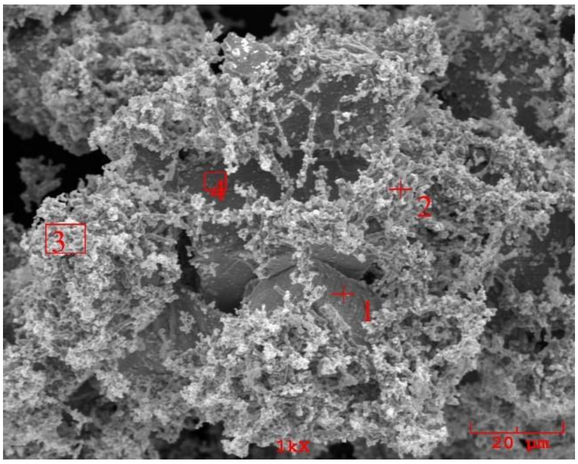
SEM micrographs of electroless nickel coated SiC powders at different magnifications are shown in Figure 7. SiC grains are dark grey and light grey/white flashes indicate nickel coating. It is seen that SiC grains are homogeneously coated with Ni. Ni coated SiC powder helps to reduce the Kirkendall voids between SiC-Cu when it is added to copper matrix. Nickel/Copper (Ni/Cu) interfaces are applied in various fields such as the fabrication of thermoelectric modules and soldering applications [17]. At Cu-Ni interfaces, it has been shown that nickel can inhibit dislocation slip and reduce effective dislocation velocities [18], [19].



(a) (b)

Figure 7. SEM images of electroless Ni coated SiC powders at different magnifications.

The SEM-EDS analysis result of Ni coated SiC powders is given in Figure 8. From the SEM image, it can be seen that Ni coating occurs by nucleating and stacking on top of each other. All the dots in the EDS analysis results taken from different points indicate Ni and prove that the electroless Ni coating process was successful.



Wt. %	1	2	3	4
C	1.516	2.130	0.580	0.235
Si	1.974	0.381	0.151	0.567
Ni	96.51	97.48	99.269	99.198

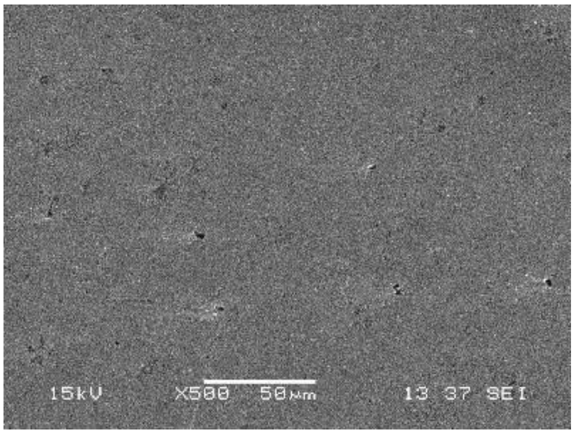
Figure. 8. EDS analysis of electroless Ni coated SiC powders.

After this section, the abbreviation Cu-Ni SiC is used when referring to Cu matrix composite specimens reinforced with Ni coated SiC sintered by ECAS method. The relative densities of the test specimens measured by the Archimedes method are given in Table 1 together with pure copper. Considering the results obtained, a relative density of more than 99% was obtained in pure copper. This value is higher than the values in the literature produced by cementation method. In addition, with the addition of Ni SiC, the relative density values of Cu-Ni SiC composite samples decreased slightly. This is thought to be due to the pre-coating of SiC particles with Ni. As a matter of fact, Cu-Ni surfaces came together instead of Cu-SiC. Since Cu and Ni atoms are fully soluble in each other, a good bonding was formed at the interface.

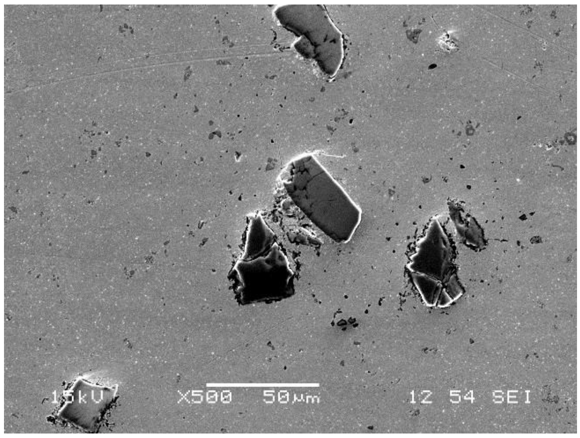
Table 1. Relative density values of cemented Cu and Cu- 0.5-1-1.5wt. % $_{Ni}$SiC composites

Wt. %SiC	Relative Density, %Number
0	99.42
0.5	98.60
1	98.45
1.5	98.35

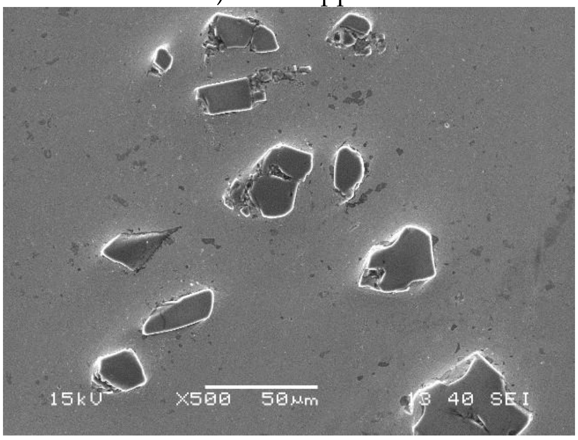
SEM micrographs of Cu-Ni SiC composite samples sintered by ECAS method at 700 °C for 2 minutes are given in Figure 9. In Figure 9, the Cu matrix is in grey color, and the reinforcement component is darker. As can be seen from the figures, Ni coated SiC particles, which are the reinforcement component, are relatively dispersed homogeneously in the matrix.



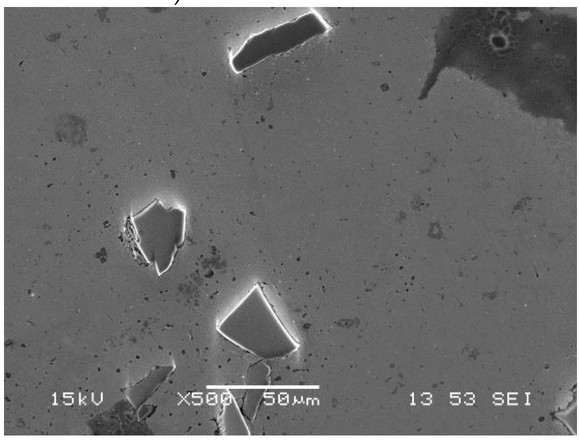
a)Pure Copper



b)Cu-wt. 0.5wt.%NiSiC



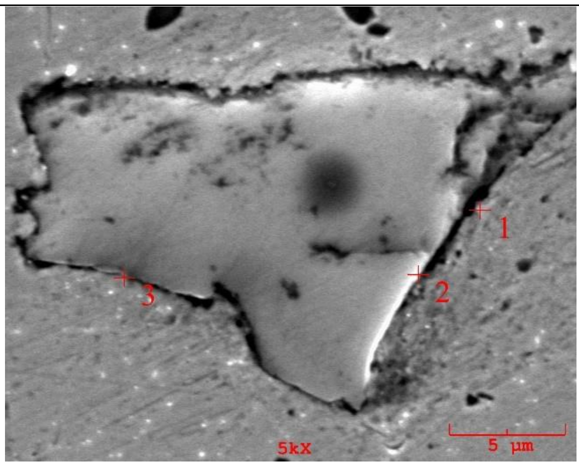
c) Cu-1wt.%NiSiC



d) Cu-1.5wt.%NiSiC

Figure. 9. SEM images of cemented Cu and Cu- 0.5-1-1.5 wt.% Ni SiC composites

SEM-EDS analysis of Cu-% 0.5wt.Ni SiC composite sintered at 700°C is given in Figure 10. The presence of Ni was detected in dot analyses obtained from Cu-SiC interfaces which shows that the Ni coating is successful.



Wt. %	1	2	3
C	1.124	12.057	9.342
O	2.705	2.906	-
Si	5.071	39.211	70.209
Fe	0.452	0.403	0.439
Ni	2.644	1.688	1.170
Cu	88.004	43.271	18.291
Here.	-	0.464	0.550

Figure 10. SEM-EDS analysis of cemented Cu- 0.5wt.%Ni SiC composite

XRD patterns of Cu-Ni SiC composites are given in Figure 11. All characteristic peaks of Cu and SiC were detected and the intensity of SiC peaks increased with the increasing amount of the SiC.

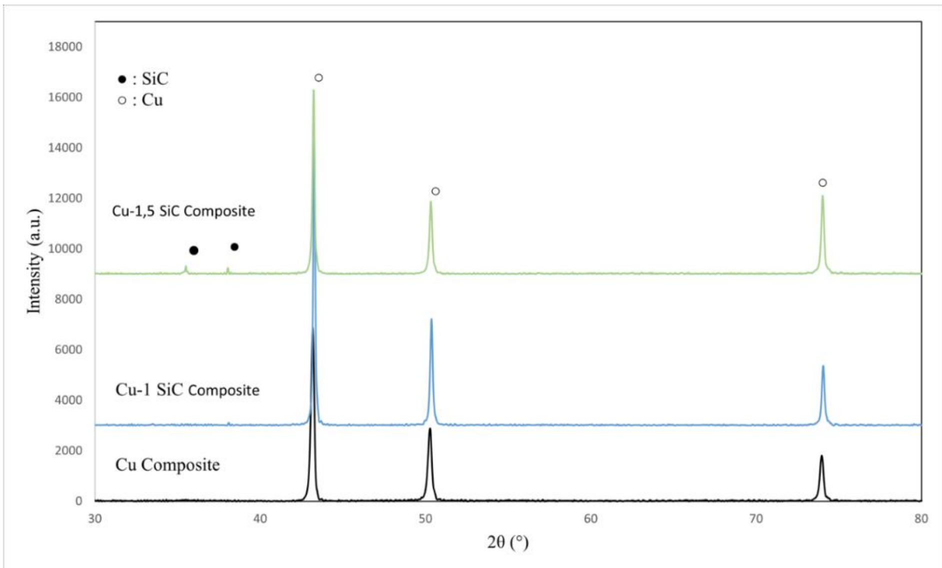


Figure 11. XRD patterns of cemented Cu and Cu 1-1.5 wt.% $_{Ni}$SiC composites

The microhardness values of the test specimens with pure copper measured by the Vickers hardness method are given in Table 2. In the microhardness measurements, care was taken to ensure relatively homogeneous coverage of Cu and SiC by the indenter. As can be seen from Table 2, the hardness of the composite samples increased with increasing reinforcement amount. In their study, M. Barmouz et al. reached a maximum hardness of 115 HV 0.1 in composite samples with SiC added to copper [2].

Table 2. Relative density values of cemented Cu and Cu-%$_{Ni}$SiC composites

wt.%$_{Ni}$SiC	Sintering Temperature 700^{o} C
	Microhardness (HV)
0	132
0.5	140
1	145
1.5	148

The electrical conductivity variation versus SiC amount is given in Figure 12 showing that the increase in the amount of reinforcement component shows that the electrical conductivity of the samples decreases compared to pure copper. The addition of SiC, which is an insulator in pure copper, prevents electron movement in the structure and decreases the conductivity since the electron movements are homogeneous and short distance distributed in the structure [3].

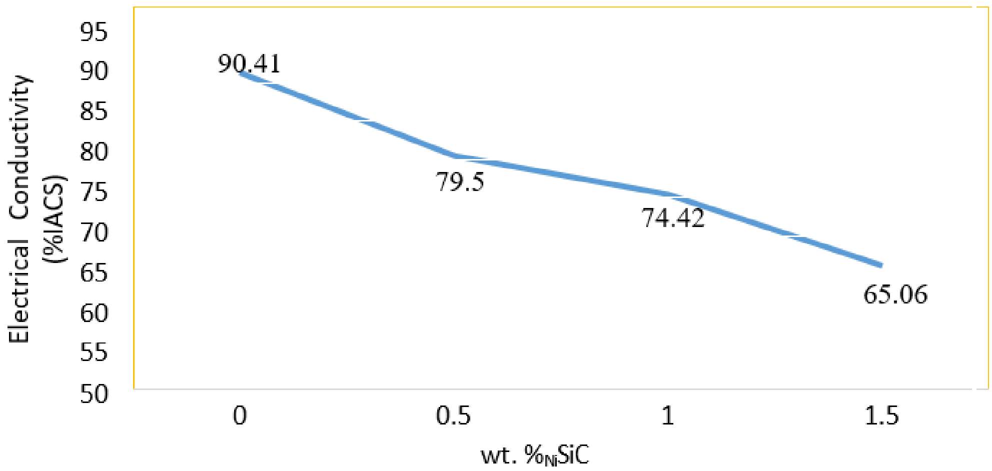


Figure 12. Electrical conductivity (%IACS) variation of cemented Cu and Cu-Ni SiCcomposites

4. CONCLUSIONS

In this study the matrix component Copper powder was successfully produced by cementation method. SiC powders were coated with Ni by electroless plating method. The production technique was ECAS while producing Cu-Ni SiC composites Sintering procedure was conducted at 2200-2500 amperes for 2 minutes. From the SEM examinations of the composite samples it was observed that Ni -SiC particles were homogeneously dispersed in the copper matrix. Hardness of the Cu-Ni SiC composites increased and the relative densities decreased slightly with the increase in the SiC ratio added to copper. Relative density results showed that the density of the Cu- 1.5wt.% $_{Ni}$SiC composite decreased by only 0.5% compared to 100% Cu without $_{Ni}$SiC. Cu and SiC were identified as the dominant components in XRD patterns. The electrical conductivity values of the composites decreased by 27% at most with an increase in the amount of reinforcing component and they are in agreement with the density and hardness results. All results show that Cu-$_{Ni}$SiC composites can be used in on-off contacts up to 1 wt%$_{Ni}$SiC reinforcement.

Declaration of Competing Interest

There is no conflict of interest in this study.

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