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Laser Surface Treatment of electroless Ni–P-SiC coating on Al356 alloy

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Abstract

Electroless Ni-P–SiC coatings are recognized for their hardness and wear resistance. In the present study, electroless Ni–P coatings containing SiC particles were co-deposited on Al356 substrate. Laser surface heat treatment was performed using 700W Nd:YAG pulsed laser. Effects of different laser operating parameters, such as laser scan rate, laser average power and defocusing distance on microstructures were investigated by optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and energy dispersive spectrometer (EDS). The results of microstructural characterization indicated that the laser treatment under different operating conditions produced composite coating contained nanocrystallined Ni-based matrix with SiC particles Ni3P, Ni12P5, Ni5P2, Ni8P3 precipitates. The microhardness measurements showed that the hardness of the coating was increased up to 60 percent, due to laser heat treatment, without effect on base metal.

Keywords: Laser surface treatment, Ni-P-SiC, Al356

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1. Introduction

Electroless Nickel-Phosphor (ENP) coatings have been widely used in the chemical, mechanical and electronic industries because of their wear, abrasion, and corrosion resistance. Co-deposition of solid particles into coatings can further improve certain properties, thus enhance their performance [1-4]. ENP–SiC coatings present excellent erosion and wear resistance. Incorporation of SiC particles in an electroless Ni-

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P matrix can improve the surface hardness, durability and tribological behavior of electroless Ni-P coatings and are usually considered as a replacement for ‘‘hard chromium” in the aerospace industry [5-7]. Many studies have been devoted to the electroless composite coatings on steel substrates [8] but little work has been reported on the surface protection of Al-based alloys [9]. The important difference between steels and aluminum alloys is that aluminum easily forms a stable passive oxide layer, reducing the adhesion of the coating. Hence, a chemical pre-treatment of aluminum substrate removing the oxide is necessary [10].

ENP–SiC coating properties can be improved further by heat-treatment of the coated specimen. Heat-treatment is an important factor that affects the hardness, structure and morphology of deposit and various investigators have reported that the microstructural properties and crystallization behavior of electroless Ni–P coatings depend on the phosphorus contents and the subsequent annealing processes [11,12]. Heat treatment of electroless Ni–P coatings is normally carried out in furnace. In a furnace treatment, coated components have to be heated up to high temperatures required for improvement of wear resistance and adhesive bonding. Obviously, it is undesirable for thermal sensitive substrate materials such as Al-based alloys. In addition, physical sizes of the components are also restricted by the use of furnace. Compared with a traditional furnace treatment, laser surface heat treatment, is a simple and versatile technique to overcome those limitations. Furthermore, novel phases in the laser processed zone can be formed because of the rapid heating and cooling rates induced by laser irradiation [13,14]. Up to date, most research on electroless Ni–P base coatings by laser treatment mainly concerns fully melting of the coatings to enhance the substrate-coating adherence and to form amorphous or nanocrystalline surfaces on pre-plated alloys due to rapid heating and cooling rates in laser processes [14].

A few publications on laser heat treatment of amorphous electroless Ni–P coatings have been found. Matsukawa, et al. [15] and Liu, et al. [16] applied a pulsed Nd:YAG laser beam to treat amorphous electroless Ni–P coatings and investigated the effect of such laser treatment on wear and corrosion properties. They found that the laser treatment improved both wear and corrosion resistance, and such result was compared with the heat-treated coatings by furnace and superior properties were obtained by laser treatment [17]. Zhang et al. [18] and Kong et al. [19] used laser to treat electroless Ni-P-Al2O3 composite coatings. The effects of laser treatment parameters on microstructure, surface hardness and frictional wear behaviour of nanocrystallized coatings were studied. Compared with furnace-annealing, improved microhardness and wear resistance resulted. Liu et al. [14,17,20] have reported the microstructural characterisation and corrosion performance of amorphous electroless Ni-W-P coatings after annealing with laser. They revealed that the laser treatment improved the corrosion resistance of the coatings compared with furnace-annealed coatings. Unfortunately, only one publication on laser heat treatment of EN-SiC coatings has been found [21] in which, Shao, et al. applied a continuous CO2 laser beam to treat amorphous electroless Ni–P-SiC coatings on carbon steel substrate and investigated the effect of such laser treatment parameters on microstructure, morphology, micro-hardness and wear properties. They found that when the laser power was 450 W with scanning speed of 0.5 m/min, the hardness of the coating was superior to the coating obtained by the conventional furnace heating, and wear resistance of the composite coating after laser treating could also improve. However, there has been no report, up to date, on Nd:YAG pulsed laser heat treatment of amorphous electroless Ni–P–SiC coating on Al base alloy substrate. Using Nd:YAG pulsed laser can be preferable because of the high absorbability of 1064nm wavelength laser radiation by metals and the short pulse duration which affords higher cooling rates and a reduced heat affected zone [22].

This work describes laser heat treatment of ENP–SiC coatings on Al356 alloy. Pulsed Nd:YAG source is used for this purpose. Microstructure, phase changes, micro-hardness of laser treated Ni–P–SiC coatings are evaluated in the paper.

2. Experimental procedures

2.1. Materials and sample preparation

The Al356–T6 alloy of the following composition was used as base material (mass %): 7% Si, 0.2% Cu, 0.1% Mn, 0.35% Mg, 0.1% Zn and 0.2% Ti. Specimens were coated with an electroless Ni–P-SiC layer whose composition was (mass %) 62% Ni, 10% P and 28% SiC. The plating bath solution containing 25 g/l nickel–sulfate and 23.2 g/l sodium hypophosphite, complexing agent and 20 g/l SiC. The bath temperature and pH was 85–95 ◦C and 4.6–4.8, respectively. Prior to coating, cleaning was performed on the specimen's surfaces in the following sequential steps. Step 1: Al356 alloy was degreased with acetone to remove oil and/or grease contamination. Step 2: Al356 alloy was treated by mild etching at 80 ◦C for 3min with an alkali cleaner containing 22.5 g/l of sodium carbonate and 22.5 g/l of sodium triphosphate to remove the alumina layer. The coating thicknesses were obtained about 60 µm.

2.2. Laser treatment

In the present work, the EN-SiC coated specimens were heat treated using Nd:YAG laser system (PIM-3475 model IQL-20) that produces a laser beam with a wavelength of 1064 nm. The incident energies of laser treatment were adjusted either with constant laser power, changing the scanning speed of laser beam, or with constant scanning speed, changing laser power. During laser treatment, argon purge at a rate of 10 l/min was used to prevent oxidation. The laser treatment parameters used in this study are listed in Table 1.

Table 1. Laser parameters used for heat treatment of EN-SiC coatings.

power (W)	Laser pulse duration (ms)	Scanning speed (mm/sec)	Frequency (Hz)	Laser spot diameter (mm)
45-120	3.4	3-7	30	0.8

2.3. Microstructural examinations

The cross-section of the test specimen was mounted and mechanically ground to 2000 mesh on SiC papers and finally polished on the cloth using a suspension of alumina powder. The microstructure of the specimens was investigated by optical microscopy and field emission scanning electron microscopy (FESEM-Vega-Tescan) equipped with an energy dispersive spectroscopy (EDS-SAMX) detector. The phase analysis was conducted by X-ray diffraction analysis system (SIFERT2000 X Pert-MPD) with Cu Kα radiation and 40 kV and 30 mA operating conditions. A Zweek microhardness tester equipped with Vickers pyramid indenter was used for microhardness measurements. A series of 100 g indents held for 15 s were made on the work piece cross-sections.

3. Results and discussion

3.1. Microstructure and phase analysis of EN-SiC coated specimen

Transverse cross-sectional view of the EN-SiC coated specimen is shown in Fig. 1. As can be observed, the SiC particles are evenly distributed throughout the matrix of electroless Ni-P alloy. Fig. 2 shows the EDS spectrum of the composite coating. The coating contains 9.74 wt.% phosphorus. The coating would produce non-crystal structure as-coated when phosphorus contents exceeded 8 wt.%[21]. Therefore, the composite coatings developed in this experiment were of non-crystal structure under as-coated condition which is confirmed with XRD pattern showed in Fig. 3.

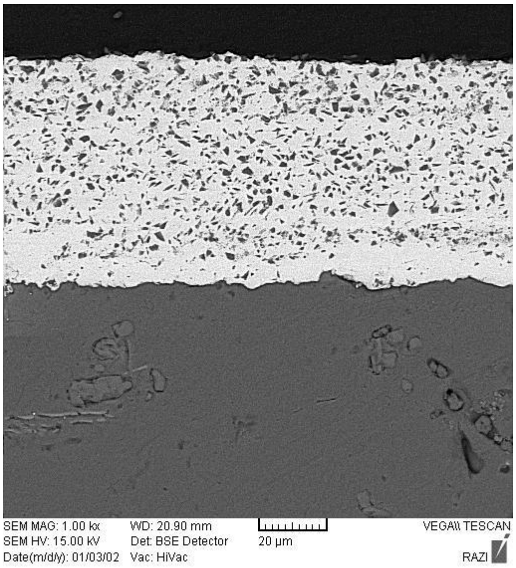
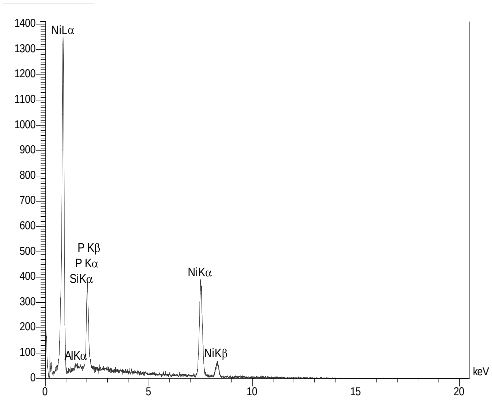


Fig.1. Transverse cross-sectional view of the EN-SiC coated specimen.



EDS analysis results of the EN-SiC coating (in wt%).

C	Al	Si	P	Ni
14.77	0.39	0.2	9.74	74.91

Fig. 2. EDS analysis of the EN-SiC coating.

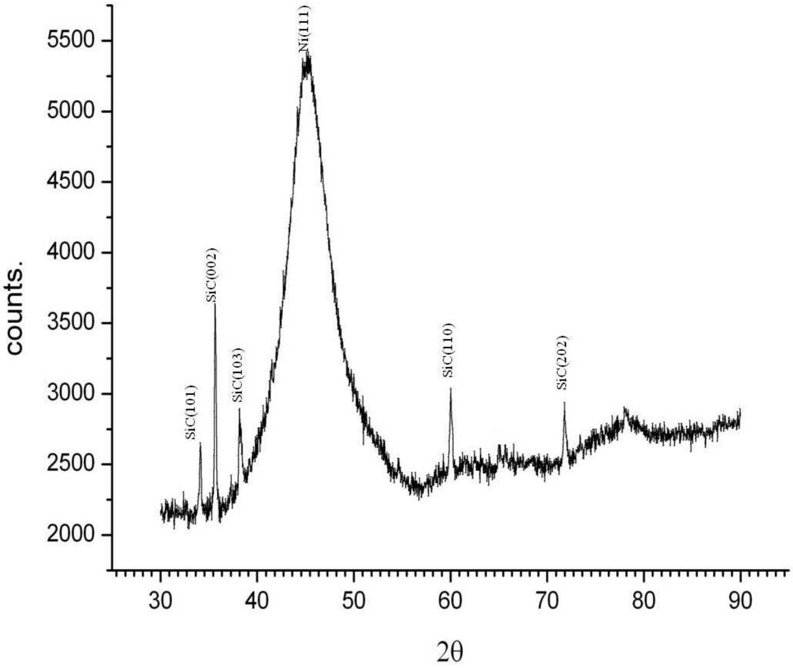


Fig. 3. XRD patterns of the EN-SiC coating.

3.2. Microstructure and phase analysis of laser treated EN-SiC coated specimens

Fig. 4. presents surface morphologies of the EN-SiC coatings after laser surface treatment in various laser parameters listed in Table 1. It can be seen that in the scanning speed of 5 mm/sec, the coating surface is melted when the laser power is increased into more than 80W. The protuberances and cracks on the surface of the coatings gradually disappeared when the laser power decreased to less than 75W. When the laser power was decreased to less than 60W no significant change could be observed in compare with EN-SiC coating before laser treatment. The structural analyses for the composite coatings after laser treatment were conducted on X-ray diffraction apparatus, as shown in Fig. 5. It can be seen that the EN-SiC coatings did make crystalloblastic transformation when the laser power reached 60 W and the diffraction pattern peaks correspond to those of Ni_3P can be recognized. It was also found that Ni-P crystal phases could not be formed in the laser power of less than 60W and the microstructure has not been changed from an original amorphous structure to a crystallized state.

3.3. Microhardness

Fig.6. shows the curves of microhardness against depth of strengthened layer for both as-plated and the laser treated EN-SiC coating. The hardness of the coating surface without laser treatment is 580 $HV_{0.1}$, but

after laser treatment can reached 740 $HV_{0.1}$. The hard Ni-P crystal phases formed in the matrix of EN-SiC deposit layer after laser treatment, could contribute to the hardening of the coating through fine-crystal strengthening and dispersion strengthening. The results show that the hardness of the composite coatings consistently improves with the increase of laser power. The reason is that when crystallization proceeded upon increasing laser power, the precipitation of Ni-P crystal phases resulted in the increase of the hardness. Besides in low laser powers, amorphous structure is kept and only a little degree of crystallization takes place and considerably, reduces surface microhardness.

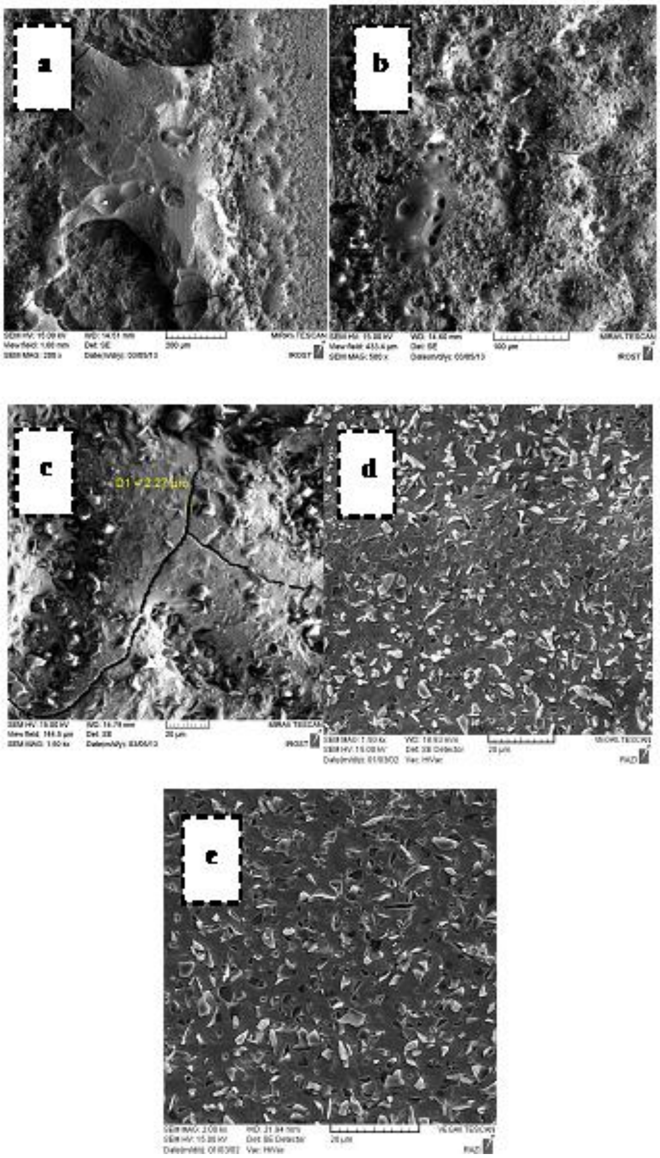


Fig. 4. SEM micrographs of Laser surface treated EN-SiC under laser scan speed of 5 mm/sec and different laser powers (a: 120W, b: 105W, c: 90 W, d: 75W, e: 60W).

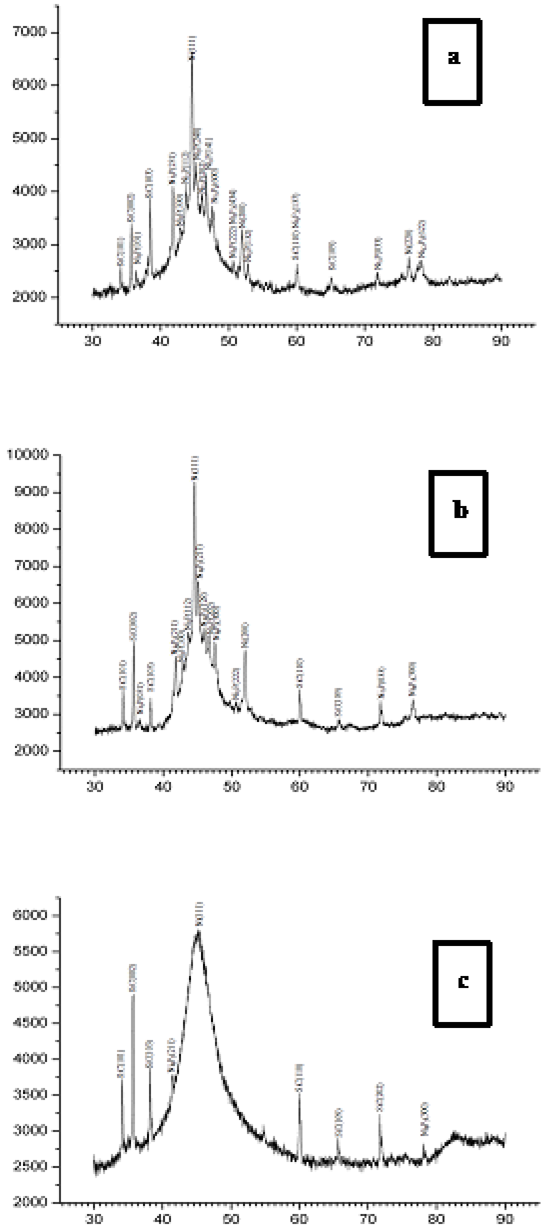


Fig. 5. XRD patterns of Laser surface treated EN-SiC under laser scan speed of 5 mm/sec and different laser powers (a: 75W, b: 60W, c: 45W).

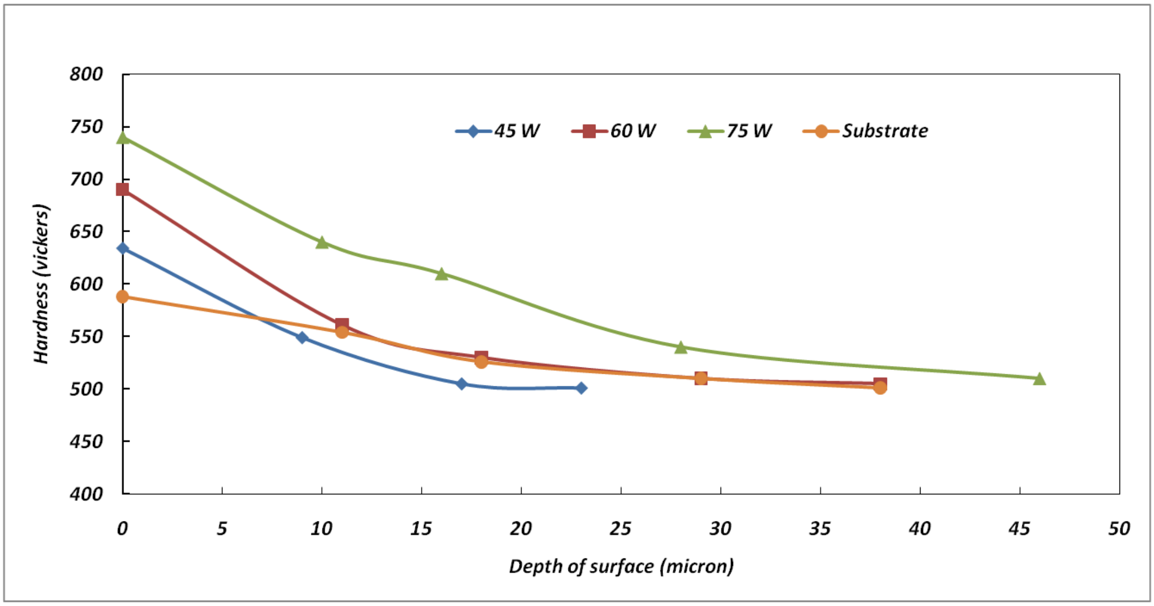


Fig. 6. Microhardness profiles of untreated EN-SiC (●) and Laser surface treated EN-SiC under laser scan speed of 5 mm/sec and different laser powers (▲: 75W, ■: 60W, ♦: 45W).

4. Conclusions

In this study, the effect of Nd:YAG laser surface heat treatment on microstructure, phase changes and microhardness of electroless Ni-P-SiC coating with Al356 substrate were investigated. Laser heat treatment was done with no major defects, melted area, protuberances and cracks. The laser treated zone structure contained mainly of Ni-P crystal phases. Moreover, the SiC particles were observed in the structure distributed homogeneously. Surface hardness of the laser treated EN-SiC coating is a function of the laser processing conditions. High laser powers (more than 90W) could due to melting of the surface and low laser powers (less than 60W), significantly, decrease the degree of crystallization. The EN-SiC coating which laser treated in 5mm/sec scanning rate and 75W laser power had the best hardness profile with no surface defects simultaneously.

References

[1] Krishnan, K. H., Jon, S., srinivasan, K., Praveen, J., 2006. An overall aspect of electroless Ni-P deposition-A review Article. Metall. Mater. Trans. A. 37, pp. 1917-1926.

[2] Palaniapa, M., and Seshadri , S.K., 2007. Hardness and Structural correlation for electroless Ni alloy deposits. J. Mater. Sci. 42, pp. 6600-6606.

[3] Keong, K.G., and Sha ,W., 2002. Crystallization and phase transformation behaviour of electroless Nickel- phosphorus deposits and their engineering properties. Surface Eng. 18, pp. 329-343.

[4] Sudagar , J., Lian , J. , and Sha, W., 2013. Electroless nickel, alloy, composite and nano coatings – A critical review. J. Alloy. Compd. 571, pp. 183–204.

[5] Apachitei , I., Tichelaar, F.D., Duszczyk, J. and Katgerman, L., 2001. Solid state reactions in low phosphorus autocatalytic NiP-SiC Coating. Surf. Coat. Tech. 148, pp. 284-295.

[6] Zhang, Sh., Han, K. and Cheng , L., 2008. The effect of SiC particles added in electroless Ni–P plating solution on the properties of composite coatings. Surf. Coat. Tech. 202, pp. 2807–2812.

[7] Wang , H. L., Liu, L. Y., Dou , Y., Zhang , W. Z., Jiang, W. F., 2013. Preparation and corrosion resistance of electroless Ni-P/SiC functionally gradient coatings on AZ91D magnesium alloy. Appl. Surf. Sci. 286, pp. 319– 327.

[8] Balaraju , J.N., Narayanan, T.S.N. and Seshadri, S.K., 2003. Electroless Ni–P composite coatings. J. Appl. Electrochem. 33, pp. 807–816 .

[9] Vojtech , D., 2009. Properties of Hard Ni–P–Al2O3 and Ni–P–SiC Coatings on Al-Based Casting Alloys. Mater. Manuf. Process. 24, pp. 754-757.

[10] Vojtech, D., Novak, M., Zelinkova, M., Novak, P., Michalcova, A., Fabian, T., 2009. Structural evolution of electroless Ni–P coating on Al–12 wt.% Si alloy during heat treatment at high temperatures. Appl. Surf. Sci. 255, pp. 3745–3751.

[11] Apachitei , I., Tichelaar , F. D., Duszczyk , J., Katgerman , L., 2002. The effect of heat treatment on the structure and abrasive wear resistance of autocatalytic NiP and NiP-SiC Coatings. Surf. Coat. Tech. 149, pp. 263-278.

[12] Chen, C.K, Feng, H.M., Lin , H.C., Hon , M.H., 2002. The effect of heat treatment on the microstructure of electroless Ni–P coatings containing SiC particles. Thin Solid Films. 416, pp. 31-37.

[13] Shao , H., Zhou ,M., Chen ,G., 2003. study on the laser treatment of electroless composite coatings. Appl. Laser. 23, pp. 194-198.

[14] Liu , H., Viejo , F., Guo , R.X., Glenday , S., Liu , Z., 2010. Microstructure and corrosion performance of laser-annealed electroless Ni–W–P coatings. Surf. Coat. Tech. 204, pp. 1549–1555.

[15] Matsukawaa , K., Kataokaa , M. and Morinushia, K., 1994. The Effect of Pulsed Laser Annealing on Wear and Corrosion Properties of Electroless Ni-P Plating. Tribol T. 37, pp. 573-579.

[16] Liu ,W.L., Chien ,W.T., Jiang ,M.H., Chen ,W.J., 2010. Study of Nd:YAG laser annealing of electroless Ni–P film on spiegel-iron plate by Taguchi method and grey system theory. J. Alloy. Compd. 495, pp. 97–103.

[17] Liu , H., Viejo , F., Guo , R.X., Glenday , S., Liu , Z., 2010. Comparative study of microstructure and corrosion resistance of electroless Ni-W-P coatings treated by laser and furnace-annealing. T. Nonferr. Metal. Soc. 20, pp. 1024-1031.

[18] Zhang , Q., Yao , J., Pan ,Y., 2010. Pulse Laser Assisted Composite Electroless Deposit to Prepare Ceramic Coating. Phys. Procedia. 5, pp. 327–332.

[19] Kong , F., Yao , J., and Dong , X., 2011. Microstructure and wear resistance of laser-treated Ni–P–Al2O3 electroless deposition composite coating. Int. J. Adv. Manuf. Syst. 10, pp. 29-35.

[20] Liu , H., Guo , R.X., Liu ,Y., Thompson , G.E., and Liu, Z., 2012. The effect of processing gas on corrosion performance of electroless Ni-W-P coatings treated by laser. Surf. Coat. Tech. 206, pp. 3350–3359.

[21] Shao , H., Jiang , X., Wang , L. and Hua , Y., 2006. Study on the laser treatment of electroless Ni-P-SiC composite coatings. Chin. Opt. Lett. 4, pp. 589-591.

[22] White, C.W, 1979. Laser and Electron Beam Processing of Materials. Academic Press Inc., pp. 721-727.