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Study of Wear Test Parameters in Micro-scale coating Nickel Base Deposited by Thermal Spraying

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Abstract. *The present study aimed to investigate the influence of the test parameters of the wear behavior of micro-adhesive coatings for wear based on nickel substrates deposited by flame spraying process in a low carbon steel. This type of testing is well established for thin coatings produceded by nitriding, but studies the applicability of thermally sprayed coatings still scarce. The experimental apparatus consisted of a wear ball type micro-computer control with fixed load and speed. We use a lot of 300N and 159.9 and speeds of 300, 400 and 500 rpm. An AISI 52100 steel ball was set in motion in contact with the coated surface of the sample, producing a crater wear semi-spherical. After the measurement, by means of optical methods, the dimensions of craters volume of material removed from each test was plotted as a function of the sliding distance. We also conducted craters using optical microscopy under a microscope with polarized light to determine the wear mechanisms of action. It was found that the charge used has low influence on the volume removed in the test, while the wear rate showed significant influence. With the increase of speed was observed an increase in volume of material removed. The abrasive wear mechanism was the predominant three bodies in all test conditions experienced. Additionally, the addition of NbC increased hardness and wear resistance of the coating Ni-Al + 25% NbC.*

Keywords: *micro-wear, thermal spray, wear mechanisms, adhesive wear.*

1. INTRODUCTION

The surface of a material is an area of maximum importance, since It is where the contact with the environment and with other mechanical or structural components occurs.

Wear is one of the main problems that leads to surface damage of materials and combating it is of great economic significance, due to the reduction of machines and equipment down time, need ed for parts substitution and maintenance. Over time, various treatments have been developed for surface protection, against wear and / or corrosion. The main treatments used are: carburizing, nitriding, boronising, PVD, CVD, electrolytic deposition and thermal spraying (GW Stachowiak 2005).

Thermal spray includes a set of processes that differ primarily in the way used to heat up the material to be deposited, either by combustion or electricity. All processes are based on the same principle: the spraying material in the form of powder or wire is atomized by a jet of hot compressed air (or other process gas) and accelerated to the substrate previously prepared, namely cleaned, preheated and with a certain roughness to ensure the mechanical anchoring to the piece to be coated (MELLALI, M., FAUCHAIS, P., GRIMAUD, 1996). The particles collide with the substrate in a molten or semi-molten state, plastically deforming and giving rise to a lamellar structure. The particles impacted and deformed are called "splats" (ASM, 2004). Coatings deposited by thermal spray are microstructurally heterogeneous, with the presence of oxides, pores and un-melted particles (ASM, 2004). The thermal spray stands out in relation to other treatments cited by the following factors:

• The spray processes do not impose excessive heating the substrate, thus avoiding undesirable metallurgical transformations;

• It has a great number of possible applications and can spray virtually any type of material (polymer, metal, ceramic or composite) on any type of substrate;

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• Allows the coating of large pieces and at the workplace what many experts consider to be the biggest advantage of this process.

• For parts that have undergone wear, allows the reconstruction of the damaged area avoiding the replacement of the entire piece (AWS, 1985).

In 1995, a test method for micro-scale wear evaluation of coatings called "calotest" was developed, ideal for small thicknesses coatings (RUTHERFORD KL; HUTCHINGS IM 1996).In this test a sphere rotates on its axis and in contact with the surface of the sample to be tested with a specific contact force. During the test an abrasive solution is dripped in the region of contact between the sample and the sphere. In the contact region a wear cap is created that may have its diameter measured by optical methods and its volume calculated (RUTHERFORD, KL AND HUTCHINGS, IM, 1997). The result of this test may be provided in the form of a graph of the volume removed against the distance traveled. There are several factors that influence the results of these tests, such as: load used, the relative velocity of the surface, ball material and surface condition of the ball and the test specimen (GEE, MG 2005). This test is well established for homogeneous layers as those produced by nitriding but has received little attention for the evaluation of thermally sprayed wear resistance coatings.

The objective of this work was to study the influence of the addition of 25 wt% NbC in the wear resistance of a nickel based alloy, as well as the effect of the applied load and test speed in the micro-adhesive wear test method.

2 - Materials and methods

The flowchart in Figure 1 illustrates the steps followed in the execution of this work.

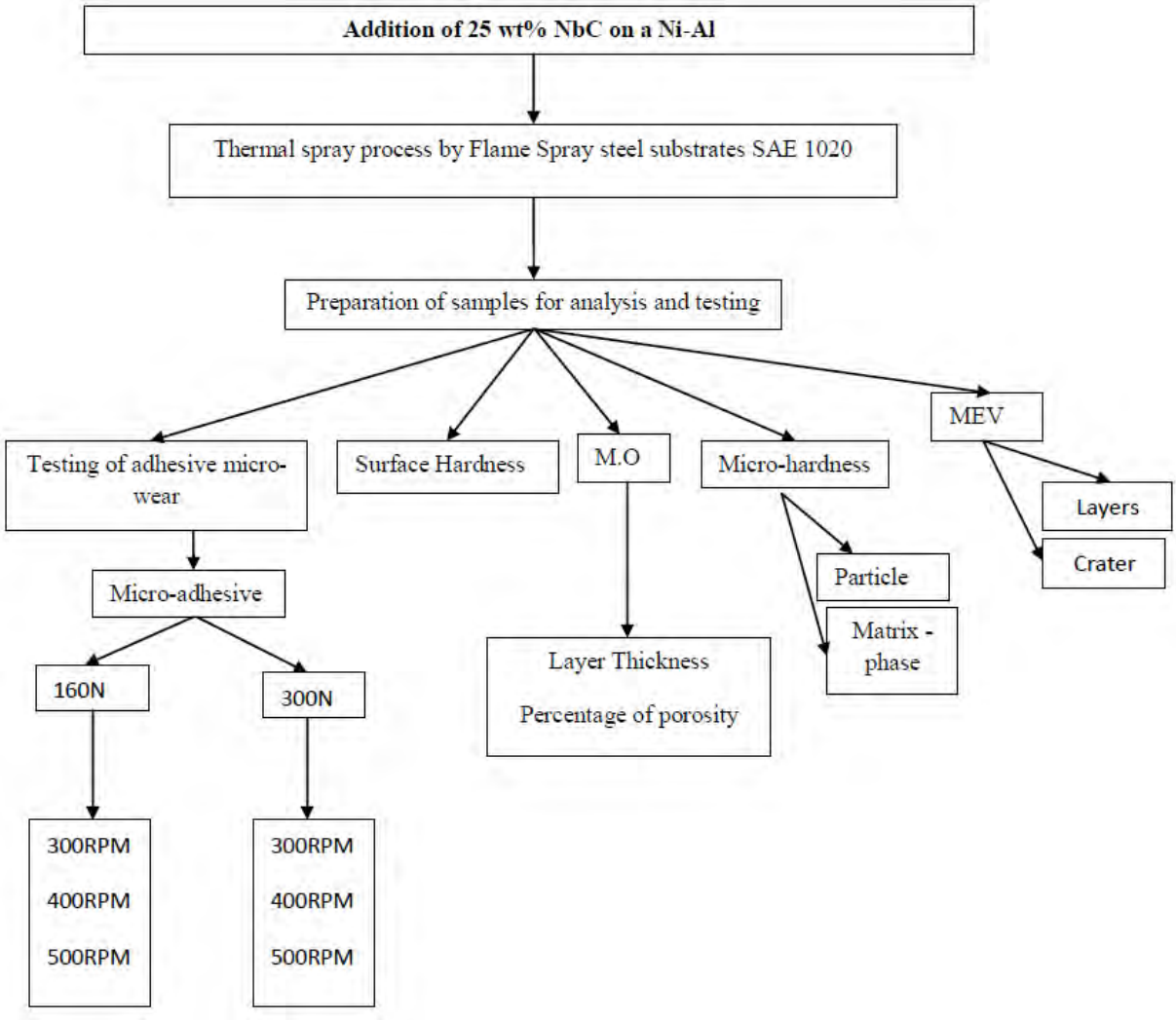

Figure 1. Flowchart of experimental work.

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This study used a nickel based alloy (L_1) whose composition is shown in Table 1.

Table 1. Composition of the base alloy used (%wt.).

B	Al	Mo	Ni
0,004	5,5	5,13	89,33

The L_1 alloy was mixed with 25wt% of niobium carbide powder, forming the L_2 alloy (75% Ni-25% Al + NbC). Quantities used were measured with a precision balance.

The spray process used was the Powder Flame Spray. Three plates of 100 mm x 100 mm x 5 mm had their surfaces abraded and cleaned with solvent to obtain the proper surface roughness suitable for the mechanical anchoring of the "splats" and to remove grease, oil and contaminants in general. The edges of the plates were rounded to avoid detachments after spraying. The spray parameters used are shown in Table 2.

Table 2. Parameters used in the coating.

Spraying Parameters	
Oxygen (bar)	4
Acetylene (bar)	0,7
Compressed air (bar)	1
Spraying distance (mm)	150
Pre-heating (° C)	70

After spraying, the plates were sectioned using a SiC abrasive disc to obtain the samples for the tests reported in the flowchart of Figure 1.

Optical microscopy observations were performed using a Zeiss Axiolab microscope with integrated camera and image analysis software AxioVision. Analysis of the images were performed to determine the thicknesses and porosities percentages of the coatings. Analysis by scanning electron microscopy were performed to study the morphologies and compositions of the coatings, to determine the prevalent wear mechanisms as well as the possible mechanism changes due to the variation of the test parameters. The samples for microscopy analyses, micro-hardness testing and for hardness and wear testing were prepared by grinding to 1200 mesh sandpaper followed by polishing with 0.3 and 0.05 μm alumina.

The surface hardness was measured on a universal Leco Model RT-240 durometer tester with a load of 15N and diamond indenter. 20 measurements were performed in the longitudinal section of each coating. The micro-hardness evaluation used a digital micro-hardness tester with a load of 100 gf .The matrix was measured 20 times and the phase particulate present in the alloy L_2 was measured 10 times.

The micro-scale adhesive wear tests were performed in a testing equipment of the type calottes with fixed sphere, as shown in Figure 2. The parameters used were: loads of 160 e 300N ; rotation speed of 300, 400 and 500RPM with test times of 5, 10, 15, 20, 30 and 40 minutes.

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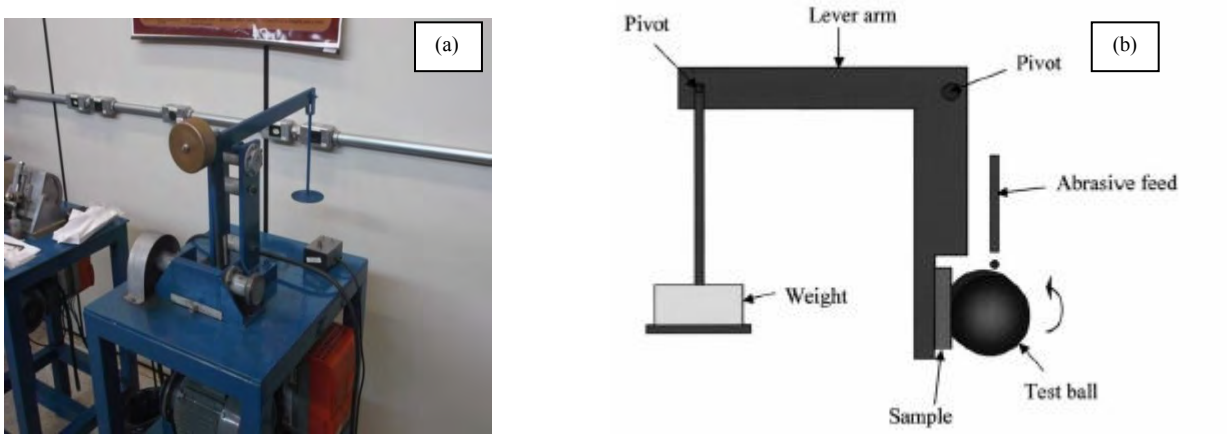

Figure 2. (a) Testing machine for micro-adhesive wear, Department of Materials Engineering - EESC-USP. (B) Illustration of the micro-adhesive wear equipment.

For each alloy tested and for each combination of parameters, the test was repeated three times and the volume removed was calculated based on the average diameters of each series of tests.

The diameters of the formed caps were measured using an optical microscope and equation (1) was used to determine the volume of material removed during the test.

$$V \approx \frac{\pi \cdot b^4}{64 \cdot R} \quad \text{equation (1)}$$

Where:
b: diameter of the cap (mm);
R: radius of the sphere (mm);
V: volume of material removed (mm ³).

3 - Results and Discussion

3.1 - Morphological characteristics of the layers and powders, and layer thicknesses

In the micrographs of figures 3 (a) and 3 (b) are shown the aspects of the powders used in the production of coatings L_1 and L_2.

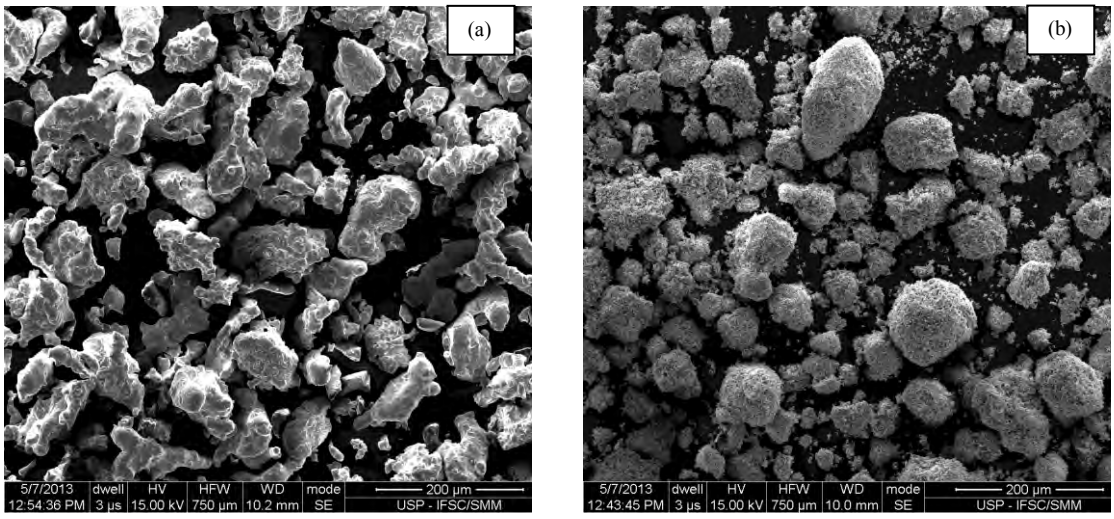

Figure 3. (A) Scanning electron micrograph of the powder alloy L_1. (B) Scanning electron micrograph of the powder added for the production of alloy L_2.

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Table 3 shows the measurements of the average diameter of the particles.

Table 3. Measurements of average particle diameter of alloy powder and the powder added for the L_2 alloy production.

	Particle size - Alloy L_1 (μm)	Particle size – NbC Powder (μm)
Average	98,9	107,3
Deviation P.	19,5	26,9

Next the alloy L_1 and the NbC powder were measured with alloy L_1 showing a greater uniformity in the particle size distribution when compared to the NbC powder that showed a relatively high variation of sizes.

Figure 4 exhibit an compositional image made by SEM. It shows that the L_1 produced layer has the characteristic aspect of the flame spray process with the presence of oxides and un-melted particles. The micro probe analysis reveals that most of the layer is composed of a nickel matrix (grayish regions) and with the aluminum being combined with oxygen (dark particles). The oxides were produced during the spraying operation when the particles reacted with the atmosphere.

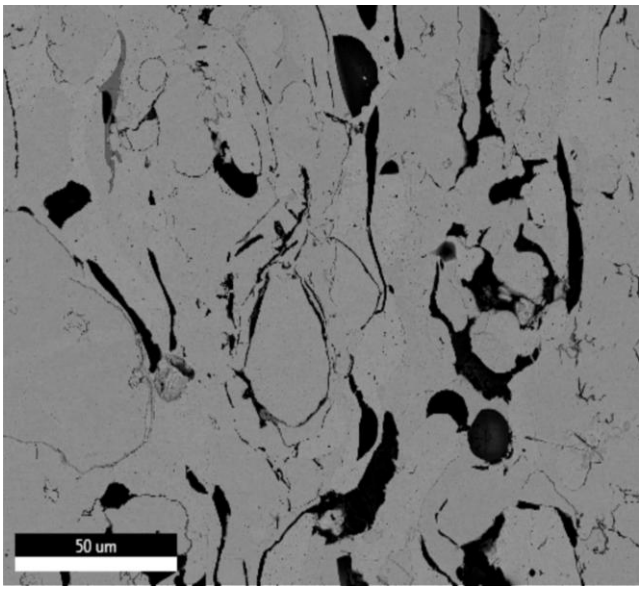

Figure 4. (a) Compositional image of the L_1 alloy layer.

Figure 5 presents the layer produced by the L_2 alloy. The image shows a nickel matrix (dark grayish regions) with the presence of un-melted NbC particles (light grayish regions). The dark regions are oxides and pores.

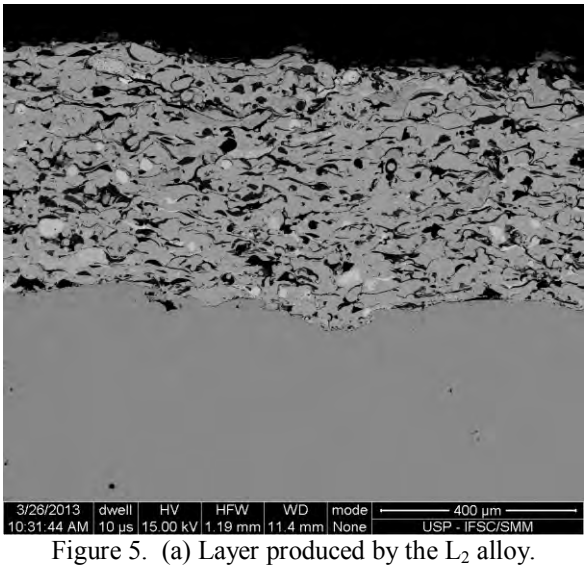

Figure 5. (a) Layer produced by the L_2 alloy.

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Table 4 shows the fraction in % of the pores and the average thickness of the L_1 and L_2 layers. The layer with NbC (L_2) showed a slightly thicker layer for a given number of spray passes and a lesser amount of porosity when compared to the L_1 alloy layer.

The porosity levels for both layers (L_1 and L_2) were considered low, once it is usual porosity levels in the order of 20% for these types of layer.

Table 4. Fraction (%) of porosity and thickness of the coatings formed by the sprayed alloys L_1 and L_2.

	Porosity (%)		Thickness (mm)	
	L_1	L_2	L_1	L_2
Average	4,3	1,8	0,54	0,77
Standard deviation	0,9	0,88	0,05	0,15

3.2 - Hardness and micro-hardness

The values in Table 5 indicate that both the hardness as the micro-hardness of the alloy L_2 were higher than that of the L_1 alloy. It is believed that this may result from the addition of the reinforcing element to form the alloy L_2. Measurements of micro-hardness of NbC particles in the coating L_2 presented average values of 1700 HV. The alloy with the addition of niobium carbide showed an17%increase in hardness measured by HR15N scale while the measurements of micro-hardness (HV) showed an increase of 40%.

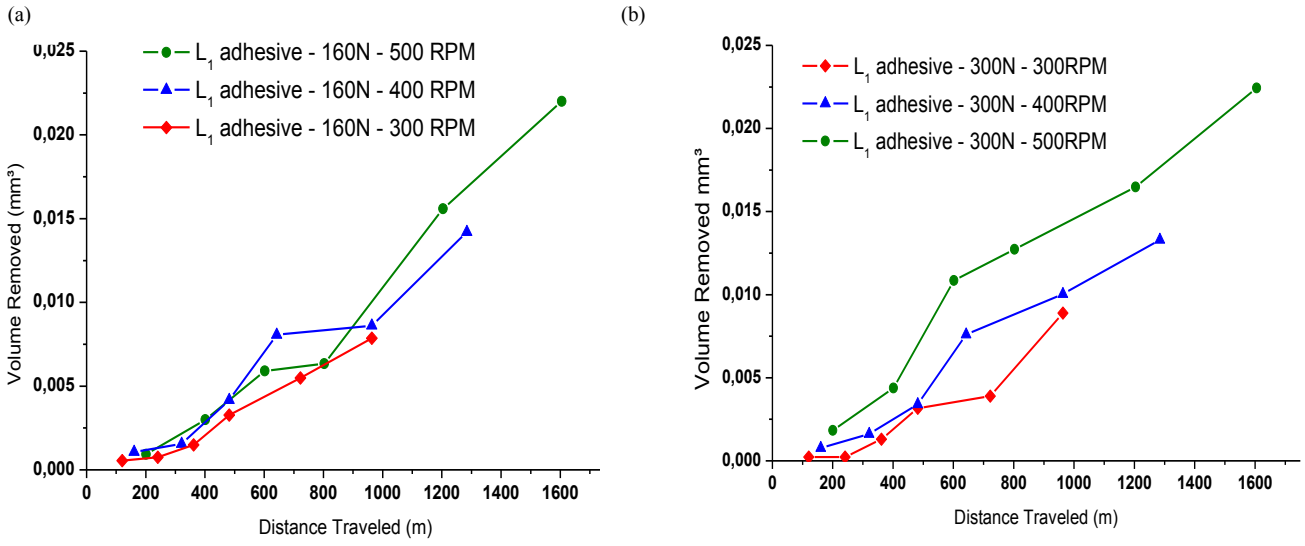
Table 5. Rockwell surface hardness and Vickers micro-hardness.

	Superficial Hardness (HR15N)		Micro-hardness (HV)	
	L_2	L_1	L_2	L_1
Average Value	58,1	51,1	269,0	161,97
Standard deviation	0,8	1,9	18,1	26,1

The largest standard deviation in the micro-hardness test arises from heterogeneity of the coatings formed by lamellae of different compositions, structures and properties. In the surface hardness test, the indenter is bigger, encompassing an larger area.

3.3 - Micro-abrasive Wear Testing

Figures 6 (a) to 6 (d) shows graphs of micro-adhesive wear for the two alloys using different combinations of the parameters tested.



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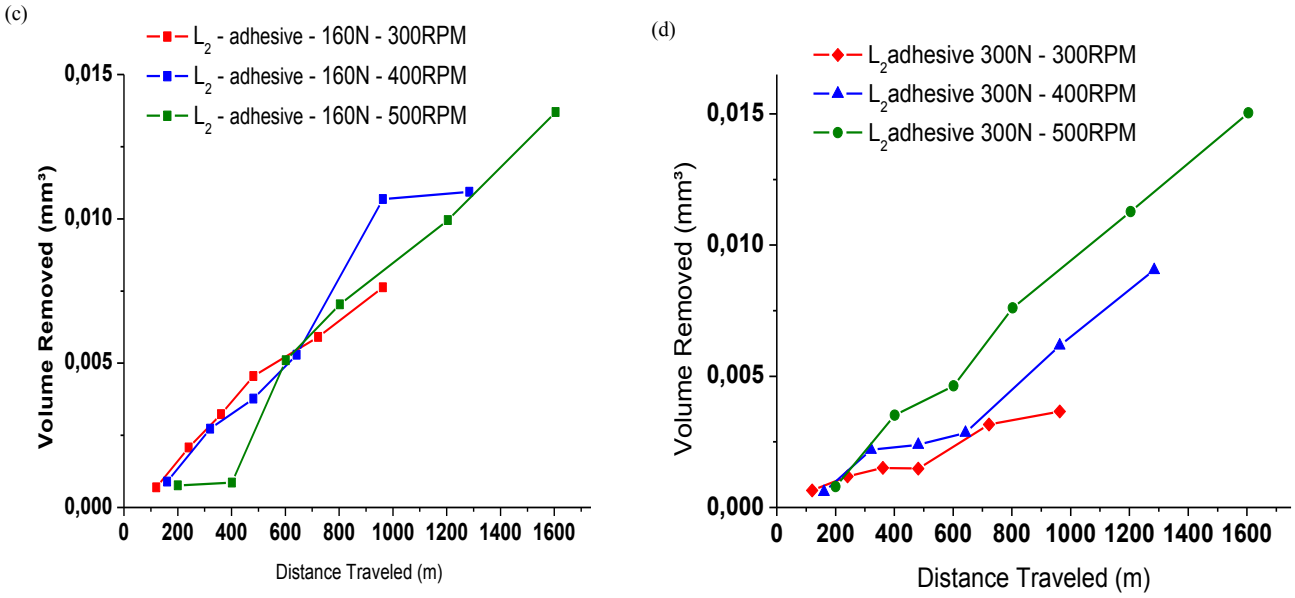

Figure 6. (a) Micro- adhesives wear tests: L_1 - 160N - 300, 400 and 500RPM. (B) L_1 - 300N - 300, 400 and 500 RPM. (C) L_2 - 160N - 300, 400 and 500 RPM. (D) L_2 - 300N - 300, 400 and 500 RPM.

The increase in rotational speed had a significant effect on the maximum volume of removed material, but only for the test load of 300N: the maximum volume removed increased with increasing rotation speed. Comparing the alloy with and without the addition of 25 wt% NbC, it is verified that for every combination of load and speed the alloy L_2 (with NbC) showed a lower total volume loss.

CONCLUSIONS

• The addition of NbC in the amount of 25wt% in the Ni-Al based alloy, L_1, altered the microstructure of the formed layer. The L_1 and L_2 alloys presented a Ni matrix with dispersed molybdenum and with all aluminum combined with oxygen to form aluminum oxide. The micro-analysis in conjunction with the micrographic analysis revealed that the coating produced by the L_2 alloy presented un-melted particles of NbC, with high hardness.

• The addition of NbC increased the surface hardness (considered to be representative of the full coating) and the micro-hardness of the matrix phase. This is due to the addition of reinforcement particles that restrict the deformation of the matrix phase and bear part of the burden which the array is being submitted.

• The addition of NbC increased the wear resistance of the L_2 alloy. This may be due to the fact that the addition of particulate phases tend to increase the wear resistance of the materials.

• The increase in testing speed causes a significant increase of the total volume removed for the higher load tests.

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