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Research Article

Experimental investigation of mechanical properties on Al 7075 using electroless Ni-P/Ni-B duplex coating with nano SiC

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Abstract
The present work deals with the formation of Ni–P/Ni–B duplex coating by electroless nickel plating process on aluminium alloy (Grade 7075) and estimation of their wear resistance, microhardness and weight gain. The Ni–P/Ni–B duplex coatings were equipped using dual baths (acidic hypophosphite and alkaline borohydride) with nano SiC as reinforcement and sodium dodecyl sulfate (SDS) as surfactant. The experimental results designated the approximate relation between the SiC content and the hardness of composite plating's. With the increasing of SiC content, wear resistance increases consistently. In particular, the influence of SiC content on 0.3% of SDS (anionic surfactant) retain better wear resistance at 70^0 bath temperature. The duplex coatings are even and compatibility between the deposits are appears to be good.

Keywords
Duplex coating, Anionic surfactant, Wear resistance, Micro hardness, Weight gain.

1.Introduction
Nowadays, lot of research work is done on aluminium and its alloys because of their fashionable properties such as great strength to weight ratios, high thermal conductivity and righteous corrosion resistance. High strength aluminium alloys, such as 7075-T6, are extensively used in aircraft structures due to their remarkable strength-to-weight ratio, machinability and low cost. Electroplating is a process that uses electric current to reduce dissolved metal cations so that they form a coherent metal coating on an electrode [1]. Electroplating is primarily used to change the surface properties of an object e.g. wear resistance, corrosion protection, lubricity, aesthetic qualities, etc. In electroless process, metal deposition is driven by the catalytic oxidation of the reductant on the substrate surface. Coatings are used on most metal products today, either for protective or for both protective and decorative purposes. In general, coatings or finishes are used for the purpose of decoration, surface. It can be developed for desired properties by choosing the compositions of coating alloy/composite to suit specific requirements [2].

As nickel melts around a temperature of about 1480^0C and is unaffected by oxidation up to 500^0C, electroless alloy/composite coatings can be applied for high operating temperatures based applications. Ni–P coatings on aluminium-based alloys may be prepared using various procedures.

Electroless nickel–boron plating is less popular than the phosphorous-based process but the properties of nickel–boron coatings are very interesting and often superior to those of nickel–phosphorous: high hardness (around 750 hv_{100} in the as-deposited state), good wear resistance, good corrosion behavior. This provides electroless nickel–boron coatings with many fields of application such as petroleum and chemical industries, plastics injection, optics, and aerospace [3]. However, using this technique for the plating of aluminium is not easy: sodium borohydride is greatly unstable except in very alkaline media, so the plating bath usually has a pH higher than 9, which is not suitable for aluminium [4]. One possible alternate approach for improving the both nickel recovery and coating efficiency of EN coating process might be the additive of some suitable surfactant in the electrolyte bath.

In recent years, electroless plating has won great popularity in preparing composite coatings, which are generally prepared by adding solid particles to the

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regular electroless plating solution to achieve co-deposition of the solid particles [5].

The codeposition improves the tribological and mechanical properties of coatings. The inclusion of ceramic particles such as SiC and TiC into the metal matrix can significantly improve the hardness and anti-wear property of the matrix. Silicon carbide (SiC) can serve as both structured and functional materials for its good thermal conductivity, electrical conductivity, chemical stability, high mechanical strength, low friction and has high material strength with excellent corrosion, erosion resistance, mechanical and physical properties[6]. SiC particles are of great technological importance for their applications as reinforcement of metal matrix composites and structural ceramics [7]. In recent years, SiC has found new applications in the electronic industry for its excellent and adjustable dielectric properties. In this research, first the incorporation of SiC nano-particles on the structure of electroless Ni–P matrix was studied [8]. Then the effects of Ni–P/nano-SiC and Ni-B coatings on the wear behaviour of Al 7075, in the presence of SDS surfactant were investigated.

2.Experimental details

A warming bath is used in the laboratory to permit the chemical reaction to occur at an eminent temperature around 100^0C. The beaker is a glass vessel which comprises the chemical solutions for the persistence of coating. It is placed in the electroless bath where the chemicals are being fiery up. The glass rod is used to mixing the chemical where the magnetic agitator is not necessary. The tongs are used to lift the bath after the plating is completed. Tri-sodium citrate was used during plating process which acts as a stabilizer for the bath [9]. It steadies the reaction occurring in the bath. Laboratory mark distilled water is used for the scrubbing process and also to blend up with the chemicals. Nickel chloride acts as the source for the plating process as the nickel is being glazed upon the surface of the substrate. Ethylene di amine is used as a complexing agent which creates a bond amongst the nickel and aluminium and causes the plating. Sodium borohydride is the reducing agent which eliminates the hydrogen ions which are being moulded up inside the bath during the plating process.

The nickel chlorides are fascinating moistness from the air and made into it as a solution. Sodium borohydride, also known as sodium tetrahydrido borate, and sodium tetrahydroborate is an inanimate compound usually encountered as a powder, and also it is a resourceful reducing agent that catches wide application in chemical industry. Ethylenediamine is the organic compound with an ammonia-like odor is a powerfully basic amine. Ethylenediamine readily reacts with moisture in humid air to generate a biting, noxious and annoying film, to which even short exposures can cause serious damage to health [10]. Ammonium chloride, an inorganic compound is a white crystal-like salt, highly doable in water. Sodium hypophosphite is the sodium salt of hypo phosphorous acid and is often faced as the monohydrate which is solid at ambient temperature, seeming as odourless white sparklers. Ammonia or azane is a composite of nitrogen and hydrogen with the formula NH_3.

Surfactant is used for reducing the surface tension of the liquid, which is classified as non-ionic surfactant and ionic surfactant [11, 12]. Non-ionic surfactant does not contain any charge in it hydrophobic position. Ionic surfactant contain positive and negative charges, they are classified into two types namely anionic and cationic surfactants. The anionic surfactant contains negative charge in hydrophobic position and the cationic surfactant contains positive charge in hydrophobic position [13].

Aluminium 7075 was used as the substrate for the preparation of electroless Ni–P/Ni–B duplex coatings. It can easily attract with oxygen, hence the fine layer is allow to form on the surface. Generally, aluminium has negative kinship towards nickel coating and hence it needs preliminary processes for coating nickel on the aluminium surface. Their high chemical reactivity with air will result in the establishment of an oxide film on their surface that has an unfavourable effect on the coatings adherence and uniformity [14]. Therefore, air interaction of specimens must be minimized throughout this process. The acidic solution oxidizes the surface, making it rougher but more chemically vigorous, providing surface pits to turn as spots for automated meshing of the EN coating.

Initially, the substrate was prepared to polish by different emery sheets in order to gain the fine smooth surface. After the powered polishing of substrate it was cleaned using the acetone, then soaked it with de-ionized water and pickled in nitric acid for 30 sec [15]. Likewise, the substrate was thoroughly washed with de-ionized water and then dunked in zincate solution for 1 min. The composition of solutions used for zincate process was

given in *Table 1*. After following this first zincate process, the substrate was soaked with de-ionized water and again immersed the same in nitric acid for 30 sec and cleans. It with the use of de-ionized water. Finally, the substrate was dipped in zincate solution for 3 mins followed with scrubbing process. The purpose of double zincate conversion is to develop the electroless deposit adhesion on the plating surface [16].

The step by step process for carrying out the zincate pre-treatment process was given in *Figure 1*. Instantly after the zincate pre-treatment process, the substrate was sensibly placed in electroless nickel phosphorous solution bath, which is readily primed and sustained at a temperature of 85^0C. The composition of Ni-P bath along with circumstances are given in *Table 2*.

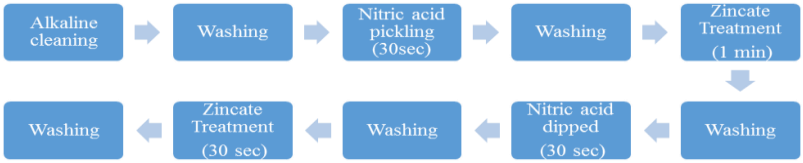

Figure 1 Zincate pre-treatment process

Table 1 Composition of Zincate

Sodium hydroxide	400 g/l
Zinc sulphate	120 g/l
Sodium potassium tartarate	6 g/l
Temperature	Room Temperature
Immersion time	3 mins

Table 2 Nickel phosphorous and Nickel boron composition and conditions

S.No	Chemicals used & conditions	Nickel phosphorous	Nickel boron
1	Nickel chloride	30 gl^{-1}	30 gl^{-1}
2	Sodium hypo phosphate	25 gl^{-1}	-
3	Ammonium chloride	50 gl^{-1}	-
4	Sodium borohydride	-	90 gl^{-1}
5	Tri sodium citrate	40 gl^{-1}	40 gl^{-1}
6	Liquid ammonia	Maintaining pH level	-
7	Ethylene di amine	-	0.8 gl^{-1}
8	pH	8	12
9	Temperature	80-90^{o}C	80-90^{o}C

The substrate (sample A) was gradually plunged and placed on the Ni-P bath with desired position, so that the confession of nickel around the surface becomes good. The temperature should be kept as constant for better reaction in between metal surfaces and solutions. The pH level was repeatedly checked with the aid of pH indicator paper for satisfactory coating thickness [17]. Whenever the coating starts a drop of SDS surfactant was plunged into the Ni-P bath exactly on the coating zone. Simultaneously nano SiC was stirred well in the separate beaker using magnetic stirrer for 1 hour. Fine stirred nano SiC was injected on to the Ni-P bath using injector with uniform time interval. After coating of the first layer (nickel phosphorous) for 30 mins, the sample was made to react in nickel boron bath for the deposition of boron which is acting as second layer. Here, the pH of 14 is maintained for better deposition of nickel boron. After the desired time given in *Table 3*, the sample A was taken from the bath and rinse it with de-ionized water. The photographical view of Ni-P/Ni-B baths were shown in *Figure 2*. The weight difference of sample before and after coating was determined using precision weighing machine.

Figure 2 Photographic view of Ni-P and Ni-B bath

Table 3 Coating time and weight difference for the samples

	Sample numbers														
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
Initial weight	4.44	4.27	4.11	4.25	4.22	4.24	4.20	4.20	4.23	4.25	4.18	4.27	4.20	4.24	4.24
Final weight	4.48	4.33	4.19	4.34	4.30	4.36	4.27	4.33	4.32	4.35	4.23	4.36	4.29	4.36	4.36
Weight difference	0.04	0.06	0.08	0.09	0.08	0.12	0.07	0.13	0.09	0.10	0.05	0.09	0.09	0.10	0.10
First layer	30 mins														
Second layer	1 hour														

The purpose of using SDS surfactant was clearly recognized in weight difference of the samples [18]. Based on the weight gain of the substrate sample 8 possesses better deposition of nickel with addition of 0.1 g/ltr of SDS surfactant than other samples. Sample 6 having a weight gain of 0.12 g with 0.3 g/ltr of surfactant on Ni-P bath. Beyond this other samples possesses minor proportion of weight gain with their corresponding composition of nano SiC, SDS surfactant and temperature given in *Table 4*.

Table 4 Electroless coating process parameters actual values and composition

	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
Nano SiC (g/ltr)	0.4	0.2	0.2	0.6	0.6	0.6	0.4	0.4	0.2	0.4	0	0.2	0.4	0.6	0.8
SDSSurfactant (g/ltr)	0.2	0.3	0.1	0.3	0.3	0.1	0.4	0.2	0.3	0	0.2	0.1	0.2	0.1	0.2
Temperature (^{0}C)	70	65	65	75	65	65	70	60	75	70	70	75	80	75	70

3.Results and discussion

3.1Micro hardness Test

The micro vickers hardness testing machine has a load range of 10 grams to 1 kg load with a least count of 0.01 mm and a diamond indentor cone angle of 136^0 [19]. Micro hardness test is taken for each sample, the value for the each sample is shown *Table 5*.

Table 5 Micro-hardness test values

Samples	**Value 1**	**Value 2**	**Value 3**	**Average value**
1	976.6	965.3	993.8	978
4	572.3	561.1	573	568
5	968	1101	1113	1060
9	973.7	988	1003	988
11	474.0	545.4	520.2	513
12	839.8	804.7	827.5	823
13	344.5	361.5	342.8	349
14	470.2	484.7	477.8	477

The above average values are made to plot in a graph for obtaining the range of the hardness for each sample that is made to test in Vickers Hardness test. The range of hardness differs for each sample due to its variation of nano SiC, surfactant and temperature. From the *Figure 3*, the sample 5 possesses better hardness compared with the other samples. Since the sample 5 having 0.06% of nano SiC and 0.3% of SDS surfactant with the temperature of 75^{0}C. whereas the sample 13 possess very less hardness compared with all other remaining sample which is having 0% of nano SiC and 0.2% of SDS surfactant. Hence from the above listed values, the ratio of nano SiC deposition rate decides the hardness of sample.

3.2Wear test

Immediate prior to wear test and weighing of sample, clean and dry the specimens thoroughly and take care to remove all dirt's and foreign matter from the specimens. Always use non chlorinated, non-film-forming cleaning agents and solvents to clean and dry it. Steel (ferromagnetic) specimens having residual magnetism should be demagnetized for better results [20]. Report the methods used for cleaning and insert the disk securely in the holding device so that the disk is fixed perpendicular (61°) to the axis of the resolution. Insert the pin specimen securely in its holder and made it to contact on rotating disc. The mass is added and the beam is hanged and the pin mounted is tightened and the setup is switched on, so that the disk rotates and due to frictional force wear occurs and the graph is plotted automatically.

Wear rates are intended results imitating wear mass loss, volume loss or linear measurement change under unit applied normal force and or unit sliding

distance [21]. Wear rate can be conveyed in many different ways. Here the wear rate is calculated using volume loss method and the respective values of initial and final weight of the samples and the rate of volume loss is calculated as given in *Table 6*. Similar to the hardness value range, the sample 5 possess minimum wear rate compared with the other samples. Meanwhile the wear resistance of sample 5 consists of 0.06% of nano SiC and 0.3% of SDS surfactant with the temperature of 75^0C as shown in *Figure 4*. Also the same sample shows better surface finish matched with remaining samples.

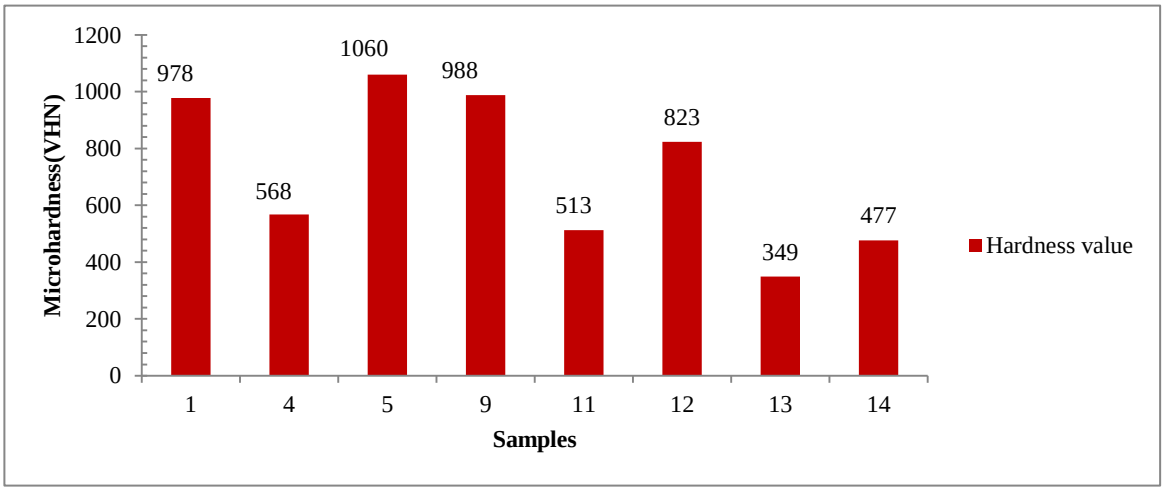

Figure 3 Microhardness of various samples using Vickers hardness

Table 6 Wear rate values by volume loss method

Sample number	Initial weight (X)	Final weight (Y)	Z=X-Y	Weight loss (A) X 10^{-3}	Area	Volume loss X 10^{-4}	Wear rate X 10^{-9}
1	4.18	4.23	0.05	5.09	48.12	1.06	5.402
4	4.25	4.34	0.09	9.17	48.12	1.91	9.734
5	4.44	4.48	0.04	4.07	48.12	8.47	4.317
9	4.23	4.32	0.09	9.17	48.12	1.91	9.734
11	4.27	4.36	0.09	9.17	48.12	1.91	9.734
12	4.22	4.30	0.08	8.15	48.12	1.69	8.613
13	4.24	4.32	0.08	8.15	48.12	1.69	8.613
14	4.20	4.29	0.09	9.17	48.12	1.91	9.734

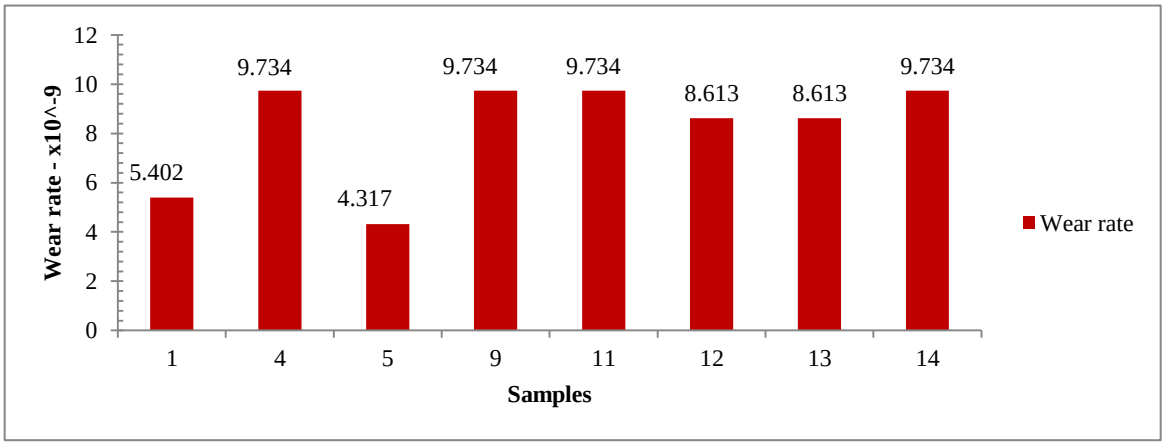

Figure 4 Graphical representation of wear rate using volume loss method

4.Conclusion

The plating rate of the electroless Ni-P and Ni-B deposits is estimated by evaluating the gain in weight after coating and using the density of the bond. The thickness of the Ni-P and Ni-B deposits rises with increase in coating time. However, the amount of increase in thickness is not linear throughout the complete period of coating and it marinates after sometime. This is due to the gathering of oxidation products of hypophosphite and borohydride in their corresponding baths. A strategy of thickness of the deposit versus coating time is obtained to prepare Ni-P/Ni-B coatings having unpredictable thickness so that they can be used for preparing Ni-P/Ni-B duplex coating. The weight gain of duplex coating has average weight gain of 0.08g.

The hardness of the coating sample are tested using the vickers hardness tester, its shows that the hardness is high for the sample 5, which has coated under low temperature (i.e) 65°C and the reinforcement added was under 0.06% of nano SiC and the time duration is high in the Ni-B coating, so that the coated layer will be strong and more.

The wear resistance taken for the various samples is allowed to be tested, The wear rate is calculated by the difference between initial weight and the final weight, and then the graph shows that the sample 4,9,11 and 14 has high wear rate and these samples were coated under 75°C and the time duration is high in the Ni-P coating, so that the rate of deposition of Ni-P and the reinforcement will be more.

Although Ni-P/Ni-B electroless duplex code position of nano SiC particles along with SDS surfactant on Ni-P and Ni-B plating bath was investigated for finding the optimum parametric condition to attain higher incorporation in non-agglomerated form, there still remains scope for further research like.

- Parameters such as the electroless bath composition, agitation and pH value of the bath may be investigated.
- By using different pre-treatment process, the optimum coating thickness of the electroless codeposition process could be identified.

Acknowledgment

None.

Conflicts of interest

The authors have no conflicts of interest to declare.

References

[1] Mukhopadhyay A, Barman TK, Sahoo P. Tribological behavior of sodium borohydride reduced electroless nickel alloy coatings at room and elevated temperatures. Surface and Coatings Technology. 2017; 321:464-76.

[2] Subramanian C, Palaniradja K. Influence of nano Al2O3 on Ni–P/Ni-B electroless duplex coating. ARPN Journal of Engineering and Applied Sciences. 2016; 11(16):10084-90.

[3] Subramanian C, Palaniradja K. Effect of surfactant on the electroless Ni-P/Ni-B duplex coatings on aluminium 7075. International Journal of Metallurgical Engineering. 2015; 4(2):25-32.

[4] Chen XM, Li GY, Lian JS. Deposition of electroless Ni-P/Ni-WP duplex coatings on AZ91D magnesium alloy. Transactions of Nonferrous Metals Society of China. 2008; 18:s323-8.

[5] Yang D, Qiu F, Zhao Q, Jiang Q. Excellent compressive strength and ductility of Ti5Si3–coated SiCP/Al2014 composites. Journal of Alloys and Compounds. 2017; 698:1086-93.

[6] Georgiza E, Novakovic J, Vassiliou P. Characterization and corrosion resistance of duplex electroless Ni-P composite coatings on magnesium alloy. Surface and Coatings Technology. 2013; 232:432-9.

[7] Delaunois F, Lienard P. Heat treatments for electroless nickel-boron plating on aluminium alloys. Surface and Coatings Technology. 2002; 160(2-3):239-48.

[8] Balaraju JN, Narayanan TS, Seshadri SK. Structure and phase transformation behaviour of electroless Ni–P composite coatings. Materials Research Bulletin. 2006; 41(4):847-60.

[9] Krishnaveni K, Narayanan TS, Seshadri SK. Electroless Ni–B coatings: preparation and evaluation of hardness and wear resistance. Surface and Coatings Technology. 2005; 190(1):115-21.

[10] Novak M, Vojtěch D, Vítů T. Influence of heat treatment on tribological properties of electroless Ni–P and Ni–P–Al2O3 coatings on Al–Si casting alloy. Applied Surface Science. 2010; 256(9):2956-60.

[11] Vijayanand M, Elansezhian R. Effect of different pretreatments and heat treatment on wear properties of electroless Ni-B coatings on 7075-T6 aluminum alloy. Procedia Engineering. 2014; 97:1707-17.

[12] Sahal M. Characterization of Ni–P coating on AZ91D magnesium alloy with surfactants and nano-additives. Journal of Magnesium and Alloys. 2014; 2(4):293-8.

[13] Kumar SM, Pramod R, Kumar MS, Govindaraju HK. Evaluation of fracture toughness and mechanical properties of aluminum alloy 7075, T6 with nickel coating. Procedia Engineering. 2014; 97:178-85.

[14] Gadhari P, Sahoo P. Effect of process parameters on microhardness of Ni-P-Al2O3 composite coatings. Procedia Materials Science. 2014; 6:623-32.

[15] Sahoo P, Das SK. Tribology of electroless nickel coatings–a review. Materials & Design. 2011; 32(4):1760-75.

[16] Narayanan TS, Krishnaveni K, Seshadri SK. Electroless Ni–P/Ni–B duplex coatings: preparation and evaluation of microhardness, wear and corrosion resistance. Materials Chemistry and Physics. 2003; 82(3):771-9.
[17] Vitry V, Sens A, Kanta AF, Delaunois F. Wear and corrosion resistance of heat treated and as-plated Duplex NiP/NiB coatings on 2024 aluminum alloys. Surface and Coatings Technology. 2012; 206(16):3421-7.
[18] Vitry V, Bonin L. Increase of boron content in electroless nickel-boron coating by modification of plating conditions. Surface and Coatings Technology. 2017; 311:164-71.
[19] Vitry V, Bonin L. Formation and characterization of multilayers borohydride and hypophosphite reduced electroless nickel deposits. Electrochimica Acta. 2017; 243:7-17.
[20] Zhang WX, Jiang ZH, Li GY, Jiang Q, Lian JS. Electroless Ni-P/Ni-B duplex coatings for improving the hardness and the corrosion resistance of AZ91D magnesium alloy. Applied Surface Science. 2008; 254(16):4949-55.
[21] Zhang S, Han K, Cheng L. The effect of SiC particles added in electroless Ni–P plating solution on the properties of composite coatings. Surface and Coatings Technology. 2008; 202(12):2807-12.

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