POLİTEKNİK DERGİSİ

JOURNAL of POLYTECHNIC

ISSN: 1302-0900 (PRINT), ISSN: 2147-9429 (ONLINE)

URL: http://dergipark.org.tr/politeknik

Investigation of diffusion welding capability of WC ceramic based Ni doped composites

WC Seramik esaslı Ni katkılı kompozitlerin difüzyon kaynağı kabiliyetinin araştırılması

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Bu makaleye şu şekilde atıfta bulunabilirsiniz(To cite to this article): Yönetken A., Erol A., "Investigation of diffusion welding capability of WC ceramic based Ni doped composites", ***Politeknik Dergisi***, 25(3): 1031-1039, (2022).

Erişim linki (To link to this article): http://dergipark.org.tr/politeknik/archive

DOI: 10.2339/politeknik.823822

Investigation of Diffusion Welding Capability of WC Ceramic Based Ni Doped Composites

Highlights

- *Electroless Nickel Plating*
- *Solid State Diffusion Welding*
- *Shear Test*
- *Ceramic Based Composite*

Graphical Abstract

In this study, composite was produced with two different production methods after sintering using WC powders that were coated and not coated with Electroless Nickel (EN). The mechanical strengths of the composites (35.4 MPa) were determined by applying tensile shear test to the produced composites. After the characterization of the samples, it has been determined that the samples produced by electroless Ni coating have a higher solid state diffusion source than the others.

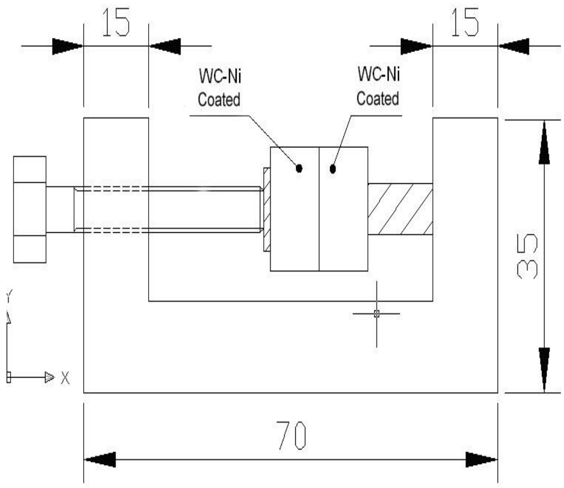

Figure 2. *Diffusion bonding unit where the samples were pressured with screw system.*

Aim

Combining Ni-doped ceramic matrix composites by solid state diffusion welding.

Design & Methodology

Composite production with powder metallurgy technique and tensile shear test, determination of mechanical and metallographic properties

Originality

The use of electroless Ni coating technique in composite production

Findings

Determination of tensile and hardness values as well as mechanical and metallographic properties of produced samples.

Conclusion

Better mechanical properties of solid state diffusion welding have also been obtained in composites produced by the electroless Ni coating method.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

WC Seramik Esaslı Ni Katkılı Kompozitlerin Difüzyon Kaynağı Kabiliyetinin Araştırılması

(Bu çalışma ICMATSE 2020 konferansında sunulmuştur. / This study was presented at ICMATSE 2020 conference.)

Araştırma Makalesi / Research Article

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(Geliş/Received : 11.11.2020 ; Kabul/Accepted : 02.04.2021 ; Erken Görünüm/Early View : 13.04.2021)

ÖZ

Bu çalışmada, Ni kaplanmış WC tozlarından üretilen (WC)Ni kompozit ve kaplanmamış WC-Ni tozlardan oluşturulan (WC)Ni/(WC)Ni ve WC-Ni/WC-Ni numune çiftleri difüzyon kaynağı ile birleştirme için kullanılmıştır. Difüzyon kaynağı Ar atmosferi altında önceden yüklenmiş sıkıştırma sistemi ile yapılmıştır. Bağlama basıncı ve sıcaklığı değiştirilerek difüzyon kaynaklı çiftler elde edimiştir.. Difüzyon kaynaklı çiftlerin çekme makaslama testi için Schimadzu 100kN üniversal çekme makinesi kullanılmıştır. Çekme makaslama testi sonuçları, akımsız nikel kaplanmış WC(Ni) kompozit difüzyon çiftinin diğer difüzyon çiftine kıyasla en iyi çekme makaslama dayanımına 35,4 MPa sahip olduğunu göstermiştir. 200 ila 400HV arasında değişen sertlik değerleri belirlenmiştir. Çalışma da elde edilen kompozitlerin ve difüzyon kaynaklı numunelerin mikro yapısal incelenmesi ve kaynak ara yüzeyinde oluşan parçacıkları analiz etmek için SEM ve XRD analiz sisteminlerinden yararlanılmıştır.

Anahtar Kelimeler: Difüzyon kaynağı, malzeme özellikleri, toz metalürjisi, elektrik akımsız Ni kaplama verim.

Investigation of Diffusion Welding Capability of WC Ceramic Based Ni Doped Composites

ABSTRACT

In this study, sample pairs of (WC) Ni / (WC) Ni and WC-Ni / WC-Ni formed from (WC) Ni composites produced from Ni coated WC powders and uncoated WC-Ni / powders were used for diffusion welding. Diffusion welding was carried out by preloaded compression system under Ar atmosphere. Diffusion welding was obtained by changing bonding pressure and temperature. Schimadzu 100kN universal tensile machine was employed for tensile shear testing of diffusion welded couples. The tensile shear test results showed that electroless nickel coated WC(Ni) composite diffusion couples yielded best tensile shear load bearing capacity 35,4 MPa compared to other diffusion couples. Hardness values ranging from 200 to 400HV were determined. SEM and XRD analysis systems were used to analyze the microstructural analysis of the composites and diffusion welded samples obtained in the study and to analyze the particles formed at the weld interface.

Keywords:Diffusion bonding, mechanical specification, powder metallurgy, electroless nickel plating

1. INTRODUCTION

Powder metallurgy method is a manufacturing method performed by mixing ceramic and metal powders under temperature, pressing in a mold with a shape and specified dimensions, and sintering at a certain temperature after shaping [1]. Diffusion welding is an important joining technique used in electrical, electronic devices, and industries [2]. Similar or different metals are combined by solid state diffusion welding technique. Diffusion welding is an optimized solid state bonding process under pressure, various temperatures, and different dwell times. Diffusion welding is formed by controlled diffusion by applying heat and pressure for a certain period with the minimum of macroscopic deformation between mating surfaces under relevant atmosphere protection. This welding method is more widely used in material couples where bonding of different material couples through traditional methods is problematical. This method was developed for airplane and space industry and nuclear technology. It is possible to reach the strength of values of the actual material approximately in the diffusion welding of the same material couples. Materials, metal and non-metal materials that form intermetallic phases and have various melting temperatures and elastic limits can be welded by this method.

The standard connection process for the production of cermet composite parts is the welding of ceramics and metals. While welding, solid state diffusion welding or soldering method may be preferred. Composites are constantly evolving with their use in every field. One of the most important innovations that have emerged is replacing ordinary materials due to the mechanical and physical properties of innovative composite materials [3]. When composite materials are exposed to environmental influences (temperature, humidity, etc.)

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like other materials; they cause a decrease in material properties and service life [4-7]. However, the solid-state diffusion weld ability of Ceramic-Metal composites gives hope for future studies, although their mechanical strength values are not high. Diffusion welding can be applied with or without interlayer. In the welding without interlayer, the joining takes place by micro-deformation mechanism, and high temperature and pressure are required for the joining process [8-9]. Diffusion welding is a solid state welding that makes it possible to combine many ferrous and non-ferrous metals and alloys, including ceramics and composites, which cannot be joined together by fusion welding. [10-15]. Parameters such as contact temperature, pressure and time significantly affect the strength at the interface while forming the solid state bond [16-17]. P/M enables easy production of materials with different sizes and different geometric shapes. It is the transformation of powders into parts with high mechanical strength after being sintered after pressing [18-25].

In this study, the composite produced from Ni coated WC powders and uncoated WC-Ni powders were cold pressed in the mold after 24 hours of mixing and then sintered at the temperature of 1200oC. The mechanical properties of the composites obtained after the sintering process were determined. Afterwards, the weldability of WC-Ni/WC-Ni ceramic-based metal-reinforced composite sample obtained from electro-less nickel-coated tungsten carbide powders (WC) Ni / (WC) Ni and non-nickel-coated tungsten carbide powders was investigated by diffusion welding method. For this purpose, ceramic-based composite couples were subjected to diffusion welding process at 1200C in argon atmosphere under 10 kg load for 30 minutes. The tensile shear strength and hardness distribution of the welded samples were determined. In addition, the microstructure of welded joints was examined.

2. MATERIAL and METHOD

In this study, WC powders at 99% purity with a size of 20 micron were used. Ceramic based Ni reinforced metal composite samples were produced by using electroless nickel coated and uncoated Electroless Ni coated and uncoated WC powders was mixed and then cold pressured in the mold. The green products were then sintering at 1200ºC. Two different ceramic based composites were produced in the experiments. The 1st Group was (WC)Ni composite samples using by electroless nickel plating, 2nd Group WC-Ni composite samples Ni reinforced WC composite material samples were produced by powder metallurgy.

In the coating process, 20% diluted hydranize hydrate (N2H4, H2O) and 36gr Nickel Chloride (NiCl2.6H2O) baths were prepared and when the temperature reached 90oC, WC powders were added to the process and the coating process was carried out until the reaction was completed. The powders of the capping process were washed 3 times with distilled water. In addition, bath residues were removed by washing with alcohol. It was made ready for sintering by drying in an oven at 105oC for 24 hours. The prepared compositions were shaped in a uniaxial mold with 6x8x12 mm dimensions and sintered in an argon atmosphere in a tube furnace at 1200°C. Mechanical and metallographic tests were applied to the produced samples.

2.1 The Determination Mechanical and Microstructural Properties of WC-Ni/(WC)Ni Composite sample

The strength of WC-Ni / WC-Ni ceramic based metal reinforced composite samples obtained from non-electroless nickel coated tungsten carbide powders and (WC) Ni/(WC) Ni composite obtained by electroless nickel coated tungsten carbide powders was determined by applying the compression test. Compression test was carried out in Shimadzu AG-XD 100kN brand test device at a feed rate of 0.001 mm.

The compression test samples were carried out in the compression test machine. They were placed within the compression test machine in vertical position. The plastic deformation amounts of the test samples under gradually increased compression loads were determined. Compression load reached the maximum load value that the samples could withstand and then fell down, and there occurred a crack on the material from which P/M was produced due to a visible deformation along the height of the sample.

The hardness measurement of the composite materials was carried out in the Shimadzu - Hmv-2l Assy tester. A load of 0.2kg was applied to the penetrating tip.

In order to determine the microstructure of the composite materials, the samples prepared by using classical metallography methods were etched in 10ml H2O + 10ml Acetic Acid + 10ml HNO3 at the end of 1 min. LEO 1430 VP model brand scanning electron microscope was used for microstructure analysis. In addition, Bruker Brand D8

Table 1. Sample sizes of compression experiment

Samples	Thickness (mm)	Width (mm)	Height (mm)
WC-Ni	6	8	12
(WC)Ni	6	8	12

Table 2. The parameters applied to samples in the diffusion welding.

Sample name	Temperature ºC	Load (Kg)	Time (min)
WC-Ni/WC-Ni	1200	10	30
(WC)Ni-(WC)Ni	1200	10	30

Advance XRD system was used for the characterization of the composite materials obtained in the study.

2.2. Preparation and Joining of Composite Materials for Diffusion Welding Process

The dimensions of the composite materials obtained by sintering Electroless Nickel coated and uncoated WC powders in the study are given in Table 1.

The surface of composite samples was cleaned for diffusion welding The schematic diagram of temperature, pressure and time in diffusion bonding is shown in Figure 1. The welding parameters applied to composite samples the in diffusion welding is given in Table 2.

The device used for diffusion welding of composite samples is shown in Figure 2. The samples were joined by means of the mechanism by controlling the deformation rate of the pressure on the material.

The composites samples were compressed with 10kg load pressure through diffusion welding at the temperature of 1200ºC. The diffusion welding process was performed in a tube oven under argon atmosphere for a 30 minute period. The samples were left to natural cool down in the oven after welding process.

2.3 Determination of Mechanical Properties of Diffusion Welded Composites

The strength of the diffusion welded samples was determined by tensile shear test. Tests were carried out with shear test equipment shown in Figure 3 which was prepared specially in order to establish the shear strength of the samples after welding.

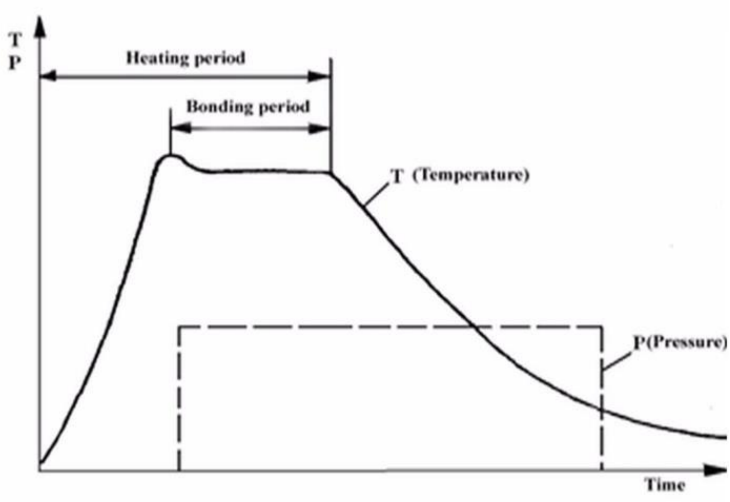


Figure 1. Konik Diagram of Temperature, Pressure and Time

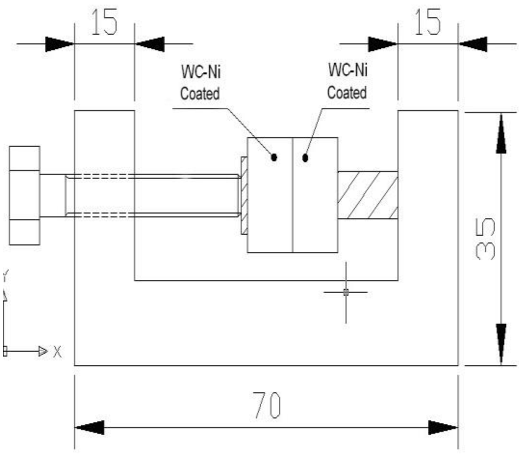


Figure 2. Diffusion bonding unit where the samples were pressured with screw system.

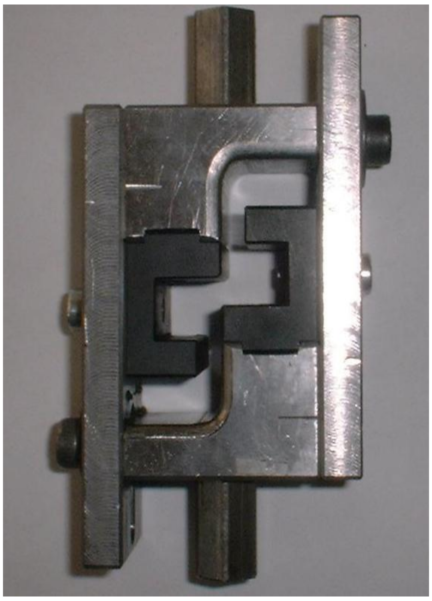


Figure 3. Tensile shear test sample apparatus

The hardness measurement of diffusion welded sample was carried out on the cross section of welded sample by using Schimadzu Hardness test machine.0,5 kg load applied to the intender of the test machine. At least five measurements were carried out to minimize the error. The average of the hardness values was used in the graphic drawing.

The surfaces of the diffusion welded samples were sanded with abrasives of 1000 meshes to examine the metallographic structures of the samples. In order to determine the microstructure of the composite materials, the samples prepared by using classical metallography methods were etched in 10ml H2O + 10ml Acetic Acid + 10ml HNO3 at the end of 1 min. At the end of the classical metallographic sample preparation processes, the microstructures of the samples were examined by optical microscopy, SEM and EDX.

3.RESULTS AND DISCUSSION

3.1 WC-Ni / (WC)Ni Composite samples properties

In this study, EDX analysis was performed to determine whether the electroless nickel coated powders used in the production of composite materials are coated or not, the result is shown in Figure 4.

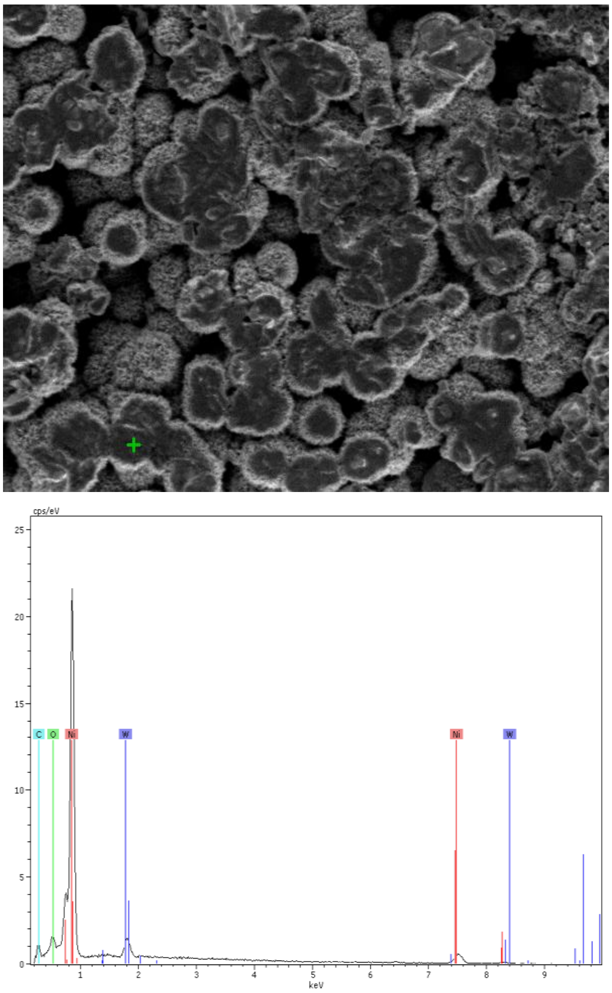


Figure 4. EDX analysis of Ni coated WC powders

The result of the analysis indicates that the WC powders were successfully coated with the electroless Nickel coating method. The strength of composite materials obtained by using these powders was determined by the compression test and the results are graphically shown in Figure 4.

sintering temperature. Since electroless Ni coating facilitates bonding between particles, the coated samples show better properties. WC particle sizes used in composite production also affect the closing of pores. As the particle size decreases, post-compression porosity will decrease. This will further reduce the porosity after

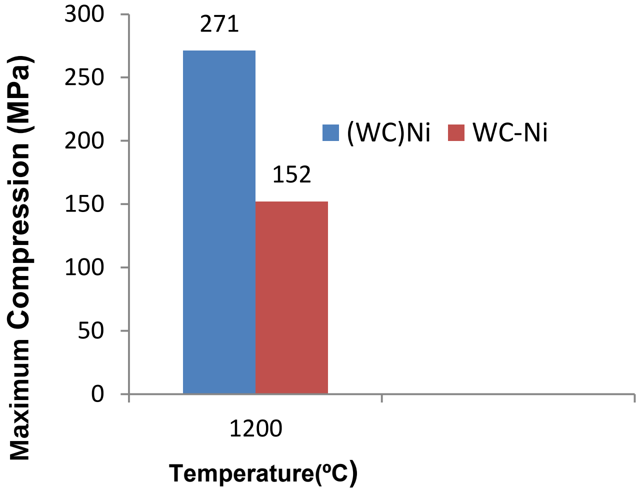


Figure 5. Compression test curve of composite samples

After sintering the material with WC matrix, which is electroless Ni coated and produced by powder metallurgy method, at 1200 ℃, the compressive strength tests were performed in Shimadzu AG-XD 100kN device. The compressive strength of the sample produced by classical powder metallurgy method was measured as 152MPa. The compressive strength of the ceramic matrix composite produced by plating electroless Nickel was measured as 271MPa.

sintering. The decrease in porosity will also reflect on the mechanical properties of the material.

Among the composite samples produced with electroless Ni coated WC powders, the highest hardness value was measured as 271HB in the composite produced from powders coated at 1200ºC. The hardness of the composite sample produced from WC-Ni mixed powders was measured as 152 HB on average. As electroless Ni coating contributes to the reduction of porosity, it has

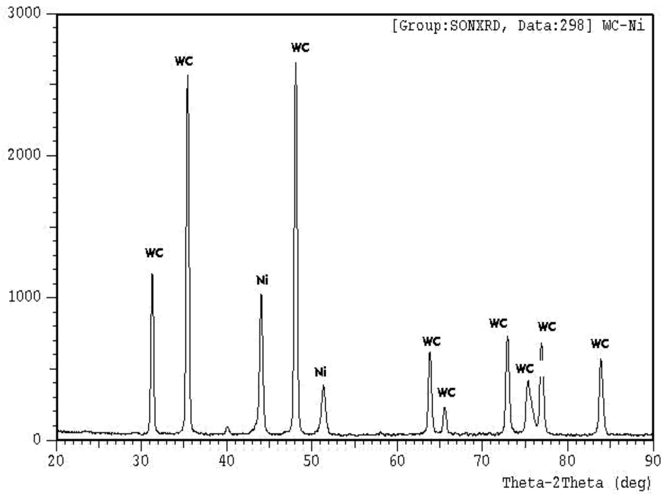


Figure 6. XRD analysis graph of WC-Ni composite sintered at 1200 °C in tube furnace

The compressive strength of the composites is obtained from electroless Ni coated and an uncoated powder was measured as 271MPa and 152MPa respectively. It has been determined that the ceramic-metal composite produced by making Ni coating shows higher strength than samples produced without coating at 1200˚C

been determined that the properties such as hardness and strength are better in samples with electroless coating. The samples sintered in a tube furnace at 1200ºC without electroless coating were examined and characterized by XRD analysis. The XRD analysis chart of the composite sample produced by adding Ni powder into WC ceramic

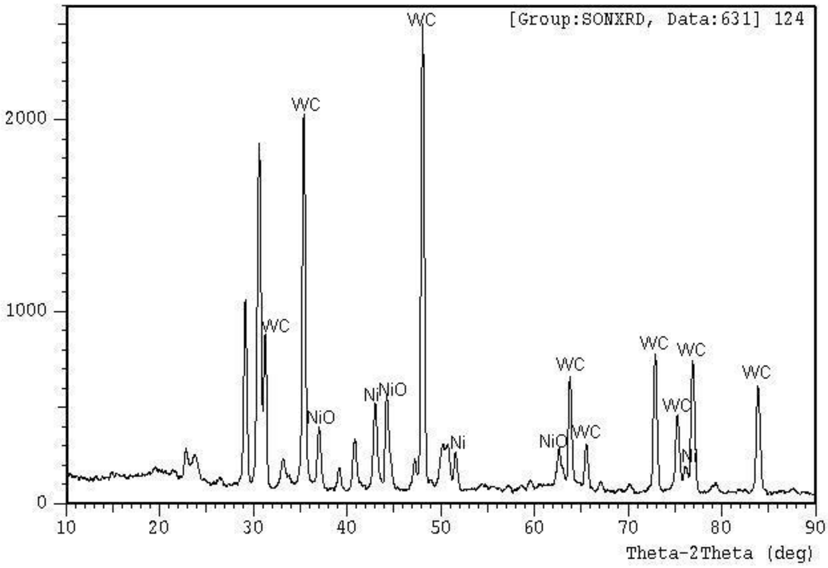
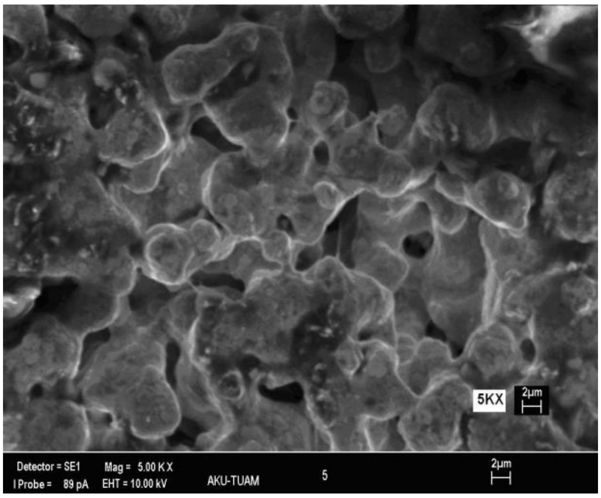


Figure 7. XRD analysis graph of (WC)Ni composite sintered at 1200 °C in tube furnace

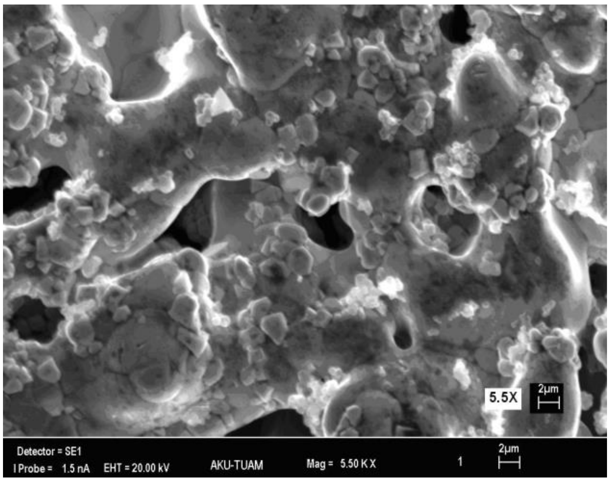
powders without applying the coating process is given in Figure 6.

As seen in Figure 6, the X-ray graph of the WC-Ni ceramic metal composite sintered at 1200 ° C is given. Peaks of WC and Ni phases are seen. The low content of Ni in the composite and the low intensity of the Ni peaks confirm the composition. In addition; the XRD analysis graph of (WC)Ni composite sintered at 1200 °C in tube furnace is shown in Figure 7.

As seen in Figure 7, the X-ray graph of the Ni ceramic metal composite (WC) produced from Electroless Ni coated WC powders at 1200 ° C is given. Peaks of WC, Ni and NiO phases are seen. It has been observed that the intensity of the Ni peaks is almost the same as the composite produced by classical powder metallurgy, due to the obtaining of the composite through Ni coating in its composition. Moreover, the microstructure of sintered composites are evaluated. The microstructure images of composites are shown in Figure 8 a and b.



(a) Ni coated WC composite sample



(b) Ni mixed WC composite sample

Figure 8. SEM image of composite samples produced by WC-Ni system sintered at 1200 ° C in tube furnace

In the sintering process, it is seen that the powders are connected to each other by forming a neck (Figure 8a). It is seen that the porosity rate of powders sintered by electroless Ni coating is lower than the composite sintered by mixing Ni powder. It is seen in SEM photographs that composite particles produced from electroless Ni coated powders exhibit a more homogeneous microstructure than the others.

3.1 Diffusion Welded WC-Ni / (WC)Ni Composite Sample Properties.

Table 3. The tensile shear strength values of the diffusion welded composite samples.

Diffusion Welded Composite	Temperature °C	Time (min)	Tensile Shear Strength (MPa)
WC-Ni/ WC-Ni	1200	30	22,4
(WC)Ni-WC)Ni	1200	30	35,4

The strength of diffusion welded composites are determined by tensile shear test. The tensile shear strength values of the diffusion welded samples were given in Table 3.

Shimadzu universal pulling device was used for shear test of diffusion welded pairs. (WC) Ni / (WC) Ni diffusion welded sample is due to the higher shear strength of the WC-Ni / WC-Ni diffusion welded pair, due to the fact that the Nickel particles accumulated on the surface parts of electroless nickel coated WC powders provide stronger intergranular bonding. is considered. Although the shear test results show that the (WC) Ni composite diffusion pair has the best shear strength load capacity of 35.4 MPa, diffusion weld pair welding is not very good. However, it gives hope for studies that will increase the power of the interface.

The hardness measurement was carried out at certain intervals (0.5mm) in both directions from the weld interface of the diffusion welded joints to the main material. The hardness results are graphically shown in Figure 9.

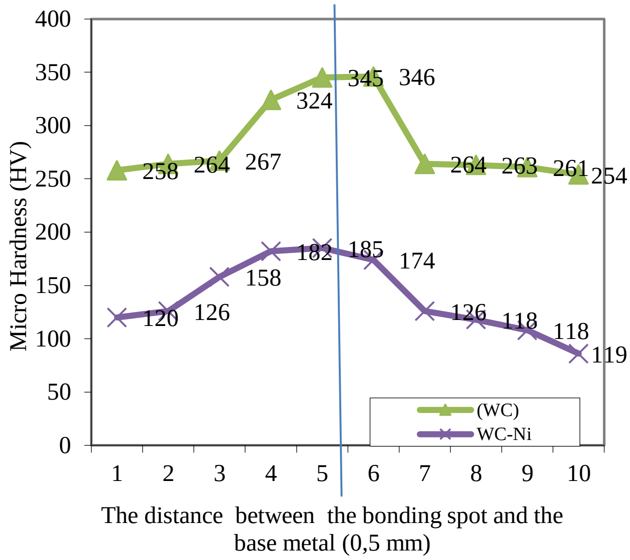


Figure 9. The diffusion bonding micro-hardness values of the samples

As seen Fig.9 In both diffusions welded samples, the hardness values increased as they approached the bonding zone. The hardness in the composite diffusion weld joint area produced by the coating method has a higher hardness value of 80-90 HV compared to the base sample. In composite produced with classical powder metallurgy, this value was measured as 50-60HV The fact that the hardness of the weld zone of the diffusion welded sample obtained from Electroless Ni coated WC powders is higher than the other diffusion source sample supports the high tensile strength.

3.1.1. Microstructural Evaluation of Diffusion Welded WC-Ni / (WC)Ni Composite sample

The macrographs of diffusion welded composite samples were shown in Figure 10a and b.

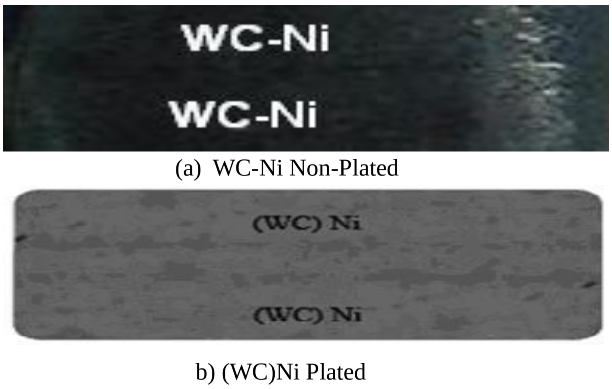
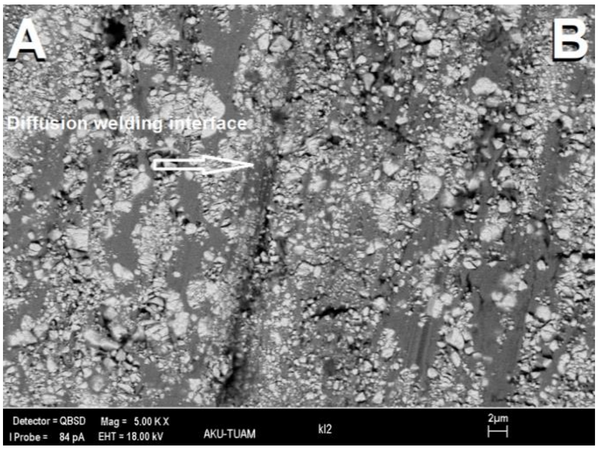
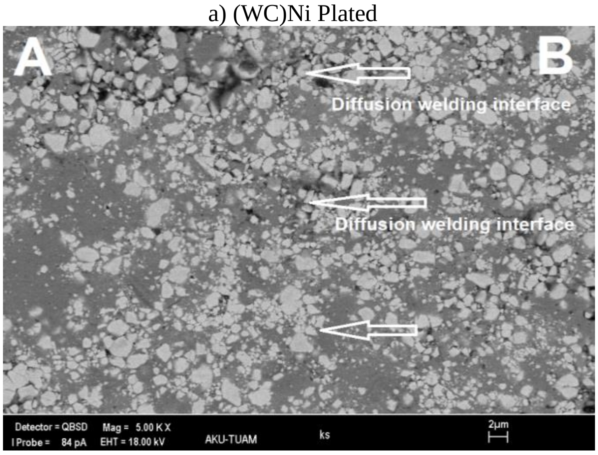


Figure 10. The photographs of diffusion bonded samples

The microstructure of diffusion welded composite samples were also examined and results are shown in Fig.11 and and b.



a) (WC)Ni Plated



b)WC-Ni Non-Plated composite

Figure 11. The SEM 10kX photographs showing the bonding spots of the composite produced by Ni Plated WC powders and Non-Plated WC powders.

Solid state diffusion weld interface of composite samples produced from electroless Nickel coated powders is given in SEM Figure in 11a. Although the sample has a homogeneous structure, it contains pores. The diffusion weld interface is clearly seen in the picture. Although its mechanical strength is lower than the strength of the main material, it is considered to be improvable.

Solid state diffusion weld interface of composite samples produced from Wolfram carbide and Nickel powders is given in SEM Figure in 11b. It is understood that homogenization of micro-structured powders takes place. It is seen in the picture that the diffusion weld interface is not very clear. Mechanical shear strength has been measured lower than composite produced from electroless coated powders. The reason for the low shear strength is due to the weak neck formation between ceramic metal powders

6. CONCLUSION

- The following conclusions have been drawn from the solid-state diffusion welding study in composite materials.
- The ceramic matrix composite and Electroless Ni coated Ceramic matrix composites joined by diffusion welding method. Welding process was applied at 1200 °C for 30 minutes dwell time in argon atmosphere by using 10 kg loads. and a good solid-state welding has been formed.
- The tensile shear test results showed that the diffusion welded WC(Ni) composite couples yielded best tensile shear strength 35,4 MPa compared to other diffusion couples. Breaks occurred at the joint interface during the shear test.
- The solid-state diffusion welded composite samples produced by using Electroless Ni coating technique has measured better mechanical properties than the other. Electroless Nickel coating method provides advantages in joining of samples.

DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods they use in their studies do not require ethics committee approval and / or legal-specific permission.

AUTHORS' CONTRIBUTIONS

Ahmet YÖNETKEN: Perofrmed the experiments and analyse the results. Wrote the manuscript.

Ayhan EROL: Perofrmed the analyse the results.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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