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(Research Article)

Micro abrasion in Fe-Cr-C-Nb alloys samples: The role of niobium

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Abstract

This paper addresses to analyze the role played by Niobium (Nb) in the microstructure of Fe-Cr-C alloys being tested in micro abrasion conditions (ball abrasion test). Three alloys with differences in Cr content and one of them with 5% Nb have been prepared as weld tracks under Fe-C substrate. The resulting microstructures contain carbides embedding in a metallic matrix (MMC). Significant changes in mechanical properties are expected depending on the morphology, size and distribution of carbides. Results pointed out to an improvement in wear resistance of Fe-Cr-C-Nb alloy although it showed lower values of hardness and volume fraction of carbides in comparison to the other alloys. The coefficient of friction was also affected by Nb addition. Such results evidence the possibility of using such alloy in applications that require high performance in wear resistance as mining and agriculture equipment.

Keywords: Niobium; Chromium; Wear; Ball Abrasion Test; Friction Coefficient**.**

1. Introduction

Engineering solutions for materials applications require appropriate decisions on designing and selection of materials. This is an important step when a given set of operational conditions is known.

There is a growing number of research dedicated to metal matrix composites (MMC) microstructures consisting of hard and brittle carbide particles embedded in a tough metal matrix. NbC is a kind of stable carbide and presents a number of interesting properties for its use in wear applications, such as high hardness, high toughness, extremely high Young's modulus, excellent adherence to matrix and high melting temperature.

In surface engineering, Fe-Cr-C alloys are useful as coatings. Sometimes with others elements, providing a great variety of microstructures showing better response to wear, especially under abrasion. Often, these alloys have carbides of MC, M_6C, M_7C_3, $M_{23}C_6$ and Cr_2C_3 type depending on the composition, solidification and cooling rates. [1, 3] Berns [2] reports how size, distribution, adherence to matrix, volume fraction and morphology can affect the wear rate of microstructures. Accordingly, to [2, 3], Nb in Fe-Cr-C systems provides NbC carbides resulting in better wear resistance. However, hard micro constituents bring some limitations as coarse morphologies and poor adherence.

Several studies on the effects of Nb addition on the microstructure and wear behavior of various classes of steels and hardfacings have been conducted. It is notorious the positive achievements that can be attained by the addition of alloying elements on mechanical properties of steels. Nb additions promotes great changes in mechanical properties, even in lower percentages [4 – 8]. Additionally, an increased fraction of carbides can result in improved tribological properties such as lower friction and wear rates. The presence of niobium carbides in iron alloys can enhance the

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formation of a protective oxide layer on the sliding surfaces, which can reduce friction and wear. The effect of niobium carbides on friction coefficient can also be influenced by other factors such as the presence of other alloying elements, the microstructure of the alloy, the roughness of the sliding surfaces, the sliding speed and load, and the lubrication conditions. These factors can interact and influence the overall tribological behavior of the niobium alloy. [9-15]

Limin Zhang, Dongbai Sun and Hongying Yu [16] investigated, in details, the role of Nb addition in iron base alloys produced by plasma cladding. They performed an accurate investigation on the influence of Nb carbides on the wear behavior of coatings deposited upon 0.45% C carbon steel. The effects of niobium element on the microstructure and wear resistance property of the iron-based coatings were investigated by these authors using scanning electron microscopy (SEM) with energy dispersive spectrum (EDS), X-ray diffraction (XRD) and ball-on-disc wear tester. Special attention was done to the role of debris, issued from the wear mechanisms, on the decreasing of friction coefficient. The morphologies of the worn surface and its debris for the 0.45% C carbon steel, the Nb-free and Nb-contained clad coating were observed.

With respect to the size and amount of debris they reported that particles entrapped on the worn surface decrease in the following serials: 0.45% C carbon steel, Nb-free clad coating and Nb-contained clad coating. In the Nb-free clad coating, the debris become smaller than that of the 0.45% C carbon steel, but the morphologies are irregular. With the Nb addition, the size and morphologies of debris become small and regular.

They found that the friction coefficient of Nb-free clad coating oscillates around 0.48, which is smaller than that of the 0.45% C carbon steel specimen which oscillates around 0.7. With the niobium addition, the friction coefficient of clad coating oscillates around 0.35.

This is an experimental proof that the addition of niobium decreases friction coefficient.

The Nb-free and Nb-contained clad coatings both owned an excellent wear resistance property compared to 0.45%C carbon steel under dry sliding wear tests. The wear resistance of Nb-contained clad coating was higher than that of the Nb-free clad coating probably due to the high hardness carbide - NbC - distributed in clad coating. The results showed that the composite coating had high hardness and excellent wear resistance under dry sliding wear test conditions. [16]

Our contribution to this area had been done measuring specific wear rate and wear coefficients of thick coatings deposited upon ASTM A36 steel plate - (150 x 50 x 11,7) mm. The deposition was performed by means of welding methods providing thick tracks of Fe-Cr-C and Fe-Cr-C-Nb. The wear tests have been done in a ball abrasion tester in wet conditions (suspension of diamond particles in distilled water). This test provides craters which diameters` variations allow to follow the loss of mass evolution during tests, among other features.

2. Materials and methods

Fifteen samples of the ASTM A36 carbon steel of dimensions (150x50x11.7mm) were produced for the coating's application of the alloys A, B and C by welding process. In each sample, four weld beads of 5mm high and 150mm length were deposited in a single layer. Micro abrasion wet tests have been performed in order to verify the influence of the addition of Nb on the microstructure and consequently on the wear resistance and friction coefficient. The micro wear behavior of these microstructures has been verified by means of ball abrasion wet test coupled with optical microscopy and SEM/EDS observations.

Our contribution had been done measuring wear rate, wear coefficients, friction coefficients evolution during long duration tests of thick coatings deposited upon ASTM A36 steel samples. The deposition was performed by means of welding methods, for instance SMAW (coated electrode welding process) and GMAW (MIG welding), providing thick tracks upon ASTM A36 steel plates. The wear tests have been done in a ball abrasion tester built accordingly to ISO 26424-2008. This test provides craters which diameters allow to follow the loss of mass Evolution. ASTM A36 steel plates (150 x 50 x 11,7) mm were taken as substrate to be coated with Fe-Cr-C and Fe-Cr-C-Nb by welding. From these plates five samples were prepared (25,0 x 25,0 x 16,7) mm to the abrasion tests taken from regions without any transitory effects of the welding process as existing in the beginning and in the end of the tracks. The preparation of samples to abrasion tests followed the metallographic recommendations aiming to have surfaces with roughness provided by diamond paste of 0,25μm after polishing.

2.1. Ball abrasion wet tests

The recommended design to a ball abrasion machine has been upgraded by introducing sensors to capture in real time the signals from normal and tangential forces allowing the calculation of friction coefficients by means of a software developed to do this. The evolution, in real time, of these three parameters (normal force, tangential force and friction coefficient) can be visually followed (monitored) in the computer screen. Table 1 summarizes the parameters of the experiments. A suspension of diamond particles (3µm mean diameter) in distilled water in a concentration of 0,35g/cm^3 has been prepared to acts as abrasive medium. [3]

Table 1 The parameters of the experiments. [18]

TEST TIME (s)	**900**	**1200**	**1800**	**3600**	**7200**	**10800**
NORMAL FORCE (N)	5.2	5.2	5.2	5.2	5.2	5.2
SPHERE ROTATION (rpm)	136	136	136	136	136	136
SLIDING DISTANCE (m)	163.43	217.90	326.85	653.70	1307.41	1961.11
ABRASIVE DRIP (drop/s)	1/15	1/15	1/15	1/15	1/15	1/15
AQUISITION TIME (s)	0.5	0.5	0.5	0.5	0.5	0.5
NUMBER OF TESTS	4	4	4	4	4	4

To minimize the influence of roughness during the micro abrasive wear test, the samples were polished with 180, 400 and 600 sandpapers (SiC) in order to have roughness (Ra) as similar as possible. The roughness measurements were performed using a Time-TR 200 model roughness meter. The average values of (Ra) for each surface are shown in the Table 2.

Table 2 Roughness Ra (µm). [18]

Coating	**Mean Value**	**Standard Deviation**
A	0.346	0.063
B	0.503	0.021
C	0.473	0.089

The geometric parameters of the weld bead, width, penetration and the areas to determine the dilution were measured using the software "ImageJ". The dilution evaluation was obtained by performing the relationship between the melt area below the sample surface and the total area of the weld bead from images obtained with a 6.3X magnification. Samples have been attacked chemically with 5% Picral reagent. Rockwell C hardness evaluation was performed according to the ABNT-NBR 6671-198 standard using a 10kgf preload and 150kgf load doing five measurements in each region of the samples. The Vickers micro hardness test was performed using a 100g load for 10 seconds on the samples chemically attacked with 5% Nital for 20 seconds [3].

Samples addressed to microstructural characterization were attacked with Murakami reagent at 60°C for 20 seconds to highlight the chromium and niobium carbides present in the coatings. Analyzes were performed by an optical microscope LEICA DM2700M with image capture camera, with increases of 500 and 1000X. To confirm the presence of the constituents detected by optical microscopy, SEM/EDS in a Tescan Microscope were performed. The percentages of carbides were evaluated with ImageJ software.

Micro abrasive wear tests allow to calculate the instantaneous volume/mass loss of samples from the crater's diameters. The evolution of wear is presented as a function of the slip distance. For this analysis, the distance of 651m was considered as reference, that is, 60 minutes of test time for which the three coating attain the permanent regime of wear. From SEM analyzes, the diameter of craters can be measured and the mechanisms of wear interpreted. [3]

3. Results and Discussion

It was possible to measure and interpret the wear behavior of each alloy following the evolution of the diameters of the craters and observing the microstructures and the features of the inner crater surface. Our discussion takes into account the correlation between micro hardness, microstructure arrangement, the volume loss of the samples and the evolution of friction coefficient.

3.1. Dilution

The dilution rate is important in studies involving wear resistance of coatings deposited by weld methods due to its relationship with the chemical composition of the fusion zone, which may change the microstructure. The values of the dilution rate of each alloy are shown in Table 3. It is observed that the alloy A presented the highest dilution rate compared to alloys B and C.

Table 3 Dilution rate of coatings A, B and C. [18]

Coating	Alloy A	Alloy B	Alloy C
Dilution (%)	36.54	22.28	22.99

3.2. Hardness

In order to have an overall information about the surface resistance to abrasion Rockwell C indentations have been done. In this essay a great amount of material is deformed providing a macro response representative of the coating hardness. Macro hardness tests have been performed according to the procedure explained in Figure 1 and results are summarized in Table 4. Hardness (HRc) of these coatings were higher than the substrate for all samples. Coating A, Fe-Cr-C (45% Cr) showed the mean highest value.

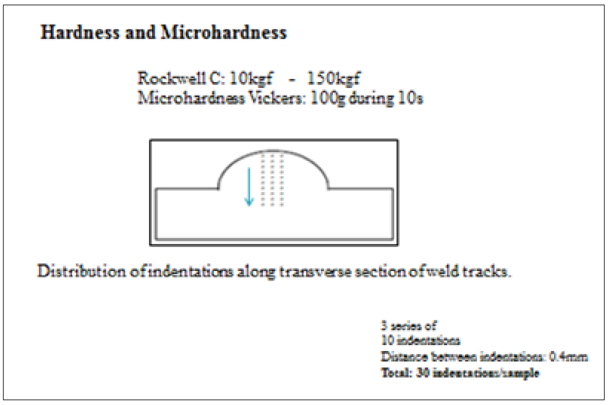

Figure 1 Schematic hardness indentation procedure. [18]

Table 4 HRc results for the coatings. [18]

Coating	Hardness (HRC)	Standard Deviation
A	53.50	3.07
B	50.25	1.58
C	40.00	1.51

Our discussion deals with the correlation between microhardness, the microstructures and the volume loss of the samples.

Cross sectionals profiles of Vickers microhardness showed in Figure 2 have been obtained after 10 indentations in three samples. Each indentation displaced 0,4mm from the other. To increase the statistics the same procedure has been repeated three times, so each profile represents 30 indentations.

The evolution of the microhardness along the cross section corresponds to the behavior of surface hardened alloys showing a significant increase in top of the coating due to the carbides in the microstructure. Local variations come from the distribution of carbides and the volume fraction in the microstructure as reported by Buchely et al. [17]

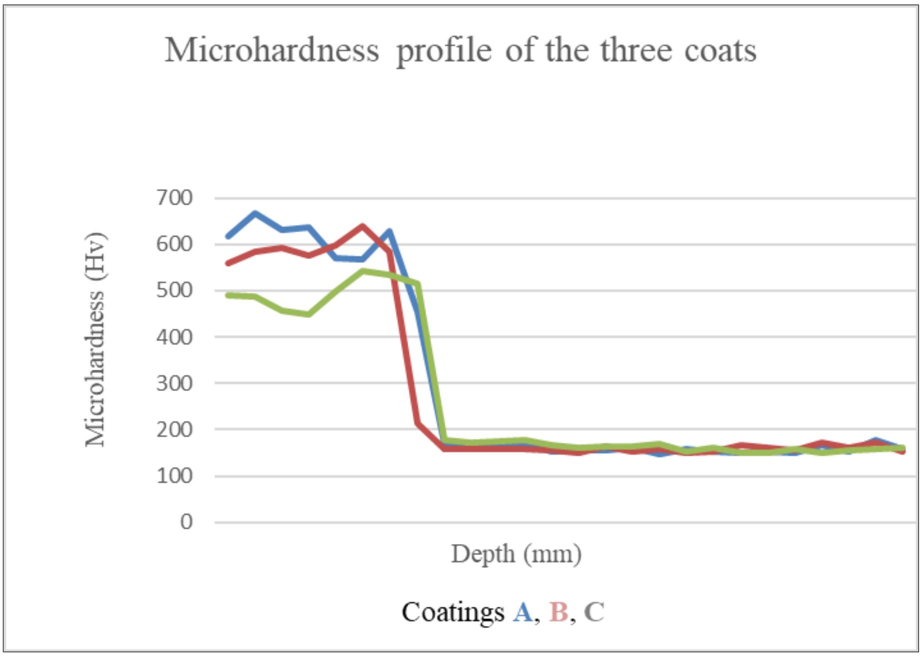

Figure 2 Evolution of coating microhardness across the transverse section Coating-Substrate. Comparison of the three types of coatings (A, B, C).

3.3. Microstructural analysis

Murakami reagent at 60ºC during 20 seconds provides good contrast to optical microscopy as well as to SEM/EDS observations because of its selective reactions. Figures 3, 4 and 5 are representative of the overall microstructure of each material. Figure 3 shows dispersed chromium carbides in the Fe-Cr-C (45%Cr) - alloy A- and the eutectic chromium carbide - austenite as found by [19, 20, 21]. According to Ogi et al. [22] as the chromium content increases, the size of the eutectic carbides crystals increases but their uniformity (regularity in shape) become worst resulting in a coarse distribution sometimes surrounded by fines carbides crystals.

Searching for information about chemical composition and phases in the microstructure SEM/EDS investigations had been performed. Results from alloys A, B and C are shown in Fig. 6, 7 and 8. Microscopic analyzes showed that all the coatings presented M_7C_3 carbides with peculiar characteristics for each morphology. Alloys A and B showed the coarsest forms of carbides: microstructure of the alloy A, Fe-Cr-C (45% Cr), consists of chromium carbides in addition to the eutectic constituent formed by chromium carbides and austenite; microstructure of the alloy B, Fe-Cr-C (25% Cr), showed dendritic growth and proeutectoid primary M_7C_3 carbides, concentrated in the matrix less coarse than the phases found in alloy A. Carbides in alloy B are distributed in the matrix as colonies and with great spacing between them. The microstructure of the alloy C, Fe-Cr-C-Nb, showed M_7C_3 carbides and niobium carbides (NbC) arranged in elongated branches named "Chinese Writing". The chromium carbides are distributed between the dendrites, arranged in colonies and with refined morphology, in comparison to the other found in alloys A and B.

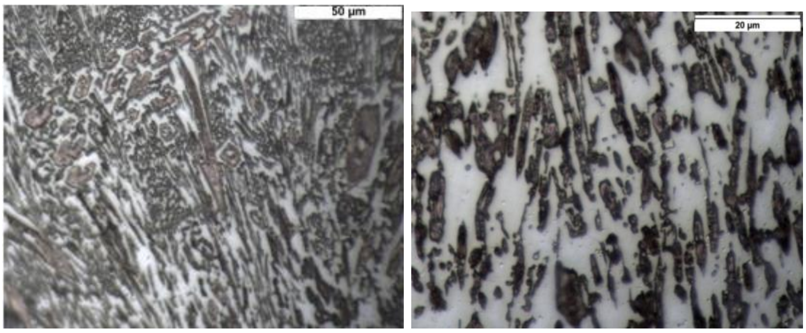

Figure 3 Microstructure in the surface of coating A. (a) 500x; (b) 1000x. (Murakami at 60ºC) (OM).[18]

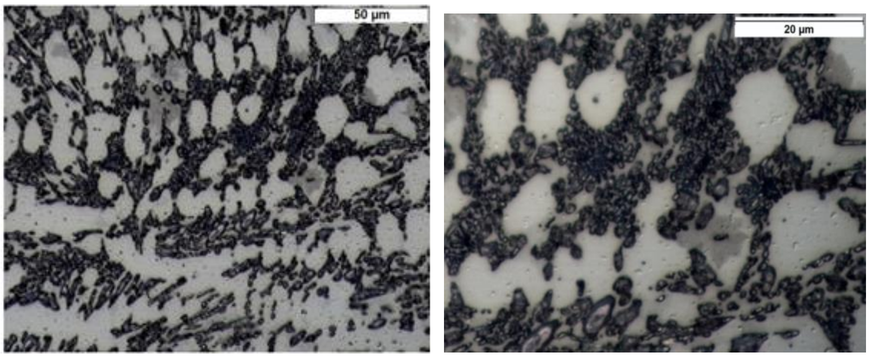

Figure 4 Microstructure in the surface of coating B. (a) 500x; (b) 1000x. (Murakami at 60ºC) (OM).[18]

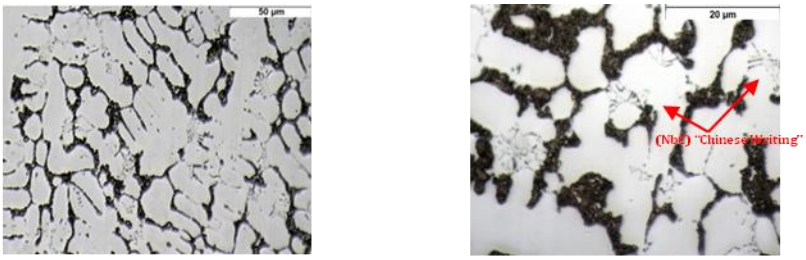

Figure 5 Microstructure in the surface of coating C. (a) 500x; (b) 1000x. (Murakami at 60ºC) (OM). [18]

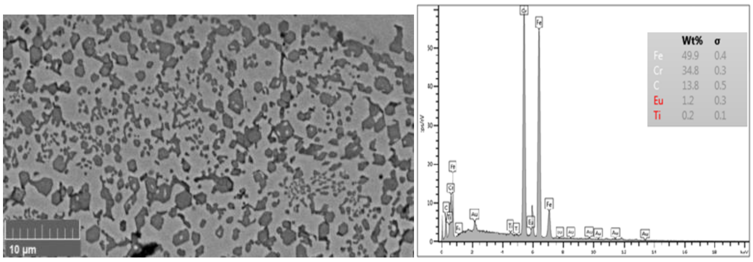

Figure 6 (a) Microstructure of coating A. (4000x). (b) EDS spectrum.[18]

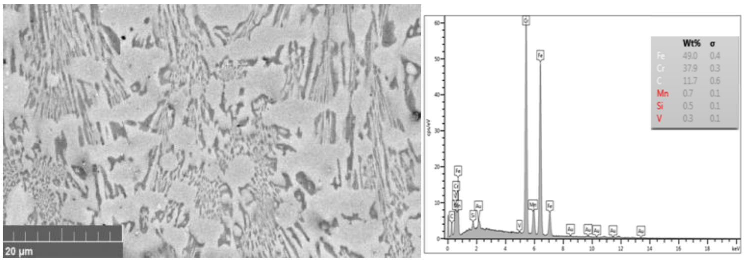

Figure 7 (a) Microstructure of coating B. (4000x). (b) EDS spectrum.[18]

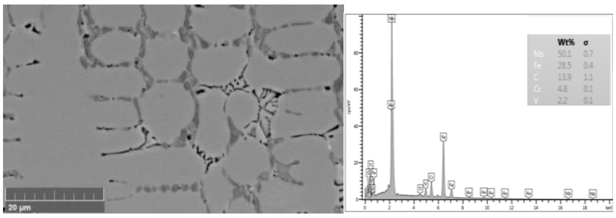

Figure 8 (a) Microstructure of coating C. (4000x). (b) EDS spectrum.[18]

SEM / EDS analysis confirmed the presence of M_7C_3 carbides in all alloys and niobium carbide (NbC) in C alloy as shown in Figures 6,7 and 8. Due to the chemical composition alloy A showed the highest percentage of carbides followed by alloy B and alloy C as presented in Figure 9.

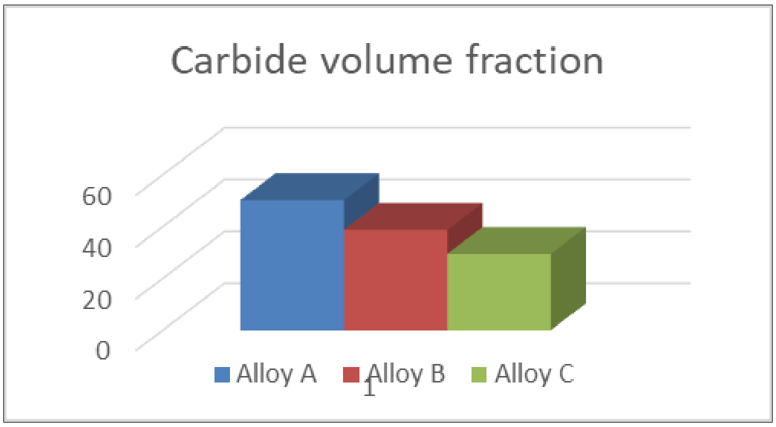

Figure 9 Carbides volume fraction in A, B and C.

3.4. Micro abrasion wear

A suspension of diamond particles (3µm mean diameter) in distilled water in a concentration of 0,35g/cm^3 has been prepared to acts as abrasive medium. The micro abrasion tests carried out with a ball abrasion micro machine by rotary ball, showed that the alloy C presented smaller dimensions of craters, lower worn volume and lower wear coefficient, although it presented lower hardness value in relation to the other alloys.

According to many authors hardness should not be considered as the unique indicator of wear resistance (Kotecki et al.) [23], 1998; Lemm et al. [24]), 2015) as proposed in Archard Equation. This work provides information in the same way. Figure 10 shows the crater diameters formed as a function of the slip distance and the Figure 11(a, b, c) shows the volume loss of each coating, the carbides volume fraction and the friction coefficient evolution for the three alloys during tests. Following the evolution of craters diameters, the worn volume for each alloy can be evaluated. The prevalent mechanisms of wear A, B and C are similar: well-defined parallels lines, which indicates the occurrence of micro-scratch.

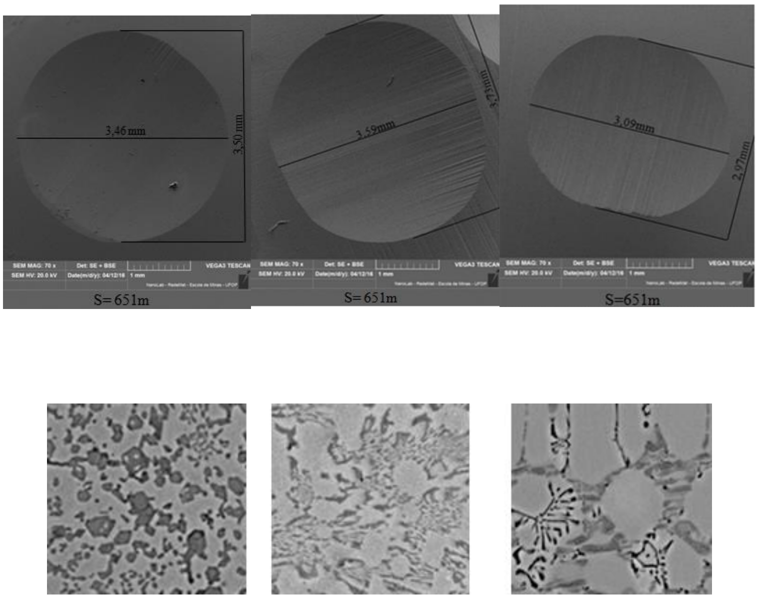

Figure 10 Wear craters and the corresponding microstructures for A, B and C coatings. SEM (4000x) [18]

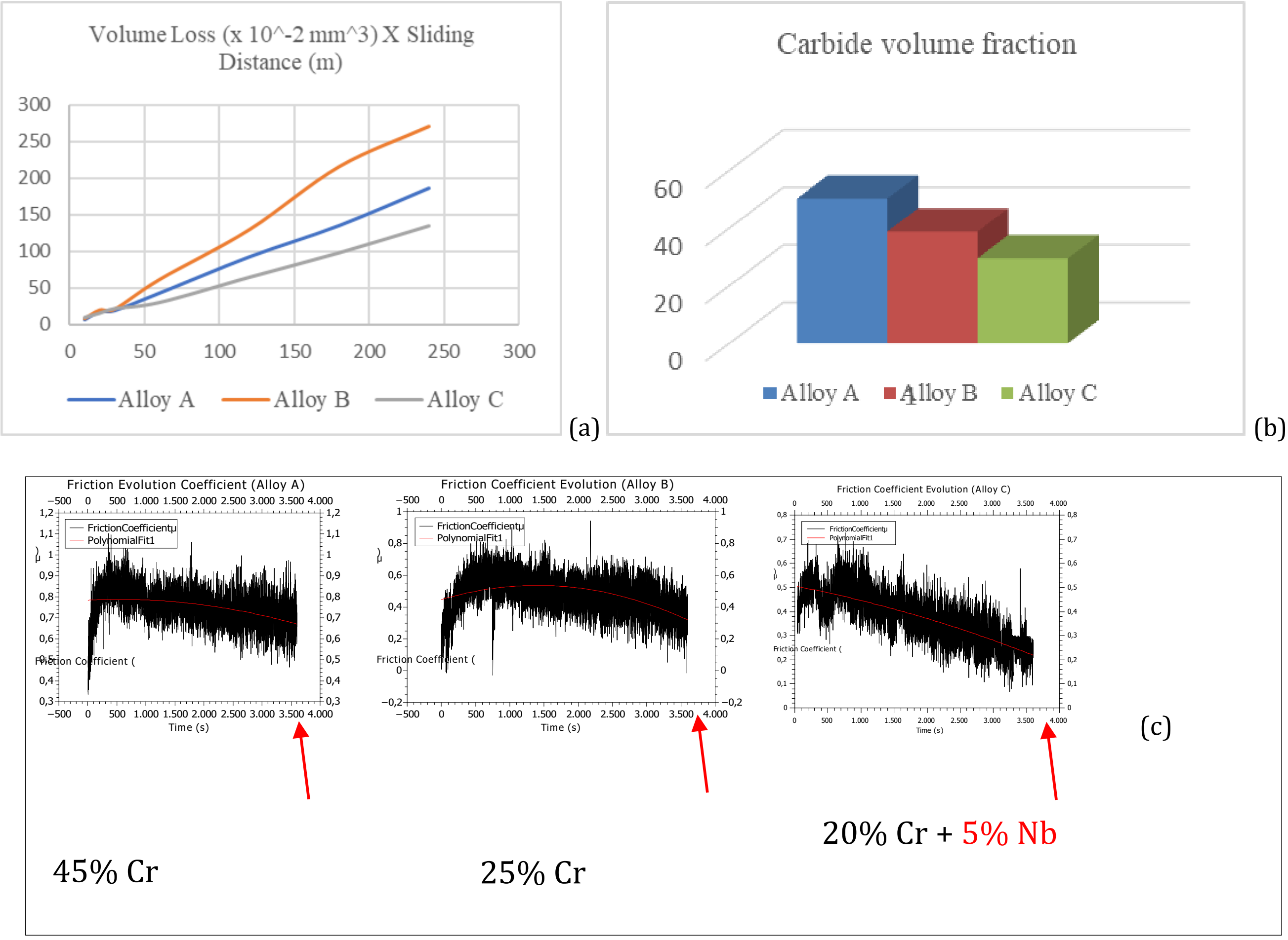

Figure 11 (a, b, c) Volume loss x Sliding distance (a), carbide volume fraction (b) and (c) the evolution of friction coefficient for A, B and C. In (c) Each plot corresponds to 7.200 collected points by the sensors (Normal and Tangential forces) during one hour of each test.

An interesting discussion comes from the analysis of the data summarized in Figure 11(c): the lowest and decreasing friction coefficient, has been measured for the coating containing NbC. For this coating, friction coefficient decreases faster than for the two other microstructures suggesting a more effective self-lubricant process probably provided by debris accumulation.

Comparing the worn volume as a function of carbides content (Figure 11(b)) the alloy C presented the lowest percentage of carbides; however, it presented the best resistance to wear. This may be related to the characteristics presented by these phases, such as the shape, size and adhesion of the carbides to the matrix (Zum Gahr [25], 1987). One can assume that the greater refinement of niobium carbides presents in the C alloy, made it difficult to remove them from the matrix increasing the resistance to wear. The M_7C_3 chromium carbides in the A and B alloys are coarser than those founded in C alloy.

Comparing the alloys A and B (both without niobium), coating A presented the higher percentage of carbides and less worn volume than B. This may be related to the more effective protection of the matrix by the carbides in A, since in alloy B carbides distribution provides greater free path for the movement of abrasive particles [26, 27, 28]. The volume fraction of carbides combined with their distribution influence the abrasion resistance.

Hamid Pourasiabi, J.D. Gates [29] and Tecza, G. [30] have done investigations with alloys containing Cr and Nb. Their investigations pointed to changes in topography of surfaces due to the formation of a protruding surface of carbides over the ductile matrix abraded during the wear tests. This surface configuration (tridimensional profile of protruding carbides) leads to a beneficial effect of carbides shielding and protecting the matrix for further abrasion/wear. Pourasiabi and Gates [29] doing micro-mechanistic observations of worn surfaces in high chromium white cast iron, alloyed with Nb, showed that NbC particles not only protrude from the matrix but do so more than Cr-rich M_7C_3 carbides. Salient NbC crystals in C alloy, can noticeably hinder progress of the abrasion event.

According to the observations done by [30] on the abrasive wear response of Hadfield cast steel the surface of this alloy shows deep scratches and grooves with embedded abrasive particles. On the contrary, the addition of niobium makes the wear of the samples more uniform: the surface of the samples is flat, the grooves and scratches disappear, and niobium carbides formed in the structure are protruding from the alloy matrix.

It was also observed that the abrasive wear resistance of the tested high-manganese niobium-containing cast steel is at least 3 times higher than that of Hadfield cast steel.

The addition of Nb makes the sample wear uniform without any grooves and scratches.

The results of our work point to the existence of similarities with the aforementioned works, allowing us to propose a mechanism for wear behavior of the alloy C and the friction coefficient reduction: the worn particles (third-body) were retained and accumulated between the steps of NbC act as a protective mechanism against wear. A tribolayer of hard particles becomes operative as a protective barrier against wear. This dense self tribofilm acts as a solid lubricant film between the sliding bodies reducing the friction coefficient and the wear rate. The previous observation is in accordance with the beneficial effect of the NbC as seen in the quantitative wear performance data. Because of the highest hardness of NbC, compared to chromium carbides, it can be assumed that during abrasion tests NbC remains attached in the matrix acting as protuberant barriers against the movement of the third-body (debris) (Figure 12).

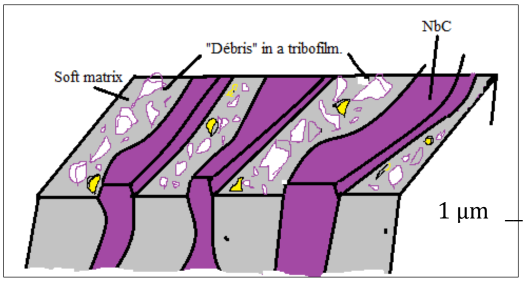

Figure 12 Illustration of the proposed mechanism for particles (microchips, oxides, ...) accumulation and formation of a thin layer responsible for wear reduction.

So, in our investigation, the lowest friction coefficient which was presented by coating C and its fast decreasing compared to A and B coatings may be justified considering the role played by NbC carbides as a micro constituent able to entrap debris providing a self-lubricant mechanism as illustrated and suggested in Figure 12. This hypothesis supposes the formation of a tribolayer of hard particles operating as a protective layer against wear. This dense self tribofilm acts as a solid lubricant film in the interface of sliding bodies reducing the friction coefficient and the wear rate.

4. Conclusion

The best performance in terms of abrasion resistance in the realized tests was observed to Fe-Cr-C (5%Nb) (Alloy C).

Results pointed out to an improvement in wear resistance of surface containing Nb carbides particles. The mass loss decreases as consequence. Fe-Cr-C-5%Nb alloy, exhibited better wear resistance, instead of lower values of hardness and volume fraction of carbides allowing it to candidate to hardfacing material.

For the Alloy C, a self-lubricant effect was observed and it is more pronounced compared to A and B alloys.

The carbide volume fraction is lower in the alloy with better performance in terms of mass loss (C). So, it indicates that the nature, morphology and distribution of these particles play a decisive role because the overall hardness is low compare to the others. This is not in accordance with Archard`s Equation and therefore suggests that refined aspects describing microstructures should be introduced into that model.

Continuously decreasing in friction coefficient for alloy C, as the test progresses, suggest a self-lubricant phenomenon more effective than the one observed for A and B alloys encouraging observational studies and comparative characterization of the different tribolayer of hard particles (debris).

Alloy A showed the highest percentage of carbides followed by alloys B and C. However, the wear resistance of alloy A was lower than that presented by alloy C. This behavior may be related to the fact that in A, carbides are coarser than in B and C providing less adherence in the matrix.

Comparing the alloys A and B, whose carbides have similar characteristics, the higher wear resistance was observed to the alloy A in which the highest percentage of carbides has been measured.

The presence of niobium carbides in the C coating should be considered the reason for the higher resistance to abrasion. It is known that these carbides present strong adhesion to the matrix. Moreover, because of the niobium presents a greater affinity for carbon, it may have contributed to the enrichment of matrix in chromium carbide that is more resistant to wear than other carbides.

For all alloys the prevalent mode of wear was scratching.

Compliance with ethical standards

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Disclosure of conflict of interest

No conflict of interest to be disclosed.

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