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Studying Mechanical Properties of Ni-P-B_4C-PTFE Hybrid Coatings by Electroless Method

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Abstract – **In this study, mechanical properties of Ni-P-B4C-PTFE hybrid coating produced by Electroless method were investigated. Ni-P, Ni-P-PTFE, Ni-P-B4C and Ni-P-B4C-PTFE coatings were created on Aircraft Grade Aluminum samples. Boron carbide particles 4 grams per liter and PTFE Suspension 10 grams per liter were added to Electroless baths with average percentage of phosphorus. Pin on disc was used in order to determine the wear resistance and friction coefficient. Wear and corrosion resistance of samples was tested. Finally, results of four types of coatings were compared. The results showed that adding boron carbide particles resulted in increased corrosion resistance, but adding PTFE particles resulted in decreased corrosion resistance. Ni-P-PTFE composite coating hadthe lowest friction coefficient and Ni-P-B4C composite coating had the highest corrosion resistance and hardness, and Ni-P-B_4C-PTFE hybrid coatings showed the best wear resistance.**

Keywords – **Electroless, B_4C- PTFE, Wear, Corrosion, Hardness.**

I. Introduction

Using surface technology is inevitable due to improve the surface properties and protect surface of parts in various applications. Applying coating on surface of parts is one of the most important ways to improve surface properties and protect surface against damages such as wear and corrosion. For example, using aluminum samples without coating will result in exacerbate part corrosion [1]. In Electroless coating, external current source is not used and deposition is the result of metal ion recovery to metal atoms in the presence of reducing agents. Electroless coating has found special industrial importance because of advantages such as creating a uniform layer, no need to electrical equipment for electroplating and no limit in systems size [2]. The Ni-P Electroless plating has a variety of applications in various industries, including electrical and electronics, oil, gas and air and space industries [3]. The latest advances in Electroless Ni-P coating is simultaneous deposition of solid particles during coating that can be hard or soft particles depending on type of particle [4]. Hard particles such as SiC, Al2O3 and B4C are used to increase hardness and wear resistance of coatings and soft particles such as PTFE, MoS2 and graphite reduces the friction coefficient and increase coatings lubrication as a dry lubricant through placing in Electroless coating of Ni-P [5-12]. Boron carbide is a black gray ceramic that is considered as a very hard abrasive material with fine hardness 3000 kg/ mm3 after diamond with hardness of 9000 kg/ mm3 and Cubic BN with hardness of 4400 kg/ mm3. Boron carbide hardness in high temperature is more than diamond and cubic BN. Another strategic property of boron carbide is its high neutron absorption that combined with the high refractory result in its usage in construction of nuclear facilities [13 and 14].

Ebrahimian et al examined hardens of Ni-P-B4C coating foe various quantities of boron carbide particles with average size of 10 microns. The highest hardness (about 1300 Vickers) has been reported increasing boron carbide particles with amount of 8 grams per liter [15]. Iraqi et al studied on wear resistance of Ni-P-B4C coating on magnesium substrate for 2 g per liter with an average size of 2 microns compared to magnesium [16, 17].

Solid lubricants are typically used in places where liquid lubricant cannot be used. These include high temperatures, extremely low temperature or high vacuum [18]. The only polymer that is widely used as coating and lubricant on hard materials is PTFE. These particles are preferred as solid lubricants because of properties such as proper lubrication, inability to Solubilisation in most solvents, good chemical stability and lower friction coefficient compared to other polymers because of lower surface energy. These lubricants provide a layer between sliding surfaces that has less shear strength than two surfaces or in some cases it may be keep totally separate surfaces [19-21].

In this study, the properties of hardness, wear and corrosion resistance of Ni-P-B4C hybrid coating with an average size of 2 Micron B4C particles and 200 nm PTFE particles on Al 7073.

II. Method

In this research, aluminum alloy 7023 plates in 20*20mm dimensions have been used for coating. Chemical analysis of prepared samples is shown in Table 1.

Table 1: Chemical analysis of sample Squanto meter made of aluminum alloy7023

Component	Percent
Al	89.973
Cu	1.4
Mg	2.2
Si	0.7
Fe	0.53
Mn	0.3
Ni	0.02

Zn	4.7
Pb	0.05
Sn	0.007
Ti	0.02
Cr	0.1

Before any plating operation on metal sample, surface preparation operation must be performed so that coating sits on it. To achieve this goal, one or more preparation actions must be done in order to clean up the surface pollution and it activation. Samples were firstly sanded and polished in order to clean up pollutions and varnish surface. The preparation of sample surface was done as follows, explaining that parts were washed with distilled water for two minutes each step:

In the first stage, surface fats were washed with soap and water. In the second stage, a solution was prepared containing 15 grams per liter of sodium carbonate and grams per liter of sodium phosphate. Degreased prepared solution was heated at 70°C led and samples were placed within solution for 4 minutes. Then, samples were immersed for 2 minutes in a solution of 2% volume and were entered into 15%nitric acid solution in order to activate surface and was kept for 1 min. After surface activation, the samples were put in commercial Zincates solution for 1.5 min and then were placed in activation solution in order to create higher quality Zincates (15% nitric acid) (this time for 30 seconds) and finally, they were immediately entered into Electroless solution.

A commercial Electroless bath made of German company AHC-Surface (RIAG) was used with average percentage of phosphorus for coating samples. The Electroless bath is prepared through combination of three separate components that final composition includes 5g/l nickel and 20g/l hypo phosphate sodium and 10 g/l Nano-particles of PTFE. It must be noted that Nano- particles of PTFE in suspension form contains 60 wt% Nano- particles with size of about 200nm.

Nano- particles of PTFE were stirred for 5 hours with a magnetic stirrer and 1000 rpm round after they were added to solution and before coating. B4C particles were then added to the solution and prior to coating they were stirred firstly using a magnetic stirrer for one hour with 1000 rpm round and then using an ultrasonic mixer for 10 minutes in order to prevent particles from agglomeration. Bath volume was considered 250 ml and after the time of coating, samples were removed from coating solution and were washed thoroughly with distilled water. Ammonia and diluted sulfuric acid were used to adjust the pH. Magnetic mixer with speed of 100 rpm was used during the coating. It must be explained that pH and temperature of coating were pH=5 and 85oC provided by manufacturer. Created thickness on samples was about 40 µm. Optical microscopy and magnetic Thickness Gauge of FISCHERSCOPE MULTI 650/ 750C model were used to determine the thickness of coating. The samples were put under heat treatment of 300°C for two hours after coating in order to evaluate the effect of heat treatment on hardness and wear resistance. Pin on disc apparatus was used in order to determine the wear resistance and friction coefficient, according to standard of ASTM G0099-04A. Before and after heat treatment, the samples were tested in abrasion test in the same conditions with force of 500 g and distance graphs were recorded based on weight reduction and friction coefficient. Coating morphology was studied by TESCAN scanning electron microscope VEGA II model and its chemical composition was studied by EDX energy resolution analysis and X-ray diffraction spectrometer, Philips X'Pert Pro model PW 3040/60. Substrates Quantometre analysis was done by BelecVario Lab apparatus. Linear polarization experiment was done by Autolab PGSTAT 302N in 3.5 wt% solution of sodium chloride in order to evaluate the corrosion resistance of coatings according to standard of ASTM G69. For this purpose, each sample was placed in solution for 10 minutes and then corrosion test was performed in the range of 0.8-1.2 volts of open circuit potential. Three electrode cells were used in order to do polarization corrosion test. Counter electrode was made of Platinum and fully saturated reference electrode was used. Each experiment was implemented three to five times with a potential sweep rate of 1 mv per second [22]. Hardness gauge of Vickers KOOPA MH3 model was used to measure hardness (the amount of force applied to measure hardness was 200 grams and its duration was 10 seconds). TESCAN scanning electron microscope VEGA II model was used to study coating morphology and its chemical composition was analyzed using EDX energy resolution analysis and X-ray diffraction spectrometer, Philips X'Pert Pro model PW 3040/60. Substrates Quantometre analysis was done by BelecVario Lab apparatus.

III. Results and Discussion

A. Studying coating morphology and composition:

PTFE and B4C powder images shown in figures (1) and (2).

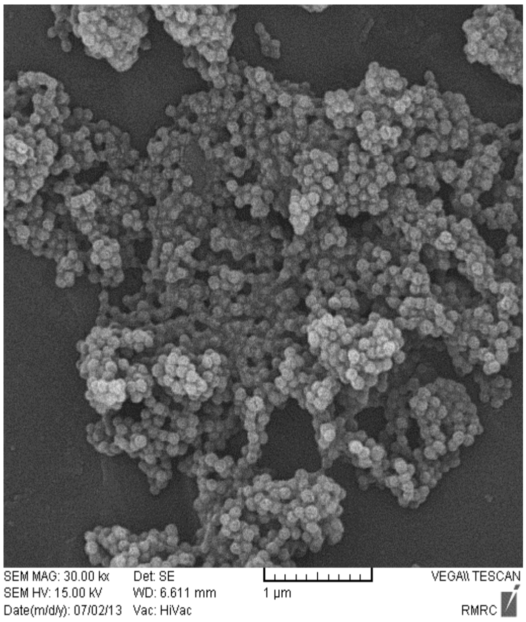

Fig.1. Illustration of PTFE powder

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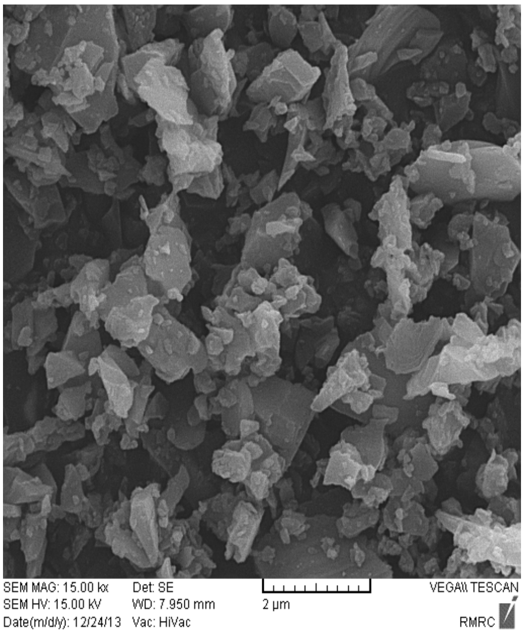
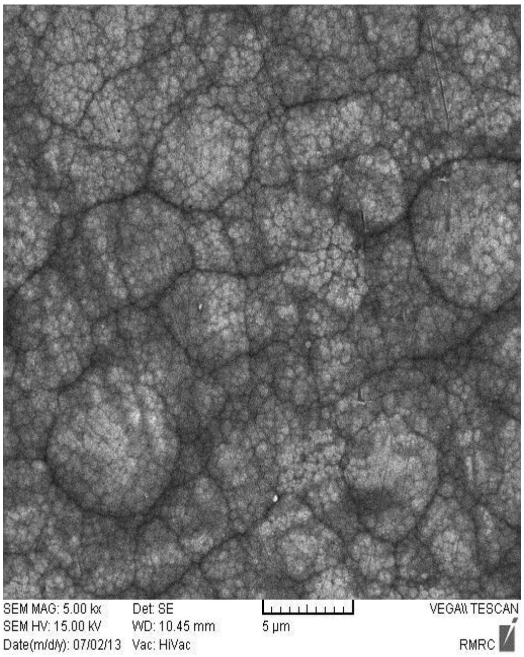


Fig.2. Illustration of Boron Carbide powder

Coatings of Ni-P and Ni-P-B4C-PTFE are observed in figures (3) and (4). Observing hybrid coating and comparing it with Ni-P coating present significant differences in morphology of coating. Also, it is observed that its structure is like disk but PTFE and B4C particles have led to increased growth of grains (nodules) and finer grain structure. PTFE particles does not allow to sedimentation of B4C powder with a particle size greater than 0.5 micron. A similar phenomenon of Ni-P-TiO2-PTFE hybrid coatings has been observed [23].



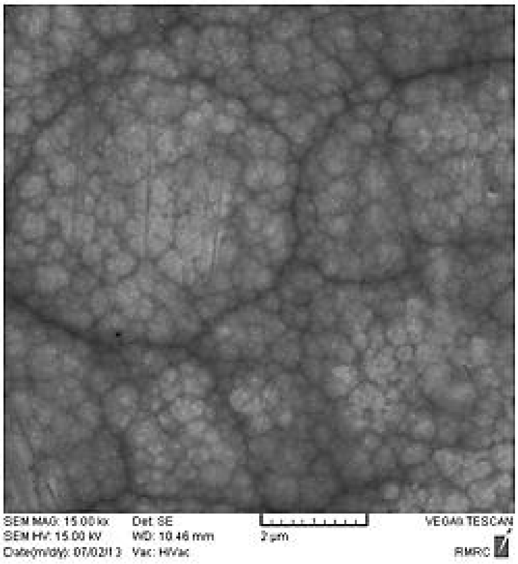


Fig.3. Illustration of Ni-P coating

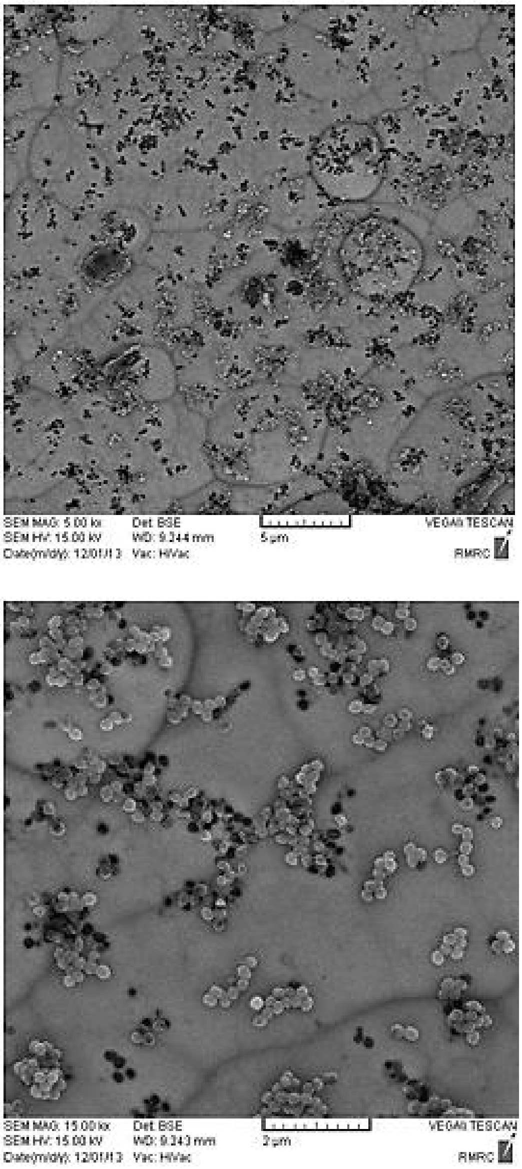


Fig.4. Illustration of Ni-P-B4C-PTFE coating

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Elemental analysis diagram of Ni-P and Ni-P-B4C-PTFE coatings have been presented in figures (5) and (6) and indicates the presence of particles like boron, carbon and fluorine in coating. Boron element represents the presence of B4C particles and flora element represents the presence of PTFE particles.

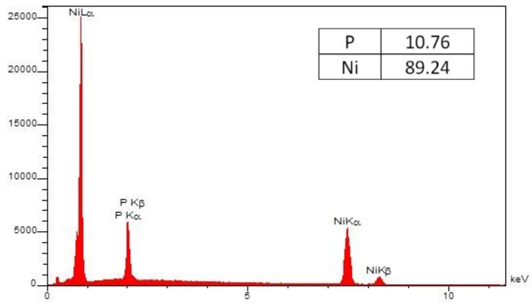

Fig.5. Elemental analysis of Ni-P coating

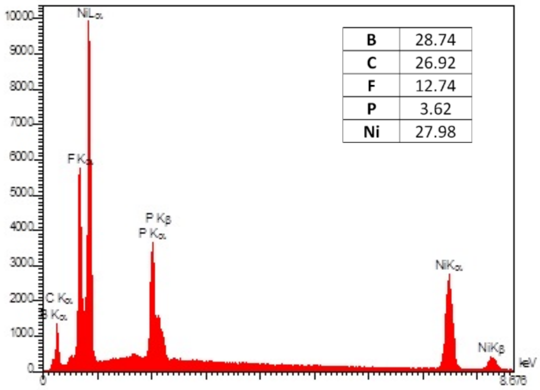

Fig.6. Elemental analysis of Ni-P-B4C-PTFE coating

B. Corrosion Resistance

For comparing the process and corrosion potential of coatings, polarization curves of Ni-P coatings, Ni-P-PTFE composite coating, Ni-P-B4C composite coating and Ni-P-B4C-PTFE hybrid coating are given in figure (7) and final results of Table 2. It was observed that corrosion process of composite coating has been decreased because of Ni-P composite coating by B4C particles (4 grams per liter), due to the cathode nature of particles and adding them to Ni-P structure. Adding 10 grams per liter of solution containing PTFE particles to Ni-P coating due to their nature leads to increased corrosion of particles to 4.5 times more than Ni-P coating. Also, because of Ni-P coating tendency to corrosion, it is increased by adding PTFE particles. Adding PTFE particles to Ni-P coating increases process and tendency to corrosion, unlike the particles B4C particles. Adding B4C particles to Ni-P-PTFE composite coating, as expected reduces corrosion of this coating; obtained corrosion in hybrid coating is like Ni-P-B4C coating corrosion, the difference is that hybrids coating tendency to corrosion has been slightly increased that is due to the presence of PTFE particles. Finally, given the required properties (improved hardness or corrosion resistance) of Ni-P coating with B4C or PTFE particles we can conclude that adding PTFE particles to Ni-P coating leas to anodic areas on Ni-P coating surface and increases process and tendency to corrosion; but adding B4C to Ni-P coating or Ni-P-PTFE composite coating improves the corrosion resistance of this type of coating. Finally, using Ni-P coatings containing only PTFE particles is not recommended in areas prone to corrosion.

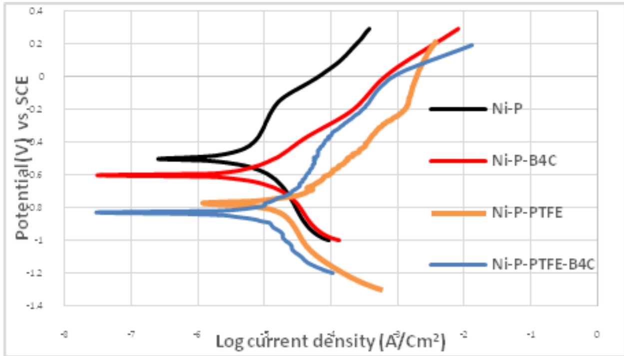

Fig.7. Polarization curves, Ni-P, Ni-P-PTFE, Ni-P-B4C coating and hybrid coating.

Table 2: Corrosion process potential, Ni-P coating, Ni-P-PTFE composite coating, Ni-P-B4C coating and hybrid coatings

Corrosion Potential Compared to SCE (mV)	Corrosion process (A/cm2µ)	Coating type
-500	0.4	Ni-P
-770	1.8	Ni-P-PTFE
-600	0.03	Ni-P-B4C
-830	0.03	Hybrid coating

C. Hardness

Micro hardness test was taken from different parts of surface. It is noteworthy that measuring hardness of soft context coating like aluminum is not recommended unless with thickness of 20 microns, it is because of soft context influence on fine hardness. Hardness of samples taken from cross section was measured and result was similar to result obtained from sample without cutting. Increasing thickness, hardness increases. However, after reaching a certain thickness (20 microns) hardness increase becomes almost zero which means the actual hardness of coating. In other words, in thicknesses less than mentioned thickness, the hardness is influenced by substrate plastic deformation. While thickness is increased more than this amount, the substrate role is removed in determination of overall hardness and actual hardness of coating will be documented [24]. Micro hardness test report on samples surface is given before and after operation in Table 2.

Entering PTFE particles to coating, hardness decreases and entering hard particles of B4C, hardness increases. Recent behavior is easily predictable. The increased amount of PTFE particles in coating is equivalent to a reduction in background nickel phosphorous alloy and finally, the hardness will drop. According to mixture rule in composites, adding composite component, the final properties of composite will change, therefore it is expected that increasing the presence of material with low

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hardness will result in composite hardness drop. In fact, increasing PTFE in coating, the coating becomes foam and amount of metal that is put under indenter of hardness gauge is reduces and hardness drops.

When force was exerted on coating surface by hardness gauge wedge, dislocations move and encounter PTFE particles in their path, since fuzzy PTFE particles are very soft they cannot prevent dislocations like other ceramic particles such as boron carbide cannot prevent dislocations and dislocations easily peel particles from surface or cut them and in both cases continue their path. Therefore, plastic deformation (in micron-scale) in coatings that contain PTFE particles is done easily than coatings with no particles and hardness of coating is less. It is seen that hybrid coating hardness of Ni-P-B4C-PTFE Electroless is between composite coating of Ni-P-PTFE and composite coatings of Ni-P-B4C. As can be seen, coating hardness is increased after heat treatment. The reason can be attributed to formation of Ni3P phase.

Table 2: The amount of coating hardness (Vickers)

After heat treatment	Before heat treatment	Coating type
1076	454	Ni-P
375	190	Ni-P-PTFE
1260	804	Ni-P-B4C

D. Wear Resistance

Figure 7 presents diagram of hybrid coatings friction coefficient under load of 500 grams compared to distance. Applying load of 500 g, mean friction coefficient of 0.48 was reached obtained. As can be seen, friction coefficient begins from 0.36 and then it is increased. It seems that PTFE particles are reduced into the coating and therefore friction coefficient is increased.

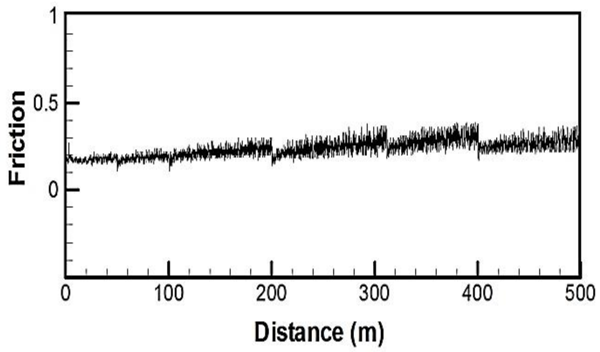

Fig.7. Diagram of hybrid coatings frictional coefficient under500g loads compared to distance

The samples were re-tested after heat treatment in order to investigate the effect of heat treatment. Figure 8 shows diagram of hybrid coatings friction coefficient compared to distance under load of 500 g after heat treatment. The figure (9) shows weight loss of hybrid coating compared to distance, before and after heat treatment under a load of 500 grams. The results showed that the mean friction coefficient (before and after heat treatment) is decreased from 0.48 to 0.32 and as a result improved wear resistance by 50%.

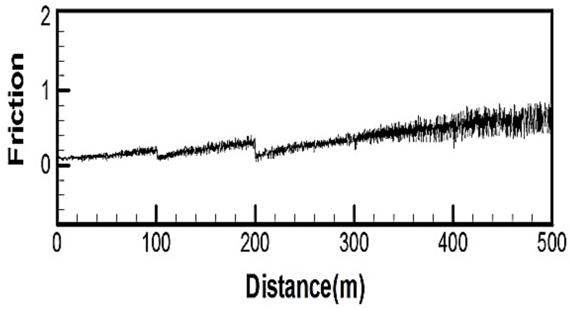

Fig.8. Diagram of hybrid coatings frictional coefficient compared to distance, after heat treatment

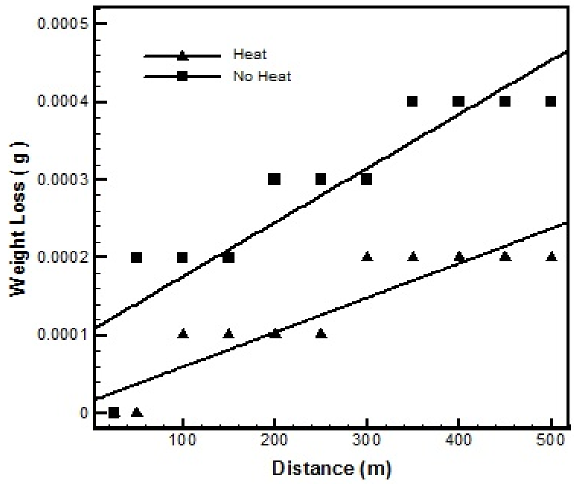

Fig.9. Diagram of hybrid coating weight reduction compared to distance, before and after heat treatment

Wear tests were performed for four types of coating after heat treatment. For comparison, in Table 3 mass reduction and mean coefficient of friction for four types are given. As can be seen, the mean friction coefficient of hybrid coating is among composite coatings. Hybrid coatings showed wear resistance. Adding Teflon particles leads to reduced friction coefficient and thus it is useful for slippery applications with less force. If too much force is exerted on the slippery surface of the Teflon particles would be useful. If too much force is exerted on the slippery surfaces, using Teflon soft particles will not be useful; however hardened particles such as boron carbide must be used. It was observed that with the addition of boron carbide micro particles, coating hardness was increased to 1260 Vickers; as a result, it resulted in improved wear resistance. As can be seen, Ni-P-B4C composite coating slippery is less than Electroless nickel coating and nickel-Teflon composite coating.

Table3: Values of mass reduction and mean friction coefficient of coatings under 500g load

Disk mass reduction (g)	Mean friction coefficient	Coating type
0.0012	0.44	Ni-P
0.0005	0.18	Ni-P-PTFE
0.0005	0.48	Ni-P-B4C
0.0002	0.32	Ni-P-B4C-PTFE

However, using Ni-P-B4C-PTFE hybrid coatings, we can increase Ni-P-B4C composite coatings slippery, increasing Teflon- Nickel coating.

IV. Conclusion

1) Entering PTFE particles into Nickel-phosphorus context, the mean friction coefficient and hardness was reduced.
2) Entering B4C particles into Nickel-phosphorus context, the mean friction coefficient and hardness was reduced.
3) With the addition of boron carbide particles, corrosion resistance was increased and but with the addition of PTFE particles, corrosion resistance was reduced.
4) Heat treatment improves the hardness and wear resistance by 50 percent.
5) The mean friction coefficient of hybrid coating is among composite coatings.
6) Ni-P-PTFE composite coating had the lowest friction coefficient and Ni-P-B4C composite coating had the highest corrosion resistance and Ni-P-B4C-PTFE hybrid coatings showed the best wear resistance.

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