

Investigation of Mechanical Properties of Boronized Composites Produced by Electroless Ni Coating

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Abstract: In this study, ceramic-metal composite production and characterization have been made in order to increase the service life of plotter tools used in soil industry. In the study Ni coated (36% AlN + 3% WC) powders were circularly shaped in uniaxial hydraulic press. These samples were sintered at 1000-1100-1200-1300-1400 °C with argon atmosphere in conventional furnaces. The highest hardness value of 112.9 Hv was obtained in the composite samples (36%AlN + 3%WC) at 1400 °C. Powder metallurgy was applied to ceramic-metal composites by using box boring in dry air atmosphere at 950 °C for 4 hours. The samples were characterized by mechanical and metallographic examination. After box boronization, hardness values were measured, their densities were calculated, the current phases were determined by X-ray diffraction (XRD) method and microstructures were examined by scanning electron microscopy (SEM). The highest hardness value of 1150.80 Hv was sintered at 1400 °C and 61% Ni was obtained in the box boron composite sample (36% AlN + 3% WC). Better mechanical and metallographic results were obtained from the samples by using box boring method. The porosity decreased in the microstructure examination of the sample. Considering the advantages of non-electric Ni coating method in sintering, it can be said that improving mechanical properties is a good method. Boronizing process was effective in reducing surface porosity and increasing surface hardness up to 10 times.

Keywords: Composite, Electroless Nickel Plating, Sintering, Box Boronizing.

INTRODUCTION

Nowadays, the rapid progress of technology and the use of new materials in automotive, aerospace and defense industry and industrial applications improve the properties of the produced machinery and equipment. In recent years, studies have been carried out to improve the mechanical properties along with the lightening of the materials. Plotter pens used in soil industry are used as shaping and strength enhancer.

Composites play an important role in the production of such materials.

With the introduction of modern technologies in the soil industry, the need for new engineering materials is rapidly increasing. It can be designed to have superior properties in ceramic-metal matrix composites. A number of studies have been carried out on ceramic materials in the form of finely dispersed powder [1, 2]. Direct fabrication methods have been developed for the production of high-strength ceramic materials [3-6].

In order to improve the mechanical and functional properties of ceramic materials, ceramic matrix composites with metal particles have been developed.

It includes the wetting properties of metals and ceramic materials as well as the magnetic, electrical and chemical properties of ceramic-metal composites [7-11]. The electroless nickel plating process was used to deposit the Ni-P layer on the surface of alumina particles [12, 13]. The boron layer formed in the produced samples is very resistant to abrasion compared to the composite material and can be preferred where abrasion resistance is required [14]. High technology materials produced with high specific strength and hardness, high temperature performance and better thermal and mechanical fatigue and creep resistance are required [15]. Aluminum matrix composites play a major role in the application of advanced engineering materials [16-18].

In the production of ceramic metal composites, electroless Ni coating management is advantageous in sintering. The positive effect of abrasion resistance after boring of AISI 316 stainless steel was investigated and it was stated that a boride layer consisting of FeB and Fe₂B phases were obtained. At the end of the study, the hardness and Ni₃B, Ni₄B₃, Ni₂B and NiB phases of the samples were obtained [19]. The free circulation of boron atom from heat from the Ekobor source and reactions occurring during the diffusion of boron element to the substrate material leads to the formation of boride compounds [20]. Boronization is recommended as a method of increasing oxidation resistance [21].

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In this study, the plotter pencils used in soil industry are changed periodically because they are short. Ceramic-metal composite production and characterization has been carried out in order to reduce the cost of production economically and increase the service life of the plotter pens. Considering the results of the study, it was proposed to be used in the sector.

MATERIALS AND METHODS

In this study; AlN and WC powders were used without Ni coating. The coated powders were shaped in a cylindrical mold under a pressure of 300 bar and sintered in a conventional sintering furnace. X-ray and SEM images were taken for characterization of the samples.

In addition to WC, AlN, Ni phases, Ni₃B, Ni₄B₃ and Ni₂B were determined as metallic phases. Study; WC (99.8% purity and 10 µm size, Aldrich) and AlN (99% purity and -325 mesh size, Aldrich) powders were used. WC and AlN powders were subjected to Ni coating process by using Electroless Ni coating technique by using Hydrazin bath. Nickel chloride hexahydrate (NiCl₂.6H₂O) hydrazine hydrate (N₂H₄.H₂O) and 30 % vol. Ammonia were used in the plating bath.

During the coating, the bath temperature was kept constant at 90-95 °C and the pH was measured between 9-10 with a Philips brand PW 9413 Ion-Activity Meter. The coated powders were washed several times with distilled water and alcohol and filtered off the residues from the coating. The coated powders were dried in an oven oven at 105 °C for 24 hours (Table 1).

Table 1: Nickel Coating Bath and Features

Chemicals	Conditions
%36 AlN powders	10.8g
%3 WC powders	0.9g
Nickel Clorid Hegzahidrat NiCl ₂ .6H ₂ O)	49.2g
Hidrazin Hidrat (N ₂ H ₄ .H ₂ O)	%20
Distile water	%80
Application Temperature(°C)	90-95°C
pH	9-10

Coated powders are shaped in cylindrical mold under 300 bar pressure in uniaxial hydraulic press. The shaped samples were sintered in a conventional sintering furnace for one hour in Argon atmosphere at

temperatures of 1000-1400 °C and composite was produced by powder metallurgy. Composite samples Boring experiment was carried out using box boring method for 4 hours at 950 °C. As a borer, a powder mixture consisting of SiC, B₄C and KBF₄, whose commercial name is Ekabor 2, was used after physical and metallographic examinations. Ecrin powder was used as oxide inhibitor. Composite samples (36% AlN + 3%WC) Ni, density (d = m / V) and hardness were measured before and after boronizing. Boronized specimens were used metallographically in LEO 1430 VP model Scanning electron microscopy and h Oxford EDX analyzer microstructure studies.

EXPERIMENTAL FINDINGS

Density

After ceramic-metal composite samples produced by powder metallurgy were sintered in argon atmosphere at temperatures of 1000-1400 °C, density of sintered samples was calculated according to Archimedes principle. The samples were then boronized by box boring at 950 °C for 4 hours. Densities of boronized samples were calculated according to Archimedes principle. Using these data, the density-temperature change graph was plotted (Figure 1).

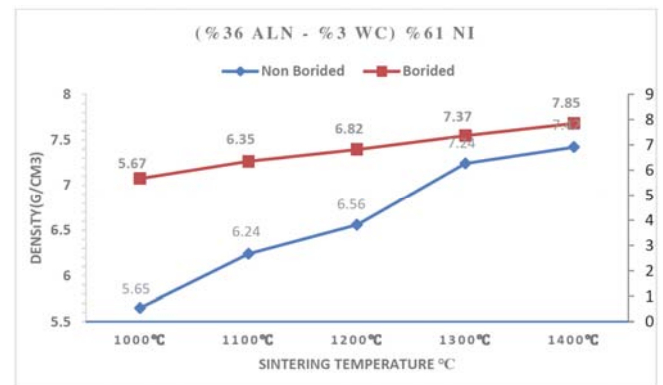


Figure 1: 36% AlN + 3% WC) Graph of density-temperature change of 61% Ni composite.

Microhardness

After the ceramic-metal composite samples produced by powder metallurgy were sintered in argon atmosphere at temperatures of 1000-1100-1200-1300-1400 °C, Vickers Hardness Measurement method was used to determine the hardness changes of the boronized composite samples. The measurements are 100 g from the surface to the matrix at 5 µm intervals. made using wight. Micro hardness measurements were

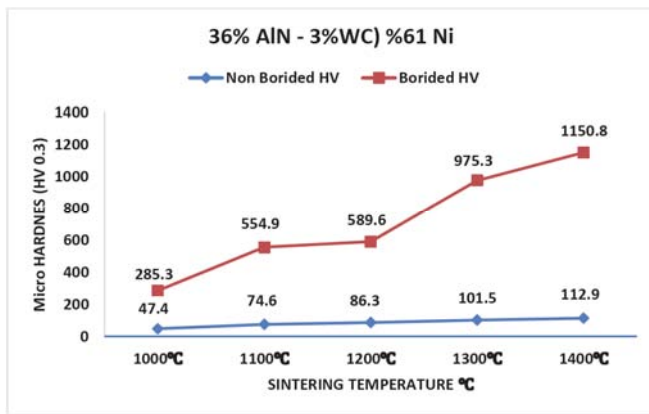


Figure 2: 36% AlN + 3% WC) Graph of microhardness-temperature change of 61% Ni composite.

made at 10 different points and the mean values were transferred to the graph. The microhardness-temperature change graph was drawn using the measured data (Figure 2).

SEM Analysis

It has been observed that composite samples contain a smooth morphology structure. The thickness of the boride layer was measured as 18-22 μm according to its chemical composition. It is known that temperature and time affect the thickness of the boride layer. SEM images of the surface microstructures of the samples boronized at 950 $^{\circ}\text{C}$ for 4 hours to the

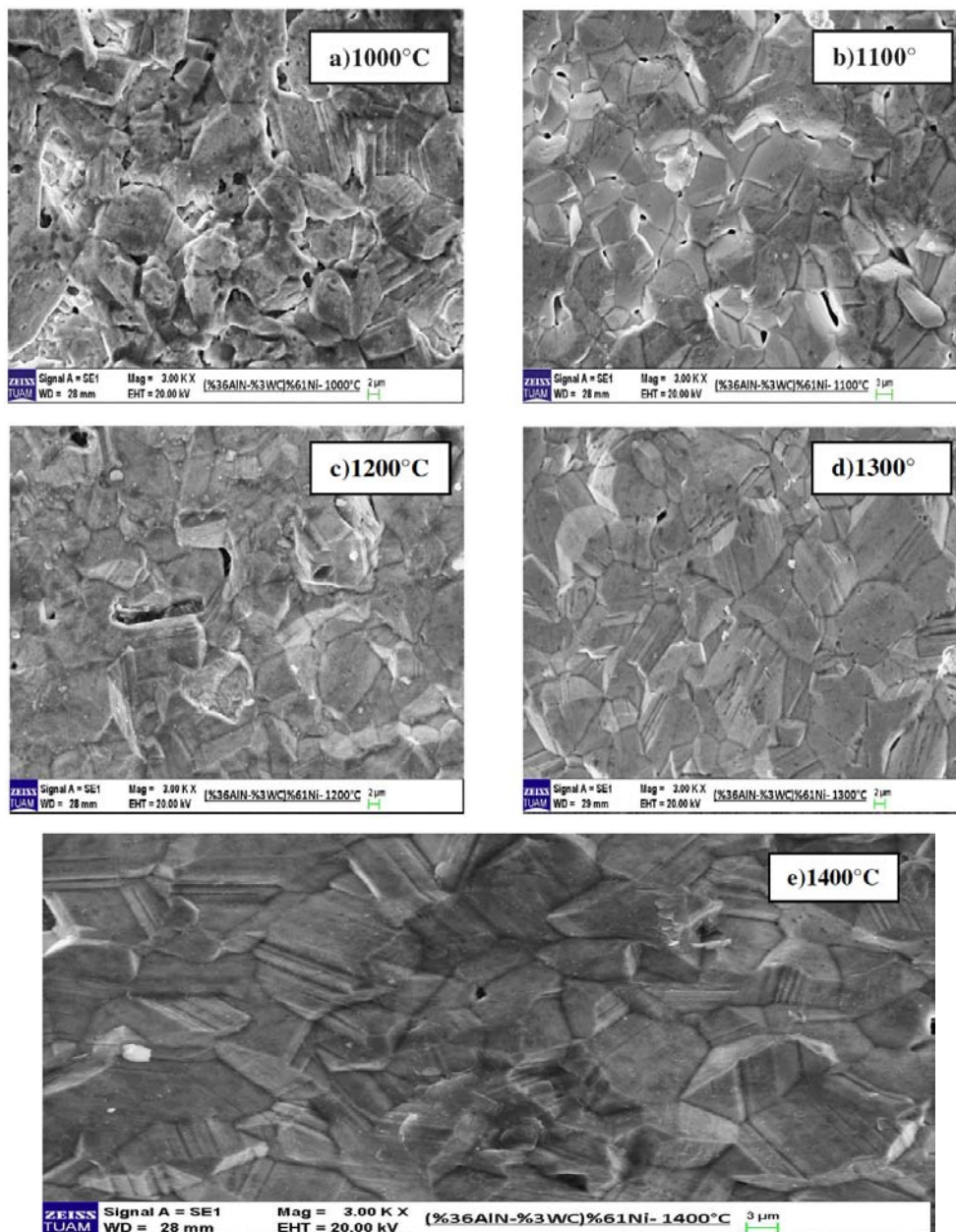


Figure 3: SEM pictures of the composite (36% AlN- 3% WC) 61% Ni).

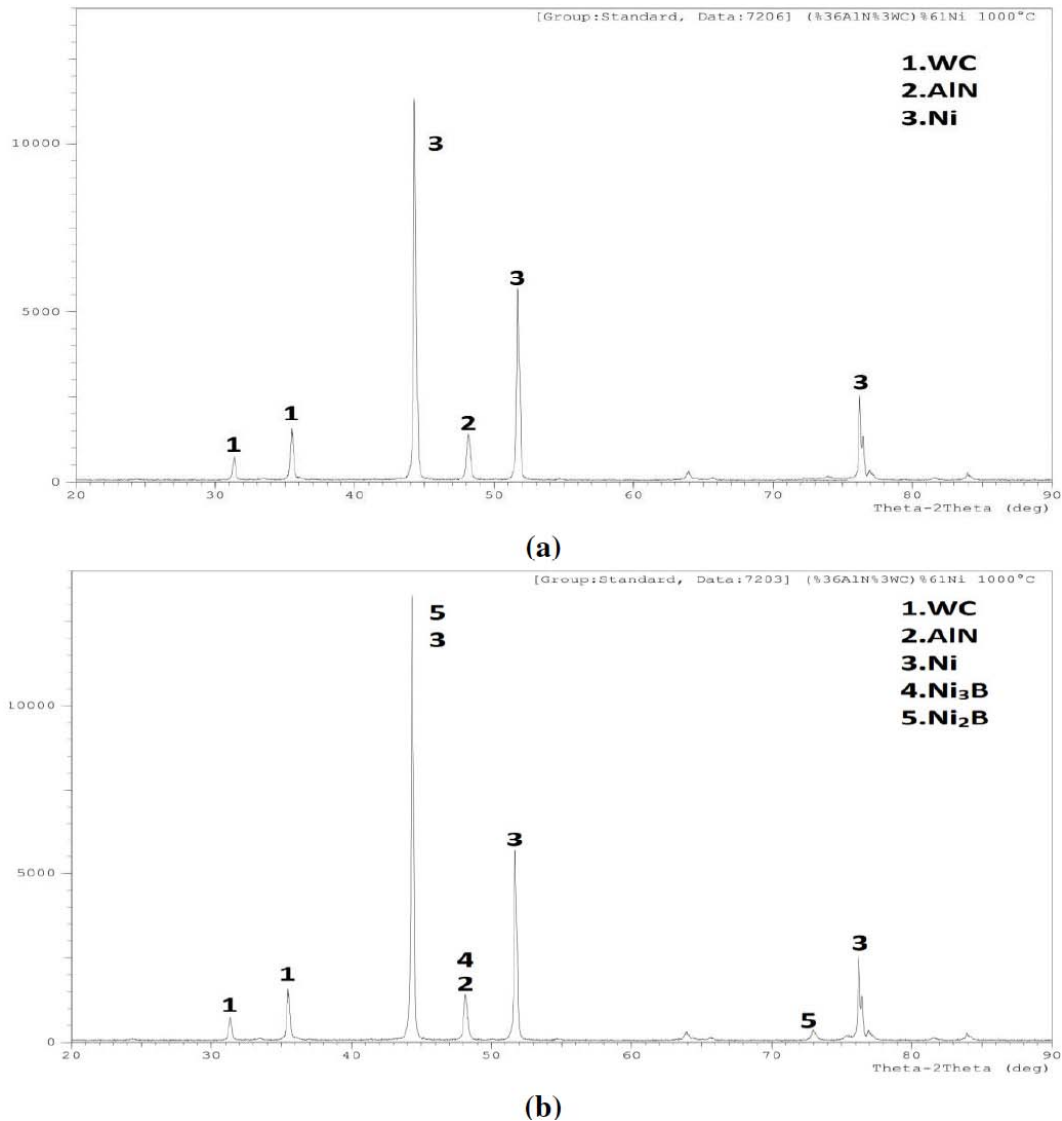


Figure 4: a) X-ray analysis of the composite (36% AlN- 3% WC) 61% Ni 1000 °C). b) X-ray analysis of the boronized composite (36% AlN- 3% WC) 61% Ni 1000 °C).

composites produced by powder metallurgy method (36% AlN + 3% WC) 61% Ni are presented in Figure 3.

X-Ray Analysis

As a result of XRD analysis applied after sintering, the phases seen in Figure 4 were determined. (36% AlN + 3% WC) 61% Ni 1000 °C composite phases respectively WC, AlN ceramic phases as well as Ni metallic phase were obtained. In addition to the ceramic-metal phases, Ni₃B and Ni₂B, which are metallic borides, were determined.

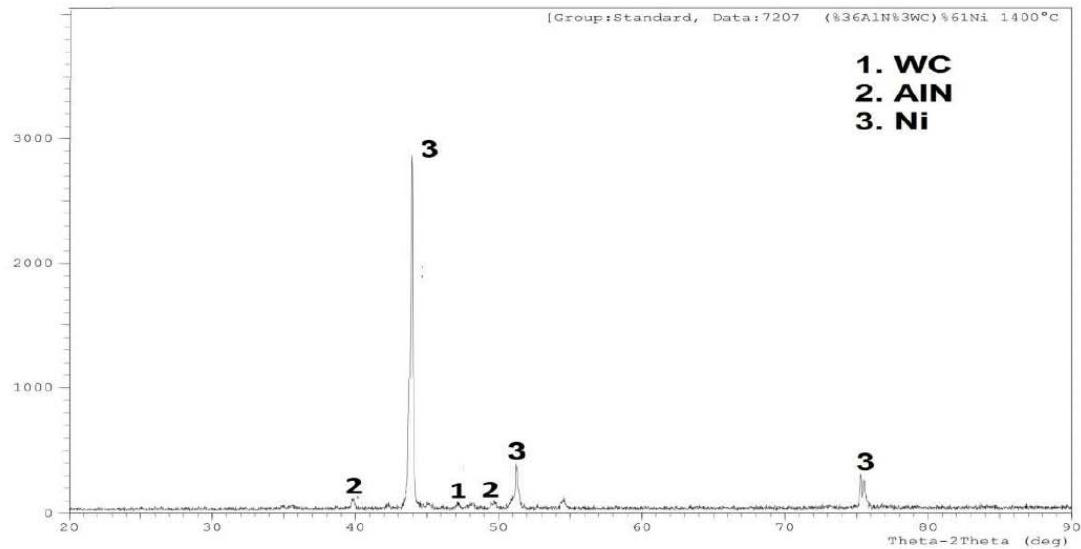
As the sintering temperature increased, grain boundaries became apparent and surface open porosity decreased. (36% AlN + 3% WC) 61% Ni 1400 °C composite X-ray analysis results, respectively, WC, AlN ceramic phases as well as Ni metallic phase was

obtained. In addition to the ceramic-metal phases, metallic borides, Ni₃B, Ni₄B₃ and Ni₂B were determined.

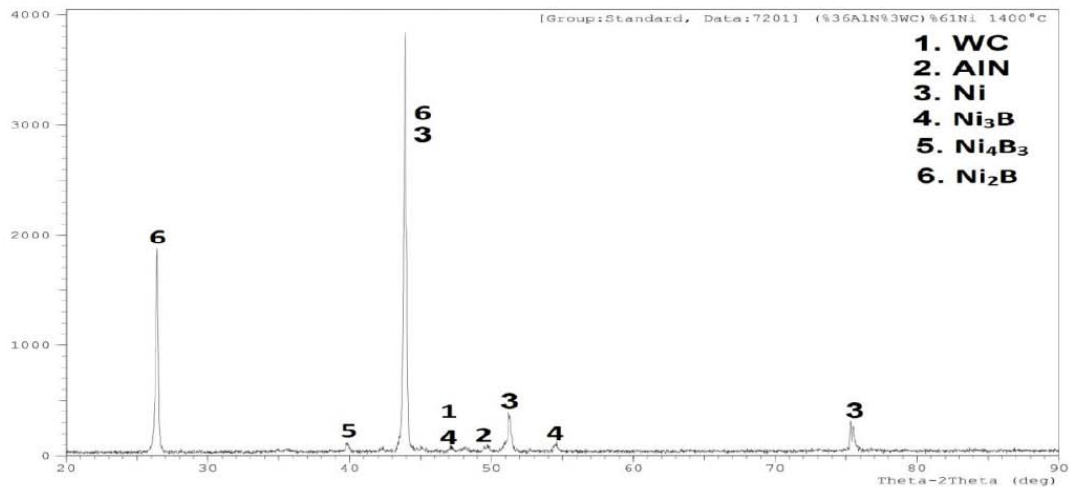
RESULTS AND DISCUSSION

The ceramic-metal composite samples produced were characterized after the box boring process and the following results were obtained. The highest density was obtained from the composites produced from sintered (36% AlN + 3% W) 61% Ni powders in conventional sintering furnace at 1400 °C. The highest density values were calculated as 7.42g / cm³ before boronizing and 14.85g / cm³ after boronizing at 1400 °C.

The highest micro hardness was measured at 1400 °C before boron 112.9 HV and after boron 1150.8 HV.



(a)



(b)

Figure 5: a) X-ray analysis of the composite (36% AlN- 3% WC) 61% Ni 1400 °C). b) X-ray analysis of the boronized composite (36% AlN- 3% WC) 61% Ni 1400 °C).

Ceramic-metal composites produced with electroless nickel plating technique show better mechanical properties besides sintering advantage.

It is less porous than the composites produced without coating and is expected to increase corrosion resistance. There is no need for electrical energy for chemical nickel plating in ceramic-metal composite production. This provides an efficient and cost effective coating process. Production with powder metallurgy does not require finishing. With electroless nickel plating technique, the thickness of the coating and the size of the coating volume provide flexibility in the samples produced. The recesses and pits of the particles to be coated can be easily filled.

Composites produced by conventional powder metallurgy, such as post-production boronizing and so on. The processes are advantageous in improving the mechanical properties of the materials.

CONCLUSIONS

At the end of this study, the surface hardness of the pencil pencils used in the soil industry was increased by applying box boring. Application of boronizing process brings additional cost to material production as disadvantage. Hardness values of commercial plotter pens used in the industry were measured in the range of 700-750HV. With the increase of surface hardness, the resistance of the exposed surfaces was increased to 1150HV. It is thought that better results can be

obtained at lower temperatures and in a shorter time if the box boring process is applied to composite samples in a microwave sintering furnace. As a result, boronizing of samples produced by powder metallurgy method, box boronizing method was performed. The borid layer formed on the composite has smooth surface morphology and Ni₃B, Ni₄B₃ and Ni₂B phases. The layer thicknesses are uniformly distributed over the sample surface. A hard borid layer has formed on the surface of the boron composites, so it is possible to use it in places that require high surface hardness. Borid layer is an abrasion resistant structure compared to composite material and can be preferred in places where abrasion resistance is required.

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REFERENCES

- [1] Yönetken A. Fabrication of Electroless Ni Plated Fe–Al₂O₃ Ceramic–Metal Matrix Composites. *Trans Indian Inst Met* 2015; 68(5): 675-681. <https://doi.org/10.1007/s12666-014-0497-1>
- [2] Yönetken A, Erol A. Sintering and Characterization of SiC Reinforced Ni Powders in Microwave Furnace. *International Journal of Engineering Research and Development* 2020; 12(1): 83-89. <https://doi.org/10.29137/umagd.474003>
- [3] Rajan TPD, Pillai RM, Pai BC. *Journal of Materials Science* 1998; 33: 3491. <https://doi.org/10.1023/A:1004674822751>
- [4] Lee DW, Yu JH, Yun JY, Turaev FR, Lim TH, Jang T. Preparation of TiC-Ni nanocomposite powder by a metallothermic chloride reduction. *Journal of Ceramic Processing Research* 2008; 9(4): 407-410.
- [5] Frage N, Froumin N, Dariel MP. Wetting of TiC by non-reactive liquid metals. *Acta Materialia* 2002; 237-245. [https://doi.org/10.1016/S1359-6454\(01\)00349-4](https://doi.org/10.1016/S1359-6454(01)00349-4)
- [6] Dong X, Hu J, Huang Z, Wang H, Gao R, Guo Z. Microstructure and Properties of Boronizing Layer of Fe-based Powder Metallurgy Compacts Prepared by Boronizing and Sintering Simultaneously. *Science of Sintering* 2009; 41: 199-207. <https://doi.org/10.2298/SOS0902199D>
- [7] Michalski J, Wejrzanowski T, Gierlotka S, Bielinski J, Konopka K, Kosmac T, Kurzydowski KJ. *J Eur Ceram Soc* 2007; 27: 831. <https://doi.org/10.1016/j.jeurceramsoc.2006.04.034>
- [8] Narayanan TSNS, Krishnaveni K, Seshadri SK. Electroless Ni–P/Ni–B duplex coatings: preparation and evaluation of microhardness, wear and corrosion resistance. *Materials Chemistry and Physics* 2003; 82: 771-779. [https://doi.org/10.1016/S0254-0584\(03\)00390-0](https://doi.org/10.1016/S0254-0584(03)00390-0)
- [9] Ling GP, Li Y. *Mater Lett* 2005; 59: 1610. <https://doi.org/10.1016/j.matlet.2004.12.054>
- [10] Luo LM, Yu J, Luo J, Li J. *Ceram Int* 2010; 36: 1989. <https://doi.org/10.1016/j.ceramint.2010.03.003>
- [11] Abe T, Akamaru S, Watanabe K. *J Alloys Compd* 2004; 377: 194. <https://doi.org/10.1016/j.jallcom.2003.12.053>
- [12] Brenner A, Riddell GJ. *F&S Nat Bur Stds* 1946; 37: 31-34. <https://doi.org/10.6028/jres.037.019>
- [13] Luoa L, Wua Y, Lib J, Zheng Y. Preparation of nickel-coated tungsten carbide powders by room temperature ultrasonic-assisted electroless plating. *Surface & Coatings Technology* 2011; 206: 1091-1095. <https://doi.org/10.1016/j.surfcoat.2011.07.078>
- [14] Iswadi J, Yusof HAM, Rozali S, Ogiyama H. Effect of Particle Sizes on the Development of Ultra Hard Surface Through Superplastic Boronizing of Duplex Stainless Steel *Journal of Solid Mechanics and Materials Engineering* 2007; 1: 539-546. <https://doi.org/10.1299/jmmp.1.539>
- [15] Xiaoyong R, Zhijian P, Ying P, Chengbiao W, Zhiqiang F, Longhao Q, Hezhao M. Ultrafine binderless WC-based cemented carbides with varied amountsof AlN nano-powder fabricated by spark plasma sintering. *Int Journal of Refractory Metals and Hard Materials* 2013; 41: 308-314. <https://doi.org/10.1016/j.jrmhm.2013.05.002>
- [16] Ravichandran M, Dineshkumar S. Experimental investigationson Al-TiO₂-Gr hybrid composites fabricated through stir castingroute. *Materials Testing* 2016; 58(3): 211-17. <https://doi.org/10.3139/120.110839>
- [17] Tarragó JM, Coureaux D, Torresc Y, Casellad D, Al-Dawerye I, Schneidere L, Llane L. Microstructural effects on the R-curve behavior of WC-Cocemented carbides. *Materials and Desing* 2016; 97: 492-501. <https://doi.org/10.1016/j.matdes.2016.02.115>
- [18] Bilici ÖV, Sarpün IH, Kılıçkaya MS. The Relationship of Thermal and Elastic Properties with Ultrasonic Wave Velocity of WC/Co-Ti Composites. *Afyon Kocatepe University International Journal of Engineering Technologies and Applied Sciences* 2019; 2(1): 20-28.
- [19] Topuz P, Aydogmuş T. Investigation Of Wear Behavior Of Boronized AISI 316 Stainless Steel. *Omer Halisdemir University Journal of Engineering Sciences* 2019; 8(2): 1105-1114.
- [20] Azaklı Y, Tarakçı M. Microstructural characterisation of borided binary Fe–W alloys. *Surface Engineering* 2018; 34(3): 226-234. <https://doi.org/10.1080/02670844.2016.1263712>
- [21] Dokumacı E, Özkan I, Önay B. Effect of boronizing on the cyclic oxidation of stainless steel. *Surface & Coatings Technology* 2013; 232: 22-25. <https://doi.org/10.1016/j.surfcoat.2013.04.047>

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