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Research Article

Akımsız Kaplama Tekniği ile Üretilen Seramik Esaslı Kompozitlerin Ultrases ile Mekanik Özelliklere Etkisinin Araştırılması

Ahmet Yönetken[1*], Vildan Özkan Bilici[2]

[1*] Afyon Kocatepe University, Faculty of Engineering, Department of Electrical Engineeirng, Afyonkarahisar, Turkey, (ORCID: 0000 0003 1844 7233), yonetken@aku.edu.tr

[2] Afyon Kocatepe University, Faculty of Arts and Sciences, Department of Physics, Afyonkarahisar/ Turkey, (ORCID: 0000-0002-3077-2103), vildanozkan@aku.edu.tr,

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Özet

Bu çalışmanın kapsamı, WC esaslı kompozitlerin fiziksel ve mekanik özellikleri ile ultrasonik hızın değişimini araştırmaktır. Havacılık ve otomotiv endüstrilerinde kullanılan malzemeler yüksek sıcaklıklara, sürtünmeye ve yüksek gerilimlere maruz kaldığından kesme diskleri ve kesici uçlar gibi imalat uygulamalarında kullanılan metal matrisli kompozit malzemelere olan ihtiyaç hızla artmıştır. WC ile güçlendirilmiş Ni-Ti metal matrisli kompozitler, tek eksenli hidrolik preste dairesel olarak şekillendirilmiştir. %30 Ni, %2 Ti ve %68 WC tozlarından oluşan karışım 1000°C-1400°C'de argon atmosferinde tüp fırında sinterlenmiş ve akımsız kaplama tekniği ile üretilmiştir. Numunelerin mekanik ve fiziksel özelliklerini incelemek için XRD (X-Işınları kırınımı, SEM (Taramalı Elektron Mikroskobu), sıkıştırma testi, sertlik ve ultrasonik hız ölçümleri kullanılmıştır. Deneysel sonuçlar, artan sinterleme sıcaklığı ile ultrasonik hız değerlerinin polinom olarak lineer bir şekilde arttığını göstermiştir. Ayrıca en yüksek sinterleme sıcaklığının 1400°C'de 429,1HV sertlik ile sonuçlanması yapının daha sağlam ve yoğunhale geldiğinin bir göstergesidir. Sinterleme sıcaklığının artmasıyla ultrasonik hızlardaki değişim yapıdaki bağlanmanın daha iyi gerçekleştiğinin kanıtıdır.

Anahtar Kelimeler: Ultrasonik hız, Sinterleme sıcaklığı, Akımsız kaplama, Darbe yankı

The Effect of Mechanical Properties on the Ultrasonic Velocity of Ceramic Based Composites Fabricated by Electroless Coating Technique

Abstract

The scope of the present study is to investigate the change of ultrasonic velocity with the physical and mechanical properties of Ni-Ti-WC composites. Since the materials used in the aerospace and automotive industries are exposed to high temperatures, friction and high stresses, the need for metal matrix composite materials used in manufacturing applications such as cutting discs and inserts has increased rapidly. Ni-Ti metal matrix composites reinforced with WC were circularly shaped in uniaxial hydraulic press. A mixture of 30% Ni, 2% Ti and 68% WC powders was sintered at 1000°C-1400°C with argon atmosphere in a tube furnace and were fabricated by electroless plating technique. XRD (X-Ray diffraction), SEM (Scanning Electron Microscope), compressive testing, hardness and ultrasonic velocity measurements were employed to investigated the mechanical and physical properties of specimens. Experimental results showed that ultrasound velocity values increased polynomial linearly with increasing sintering temperature. In addition, the fact that the highest sintering temperature resulted in 429.1HV hardness at 1400°C is an indication that the structure has become more robust and compact. The change in ultrasonic velocities with the increase in sintering temperature is proof that the bonding in the structure is better and the particle growth is well achieved.
Keywords: Ultrasonic velocity, Sintering temperature, Electroless Coating, Pulse echo

[*] Corresponding Author: yonetken@aku.edu.tr

1. Introduction

Composite materials containing a heterogeneous system with an electrodeposited metal and micrometer sized particles have ever been of great interest to engineers and sciencetists and nowadays are still important for many applications in terms of its usage areas and psycho-mechanical properties. (Anik and Korpe 2007, Mallory and Hadju 1990, Zhang et al. 2005, Bindra et al. 1984, Agarwala and Agarwala V. 2003). Electroless nickel plating technique is widely used in many fields; therefore, it provides a high-performance product with high density, wear and corrosion resistance. This method has many advantages over other methods such as easy formation of a continuous and uniform coating on the complex shaped substrate surface, low cost, homogeneity (Agarwala and Agarwala V. 2003, Chen et al. 2003, Surender et al. 2004). Today, since the quality of WC-based composite materials is generally represented by high strength and hardness criteria in powder metallurgy, it is known that they are used in places where high strength is required and in cutting tips. In addition, sintering regimes are applied on ceramic-metal composite materials with WC in order to obtain the desired mechanical properties with various additions (Mc Candlish et al. 1995, Li et al. 2006, Gaard et al. 2006). Since ultrasonic methods do not damage both the tested sample and the tissue, they can be applied to determine the strength, pores and defects in the material of composites. Many studies have revealed the relationships between the properties of solid materials (hardness, fracture strength, tensile strength, strength, etc.) and the ultrasound properties of attenuation and wave velocity. In addition, particle size measurements and the use of spreading areas have increased to determine the homogeneity in the structure of the metal (Sarpün et al. 2009, Eren and Kurama 2012, Bhaskar 2011, Mažeika et al. 2010, Bilici Özkan et al. 2021).

In this study; Ni-Ti-WC composite samples were produced by electroless nickel plating technique at different temperatures. The properties of the new produced Ni-Ti-WC composites werecorrelated with the ultrasonic velocity and mechanical properties, which is one of the ultrasonic properties, by using the pulse-echo method, which is one of the non-destructive testing methods.

2. Materials and Methods

In the study, the proportions of the composite composition were determined by considering the information in the literature. Composites were prepared by electroless nickel plating method, which is one of the catalytic plating methods. WC and Ti powders were used in electroless coating samples and they were coated separately with nickel by electroless method and then shaped again in a hydraulic press under 305.9 kg/cm^2 pressure. The samples, which were shaped with a diameter of 15 mm and a height of 3-4 mm, were sintered in an argon gas atmosphere for 2 hours in a tube furnace at a temperature range of 1000 ºC - 1400ºC. Thus, the samples were made ready for physical, mechanical and metallographic analyses. In electroless Ni plating baths, 68%WC and 2%Ti powders, Nickel chloride, ammonia, hydrazine hydrate and distilled water are used. The contents of the coating baths are given in Table 1.

Table 1. Chemicals and properties of Ni plating bath and their ratios

Chemicals	***Conditions***
Tungsten Carbide (WC)	*20,4g*
Titanium (Ti)	*2g*
Nickel Chloride ($NiCl_2.6H_2O$)	*32g*
Hydrazine Hydrate ($N_2H_4.H_2O$)	*20%*
Distilled Water	*80%*
Ammonia	*250-300 ml*
Temperature (°C)	*90-96°C*
pH value	*9-10*

Pulse-echo method, which is one of the ultrasonic measurement methods, was used to measure the ultrasonic velocity values of the prepared samples. It is a method based on measuring the reflected or transmitted parts of the sound wave in terms of amplitude and time in detecting a discontinuity with different acoustic impedance in the material by ultrasonic examination. The longitudinal ultrasonic velocity (V_L) measurements of the samples were made with the Sonatest Sitescan 150 model ultrasonic wave flow detector. A 2 MHz receiver/transmitter probe (Sonatest SLH2-10) was used for ultrasonic wave velocity measurements. The image of the ultrasonic wave sent to the sample with the transmitter/receiver transducer on the screen on the flow detector was obtained as reflection peaks and echo peaks, and the ultrasonic velocity values were measured by using the calibration and thickness measurements. SEM-EDX analyzes of the samples, which were sintered between 1000°C -1400°C in the tube furnace, were performed with the LEO 1430 VP device equipped with Röntec EDX at 2kX magnification and the images were taken. In addition, Shimadzu-AG/IS 100kN test device was used to measure the compressive strength of the samples and Shimadzu HMV 2L brand microhardness device was used to measure the microhardness of the samples. Microhardness measurements were obtained by averaging the microhardness values. The hardness values obtained from 10 different regions of each composite sample are given by taking their average. The (d=m/V) formula was used to calculate the volumetric changes after sintering of the composite samples prepared at 30% Ni, 2% Ti and 68% WC ratios, which were sintered at differenttemperatures, and Archimedes' principle was used to take the measurements of the samples.

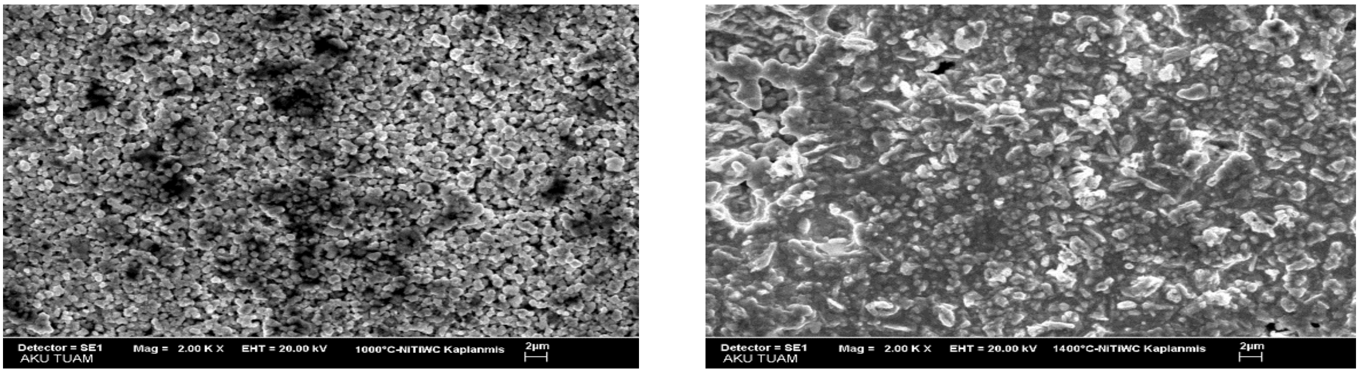
3. Results and Discussion

Sintering temperatures, density, microhardness and compressive strength of Ni–Ti–WC composite samples, depending on the devices and methods used, are shown in Table 2. In addition, ultrasonic velocity values of metalmatrix composite samples produced at different temperatures were calculated using pulse-echo technique at room temperature. (Table 2).

Table 2. Physical, mechanical properties and sintering temperature data of metalc matrix composite samples

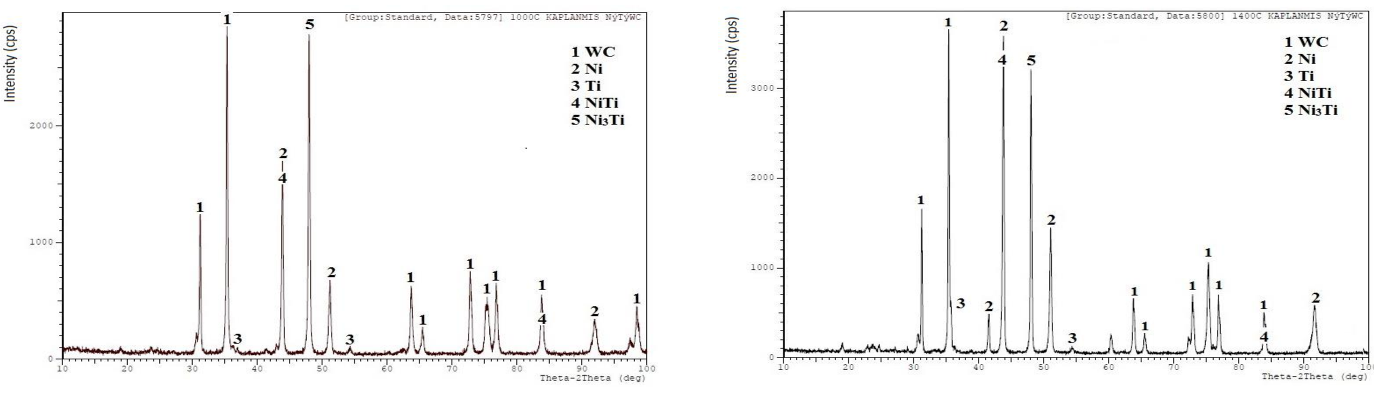
Composite Samples	***Sintering Temperature (ºC)***	***V_L, (m/s)***	***Hardness (0.05 HV)***	***Compression strength (MPa)***	***Density* (g/cm^3)**
30%Ni-2%Ti-68%WC	1000	2731.5 ± 23	252.4	2.803	6.4251
	1100	2800.5 ± 23	255.1	3.521	6.1214
	1200	3033 ± 19	281.3	13.552	6.3063
	1300	3821 ± 9	310	53.501	6.6556
	1400	4212.5 ± 12	429.1	157.787	8.4701

SEM analysis was carried out to examine whether the coating made after the nickel plating process was in the 30% Ni-2%Ti-68% WC composite sample and to examine the structural changes.SEM and XRD analysis results of the coated sample obtained from powders (30%Ni-2%Ti-68%WC) sintered only at 1000°C and 1400°C were shown in Figure 1 and Figure 2. The reason why metallographic analyzes of only samples sintered at 1000°C and 1400°C are taken is that the microstructural change between the initial and final temperature value can be seen more clearly.When the SEM images were compared, it was seen that the particles adhered to each other, the reinforcing powder particles in the structure were connected to each other with a neck tie and the particles grew. In addition, homogeneously distributed pores surrounding the reinforcement particles were observed between the grains.



(a) (b)

Figure 1. SEM micrographs of samples sintered at the temperature of a) 1000 ºC and b) 1400ºC for two hours.

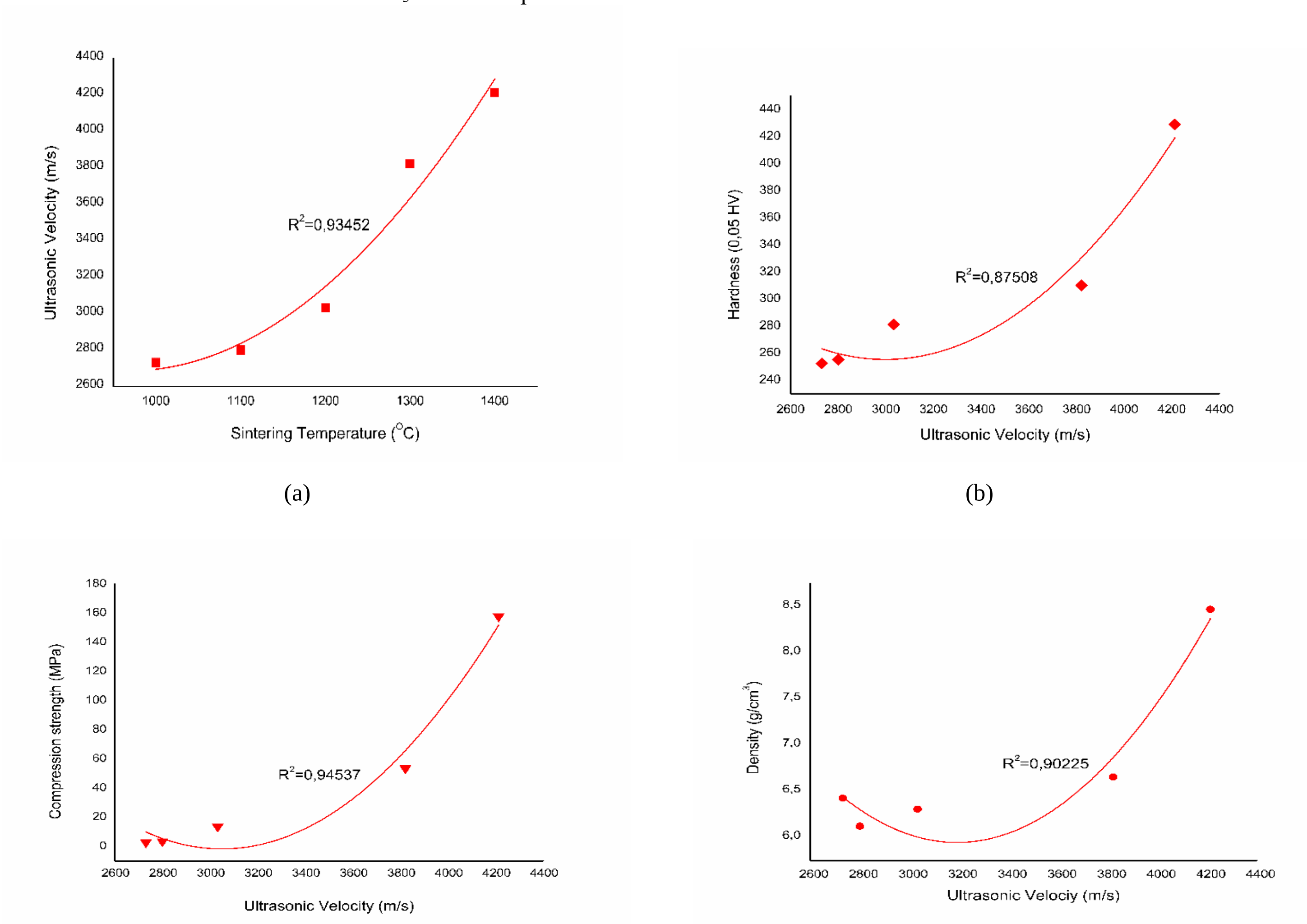


(a) (b)

Figure 2. The XRD analysis result of composite sintered at the temperature of a) 1000 ºC and b) 1400 ºC for two hours.

In Figure 2, the Ni, Ti, NiTi, Ni_3Ti and WC peaks werein the XRD analysis of the Ni-Ti-WC composite sintered in the tube furnace at 1000°C and 1400°C. XRD analysis results clarified the phases and precipitates formed in the samples. The strong nickel peak in the XRD analysis results indicated that the structure was composed of nickel matrix. It could also be seen that WC was reinforced with NiTi and Ni_3Ti metallic phases.

The relationship between the physical properties and mechanical properties of the coated samples after microstructural analysis could be seen in Table 2. Accordingly, calibration curves were drawn using the sintering temperature, ultrasonic velocity, hardness, compressive strength and density values of the samples given in Table 2 in the study (Figure 3).



(a) (b)

(c) (d)

Figure 3. Relationship between ultrasonic velocity and mechanical properties of composites

When the changes in ultrasonic velocityand sintering temperature, density, microhardness and compressive strength for coated samples were examined respectively, it was seen that there was a regular increase. Considering the temperature change of the heat-treated samples, the particles in contact with each other in the sample became more bonded to each other at higher temperatures with increasing sintering temperature. As in the SEM images, this bonding at 1400°C was manifested by necking between the contacting particles. The presence of necking means that the durability increases compared to the raw durability, which is manifested by an increase in the hardness and compression strength values. So that the wave will encounter some particle boundary in the sample, the particles sticking together and the increase in particle size with the growth of the particles should result in increased propagation velocity of the wave. The increase in ultrasonic velocity values with the increase of sintering temperature confirms this. It is a polynomial approximation since the increments are a curvature rather than linearity. Polynomial regression may give the best approximation to the relationship between dependent and independent variable. This approach can basically fit a wide range of curvature. The obtained measurement results and correlation coefficients also confirm this. In addition, it is seen in Figure 3 that as the sintering temperature increases in Ni-Ti-WC composite samples, the density increases. As the sintering temperature increases, sintering becomes easier, more dense samples are obtained (Karabulut et. al. 2013).

4. Conclusion

The aim of this study is to observe and characterize the relationship between the mechanical properties of WC matrix composites and the ultrasonic wave velocity.

The samples produced by electroless nickel plating technique at different temperatures were characterized. In the study, ultrasonic pulse-echo technique was used effectively for the characterization of 30%Ni-2%Ti-68%WC composite properties. The results of the study show that ultrasonic velocity measurement is a reliable tool for the characterization of hardness, sintering temperature, compressive strength and density in Ni-Ti-WC composites.

In addition, the highest density (8.4701gr/cm^3), the highest hardness (429.1HV) and the highest compressive strength (157.79 MPa) values in the composite consisting of 30%Ni, 2%Ti and 68%WC powders sintered at different temperatures, 1400° C was obtained. It has been determined that the best physical and mechanical properties for the 30%Ni, 2%Ti and 68%WC composition are at 1400°C. When the polynomial changes in the graphs were examined, it was observed that the ultrasonic velocity values and the sintering temperature, hardness, compression strength and density values increased. The best approach to the relationship between dependent and independent variable gives polynomial regression. The fact that the correlation coefficient is close to 1 also supports it.

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