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Investigation on the Effect of Coating Temperature on the Properties of NbC Layer Coated with Pack Cementation Technique on GGG70 Nodular Graphite Cast Iron Surface

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ABSTRACT: Nodular Graphite Cast Irons stand out with their high castability, high strength, vibration damping and high loading capacity. With today's technologies, the surface properties of materials can be improved by coating them with various methods and high-performance engineering materials can be obtained. Pack Cementation Technique is preferred among coating methods due to its relatively lower cost and applicability. In this study, it was aimed to coat NbC on the surface of GGG70 Nodular Graphite Cast Iron using the Pack Cementation Technique at 900 °C, 1000 °C and 1100 °C for 6 hours. The effect of temperature on the properties of the resulting coating layers was investigated. For this purpose, the surface morphologies of the samples were examined and their fracture toughness and hardness values were obtained. Coating morphologies were examined by XRD, optical microscope and SEM analysis and changes in coating structure and thickness were obtained. When the results were examined, it was determined that the coating thickness increased with the increase in coating temperature. Accordingly, it was observed that the fracture toughness value of the coatings first increased and then decreased slightly. Microhardness values increased approximately 5 times in the coating areas.

Keywords: Nodular Graphite Cast Iron, NbC Coating, Fracture Toughness, Microhardness.

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1. INTRODUCTION

Increasing technological needs and their corresponding developments lead to important discoveries in the field of materials (Fernandes et al., 2018). In the industry, efforts are being made to increase the service life of materials by improving their surface properties, wear resistance, corrosion and oxidation resistance and friction properties (Kan et al., 2018; Chen et al., 2021a, 2021b; Erdoğan, 2019; Okay et al., 2010; Günen et al., 2023a, 2023b, 2023c). For this purpose, creating a hard and thin film coating on the surface is a good option (Karamış and Yıldızlı, 2010; Okay et al., 2010). Carbides such as niobium carbide, vanadium carbide etc. are obtained on metal material surfaces by forming thin films through various processes such as pack cementation technique, laser coating and reactive spraying etc. (Cai and Xu, 2017; Chen et al., 2021). The pack cementation technique is relatively less complex and less costly than other methods (Cai and Xu, 2017).

Nodular graphite cast irons are used as an alternative to steels in the industry and are preferred due to their properties close to steels (Fernandes et al., 2018; Okay et al., 2010). In addition, its ability to be produced more economically compared to steels and its high castability properties provide an additional advantage (Karaca and Şimşir 2019). Nodular graphite cast irons are obtained by adding elements such as magnesium and cerium to the molten metal and the graphite solidifying into spheroids. Thus, an alloy with superior strength, fatigue resistance, toughness, ductility and vibration damping properties is obtained compared to other alloys. With these properties, Nodular graphite cast irons are used in many areas such as hot rolling rolls, aluminium melting crucibles, molds for the glass industry, gears and hydraulic presses. In addition, although they are used in many areas, their wear resistance is not sufficient in places where wear resistance is required. Therefore, in order to increase the wear resistance of nodular graphite cast iron material surfaces, the surfaces have been tried to be improved by methods such as thermal spray, laser, pack cementation technique, etc. and various heat treatments (Megahed et al. 2019), and positive results have been reported (Günen et al.,2022).

Owing to their important physical and chemical properties, carbides of transition metals have found a place in cutting tool coatings, aerospace, and many industrial applications (Peng at al.,2021).The pack cementation technique is based on the principle that the carbon atom spreads to the surface with high temperature and forms carbides on the surface with elements such as Cr, Nb, V. Thanks to this method, depending on the structure of the carbide layer formed, very hard layers can be obtained on the surface without any deterioration in the main structure of the material. Thus, while the wear and hardness values of the material are increased, its toughness properties are preserved against external impacts (Günen et al., 2022). It has been stated that in this method, transition metal carbide coatings cannot be obtained in alloys with C concentration below 0.3% (Günen et al., 2023c).

Niobium carbide (NbC), is considered an ideal candidate as a coating material thanks to its resistance to corrosion and wear (Zhong et al., 2020; Peng et al.,2021). NbC, which has high hardness (35GPa) and high melting point (3600 °C)(Cai et al., 2015a), can be deposited from the surface to the depths of Fe-based materials, and the hardness and elastic modulus values of the material are increased (Zhong et al., 2020). Various studies have been carried out on the formation mechanism and microstructure of the NbC coating layer. With the improvement of the morphology of the coating layer, the surface properties will change significantly (Cai et al., 2015b).

A few studies have been conducted in the literature involving the coating of NbC coatings on steel materials (Günen et al., 2023b.). A small number of NbC coating studies on cast irons with the TRD method have been encountered. In their study, Mariani et al. (Mariani et al., 2020) obtained 3 different coatings, namely NbC, VC and NbVC, on the surface of lamellar graphite cast iron in a salt bath at 1000 °C for 2 hours with the thermoreactive diffusion method. It has been reported that the hardness of the coating was obtained above 2300, 2500 and 3050 Hv, respectively. Cai et al. In their study, (Cai et al., 2016) formed NbC coatings on the gray cast iron surface using the in-situ method. It was reported that the coating thickness was approximately 21μm and the coating hardness was 21 GPa.

In this study, NbC layer was formed on GGG70 nodular graphite cast iron surfaces at 900 °C, 1000 °C and 1100 °C for 6 hours. The effects of coating temperature on the properties of the formed NbC layers were examined. For this purpose, the changes in coating morphology, fracture toughness and hardness values were observed and the results were compared.

2. MATERIALS AND METHODS

In this study, GGG70 spheroidal graphite cast iron samples, whose elemental content is given in Table 1, were cut into 15x15x10 mm dimensions and used as the substrate material to be coated. All surfaces of the samples to be coated were first sanded with 120-400-800 sandpaper and then washed with soap and water to remove oil and dirt.

Table 1. Elemental content of GGG70 nodular graphite cast iron samples.

	Wt%								
Material	**C**	**Si**	**Mn**	**Cu**	**Cr**	**S**	**P**	**Mg**	**Fe**
GGG70	3.47	2.41	0.17	0.07	0.03	0.01	0.02	0.05	Balance

The samples, whose surface water was removed with alcohol, were placed in steel crucibles with coating powder consisting of a powder mixture of 45% Fe-Nb + 45% Al_2O_3 + 10% NH_4Cl. In order to minimize contact with air, activated carbon powder was added to the mixture and then the mouth of the crucible was covered with ceramic clay pastes. The prepared crucibles were kept in a high temperature furnace (Carbolite CWF1200) for 6 hours at 900 °C, 1000 °C and 1100 °C, respectively. The heating rate was 15 °C/min from room temperature to coating temperatures, and then the samples were cooled in an open air environment. The samples, which had a coating layer on their surface, were cleaned of dust and dirt, their surfaces were sanded with 120-400-800-1200 mesh sandpaper, and then they were polished and etched with 3% Nital solution. The fracture toughness (K_C) of the NbC coating layer formed on the surface was estimated using Equations 1 and 2, taking into account the crack lengths developing from the corners of the micro hardness trace taken from 3 different regions (Kulka et al., 2017). Future-Tech (FM 700) microhardness device was used for fracture toughness and microhardness measurements. Nikon (MA 100) microscope and Clemex (image analysis system) image analysis system were used to determine the phase structure, crack lengths and coating thicknesses. The following equations were used for fracture toughness calculation.

$$K_C = A.P/c^{3/2} \tag{1}$$

$$A = 0.028\ (E/H)^{1/2} \tag{2}$$

where A is the residual indentation coefficient, P is the load, c is half the notch crack length, H is the hardness, and E is the Young's modulus. The obtained E_{NbC} values are approximately 435 GPa. (Zhao et al., 2017)

3. RESULTS AND DISCUSSION

3.1 Characterization of the Coating Layer

SEM microstructure photographs of samples coated with NbC by the pack cementation method at 900 °C, 1000 °C and 1100 °C for 6 hours are given in Figure 1. The NbC layer formed on the surface has a homogeneous and flat morphology. The layer/matrix interface appears quite flat. It is seen that the layer thickness increases with the increase of the coating temperature. However, in the sample coated at 1100 °C, the coating thickness reached the highest values and pore-like pits were formed. Günen et al. (2023a) observed similar pore-like pits on the coating surface due to the diffusion process when they applied niobizing process to AISI D3 steel at 1000 °C for 6 hours in their study. As a result of point EDS analysis, they determined that the pit regions had lower Nb and C ratios than other regions. (Günen et al., 2023). When the coating temperature was 1100 °C and 6h, due to the high diffusion time and temperature, it caused an indentation to grow towards the substrate material in the coating line and the formation of pore-like structures in the coating morphology (Figure 1-c).

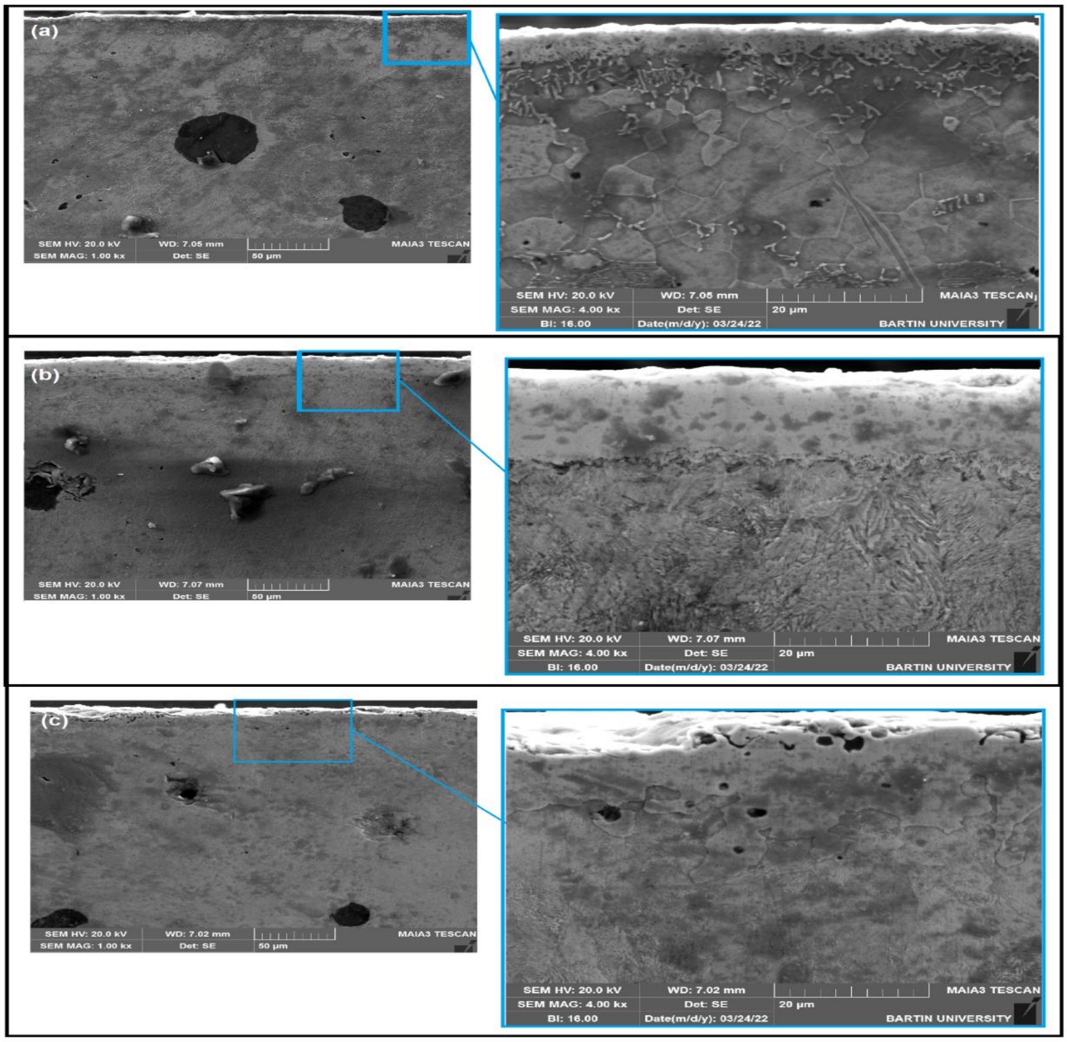

Figure 1. SEM images of NbC coated samples at a) 900 °C, b) 1000 °C and c) 1100 °C for 6 hours.

XRD analysis results taken from the surfaces of the obtained NbC coated samples are given in Figure 2. The diffraction peaks of the samples show that a layer consisting of NbC (ICDD card No. 00-038-1364) phase and α-Fe (ICDD card No. 01-087-0721) is formed, in accordance with the literature (Mariani et al., 2020; Günen et al., 2023a). In all samples, the most dominant peaks were observed between 2θ=30° and 60°. In the sample coated at 900 °C, the most dominant phase was the (111) plane NbC phase and the second dominant phase was the (110) plane α-Fe phase (Figure.2-a). In the sample coated at 1000 °C, the most dominant phase was the (111) plane NbC phase and the second dominant phase was the (200) plane NbC phase, and the (110) plane a-Fe phase intensity decreased slightly (Figure.2-b). In the sample coated at 1100 °C, the most dominant phase was the (110) plane α-Fe phase and the second dominant phase was the (200) plane NbC phase, and the intensity of the (111) plane NbC phase decreased slightly (Figure 2-c). As the coating temperature increased from 900 °C to 1000 °C, the intensity of the (110) plane α-Fe phase first decreased, but in the sample coated at 1100 °C, it increased and became the most dominant peak.

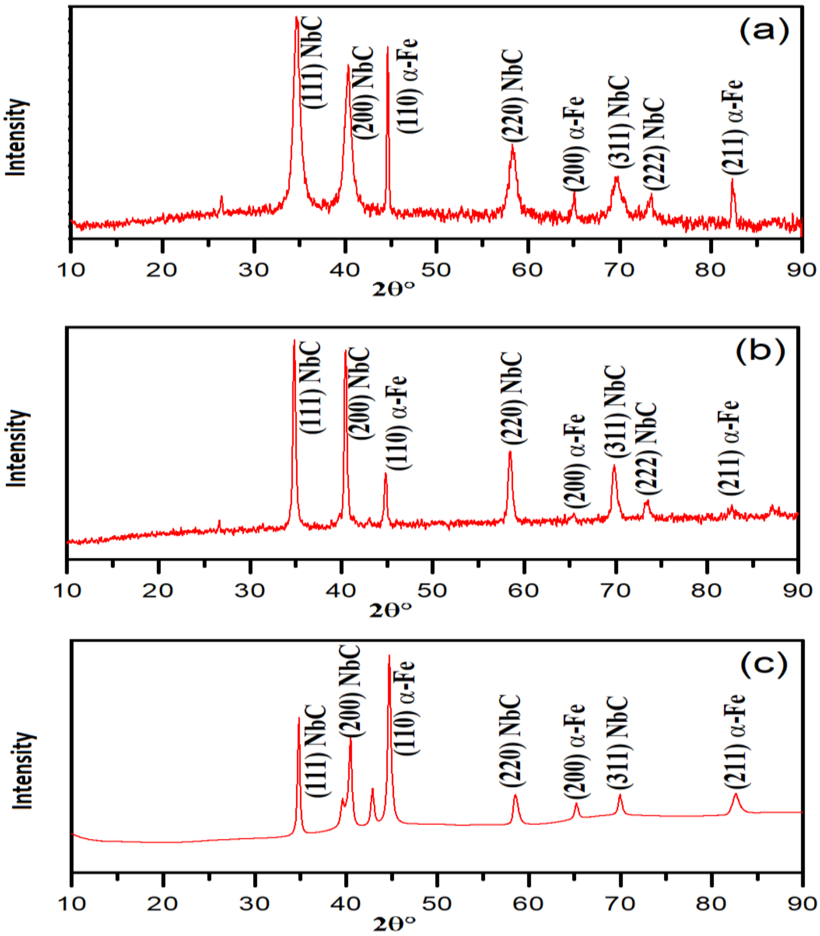

Figure 2. XRD analysis graphs taken from the coating surface of samples coated at a) 900 °C, b) 1000 °C c)1100 °C for 6 hours.

Linear elemental analysis graphs taken from the surface of the coating samples to the substrate material are shown in Figure 3. In the sample coated at 900 °C (Figure.3-a), it is seen that wt% Nb decreases from the coating to the substrate material and wt% Fe suddenly increases in the substrate material. A similar situation is observed in the sample coated at 1000 °C (Figure 3-b). However, it is observed that the wt% Nb and C ratio in the coating layer in the sample coated at 1000°C is higher than in the sample coated at 900°C. In the sample coated at 1100 °C (Figure.3-c), it is seen that wt%

Nb decreases from the coating to the substrate material and wt% Fe suddenly increases in the substrate material. However, in the sample coated at 1100 °C, it is seen that the wt% Nb ratio is lower and the wt% Fe ratio is higher at the coating line compared to the samples coated at 900 °C and 1000 °C. This is compatible with the XRD results.

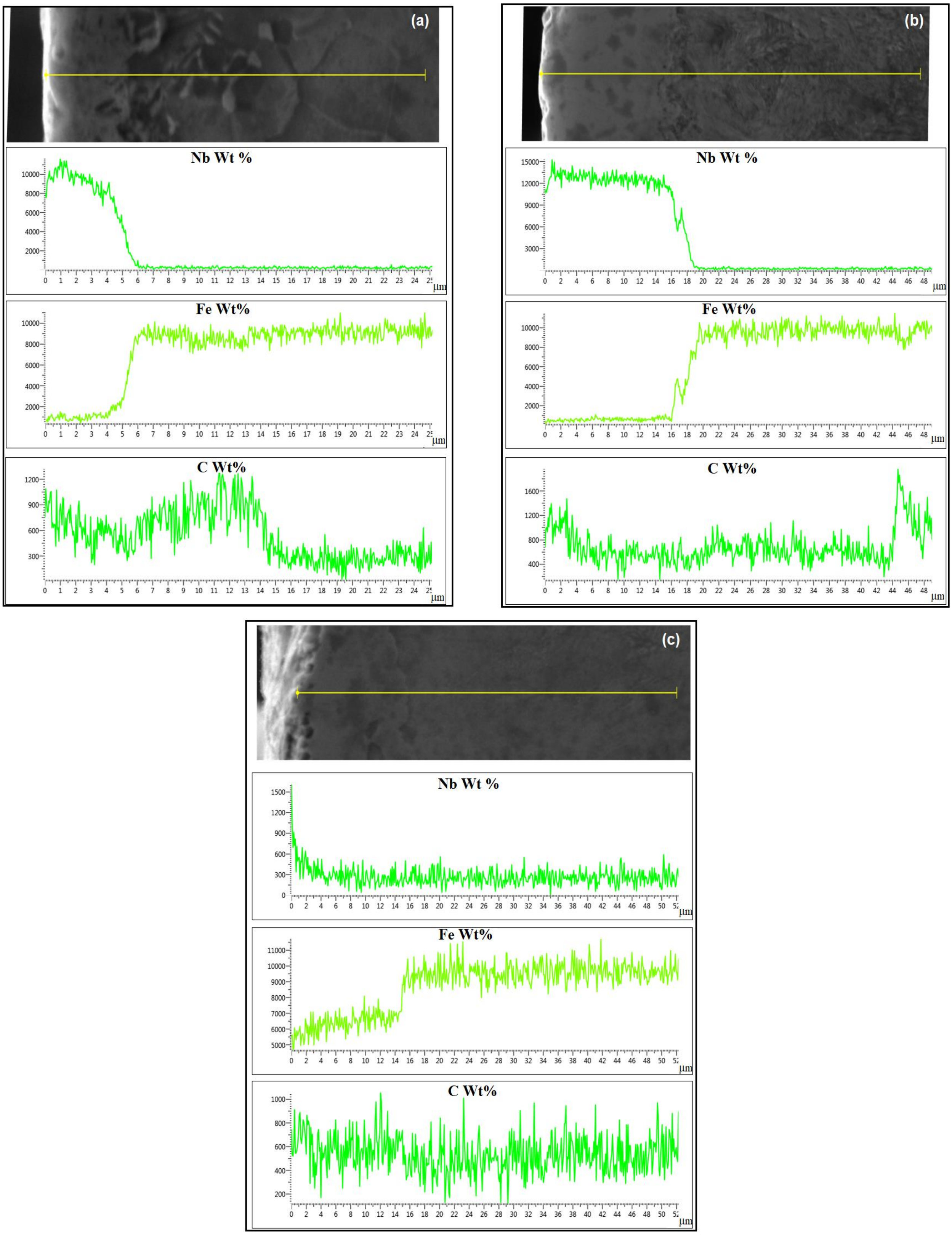

Figure 3. Linear elemental analysis images taken from samples coated with NbC at a) 900 °C, b) 1000 °C c)1100 °C.

3.2 Thickness, Fracture Toughness and Hardness of NbC Layer

NbC layer thickness values and Fracture toughness values of steel coated with pack cementation technique at different temperatures are given in Table 2. Additionally, the optical microstructures of NbC layer thicknesses are shown in Figure 4. The fracture toughness (K_C) of the NbC coating layer formed on the surface was estimated by taking into account the crack lengths developing from the corners of the microhardness trace.

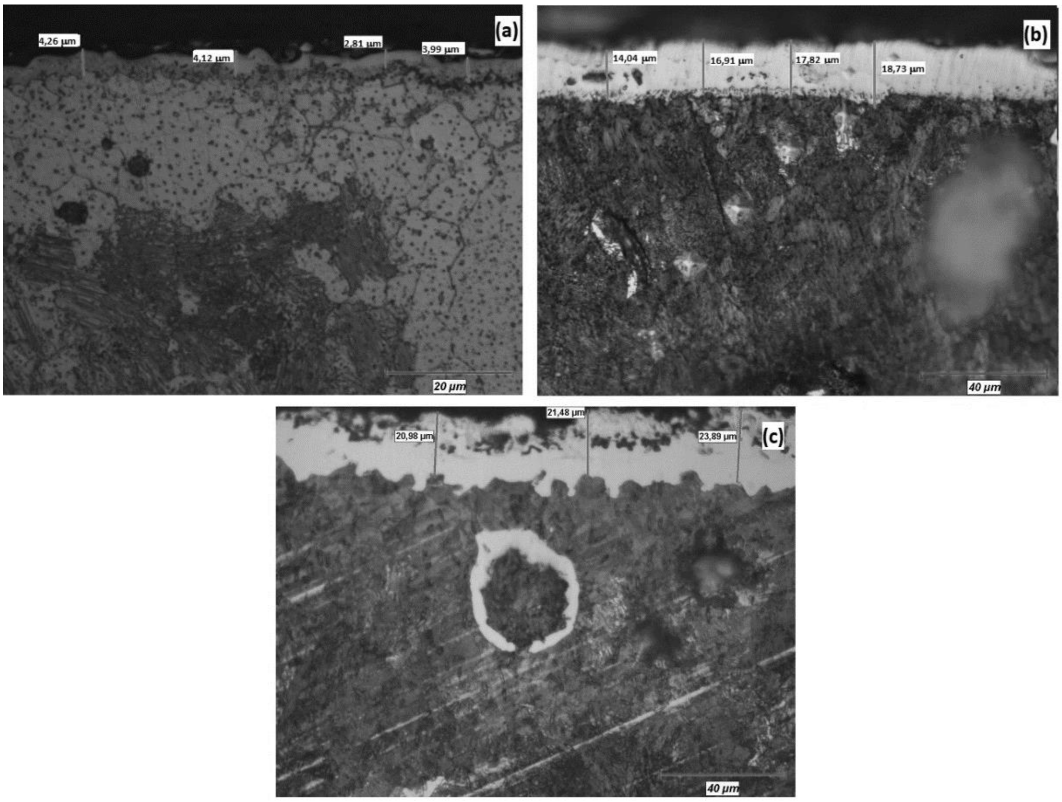

Figure 4. Optical microstructure images taken from samples coated with NbC at a) 900 °C, b) 1000 °C c)1100 °C.

With the increase in coating temperature, coating thicknesses increased and were obtained as an average of 3.79 µm, 16.87µm and 22.12 µm at 900 °C, 1000 °C and 1100 °C, respectively. In addition, fracture toughness values increased from 1.47 MPa $m^{1/2}$ to 2.61 MPa $m^{1/2}$ as the coating temperature increased from 900 °C to 1000 °C. However, even though the coating thickness increased in the sample coated at 1100 °C, the fracture toughness decreased to 2.25 MPa $m^{1/2}$.

Table 2. Thickness and fracture toughness values of GGG70 nodular cast iron coated with NbC at different temperatures.

Coating Temperature (°C)	Coating Thickness (µm)	Fracture Toughness (K_C (MPa $m^{1/2}$))
900	3.79±0.67	1.47±0.2
1000	16.87±2.03	2.61±0.3
1100	22.12±1.56	2.25±0.2

Microhardness measurements were made under a 25 gf load from the cross-sectional surface of the coated samples to a certain depth and the hardness of the NbC layer and matrix regions was determined. The microhardness measurements were determined by taking the order of hardness from the substrate material to the coating surface (Figure 5).

By coating the GGG70 nodular cast iron surface with NbC using the pack cementation method, the surface hardness increased approximately 5 times. As seen in Figure 5, the hardness distribution of GGG70 nodular cast iron towards the coating surface can be seen. In the hardness distribution of the formed NbC layer from the surface, a high hardness value is obtained throughout the NbC layer and a sudden decrease is observed when it reaches the matrix. Depending on the thickness of the coating layer, the hardness values are seen to be higher than the substrate material. It has been reported in the literature that microhardness values will increase with increasing coating temperature (Günen et al., 2023a). It was observed that the microhardness values increased as the coating temperature increased from 900° to 1000°C and 1100°C in the NbC coating layers formed on the GGG70 nodular graphite cast iron surface. Although the hardness values of the coating obtained at 1100 °C are close to the hardness value of the coating layer obtained at 1000 °C, it is seen that it decreases slightly. It can be said that this is due to pore-like pits that are poorer in Nb and C.

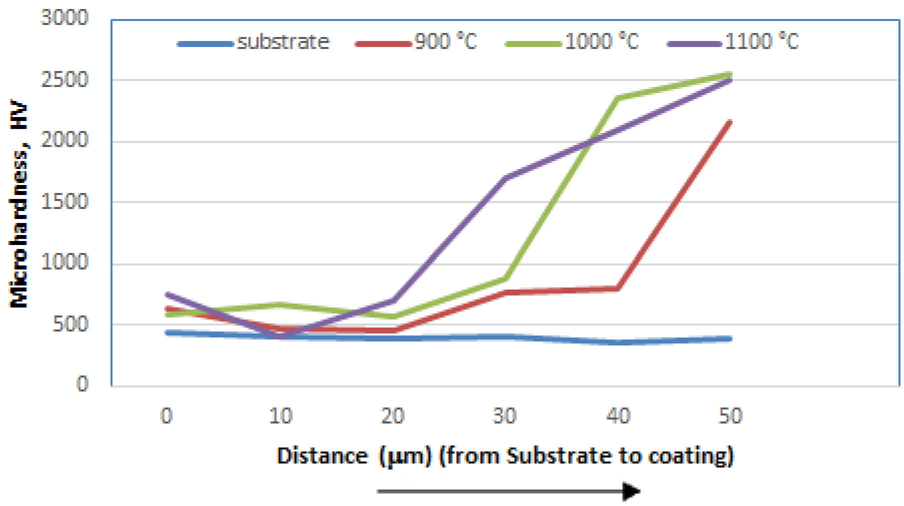

Figure 5. Microhardness change of NbC layers in the obtained samples.

4. CONCLUSION

In this study, an NbC layer was obtained on the surface of GGG70 nodular graphite cast iron using the pack cementation technique at temperatures of 900°C, 1000°C, 1100°C for 6 hours, and the effects of the coating temperature on the coating properties were investigated. In conclusion;

- NbC layer with a homogeneous and flat morphology was obtained on the surface. The layer/matrix interface appears quite flat. It is seen that the layer thickness increases with the increase of the coating temperature. However, although the coating thickness reached the highest values in the sample coated at 1100 °C, a pore-like coating layer poor in Nb and C was obtained. The most homogeneous and smooth coatings were obtained on sample surfaces coated at 1000 °C for 6 hours.
- As a result of XRD analysis, the desired NbC phase was obtained. It was observed that the intensity of the α-Fe phase increased and the intensity of the NbC phase decreased in the sample coated at 1100 °C.

- The hardness values of the coating layer increased approximately 5 times compared to the substrate material.
- When the fracture toughness values of the coatings were examined, the highest value was reached in the coating obtained at 1000 °C and this value decreased slightly in the samples coated at 1100 °C.

5. CONFLICT OF INTEREST

Author approve that to the best of their knowledge, there is not any conflict of interest or common interest with an institution/organization or a person that may affect the review process of the paper.

6. AUTHOR CONTRIBUTION

Serkan Dal: Determining the research and research method, conducting experiments, collecting and analyzing data, interpreting the analysis results.

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