EUROPEAN MECHANICAL SCIENCE

Research Article

e-ISSN: 2587-1110

The effect of niobium carbide coating on wear behavior of grey cast iron via thermo-reactive diffusion process

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Abstract: Wear is the limit for grey cast iron (GCI), which is utilized extensively in today's industries. Coating the surface of a material can enhance its ability to withstand wear. In this study, thermo-reactive diffusion (TRD) process was used to coat the surface of grey cast iron with niobium carbide (NbC). The coatings were applied for 2, 4 and 6 hours at 950°C and 1050°C. The coated samples were subjected to metallographic examination to investigate the microstructure of the coating zone. For this purpose, optical microscopy examinations were carried out. Microhardness tests were carried out to assess the mechanical properties of the samples. The coated surfaces were analyzed using energy-dispersive X-ray spectrometry (EDS), X-ray diffraction (XRD), and scanning electron microscopy (SEM). Wear tests were carried out on the coated surfaces to measure the volumetric wear loss, the wear rate and the changes in the coefficient of friction. Coating thickness rose as furnace waiting time increased, according to optical microstructures of coated surfaces. The hardness of the coated surfaces increased with a longer coating duration. Depending on the duration and temperature of the coating process, the layer thickness ranged from 6 to 52 μm. The lowest microhardness and the highest microhardness values of the coatings were determined at 950°C for 2 hours and at 950°C for 6 hours, respectively. Compared to the uncoated samples, the coated samples had a 6-9 times higher hardness value. In the abrasion tests, the loss of wear volume increased with increase in load.

Keywords: Thermo-Reactive Diffusion (TRD); Grey Cast Iron (GCI); Niobium Carbide (NbC); Sliding wear

1. Introduction

The use of cast iron is widespread in many industries. Meltability, strength and ductility are fundamental properties of this material. At heavy loads, it is utilized to replace steel castings. [1]. Chemically speaking, cast iron is a ferrous metal with more than 2% carbon content. It is an iron-carbon-silicon alloy containing about 1-3% silicon and 2-4% carbon. Cast iron contains elements such as phosphorus, manganese, sulfur, carbon and silicon [2]. Cast iron is a ternary Fe-C-Si alloy with small amounts of S and P [3, 4]. GCI is often used in engineering applications due to its features of vibration damping, machinability, and high thermal conductivity [5]. Due to its versatility, excellent castability, low cost (20-40% cheaper than steel) and strong mechanical properties, GCI is used in various industrial applications. The structure of GCI is influenced by its chemical composition, inoculants and cooling conditions [6-9]. Graphite flakes, which give them solid lubrication properties, are placed on the contact surfaces of GCI [10].

The hardness of the material, the high surface quality and the wear resistance ensure a long service life. In industrial settings, surface treatment processes are crucial for extending the life of mechanical components and tools [11]. Carbide and nitride coatings are utilized in various tribological applications, such as tools, mechanical components, and molds for processing metals, plastics, and glass. Traditionally, two different techniques have been used: physical vapor deposition (PVD) and chemical vapor deposition (CVD). However, these techniques have disadvantages such as plant invest-

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Cite this article as:
Yakut, R. (2024). The effect of niobium carbide coating on wear behavior of grey cast iron via thermo-reactive diffusion process. *European Mechanical Science*, *8*(3): 115-124. https://doi.org/10.26701/ems.1467274

History dates:
Received: 09.04.2024, ***Revision Request***: 25.04.2024, ***Last Revision Received***: 24.05.2024, ***Accepted***: 22.06.2024

ment cost, vacuum or highly controlled atmosphere [12, 13]. Moreover, CVD process produces highly toxic gases [14]. Carbide coatings are highly resistant to wear and corrosion. Therefore, they are of great industrial interest. One successful method for forming these coatings is TRD method [15]. TRD is a thermochemical process that forms dense and compact ceramic coatings on a substrate, especially steel [16-22]. The TRD method has many advantages compared to other coating processes, for example, being relatively simple, inexpensive [23], and environmentally friendly [24]. It produces consistent coatings with excellent adhesion, abrasion resistance and low friction. [16]. In TRD, elements form carbides in the salt and react with carbon atoms in the substrate. This produces an effective carbide coating that forms on the substrate surface. Niobium carbide, chromium carbide and vanadium carbide are the most commonly used single-carbide coatings [24]. Vanadium carbide (VC) and niobium carbide (NbC) are transition metal carbides. They have a wide range of industrial applications due to their high melting point, hardness, and thermal conductivity [25-27]. The layers formed by NbC coating have high microhardness [28], toughness and Young's modulus. This property is desirable for applications requiring high wear resistance. Moreover, this material is suitable for applications that require high temperatures, as its melting point is 3873.8°C [29]. The TRD technique is a high-temperature-resistant coating process that operates at temperatures between 800 and 1250°C [30]. Using a thermochemical process, a carbide-forming agent is deposited on the material's surface (Cr, V, Ti). Then a reaction occurs between the carbon decayed from the substrate to the surface and the carbide-forming agent [31-34]. This TRD technique was developed and patented by Toyota Motor Corp. in Japan [35-38]. Cai and Xu. [39] coated NbC on GCI using the in situ reaction (ISR) technique at 1085°C for 10, 20 and 30 minutes. In their study, it was discovered that the niobium carbide coating exhibited a lower wear rate, a higher load-carrying capacity, and excellent resistance to wear and corrosion.

Wear is a major problem in today's industry. Wearing is an unwanted deformation of materials caused by the mechanical actions of particles being removed from their surface [40]. Wearing causes deterioration, increased maintenance costs, pollution, energy costs and possibly accidents [41]. Corrosion and wear-resistant coatings are often required for mechanical components, such as tools that shape and machine workpieces. Selecting a suitable coating method can lead to significant performance improvements [42]. Mariani et al. [43] studied the formation of NbC and VC coatings (produced by TRD process) on Austempered Ductile Iron (ADI) specimens and their effects on the wear properties of the coatings. At 1000°C for 2 hours, they used molten salt baths containing sodium borate, aluminum, and a ferroalloy (Fe-V or Fe-Nb). As the high TRD temperature is responsible for the austenitisation of the sample, austenitisation in a further molten salt bath at 300°C occurred immediately after the TRD bath. Compared to the substrate, the coated parts were harder and more wear-resistant after these processes. The austempering process, on the other hand, increased the hardness further. The carbide coatings produced have a much higher wear performance (5-35 times) than the substrate. Soltani et al. [44] coated low-alloyed special tool steel (AISI L2) with NbC using the TRD process. The process took place in a sealed container containing a mixture of ferro niobium, ammonium chloride, and aluminum oxide powder at temperatures of 900, 950, 1000, and 1050°C over a period of 2, 4, 6, and 8 hours. Depending on the thickness of the coatings, the ratio of powders was optimized. After the TRD procedure, the samples were cooled in the air. This process resulted in NbC coatings of AISI L2 steel having a hardness of 2500±200 HV.

GCI, widely used in industry, is subject to wear. The surface hardening of GCI can increase the wear resistance. For this purpose, it is necessary to obtain a coating that effectively prevents wear and reduces the coefficient of friction. Therefore, GCI is coated, thus its surface hardness is increased. In this study, the surface of GCI was coated with NbC using TRD process. Coatings were performed for 2, 4 and 6 hours at 950°C and 1050°C. The microstructure of the coated samples was examined at the coating zone. Tests were then carried out using a reciprocal wear tester to investigate the wear behavior of the coated samples. Testing was conducted with a 6 mm Al_2O_3 ball at a 10 cm/s sliding speed, 8.5 mm sliding length, and 500 m total sliding distance, with loads of 10N and 15N, respectively. This study aims to improve the hardness and shear wear properties of coated surfaces by using the TRD process.

2. Materials and Methods

2.1. Preparation of Sample Materials

The substrate material used was GCI with a chemical composition of 3.30C-2.70Si-0.65Mn-0.016P-0.08S-0.28Cr-0.12Cu-0.001Al-0.025Ti (wt%) and equilibrium Fe. The GCI samples used as substrate material were cut with a precision metallographic cutter for microstructural analysis. All surfaces of the cut specimens were finally polished with 1200 mesh sandpaper. The polished samples were cleaned with alcohol before TRD process. For the coating process, Ferro Nb powder (45%), alumina (45%) and ammonium chloride (10%) were weighed with a precision balance and then mixed. Ferro niobium (FeNb) powder with a chemical composition of 65Nb-0.10C-2.5Si-0.05S-0.1P-1.5Al (wt%) was used in equal weight for each experiment. Melting point, purity, density, and size were 1530-1580°C, 99.5%, 8.1 g/cm^3 and 43 µm, respectively. This powder material was purchased from BC Technology (Türkiye). All surfaces of the GCI specimens used as the substrate materials were polished, cleaned and placed in a stainless steel crucible with the coating powder mixture. The crucible was then tightly sealed. Preparation of the crucibles was followed by coating at 950°C and 1050°C using the TRD process. After the process, the crucibles, which were removed from the furnace, were rapidly cooled with water, then the mouth of each crucible was opened, and the samples were quickly removed and cooled in water.

2.2. Sample characterization

The samples coated in accordance with the TRD method were molded using the cold molding technique. They were sanded through coarse and fine stages until the base material was reached. The ground specimens were polished with 3 and 1-micron diamond solutions. They were etched with a 5% Nital solution. Thus, the coating layer and layer-substrate material interface cross-sections were prepared for optical microscopy and SEM. The inverted metal microscope Nikon MA 100 and the image analysis system, Clemex, were used for the optical microscopy. EDS and XRD analyses were performed on the coated surfaces, along with SEM analysis.

In this study, microhardness measurements were performed on samples prepared for optical microscopy, such as coating cross-section and the substrate material near the interface. The hardness measurements on the samples used in the experiments were performed with the Future Tech FM -700 Vickers hardness tester. The parameters used for the microhardness measurements were 50 gf load and 10 seconds duration.

Sliding wear tests were performed according to ASTM G99. All abrasion tests were carried out with a reciprocating abrasion tester under normal atmospheric conditions (25 ± 1 °C and 60 ± 2% humidity) in a dry environment. The abrasive used was a 6mm diameter Al_2O_3 ball. The wear tests were carried out under 10N and 15N loads. The sliding speed was 10 cm/s, the sliding length was 8.5 mm, and the total sliding distance was 500 m. Friction coefficients were recorded during the test. The sliding wear was carried out on a different area of the specimen each time. The wear marks never overlapped. After the abrasion tests, the appearance of the abrasion marks was examined via SEM.

3. Results and Discussion

3.1. Microhardness test results

For the measurements at 950°C, the average hardness values were found to be 1943 HV for 2-hour coating, 2231 HV for 4-hour coating and 2551 HV for 6-hour coating. Hardness measurements at 1050°C were recorded as average hardness values of 2259 HV in 2-hour coating, an average of 2448 HV in 4-hour coating and an average of 2364 HV in 6-hour coating. At the end of the hardness measurements, it was found that the highest average value for the coating was measured at 950°C for 6 hours. The lowest average value was obtained for the coating held at 950°C for 2 hours. It was established that as the coating time increased, the hardness values increased. The hard NbC phase and the microstructure greatly influenced the hardness of the coating [39]. The results of the hardness measurements for NbC-coated samples are shown in ▶**Table 1** for various temperatures and dwell times.

Soares et al. [41] treated two ductile cast irons (with and without copper addition) in a salt bath of borax, Ferro niobium (16 wt%) and aluminum (3 wt%) at 1000 °C for 4 hours. The hardness measurements they carried out on the samples produced using the TRD process yielded NbC coatings with a hardness of more than 2000 HV. Mariani et al. [45] subjected GCI samples to two thermo-reactive niobizing processes as a substrate material. Iron-niobium powders, NH_4Cl, and Al_2O_3 were used for the first process, which took 2 hours at 900°C. In the second process of the TRD method, a liquid molten bath consisting of sodium borate and iron niobium was used for 2 hours at 900°C. After the surface coating process with NbC, the hardness measurement value was obtained as 2000 HV. When similar studies are examined, it is seen that the microhardness values obtained in this study and shown in ▶**Table 1** are compatible with those of other studies.

Table 1. Average values of microhardness of NbC-coated samples

Coating Temperature (°C)	Coating Time (hour)	Coating Thickness (µm)	Microhardness (HV)	
			Coating Layer	Substrate Material (Grey Cast Iron)
950	2	11± 2	1943 ± 182	290± 83
950	4	14± 2	2231 ± 63	
950	6	16± 3	2551 ± 129	
1050	2	7± 1	2259 ± 187	
1050	4	14± 2	2448 ± 49	
1050	6	41± 9	2364 ± 90	

3.2. Wear Test Results

In this study, the development of wear was continuously analyzed. The highest coefficient of friction was observed at a load value of 10N at 1050°C for 6 hours, while the lowest coefficient of friction was observed at a load value of 10N at 950°C for 2 hours, considering the changes in the coefficient of friction of the samples in ▶**Figure 1**. The applied load and the reinforcement ratios are shown in ▶**Table 2**. The maximum wear volume loss was found to be 6.97 × 10^{-2} mm^3 at a load of 15 N on the grey cast iron surface used as the substrate material. The maximum wear volume loss was 2.29 × 10^{-2} mm^3 at 2 hours and 15 N load for 2 hours, 4 hours and 6 hours at 950°C. The lowest wear volume loss at 950°C was 1.84 × 10^{-2} mm^3 at 6 hours and 10 N load. The highest wear volume loss for 2 h, 4 h and 6 h at 1050°C was 2.66 × 10^{-2} mm^3 for 6 h and 15 N load. The lowest wear volume loss at 1050°C was 1.54 × 10^{-2} mm^3 at 2 hours and 10 N load. When analyzing the wear volume loss (▶**Figure 2**), it was found that the wear volume loss increased with increasing load when the temperature and waiting time were kept constant. In addition, an increase in the coefficient of friction with increasing load was also observed at constant temperature and waiting time. It can be said that the wear rate (▶**Figure 3**) also generally increases with increasing load when the temperature and dwell time are kept constant. More severe wear conditions can be attributed to the increase in wear loss of the specimens with applied load and sliding speed. The tendency to crack due to dissolution at the interfaces between graphite and matrix leads to a significantly higher wear loss due to the crumbling of the material. The changing slope of the wear loss versus load graphs (▶**Figure 2**) shows that the mechanism of working wear is changing. For example, a low inclination corresponds to light wear, while a high inclination indicates heavy wear [46]. GCI's wear rate increased with increasing load during wear tests [10]. However, when the surface of the GCI was coated with NbC using TRD method and the temperatures and waiting times were kept constant, it was observed that the wear rate values (▶**Figure 3**) decreased with increasing load. This situation can be explained by the fact that the hardness of the coating surface increases with increasing temperature and waiting time. The hardness of materials is a decisive factor influencing wear properties [38, 47]. As the material's hardness increases, the wear rate decreases as it becomes more difficult to remove particles from the surface. Factors that influence wear resistance include microstructure, surface hardness, modulus of elasticity, size and distribution of hard particles [47]. In the dry sliding test, the contact area between the coating and the counterpart was reduced compared to the substrate. This led to a reduction in the coefficient of friction due to the harder NbC phase and the fine microstructure in the coating [39, 48, 49].

3.3. Optical and SEM/EDS/XRD Results

The substrate material was coated with NbC using the TRD technique for 2 hours, 4 hours and 6 hours. Optical microstructures were examined to analyze the coating layer, and SEM examinations were carried out after wear tests. When investigating the optical microstructures of NbC coatings held at 950 °C for 2 hours, 4 hours and 6 hours, it was found that the coating thickness increased as a function of the holding time in the oven. It was determined that the coating time is a determining parameter in coating thickness. Optical microstructure views are given in ▶**Figure 4**. Depending on the deposition time and temperature, NbC layers with a thickness of 6 to 52 μm formed on the substrate.

Table 2. Values of the volume loss, wear rate, and coefficient of friction for the samples

Coating Temperature (°C)	Coating Time (Hours)	Load (N)	Volume Loss (×10^{-2} mm^3)	Wear Rate (×10^{-6} mm^3/Nm)	Coefficient of Friction (COF)
Substrate Material (Grey Cast Iron)	-	10	6.62	13.25	0.511
	-	15	6.97	9.30	0.445
950	2	10	1.95	3.91	0.249
950	2	15	2.29	3.05	0.342
950	4	10	1.90	3.79	0.373
950	4	15	2.11	2.81	0.392
950	6	10	1.84	3.69	0.404
950	6	15	2.06	2.75	0.423
1050	2	10	1.54	3.08	0.518
1050	2	15	2.39	3.18	0.601
1050	4	10	1.89	3.79	0.434
1050	4	15	2.09	2.78	0.455
1050	6	10	2.23	4.45	0.630
1050	6	15	2.66	3.55	0.529

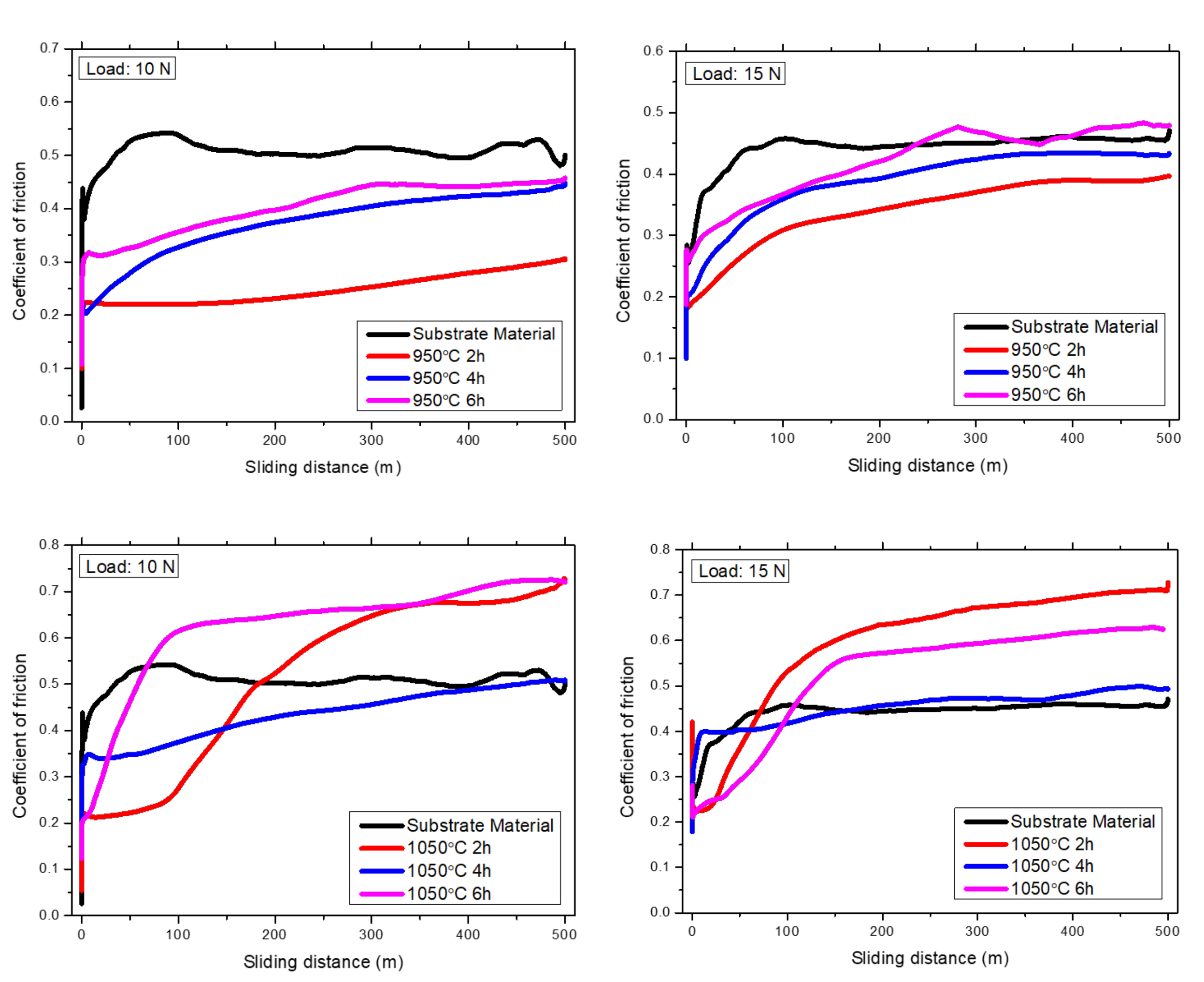


Figure 1. Comparison of friction coefficients at 10 N and 15 N loads

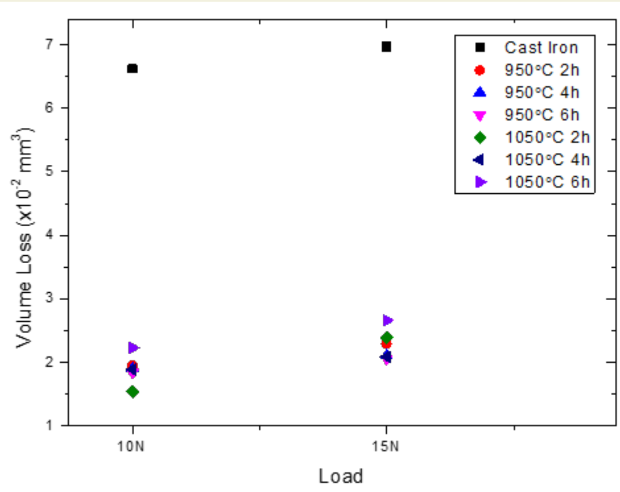


Figure 2. Wear volume loss measurement values

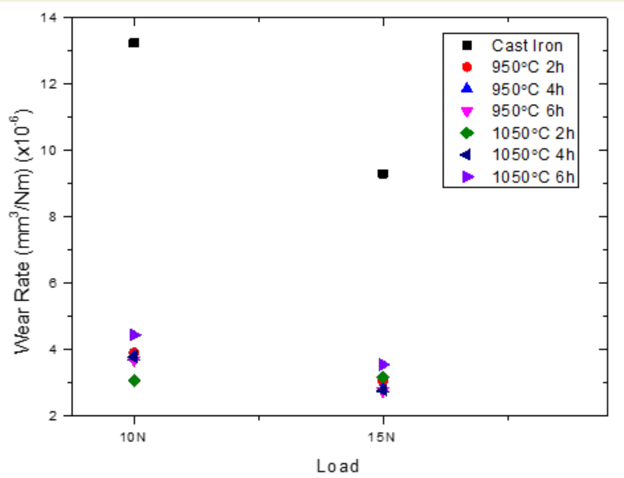


Figure 3. Wear rate measurement values

Element diffusion analysis mainly focuses on C and Nb atoms. Nb atoms can diffuse into the substrate and form a layer of solid solution on its surface. The nucleation of NbC begins at the dislocations or grain boundaries on the substrate's surface. The crystal nucleus grows and expands to form a thin, continuous layer of NbC [50]. Niobium carbide's structure is a face-centered cubic (FCC) lattice. The niobium atoms are located in the FCC phase, and the carbon atoms are in the intermediate areas, resulting in a subtly defined crystalline material [15]. The grain size is significantly influenced by the concentration of C atoms [51]. In this study, NbC

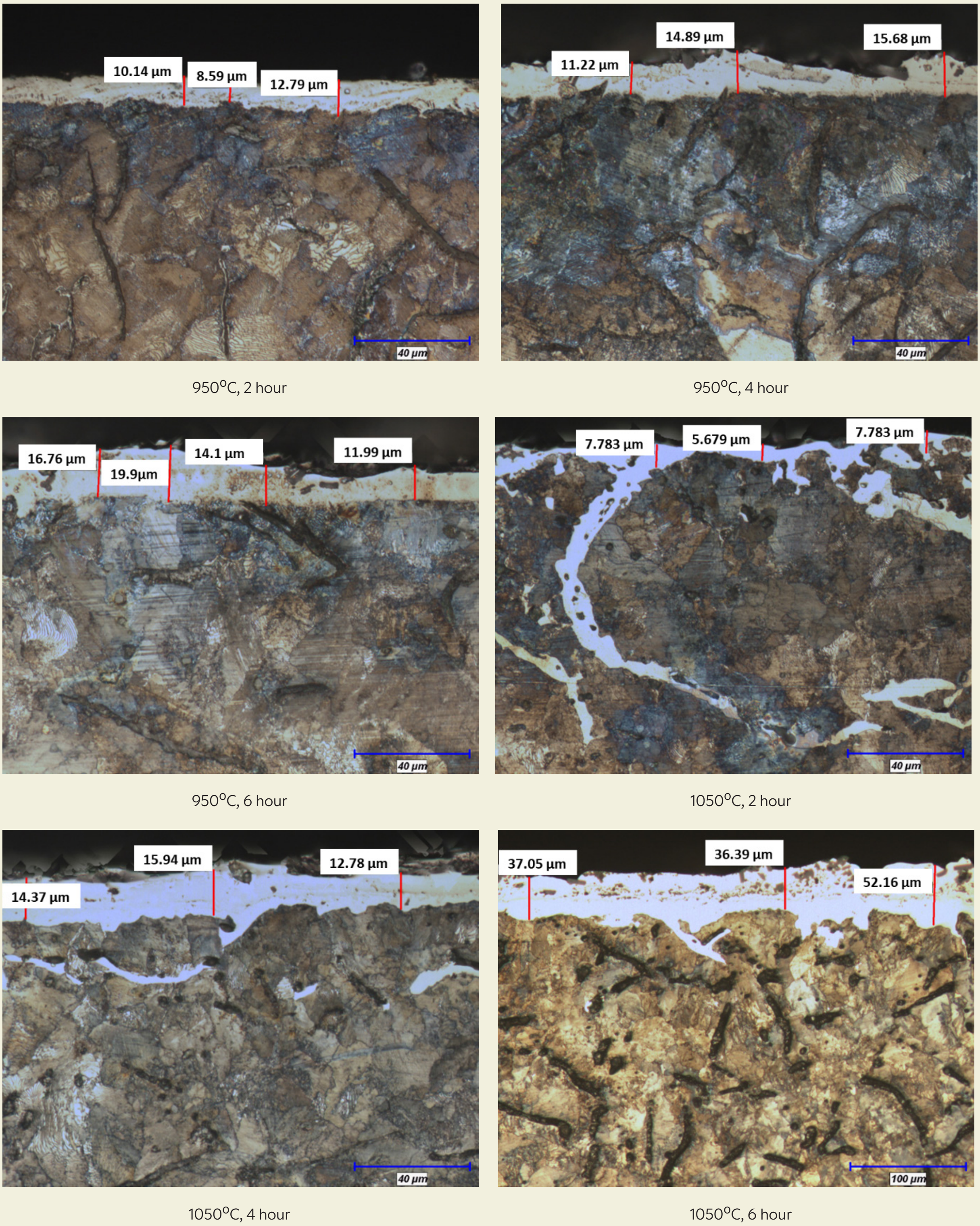


Figure 4. Optical microstructure views

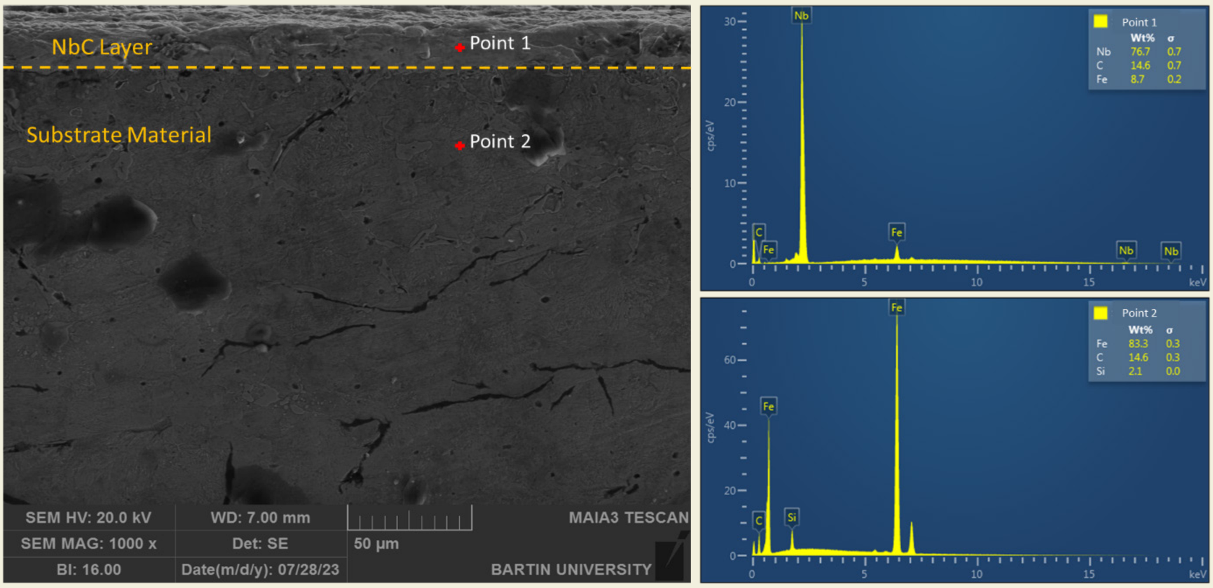


Figure 5. EDS image of NbC coated specimens at 950°C for 6 hours

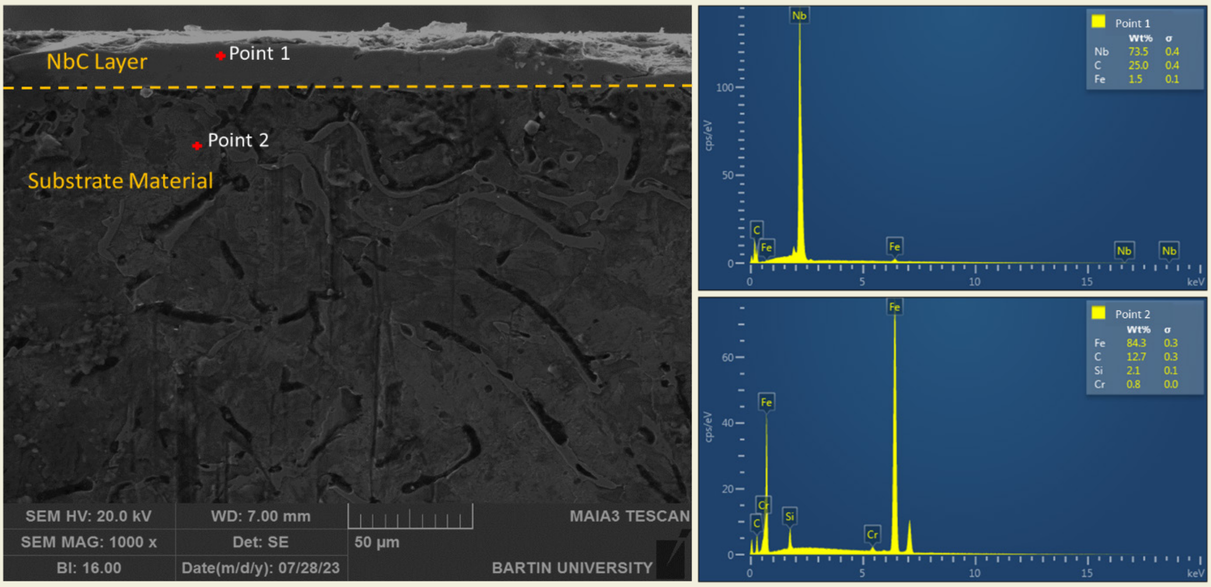


Figure 6. EDS image of NbC coated specimens at 1050°C for 4 hours

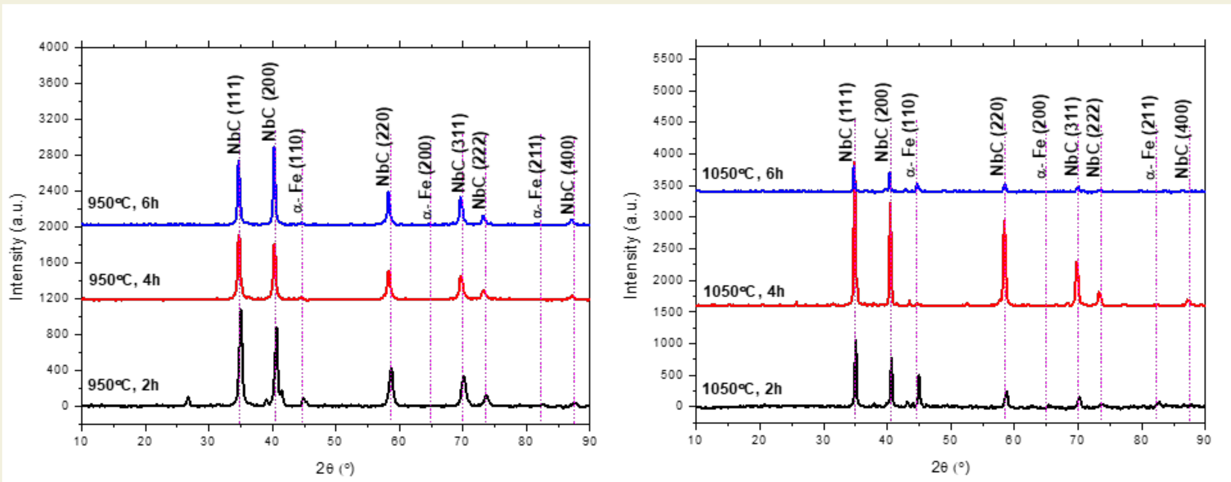


Figure 7. XRD analysis results of NbC-coated samples

extended due to the high diffusion speed of the grain boundaries and the graphite carbon density.

NbC was coated on grey cast iron by TRD method at 950 and 1050°C for 2, 4 and 6 hours. When EDS image (▶Figure 5) of NbC coated samples at 950°C for 6 hours was analyzed, wt% Nb: 76.7, C: 14.6, Fe: 8.7 in spectrum 37 (Point 1) and wt% Fe: 83.3, C: 14.6, Si: 2.1 in spectrum 39 (Point 2) were found. When EDS image (▶Figure 6) of NbC coated samples, which were kept at 1050°C for 4 hours, was analyzed, wt% Nb:73.5, C:25.0, Fe:1.5 in spectrum 10 (Point 1) and wt% Fe:84.3, C:12.7, Si: 2.1, Cr:0.8 in spectrum 12 (Point 2) were found.

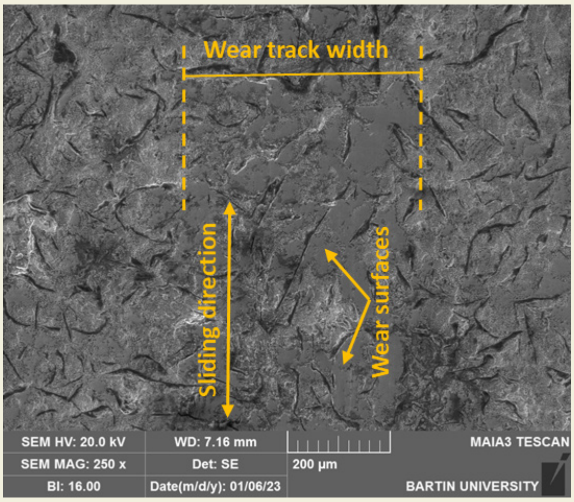
When the results of the XRD analysis of the NbC-coated samples are examined in ▶Figure 7, it can be seen that the NbC phase has been formed as the dominant phase on the surface of all the samples. Soares et al. [41] coated spheroidal graphite iron surfaces with NbC using TRD. The XRD analysis conducted on the coating layer revealed that it was made up solely of NbC phase. OrjuelaG et al. [30] coated AISI 1045 steel surfaces with NbC using TRD method. XRD analysis of the coating layer showed that NbC phase was the main phase, while the other phase was α-Fe.

After the sliding wear test under dry sliding conditions were conducted, the sample surfaces were analyzed using the TESCAN MAIA3 XMU scanning electron microscope (SEM) (▶Figure 8). When the sample coated at 1050°C for 2 hours was subjected to an abrasion test at 15 N load, the abrasion marks were clearly visible. As the applied load increases, so does the contact pressure [52]. Due to the high hardness of the coated parts, no damage to the wear surfaces due to particle breakage was observed. However, the coated samples showed higher wear resistance. As a result, fewer particles broke off the coated surfaces and smoother surfaces were achieved.

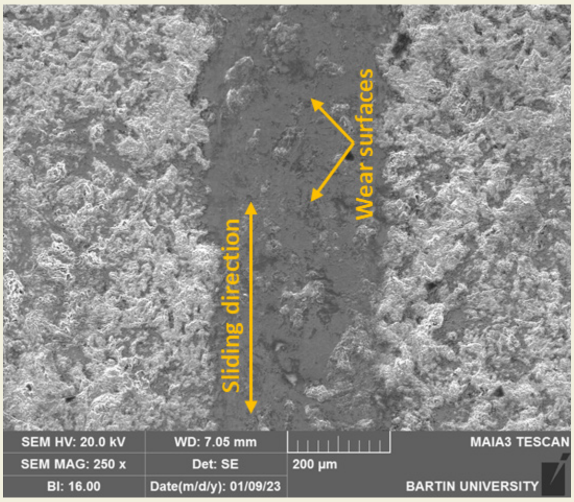
4. Conclusions

The surface of the grey cast iron was coated with NbC using the Thermo-reactive diffusion technique (TRD). The coating process was carried out at 950°C and 1050°C for 2, 4 and 6 hours. The microstructure of the coated samples was analyzed in the area of the coating. Tests were then carried out using a reciprocating wear tester to investigate the wear behavior of the coated samples. The wear tests were carried out under 10N and 15N loads. The results obtained in the study are given below:

- When examining the optical microstructures of 2, 4 and 6 NbC layers, it was found that the layer thicknesses increased with the waiting time in the furnace.
- NbC layers with a thickness of 6 to 52 µm were formed on the substrate, depending on the coating time and temperature. Hardness measurements showed the coating's highest value at 950°C for 6 hours and the lowest value at 950°C for 2 hours.
- The substrate material showed the highest volumetric wear loss at a load of 15 N, while the sample coated for 2 hours at 1050°C showed the lowest volumetric wear loss at a load of 10 N.



950°C, 2 hour, 15N



1050°C, 2 hour, 15N

Figure 8. SEM images after sliding wear tests

- The highest wear rate occurred at a load of 10 N on the substrate material, while the lowest wear rate occurred at a load of 15 N on the sample coated at 950°C for 6 hours.

Research Ethics

Ethical approval not required.

Author Contributions

The author(s) accept full responsibility for the content of this article and have approved its submission.

Competing Interests

The author(s) declare that there are no competing interests.

Research Funding

Not reported.

Data Availability

Not applicable.

Peer-review

Externally peer-reviewed.

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