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STRUCTURAL AND MECHANICAL CHARACTERIZATION OF TUNGSTEN NITRIDE COATINGS PREPARED BY DC SPUTTERING AT CYLINDER LINER

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ABSTRAK

Dinding silinder adalah tabung yang berfungsi untuk melindungi piston dan sebagai tempat berlangsungnya proses pembakaran. Proses pembakaran menyebabkan gesekan secara terus menerus membuat dinding silinder menjadi aus. Maka dari itu dinding silinder harus terbuat dari bahan yang memiliki kekerasan tinggi. Surface treatment dengan teknik DC sputtering dilakukan dengan tujuan memperoleh tingkat kekerasan yang tinggi. Surface treatment dengan teknik DC sputtering selama 120 menit dengan variasi gas Argon dan gas Nitrogen 70:30, 80:20 dan 90:10. Berdasarkan hasil uji XRD semua sampel yang di treatment terdapat lapisan WN. Struktur kristal sampel raw material adalah hexagonal dan struktur kristal semua sampel yang di treatment yaitu kubik. Karakterisasi kekerasan vickers hardness menunjukkan untuk raw material sebesar 110,93 HV. Sedangkan pada sampel yang di treatment Ar70:N30 sebesar 234,66 HV. Kekerasan optimum terjadi pada sampel yang di treatment Ar80:N20 dengan kekerasan 311,87 HV. Namun pada treatment Ar90:N10 mengalami penurunan kekerasan menjadi 130,44 HV. Berdasarkan hasil pengujiam lapisan tipis, WN berpengaruh terhadap peningkatan kekerasan material dinding silinder. Hal ini dibuktikan dengan terjadinya peningkatan kekerasan pada sampel yang dilapisi dengan WN.

Kata kunci: dinding silinder, DC sputtering, surface treatment.

ABSTRACT

The cylinder liner is a tube that protects the piston and is a place for the combustion process. The combustion process causes continuous friction to make the cylinder liner wear out. Therefore the cylinder liner must be made of materials that have high hardness. To obtain a high hardness level, surface treatment with DC sputtering techniques is carried out. Surface treatment with DC sputtering technique for 120 minutes with variations of Argon gas and Nitrogen gas 70:30, 80:20, and 90:10. Based on the XRD test results, all treated samples contained a WN layer. The crystal structure of the raw material samples is hexagonal, and the crystal structure of all processed samples is cubic. Vickers hardness characterization shows that the raw material is 110.93 HV. While the sample treated with Ar70:N30 was 234.66 HV. The optimum hardness occurred in samples treated with Ar80:N20 with a hardness of 311.87 HV. However, the Ar90:N10 treatment experienced a decrease in hardness to 130.44 HV. Based on the results of thin film testing, WN affects the increase in the hardness of the cylinder liner material. This is evidenced by the increase in hardness in samples coated with WN.

Keywords: *cylinder liner, DC sputtering, surface treatment.*

1. INTRODUCTION

The development of facilities and infrastructure over time is increasing, especially the need for two-wheeled and four-wheeled transportation facilities. Motor fuel has enormous benefits for human life, especially as a means of transportation. For the combustion engine to work perfectly, the cylinder wall must be made of a material that has a high hardness. The cylinder wall is a tube that serves to protect the piston and as a place for the combustion process to take place [1].

Gray cast iron is a material that is widely used in engine components, especially cylinder liners [2]. Gray cast iron has a high hardness, so it is widely applied to machine tools such as cylinder walls for internal combustion engines [3]. However, the friction between the piston and cylinder walls causes compression leakage [4]. This leak results in reduced engine power and incomplete combustion resulting in smoke in the motorcycle exhaust [5].

To increase the hardness of the cylinder wall material, a surface treatment method is needed to improve the surface so that it has high hardness properties. When a solid surface is bombarded with energized ions, the atoms of the solid surface are scattered due to the collision between the surface atoms and the energy particles. This phenomenon is called DC sputtering. The DC sputtering technique is a development of the coating technique, where the difference lies in the DC sputtering technique does not undergo a heating process that results in melting so that when a collision occurs on a high-voltage particle, there is an exchange of momentum which results in the material being thrown from a solid. It is very advantageous to deposit materials with a high melting point and are stronger attached because the atoms can enter deeper into the surface of the material so that the service life is longer [6]. Another advantage of the sputtering technique is that it can provide even deposition [7].

Tungsten Nitride has good hardness, chemical stability, thermal stability, high electrical and thermal conductivity, and wear-resistant protective coating on mechanical components, so it is widely used for thin coatings on substrates [8]. This research aims to perform treatment with the DC sputtering technique with variations of Argon gas and Nitrogen gas to obtain high hardness on the cylinder wall. The cylinder wall was cut with a length of 10 mm, a width of 10 mm, and a thickness of 2 mm as the research sample. Before surface treatment, the cylinder wall material is tested for X-Ray Fluorescence (XRF) composition to determine its elemental content. The cylindrical wall samples will be tested for crystal structure using X-Ray Diffraction (XRD) and hardness using the Vickers Hardness method.

2. METHODOLOGY

In this study, samples were prepared from the cylinder liner material of motorcycle, which was cut using a milling machine. Sample in the form of a cube with a length of 10 mm, a width of 10 mm and a thickness of 2 mm. The samples were smoothed using sandpaper as much as eight levels of roughness from 400 until 5000 mesh with a wet sanding technique. The next stage is the process of polishing to a shine with an aerosol on the velvet fabric. This sanding and polishing process is carried out to prevent damage and surface changes due to excessive heating [20]. The overall appearance can be seen in Figure 1.

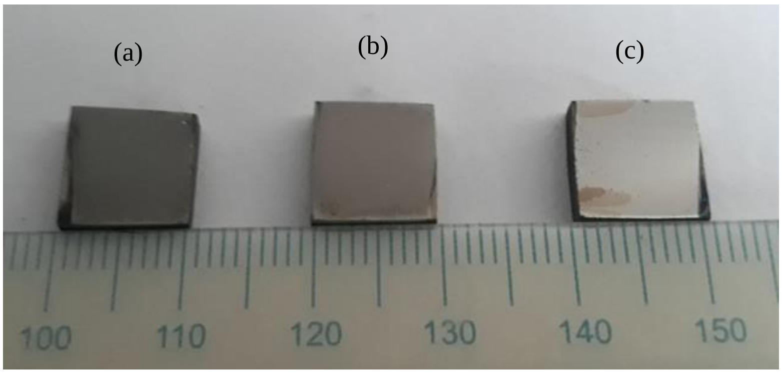


Figure 1. Samples of WN deposition at 120 minutes with variations in gas Ar:N (a) Ar70:N30, (b) Ar80:N20, (c) Ar90:N10

The prepared sample will be tested for composition first to determine the elemental composition of the cylinder liner which is the raw material using XRF equipment. This composition test is an early stage of testing before the treatment process with DC Sputtering. The prepared sample will be tested for composition first to determine the composition of the cylinder liner wall (cylinder liner) using XRF. The prepared sample was deposited with tungsten nitride using the DC sputtering technique. The sputtering process is carried out using a DC sputtering device at PSTA BATAN Yogyakarta.

(a) **(b)**

Figure 2. (a) DC Sputtering Device, (b) Reactor (Chamber)

The deposition process begins with the process of exhausting the reactor tube (chamber). Exausting is done to ensure the cleanliness of the reactor from residual gas. Vacuuming of this reactor tube to a pressure of 10-5 Torr. Argon gas and nitrogen gas flowed into the reactor tube. The gas flow rate is regulated using a flow meter. The WN deposition process uses a fixed variable form the voltage and current are also increased slowly until they reach ±5 kV and 10 mA, so that a tube pressure of ±2.1 × 10-2 Torr is obtained. Tungsten nitride (WN) deposition for 120 minutes. The variables that change in the research are variations in the ratio of Argon gas and Nitrogen gas are 70:30, 80:20, and 90:10. raw materials and samples that have been treated with the DC sputtering technique are characterized. This characterization includes crystal structure using XRD and hardness test using Vickers hardness tester. The Vickers result is shown in Figure 3.

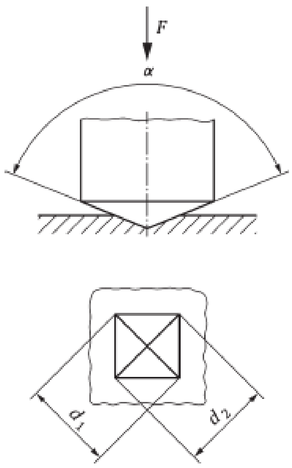


Figure 3. Vickers hardness indentation measurement (ISO 6507-1, 2018)

The hardness of the material using the Vickers method can be calculated by equation 1 below.

$$HV = 1.8544 \, . \, P/d^2 \quad (1)$$

Where HV is the Vickers hardness, P is the indenter load (KGF), and d is the diagonal length of the indent (mm) [18].

3. RESULTS AND DISCUSSIONS

X-Ray Fluorescence (XRF) is a test tool used to analyze the sample elements qualitatively. The qualitative analysis provides information on the elements in the sample. According to SAE Standard J431 on the chemical composition of gray cast iron in automotive applications, the main elements of gray cast iron must be Carbon, Manganese, Silicon, Phosphorus, and Sulfur. Table 1 is the result of the composition test of the cylinder wall sample without complete treatment. From the test results, this elemental composition can be used to predict the phases that may appear in XRD analysis.

Table 1. Test results of elemental composition

Composing Elements	Symbol	Atom Number	Percentage (%)
Iron	Fe	26	94.504
Silicone	Si	14	1.510
Manganese	Mn	25	0.330
phosphorus	P	15	0.240
aluminum	Al	13	0.324
Promethium	Pm	61	0.277
Europium	Eu	63	0.290
Gadolinium	Gd	64	0.463
Terbium	Tb	65	1.532
Holmium	Ho	67	0.145

Figure 4 is a visualization of gray cast iron taken through a microscope with the help of a camera. Based on the standard (ASTM A247-16a), the type of graphite is Type VII Class 3 Graphite type VII in the form of flakes of an iron gray color. While class 3 is categorized based on the graphite particles formed. The actual dimensions of the particles in class 3 are 320 to < 640 m. It is appropriate that the cylinder wall sample used is a gray cast iron sample. The black line in the image represents graphite flakes which will physically provide strength. The finer and more evenly distributed the graphite flakes, the stronger the metal [2].

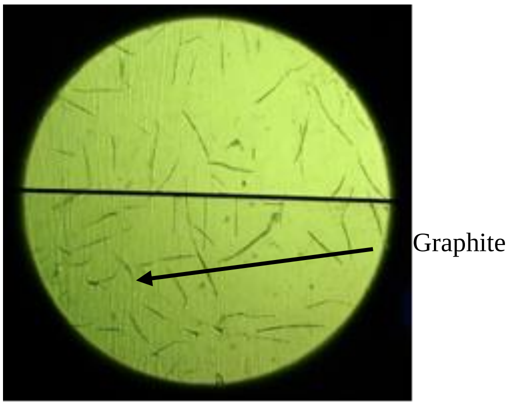


Figure 4. Visualization of gray cast iron

Crystal structure characterization has been carried out using X-Ray Diffraction (XRD) in BATAN. The source used is Co-Ka1, with a wavelength of 1.789010. Characterization was carried out on the raw material and three samples which were treated with a sputtering technique using a gas ratio of Ar:N 70:30, 80:20, and 90:10. The XRD result data was analyzed using the Match!3 application. This qualitative analysis only knows the phase formed by matching the measurement data to the database from the Crystallography Open Database (COD) Match!3 programs.

Based on Figure 5, the raw material obtained Epsilon Iron Carbide (Fe2.4C) phase with a cubic crystal structure at the highest peak obtained at the appropriate hkl plane orientation is (111) at 2θ=50.68º, then (110) with 2θ= 44.31º, and (201) at 2θ=57.52º. In the Ar10:N30 treatment, the highest intensity was obtained successively at an angle of 2θ=51.41º in the corresponding plane (200), 2θ = 44.11 at (111), and 2θ=57.98º plane (201) with a crystal structure cubic. The Ar80:N20 treatment showed the highest peak with an intensity of 147.49 in the respective orientations (111), (200), and (202). The crystal structure formed in this treatment is hexagonal. This is similar to the structure in the Ar90:N10 treatment, which shows the plane's suitability starting from the highest peak in the (200) plane with 2θ=51.32º.

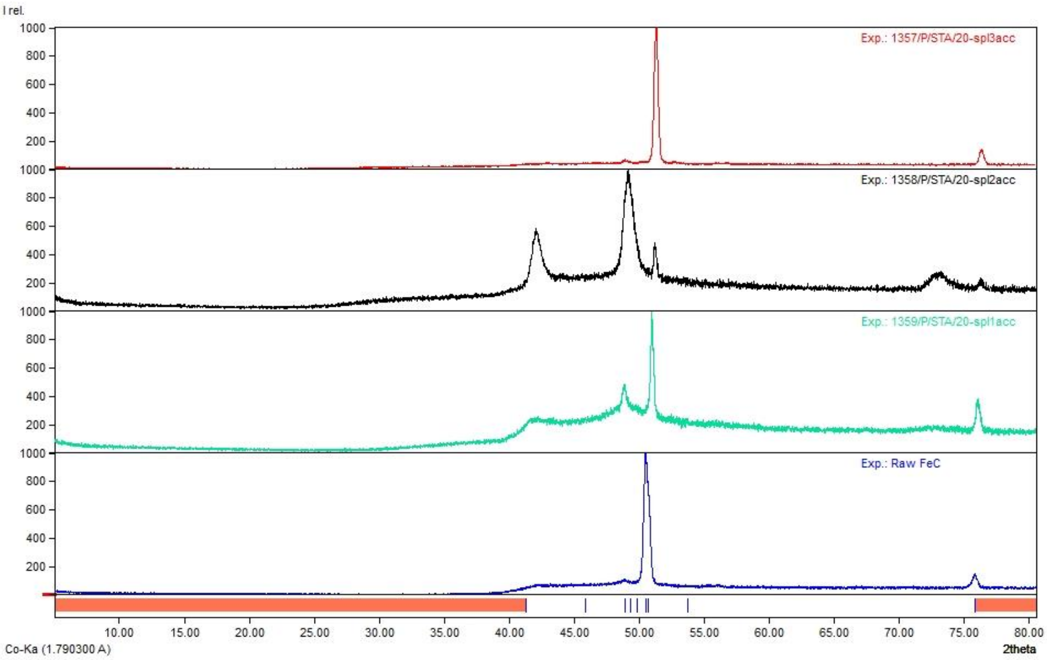
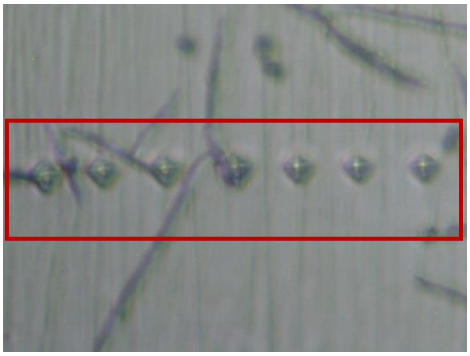
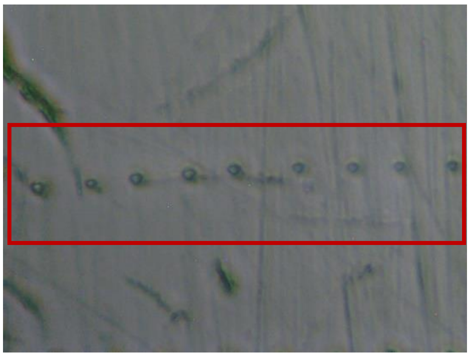


Figure 5. XRD diffractogram of test sample

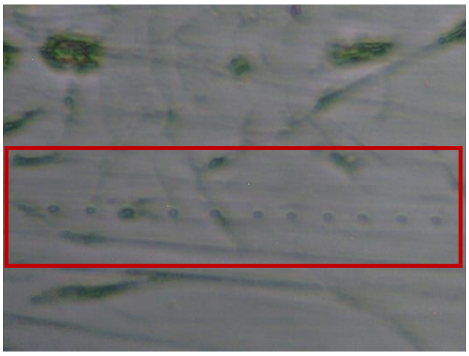
The results of the analysis of the raw material had a Fe2.4C phase, samples treated with Ar70:N30 formed a W3N4 (cubic) phase, and samples treated with Ar80:N20 and Ar90:N10 had a WN (hexagonal) phase. It can be seen in Figure 5 that there was a significant change in the samples treated with WN with Ar70:N30 and Ar80:N20. Meanwhile, the samples treated with Ar90:N10 had the same peak as the raw material. The addition of the number of peaks affects the mechanical properties, especially in this study discussing the hardness of the material. This is related to the factor that the higher the level of hardness of a material will cause the level of engine wear to smaller.



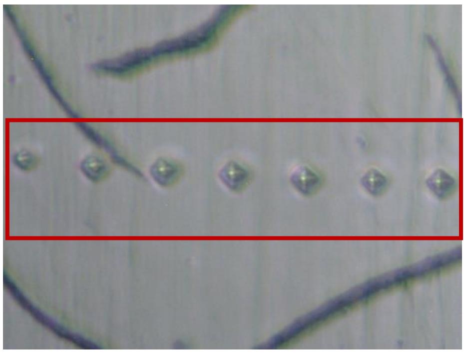
(a)



(b)



(c)



(d)

Figure 6. Vickers hardness test results indentation of WN deposition at 120 minutes with Ar: N . gas variation (a) Raw Material, (b) Ar70:N30, (c) Ar80:N20, (d) Ar90:N10

Hardness test using Vickers Hardness Tester Matsuzawa type MMT-X7. The working principle of Vickers hardness is to make a trace using a diamond pyramid on the sample so that the traces of the diagonal indenter traces from this trace will be measured and then calculated by Equation 1. The hardness test material in this study is a sample of raw material and a sample that is sputtered at 120 minutes with the variation of Argon gas versus Nitrogen gas 70:30; 80:20, and 90:10.

Figure 6. (a) and (d) have a wider footprint than Figure 6. (b) and (c). A wide indentation mark means that it has low hardness. On the other hand, small indentation traces have high hardness. Hardness data were collected at seven different points. This is because the mechanical properties, such as the hardness of the material, are not homogeneous on the surface layer of the cylinder wall material.

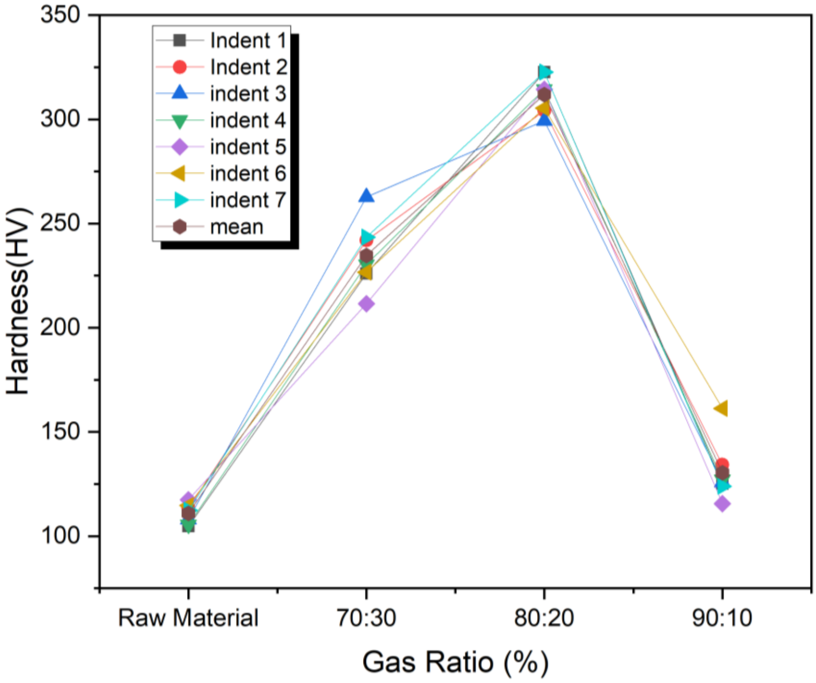


Figure 7. Graph of hardness test results

Figure 7 is a graph of the hardness test results of a cylinder wall sample using a 5 Gf load. It can be seen that the hardness increased from the raw sample of 110.93 HV to 234.66 HV in the sample treated with the sputtering technique at a ratio of Argon 70 gas and Nitrogen gas 30. The increase in hardness occurred because the cylinder wall sample formed a thin layer of WN, so The phases formed are the Fe2.4C phase and the WN phase. the formation of carbide ε (Fe2,4C) causes an increase in material hardness[25]. The optimum hardness occurred in the sample with a comparison parameter of Argon 80 gas and Nitrogen gas 20, 234.66 HV. This is because the WN layer formed is thick enough to stick to the substrate [22]. According to [23], the nitrogen atoms are arranged more closely under optimum conditions and have a perfect arrangement. In samples treated with Ar90:N10, there was a decrease in the level of hardness. This is because the deposited substrate is already saturated. This statement is supported by research conducted by [24] that samples that have passed the optimum will experience a decrease in hardness level.

4. CONCLUSION

Testing the composition using XRF showed that the cylinder wall material without treatment contains an alloy of the main elements of Iron, Silicon, Manganese, and Phosphorus, as well as other impurities. Analysis of hardness using the Vickers hardness method has increased after treatment compared to raw material. The XRD results show that the crystal structure for the raw material is hexagonal, but after treatment, the crystal structure changes to cubic.

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