Wear behavior of Ni-B electroless films on a steel S7

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Abstract

Wear resistance of materials is a very important aspect from the economic point of view, since lack of wear resistance is one of the important ways for materials to lose utility. To improve wear resistance of materials that are in relative movement, one against other, several methods can be applied. One method that is gaining importance is the application of electroless films, being the Ni-B electroless plating a relatively new way to obtain high wear resistance films on metals. In this work, the wear tests results of the film of Ni-B in its amorphous state and after different heat treatments are presented. The heat treatments were done from 200 °C to 550 °C with intervals of 50 °C. The film Ni-B was characterized by optical microscopy, scanning electron microscopy (SEM), X-ray diffraction and microhardness. Results of X-ray diffraction shown that the film without heat treatment is amorphous, and after the heat treatments, intermetallic Ni_3B precipitates are formed, which can be the cause of the increase of the hardness of the film Ni-B. Surface characterization by SEM of Ni-B films obtained at different immersion time, shown that deposits have cellular morphology and as treatment time increases, morphology becomes nodular. The greater hardness Vickers is obtained for the heat treatment at 215 °C and 36 hours.

Introduction

Since the development of commercial viable form in 1946 by Brenner and Riddell, the processes of nickel electroless, have grown since a curiosity of laboratory to an industry of several thousands of million dollars. Currently, there is a continue advance in the development of more stable bath formulations and the application of films with more predictable behavior in periods of long life and under different operation conditions. The electroless plating is produced by the autocatalityc reduction of ions of nickel of a solution, on the surface of a catalytic substrate using hypophosphite, amineborane or borohidrure as the reducing agents (1) (2) (3).

Technical literature describes electroless plating processes to apply a deposit of Ni-P, even ternary alloys of Ni-P involving as ternary element W, Mo, Co, etc.. For the case of the coatings Ni-B not information exists as abundant as in Ni-P, although apparently those coverings have properties with certain advantages such as values of hardness of to 1200 HV, which are obtained with an appropriate heat treatment, after applying the coating, and those films can applied at low temperatures (4) (5).

Of the problems that concerns the baths for Ni alloys plating, the stability of baths is one of the most important, many investigations with different stabilizers have been carried out such as those that have ions of lead, iodate, 2-mercaptobenzothiazole (2-MBT) and malic acid. According to Das and Chin (6) the stabilizers retard the velocity of deposit on the inclusions of carbon and reduce the content of phosphor in the coating, additionally it was found that sulfur and lead deposit simultaneously in presence of 2-MBT (6).

Studies of wear have been carried out with alloys Ni-P, Ni-B and Ni-Composite. For plating with Ni-P with particles of SiC, it was found that unless an increase of their resistance to wear and values of greater hardness to 1000 HV are obtained, normally they are porous coatings and they show low resistance to corrosion (7). Also baths of Ni-P adding MoS_2 have investigated, being obtained a decrease of the coefficient of friction and adhesive wear after an adequate heat treatment (8).

Actually, the deposits of Ni-P-(W, Mo, Co) and Ni-B are being used in the chemical, automobile, aerospace, petroleum, electronic and textile industry, as well as the development of medical equipment, since a great potential of applications is observed which will allow a better performance of coated materials (3).

In this work, the objective is to determine the behavior in wear test of Ni-B plating on steel S7. Steel S7 is commonly utilized to manufacture tools that are under abrasive wear.

Experimental

To determine the better conditions of application of the electroless bath of Ni-B that will provide the adequate hardness for the tests of wear, plates of steel S7 samples were prepared with dimensions of 10 mm x 10 mm x 3 mm. Samples were treated according to the procedures of surface preparation from international standard ASTM B733-97 and ASTM B322-99 (9) (10).

The samples were immersed in the bath of Ni-B, during 15, 30, 60 and 90 min. The concentration and the conditions of the bath of Ni-B electroless can be seen in the table 1. A micro structural characterization was done to samples taken the cross section of films, through an optic microscope in order to determine the thickness and revealing the microstructure of the coating Ni-B.

For the heat treatment study an immersion time of 60 min for all samples was applied. Thermal treatment was made from 200 °C until 550 °C with intervals of 50 °C, and each sample was maintained at selected temperature during 90 min. Microhardness was measured before and after heat treatments for each sample.

TABLE 1 Compositions and Conditions of the bath Ni-B electroless

Reactive	Concentration (mol/L)
Source of nickel ions Ni^{2+}	0.19
Reducing Agent	0.40
Complexing Agent	0.09
Stabilizer	0.00007
Conditions	
Temperature	60-70 °C
pH	5-6
Agitation	Constant

Additionally, other samples were treated at 215 °C and 36 hours of permanence according to suggestions found on technical literature (11).

For the tests of wear, from a cylinder of steel S7, 9 disks of wear with 101.6 mm of diameter for 6.35 mm in thickness and a hole of 6.35 mm of diameter were manufactured. Also for the material against which samples were wear; from a cylinder of stainless steel 316, wheels of 52.47 mm of diameter 12.5 mm thickness and of with a hole of 16.4 mm were manufactured. The tests of abrasion were carried out with 500 g of load and 500 cycles.

The characterization of the zone of wear for plated steel S7 samples, before and after the tests of wear, was carried out in a Jeol 580-SLV scanning electron microscope. Chemical analysis of the film of Ni-B was carried out by means of the stripping of the coating with a mixture of nitric acid and acetic acid according to method described by Riedel (1) and analysis by means of a spectrometer of inductively coupled plasma emission (ICP).

Result and Discussion

The results of the chemical analysis indicate a content of boron in the film of 4-5% in weight. In the figure 1, the thickness of the coating Ni-B for the studied immersion time treatments in the bath of can be observed, i.e., 15, 30, 60 and 90 min. As can be seen, the thickness directly proportional to immersion time. Also is observed that the appearance of the coating tends to have a nodular growth for greater times of immersion. The images also show that good adhesion is obtained for Ni-B film.

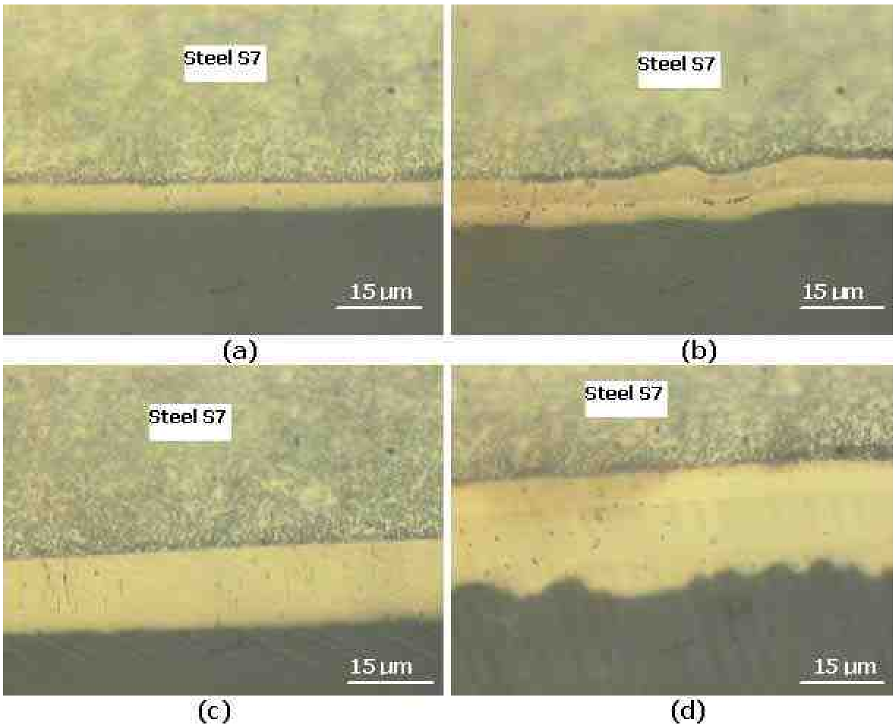


Figure 1.- Microstructure determined by optical microscopy of cross section of Ni-B films with times of immersion (a) 15 min, (b) 30 min, (c) 60 and (d) 90 min

The figure 2, shows the surface morphology of coatings and as can be observed, at low immersion times, the growth of the film is cell-like and as immersion time increases, the film presents a nodular growth. The figure 3, shows the hardness Rockwell “C” of Ni-B films after heat treatment of samples. As can be seen, major value of hardness is obtained when the heat treatment is carried out at 200 °C during 120 minutes.

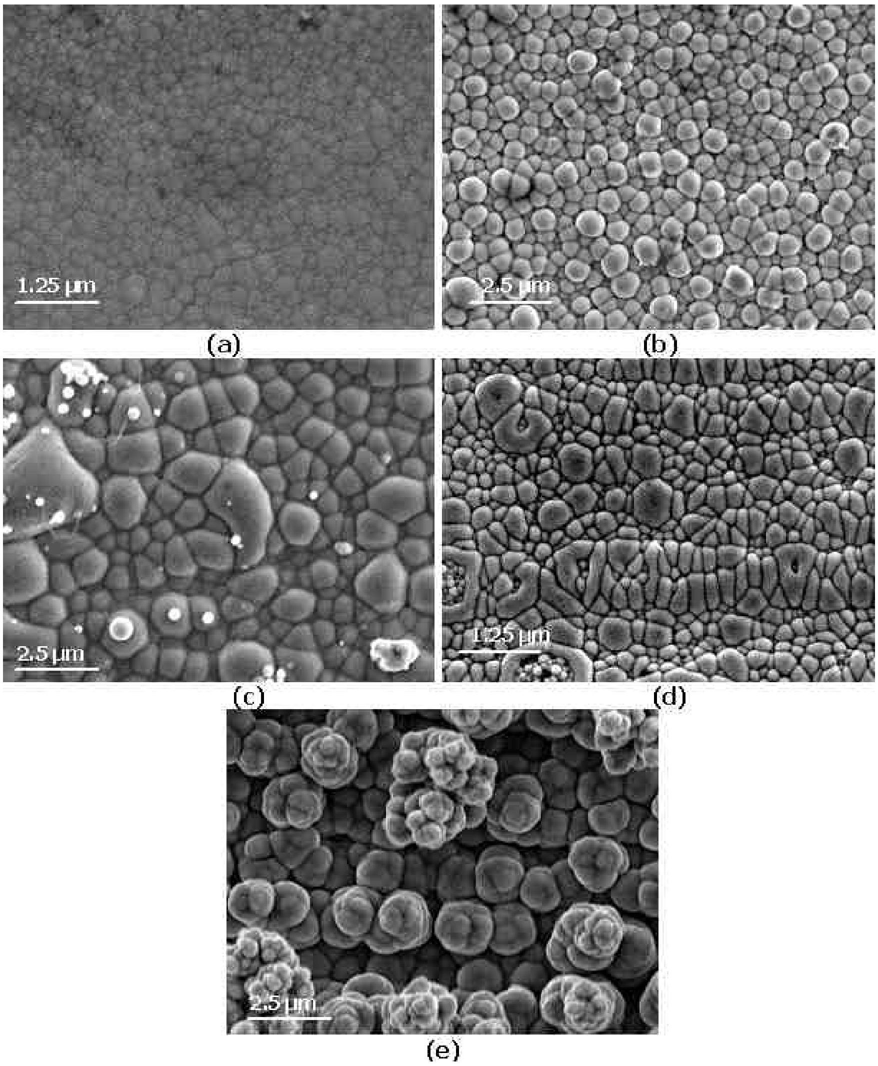


Figure 2.- Scanning electron microscope microphotographs of surface morphology for Ni-B coatings obtained with immersion time of (a) 15 min, (b) 30 min, (c) 60 and (d) 90 min

Once the best heat treatment was identified, nine steel S7 wear disks were plated with the film of Ni-B. For these samples immersion time was 30 minutes, and heat treatment at 215 ° C for 32 hours. The readings of microhardness were obtained with Vickers microhardness and values were converted to Rockwell “C” hardness. Results can be seen in the table 2. The average of Rockwell “C” hardness was 63.6, it must be observed that sample 1 corresponds to the value obtained for sample without heat treatment.

As it can be seen in the table 2, the disks with the film of Ni-B increase in weight instead of diminishing, probably due to stainless steel coming from the wheels used to wear samples. The scanning electron microscope microphotograph obtained for plated samples exposed to wear treatment is shown in figure 5. It can be observed the integrity of Ni-B plating, and how in the zone of the wear, there are particles which due to its content of Cr, Fe and Ni can be considered that they come from particles of stainless steel of wheel used to wear the studied samples.

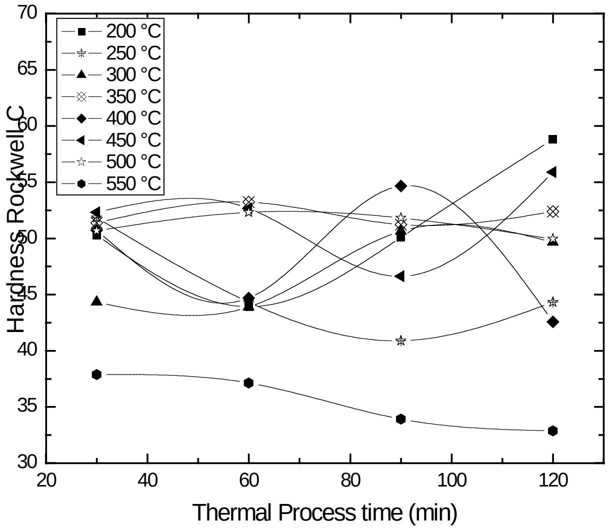


Figure 3.- Hardness of Ni-B film vs heat treatment time and temperature of heat treatment.

Table 2.- Weight loss (wt %) after wear testing and hardness of plated surface

Wheel	Hardness (Rockwell "C")	Weight lose (mg)
1	41	- 8
2	63	+12
3	63	+10
4	58	+23
5	66	+20
6	65	+26
7	65	+19
8	63	+8
9	62	+18
10	65	+17
Average	63.6	+ 14.5

Figure 4 shows x-rays diffraction pattern for samples after heat treatment. According to the XRD results, for samples treated at higher temperature, a predominance of Ni_2B is observed and after 450 °C the formation of segregated pure nickel phase is formed. The formation of these two phases does not improve the hardness, and according to the results, the starting of formation of Ni_3B phase from amorphous structure produced during plating, gives the best values for hardness.

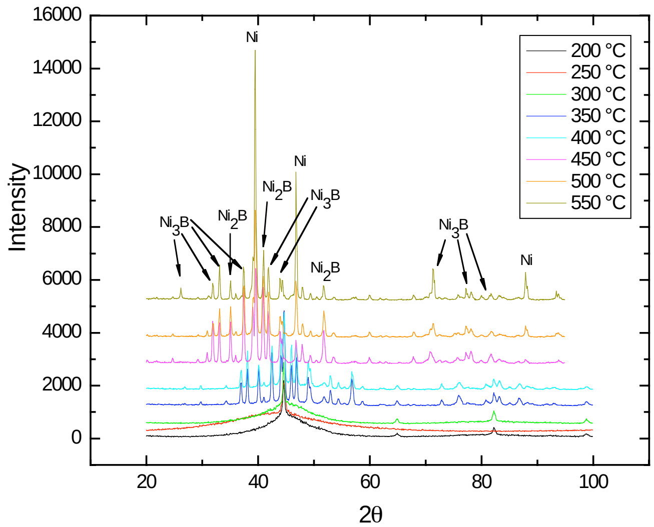
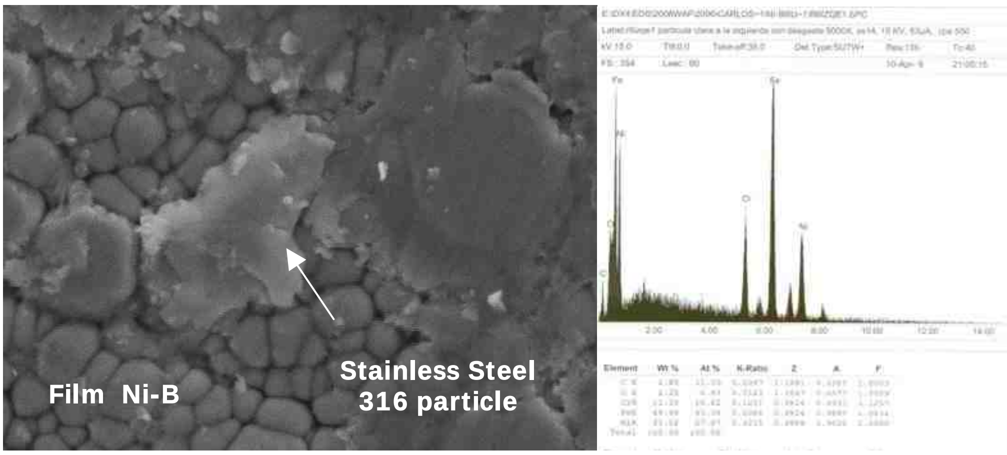


Figure 4 X-Rays of the samples of steel S7 with the coating Ni-B without heat treatment and with the heat treatments proposed



(a) **(b)**

Figure 5 (a) Microphotographs of SEM showing disk wear zone a particle of Stainless steel 316 (b) Spectrum EDS of particle of stainless steel 316

Conclusions

1. For the electroless plating of steel with Ni-B layer, the surface morphology of the deposit changes of cell to nodular with the time of immersion.
2. A hardness of 63 Rockwell "C" is obtained when heat treatment is made at 215 °C for 36 hours, which represents around 50 % of increase for an untreated piece.
3. The increase of hardness after heat treatment probably is due to the formation of the phase Ni_3B.
4. The decrease of hardness with the heat treatments above 450 °C, seems to be consequence of the formation of phase Ni_2B and Ni.
5. The Ni-B plating improve the wear resistance and at tested condition any wear was detected.

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