Influence of Treatment Sequence on Tribological Performance of Duplex Surface-Treated AISI 1045 Steel

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Received: 10 September 2018 / Revised: 12 November 2018 / Published online: 21 February 2019
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Abstract

The surface of AISI 1045 steel specimens was modified via two duplex surface treatments of (i) titanium diffusion coating followed by plasma nitriding (Ti–PN) and (ii) plasma nitriding followed by titanium diffusion coating (PN–Ti). A comparative study on the microstructure and wear properties of the fabricated TiN coatings was conducted. A scanning electron microscope equipped with energy-dispersive X-ray spectroscopy, X-ray diffractometer (XRD), and pin-on-disk wear test was utilized to evaluate the microstructures, phases, and wear properties of the coatings. XRD patterns of duplex-treated specimens illustrated that the modified layer consisted of nitride and carbo-nitride phases such as TiN and $\text{TiC}_{0.7}\text{N}_{0.3}$. The results also showed that Ti–PN treatment led to a low friction coefficient and the lowest wear factors, as compared with the PN–Ti treatment. Oxidation and mild fatigue fracture wear were found to be the main wear mechanism of the Ti–PN coating. The wear mechanism in the PN–Ti specimen involved a higher oxidative wear regime followed by plastic deformation wear regimes.

Keywords Duplex surface treatment · TiN coating · Plasma nitriding · Titanium diffusion coating · Tribological performance

1 Introduction

Various surface treatments have been widely utilized to enhance different surface properties, including wear, corrosion, and fatigue properties, all prolonging the service life of the components. Among various surface treatment techniques, duplex surface treatment is a method involving the consecutive diffusion of two or more elements into the surface of the component to fabricate a superficial layer with a given composition and the desired properties on the surface. Examples of the technique include deposition of Al [1], Cr [2, 3], Ti–C–N [4], Ti–Al–N [5] which is followed by nitriding or boriding to achieve various nitrides or borides on the surface. An important characteristic of these diffusive

layers is the presence of an intermediate layer between the top layer(s) and the substrate, which usually helps the system to resist much better against thermal stresses due to the existing thermal expansion mismatch between the top layer and the substrate.

Among various hard face layers, nitride layers most importantly improve the load-bearing capacity of the surface; also, they can enhance the wear and corrosion resistance [6, 7]. A wide range of coating systems such as AlN [8], CrN [9], TiAlN [10, 11], CrAlN [12], and TiN [13, 14] have been applied on steel using the duplex surface treating method. In addition, TiN coatings have found extensive industrial applications, such as coatings on mandrels, drawing dies, cutting tools, molds, gears, and bearings [15, 16], as they offer high hardness, wear, and corrosion resistance [17, 18]; also, good thermal and electrical conductivities should be noted [19]. The TiN coating has been deposited on steels by various methods such as chemical vapor deposition (CVD) [7], combination of plasma nitriding and plasma-enhanced CVD [13], physical vapor deposition (PVD) [16], plasma-assisted chemical vapor deposition (PACVD) [20], and the duplex treatment method of the tufftriding followed by the pack titanium coating [14]. Wear-resistant TiN

Available online at <http://link.springer.com/journal/40195>

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coatings have been investigated in different applied loads (up to 20 N [21–23] and 75 N [24]). According to the previous studies, the tribological performance of TiN coatings is closely related to their microstructure, roughness, hardness, ductility, the presence of oxide films, and the coatings process [25]. Recently, the evolution of friction coefficient in hard coatings has been considered [26, 27] to better understand the friction and wear processes and to explain the relationship between the control of parameters and the coefficient of friction. Accordingly, the TiN coating, as a hard industrial coating, should also be noticed in this respect.

In the present study, fabrication of the TiN coating on the carbon steel was investigated. Two different duplex surface treatment methods including the titanium diffusion coating followed by plasma nitriding (Ti–PN) and the reverse procedure (PN–Ti) were applied on the AISI 1045 steel, and then, their microstructure and wear properties, as well as friction coefficient evolution, were investigated.

2 Experimental

As-received AISI 1045 steel was used as substrate, with composition of 0.45 C, 0.25 Si, 0.75 Mn, $P < 0.03$, $S < 0.03$, $Ni < 0.20$, $Cr < 0.20$ (wt%), and Fe. The cylindrical coupons with 20 mm in diameter and 9 mm in height were prepared as the substrates for the later coating process. All coupons were initially ground with 60–5000 grades emery papers to produce a mirror finish before surface treatment. The coupons were then cleaned and degreased in acetone.

Some coupons were duplex-treated via plasma nitriding and pack titanium diffusion coating (PN–Ti). The others were duplex-treated via the reversed procedure of titanium diffusion coating and plasma nitriding (Ti–PN). The titanium diffusion coating was carried out at 900 °C for 5 h using a conventional pack cementation in a homogeneous mixture of 40 wt% pure Ti powder as titanium source, 40 wt% Al_2O_3 as the inert filler, and 20 wt% NH_4Cl as the activator. Plasma nitrides were performed in a pulsed direct current (DC) plasma nitriding chamber at 550 °C for 5 h in a gas mixture of 75% N_2 and 25% H_2 at the pressure of 4 mbar. Before starting the plasma nitriding process, the chamber was pumped down to 10^{-2} mbar, and then, hydrogen was introduced to the chamber. The specimens were cleaned via hydrogen sputtering for 30 min at the pressure of 3 mbar. The specimens were slowly cooled down to room temperature in a vacuum chamber.

The surface-treated specimens were sectioned, mounted, and prepared for metallographic studies. Scanning electron microscope [SEM coupled by energy-dispersive X-ray spectroscopy (EDS)] was employed for microstructural studies. From the SEM images, the volume fractions of the constituent phases were estimated in the alloys by ImageJ software

[28, 29]. The surface micro-hardness value was averaged over five measurements at different locations on the surface in Vickers scale.

The surface roughness was measured by a roughness meter (TIME, TR200), and the average of the arithmetic mean roughness (R_a) at three different locations was reported (Din 4768 standard). Room-temperature dry wear tests were performed using a pin-on-disk apparatus at sliding speed of 0.08 m/min, the constant normal load of 20 N, relative humidity of about 35%, and sliding distance of 500 m. Tungsten carbide pins with 5 mm diameter were employed as counterpart against the surface-treated specimens. The weight loss of the specimens was determined by 10^{-4} g precision. Worn volume (V) and wear factor (W) of the specimens were calculated according to ASTM G99-05(2010) standard, as follows:

$$V (\text{mm}^3) = \frac{\Delta m}{\rho} \times 1000, \quad (1)$$

$$\rho = \rho_1 \times \frac{m_a}{m_a - m_1}, \quad (2)$$

$$W \left(\frac{\text{mm}^3}{\text{N} \times \text{m}} \right) = \frac{V}{F \times d}, \quad (3)$$

where Δm is the weight loss (g), ρ is the density (g/cm^3), d is the sliding distance (m), ρ_1 is the density of water [g/cm^3] at room temperature, m_a is the weight of the experimented specimens in the air (g), m_1 is the weight of the experimented specimens in water (g) and F is the normal load (N). The wear factor values presented in this work were the average value of three repeated measurements. The wear tracks were also studied by SEM.

3 Results and Discussion

3.1 Microstructural Characterization of Superficial Layers

Figure 1 shows the surface topologies of Ti–PN and PN–Ti coatings. The superficial nitride layer on the Ti–PN specimen (with $R_a = 1.29 \pm 0.27 \mu\text{m}$) contained some cracks and pores with an almost uniform distribution, as shown in Fig. 1a, similar to the other typical surface morphologies produced by ion sputtering [30, 31]. The diffusion of nitrogen into the pre-titanized layer occurred as a result of the chemical potential gradient of nitrogen at the gas phase–nitrided substrate interface. Nitrogen diffusion into the substrate of the pre-titanized layer was a reactive diffusion promoting the formation of titanium nitrides (with $R_a = 1.58 \pm 0.25 \mu\text{m}$, Fig. 1c). The final stage of pack cementation process of the

Fig. 1 SEM surface topographs **a, c**, and EDS analysis **b, d** of **a, b** Ti–PN and **b, c** PN–Ti specimens with EDS elemental analyses of selected regions

Table 1 Top surfaces EDS analyses of duplex-treated specimens for points A–C in Fig. 1 (at%)

Element	A	B	C
N	63.05	19.63	18.40
Ti	13.95	36.79	44.00
Fe	32.28	26.62	27.79
C	12.24	16.95	9.81

PN–Ti specimen provided many spot defects on the surface layer, which was flaked off locally from the surface; this was in accordance with the other reports [8]; also, the surface accompanied sputtering damages from the plasma nitriding stage. The EDS results for the selected regions in all the specimens, as presented in Table 1, revealed that the surfaces of all specimens contained N, C, Fe, and Ti, which indicated the inter-diffusion of N and Ti (from the coating environment) and Fe and C (from the substrate). Moreover, Ti–PN

coating had a very higher nitrogen in comparison with the other coating, as well as higher iron content at the surface in comparison with the PN–Ti one; on the other hand, titanium and carbon were more in the PN–Ti coating than the Ti–PN one. The volume fractions of the TiN and TiC_{0.7}N_{0.3} phases in the Ti–PN specimen were measured to be 78% and 22%, respectively. The EDS analysis (Table 1) also showed that there was some compositional non-uniformity on the PN–Ti coating surface, as C content was increased and titanium content was reduced from B to C, as shown in Fig. 1, with similar iron and nitrogen contents. Based on the EDS results, it could be expected to have more iron nitrides on the Ti–PN coating surface rather than the other coating, while titanium carbo-nitride content became higher on the surfaces of the PN–Ti coating.

XRD patterns of the specimens are shown in Fig. 2. The titanized steel resulted in the formation of TiC on the surface

Fig. 2 XRD patterns of experimented specimens

(Fig. 2a), while the later plasma nitriding of the titanized layer (as in the Ti–PN specimen) decomposed the TiC layer and promoted the formation of TiN and $\text{TiC}_{0.7}\text{N}_{0.3}$ phases (Fig. 2b) in the surface layer. The reverse sequence, i.e., plasma nitriding followed by titanizing (PN–Ti), led to a different path, whereas plasma nitriding promoted the formation of Fe_{2-3}N and Fe_4N phases during the initial stage (Fig. 2c); on the other hand, the later plasma nitriding (PN–Ti) led to the formation of the single $\text{TiC}_{0.7}\text{N}_{0.3}$ phase (Fig. 1c). The results were in a good agreement with the EDS analyses, as shown in Table 1.

It was evident that the TiC (from titanium diffusion coating) and iron nitrides (from plasma nitriding) became unstable during the second stage of duplex treatments, and they were decomposed to form TiN and $\text{TiC}_{0.7}\text{N}_{0.3}$ phases. TiC phase was easily decomposed and replaced by TiN during

the second stage of Ti–PN duplex treatment, since more than 90% of the final layer consisted of the TiN phase in the Ti–PN specimen (Fig. 2b). However, primary iron nitrides (Fig. 2c) did not result in any TiN precipitation during the second stage of the titanium diffusion coating after the PN–Ti treatment, and the $\text{TiC}_{0.7}\text{N}_{0.3}$ phase precipitated as the final phase in the PN–Ti specimen (Fig. 2d). The structural similarity between TiC and TiN [32] could be attributed to the easy replacement of TiC by the TiN phase in the Ti–PN specimen, while the crystalline structures of Fe_4N and $\epsilon\text{-Fe}_{2-3}\text{N}$ phases [32] had less or even no similarity to the TiN structure, such that precipitation of TiN was not evident in the PN–Ti specimen. Formation of the $\text{TiC}_{0.7}\text{N}_{0.3}$ phase instead of the TiN (Fig. 2d) in the PN–Ti specimen could also be due to the inadequate time and temperature of surface treatment [33]. The XRD patterns did not show the presence of oxide in the coatings.

Micro-hardness measurements (Table 2) demonstrated that the duplex treatment of pack titanizing followed by plasma nitriding provided a harder surface layer, in comparison with the reverse treatment. The maximum micro-hardness value of $1435 \pm 4 \text{ HV}_{0.1}$ was achieved in the Ti–PN specimen containing TiN as the dominant phase. The duplex treatment of Ti–PN had gained comparable hardness in comparison with the other duplex-treated layers, such as pack boriding followed by plasma nitriding, with the hardness of 1234 HV [34], chromizing and post-plasma nitriding ($\sim 1400 \text{ HV}$) [9], plasma nitriding followed by pack aluminizing (1284 HV) [8], plasma nitriding and carbo-nitriding (1350 HV) [35], and pack nitriding and titanizing (1450 HV) [36].

Cross-sectional back-scattered electron SEM (BSE-SEM) micrographs of Ti–PN and PN–Ti specimens are presented in Figs. 3 and 4, respectively. The average thicknesses of the modified layers are given in Table 2. It could be seen that the Ti–PN coating provided continuous and compact layers, while the PN–Ti coating contained some cracks and pores; also, it suffered from inhomogeneity in the microstructure. During plasma nitriding of the titanium diffusion-coated surface (Fig. 3b), the surface layer was sputtered by impinging energetic ions and superficial atoms were detached from the plasma atmosphere [37, 38]; then, the surface reacted with nitrogen and the product precipitated on the surface. It might

Table 2 Thickness, micro-hardness, surface roughness and average friction coefficients of different surface-treated specimens

	AISI 1045	Ti	PN	Ti–PN	PN–Ti
Thickness (μm)					
Outer layer	–	5.7 ± 2.0	8.6 ± 1.5	1.88 ± 0.70	2.50 ± 1.07
Intermediate layer	–	–	–	4.50 ± 1.50	8.60 ± 1.23
Micro-hardness (HV)	270 ± 9	1049 ± 5	708 ± 9	1435 ± 4	1095 ± 8
Surface roughness R_a (μm)	0.257 ± 0.11	1.30 ± 0.28	0.59 ± 0.21	1.29 ± 0.27	1.58 ± 0.25
Average friction coefficient	1.11 ± 0.07	1.27 ± 0.02	1.27 ± 0.02	0.57 ± 0.01	0.65 ± 0.01

Fig. 3 Cross-sectional SEM micrographs of AISI 1045 steel after **a** titanium diffusion coating, **b** Ti-PN treatments and **c, d** their EDS elemental line scan profiles

be the reason for the remarkably less thickness of the Ti-PN coating after plasma nitriding of the titanium diffusion-coated layer (Fig. 3a, b). The cross sections of the Ti-PN duplex-treated coating consisted of two distinct regions: a uniform layer (i), which contained TiN and $\text{TiC}_{0.7}\text{N}_{0.3}$ phases with $\sim 2 \mu\text{m}$ thickness, and an intermediate layer (ii) with $\sim 4.5 \mu\text{m}$ thickness (Fig. 3b and Table 2). The regions were in a good agreement with the corresponding elemental depth profiles of Ti, N, Fe, and C elements used to illustrate the chemical composition distribution of the elements inside the reaction zone. According to the line scan of the titanized specimen (Fig. 3c), the first 13 μm thickness of the titanized layer was composed ~ 50 at% C and ~ 50 at% Ti, which referred to the presence of the TiC phase, as also indicated by its XRD pattern in Fig. 2. Titanium concentration was reduced sharply to zero in the diffusion layer having $\sim 4 \mu\text{m}$ thickness. The diffusion driving force was the negative gradient of the chemical potential of atoms, diffusing in or outward the material core. Thus, atoms were

usually diffused from the high concentration areas to the low ones. By plasma nitriding of the titanized layer, nitrogen was diffused into the titanized layer and titanium carbide became unstable; then, it was decomposed. So the process left a dense superficial nitride layer consisting of TiN with some $\text{TiC}_{0.7}\text{N}_{0.3}$ (Fig. 2). In this state, the concentration of Ti and N was decreased gradually at the interface between the outer layer and the steel matrix; however, N could be diffused deeper (25 μm) due to its smaller atomic size, as compared with titanium [39].

Cross-section microstructure of the PN-Ti specimen (Fig. 4b) showed that the coating contained two distinct layers of the gray zone (i) as the top layer and a darker zone (ii) as the second layer; there was also an intermediate layer (iii) beneath these layers, with non-uniform layer thicknesses. The corresponding line scan also showed that the darker layer consisted of more carbon, and the outer layer with gray color appearance contained a lower C and a higher N. Finally, the intermediate layer had a moderate

Fig. 4 Cross-sectional SEM micrographs of AISI 1045 steel after **a** plasma nitriding and **b** PN-Ti treatments and **c, d** their EDS elemental line scan profiles

C and a lower N (Fig. 4d). Comparing the XRD pattern (Fig. 2d) and line scan (Fig. 4d), results might indicate that the superficial layer in the PN-Ti coating was mainly composed of titanium nitro-carbide, while some TiC could be expected at the darker zone (ii) in the coating.

Line scan analysis of the PN specimen also indicated that the superficial layer was composed of ~80 at% Fe and ~20 at% N, which corresponded to the Fe_4N phase, as indicated by its XRD pattern in Fig. 2. The nitrogen content was gradually decreased from ~20 at% at the surface to zero at its interface with the substrate. After titanizing of the plasma nitrides surface, the Ti intensity remained constant inside the regions i and ii. It seems that Ti could be diffused easily in the superficial nitride layer due to the high temperature of the pack titanizing temperature, and even superficial white layer could not hinder the diffusion of Ti into the nitride layer. It could be seen in Fig. 4c and d

that the thickness of the PN-Ti layer was almost the same as the plasma-nitrided layer.

3.2 Tribological Studies

After the wear test using tungsten carbide as the counterpart abrasive, variation in the friction coefficient versus sliding distance was obtained for the specimens (Fig. 5). The average values of the friction coefficients are reported in Table 2. The single treated specimens (Ti and PN specimens) had the maximum friction coefficient of ~1.27. The friction coefficient of the titanium diffusion-coated (the monolayer containing the single phase of TiC) specimen was achieved to be by 1.26; then, it was decreased to 1.0 as the sliding length was increased (Fig. 5). Profiles of friction coefficients also showed that the profile in the Ti-PN specimen could be divided into three stages according to different wear behaviors

Fig. 5 Plots of friction coefficients versus sliding distance for surface modified

and mechanisms. These included stage I: low friction stage (~0–100 m), stage II: plowing friction stage (~100–220 m) and stage III: coating fracture stage (220–500 m) [25, 40, 41]. In stage I, the friction coefficient was low and stable, with the value ranged from 0.3 to 0.4. In this stage, it has been reported that only a small number of micro-cracks could be available with coating spallation [25]. As the sliding distance was increased, further micro-cracks might be developed, and wear debris could be generated due to the local spallation of the coating. In stage II, the friction coefficient started to rise rapidly from 0.4 to 0.7. The wear debris which was mostly made of TiN particles and generated during the initial stage of sliding could increase the friction coefficient remarkably and accelerate the wear of the coating [42, 43]. During stage III, the friction coefficient reached a plateau amount with an average value of ~0.7, which could be due to the self-lubricating property of the TiN/TiC_{0.7}N_{0.3} coating because of the formation of titanium oxide (TiO₂) during the wear test [44]. The fluctuation in the friction coefficient was attributed to the accumulation of wear products in the contact zone [45], or due to the adhesive friction. This frictional behavior could be due to the presence of the hard superficial nitride/carbo-nitride layer which was well supported by the intermediate layer.

Figure 6 shows the wear factors of the specimens after sliding against a WC pin under 20 N load for 500 m. The wear factor values of the TiN coating (the multilayer containing TiN/TiC_{0.7}N_{0.3} phases) were smaller than those of the PN–Ti, Ti, and PN coatings; the results were, in fact, consistent with their lower friction coefficients, as shown in Fig. 5 and Table 2. It could be seen that the higher surface hardness resulted in a lower wear factor, which was in agreement with the Achard's equation [46], which suggests the wear resistance is increased by hardness:

$$V = KW/H \quad (4)$$

$$K = k/3 \quad (5)$$

Fig. 6 Variation of wear factors and surface hardness of experimental specimens

where K is a dimensionless constant, W is the normal load, H is the surface hardness and k is the fraction of surface area in contacts with the wear debris (probability factor < 1). In addition, relatively lower friction coefficients were obtained at the higher surface hardness [47, 48]. There was an exception in the wear factor results (Fig. 6); this was since the wear factor of the PN specimen (the monolayer containing Fe₄N/Fe₂₋₃N) was similar to that of the PN–Ti one (containing TiC_{0.7}N_{0.3}), even though the surface hardness of the PN–Ti specimen (1095 ± 8 HV_{0.1}) was higher than that of the PN specimen (708 ± 9 HV_{0.1}). This could be due to the rougher nature of the PN–Ti in comparison with that of the PN specimen (see Table 2). It is well known that the surface roughness can have a significant effect on the wear factor, as contact surfaces and pressures between the asperities of both engaging sides can be increased with the roughness of the surfaces, leading to the higher wear loss. Therefore, the Ti–PN (1435 ± 4 HV_{0.1})-coated specimen with the highest hardness, which contained the mixture of TiN/TiC_{0.7}N_{0.3} phases, showed the lowest wear factor and

friction coefficient. Moreover, the relatively lower wear resistance of titanium diffusion-coated specimen, in terms of its higher hardness, in comparison with PN and PN-Ti specimens could be attributed to the presence of the hard debris of TiC and/or TiO₂ which was entrapped on the wear tracks, increasing the wear factor [49].

Examination of the wear tracks of Ti-PN and PN-Ti specimens was conducted using SE and BSE-SEM and

EDS to clarify the wear mechanisms involved (Fig. 7 and Table 3). Figure 7a, b shows the micrographs of the wear tracks on the Ti-PN coating. An overall even surface with some micropores besides few micro-cracks could be regarded as the main features of the wear track on this coating. Formation of these micropores could be related to the continuous plastic flow of the coating and debris [45]. Milosev et al. [50] reported that micropores on the wear

Fig. 7 a, c Secondary electron and b, d BSE micrographs of worn surfaces for a, b Ti-PN and c, d PN-Ti specimens

Table 3 EDS analyses of worn surfaces on duplex-treated specimens for points A–F in Fig. 7 (at%)

Element	A	B	C	D	E	F
O	18.35	11.28	48.51	23.68	13.45	3.70
Fe	41.82	72.13	18.35	24.37	13.37	66.89
Ti	20.30	20.31	28.17	39.75	52.54	23.02
N	19.53	16.28	4.97	12.20	20.65	6.39

track could be formed in the process of the release and entrapment of nitrogen within the superficial oxide layer, which caused pores or delamination. The presence of oxygen in points A and B on the wear track (Table 3) indicated that the continuous oxidation of the superficial layer could be potentially responsible for the wear mechanism of the Ti–PN specimen, wherein the oxidation of the surface layer due to the increase in temperature at the contact surface [45], the fracture of the thin and brittle oxide layer [51] and its removal from the contact zone by abrasive pin and, finally, re-oxidation of the newly fresh surface could promote the substantial wear loss that happened in the coating. The presence of the significant iron in the worn surface could express that iron oxide constituted the main part of the oxide layer; they could also reduce the friction coefficient to some extent.

Heavy micro-fragmentation process and plastic deformation were seen on the wear track of the PN–Ti coating containing the $\text{TiC}_{0.7}\text{N}_{0.3}$ phase (Fig. 7c, d), which caused the larger wear losses. Therefore, the wear resistance of this coating was lower than that of the Ti–PN coating. It could be due to the presence of some micro-cracks on the superficial layer (see Fig. 4b), which could decrease the resistance of the coating against normal and shear components of the sliding force. The EDS results (Table 3) also showed that the dark regions in the BSE micrograph (Fig. 7d) corresponded to oxygen-rich areas, while the bright regions contained the higher Fe content. Average of oxygen on the worn track was higher than the other wear tracks of the Ti–PN specimen, showing that oxidation mechanism could play the more prominent role on the wear of the PN–Ti coating.

4 Conclusions

1. XRD analysis revealed the formation of TiN and $\text{TiC}_{0.7}\text{N}_{0.3}$ phases in the Ti–PN layer, $\text{TiC}_{0.7}\text{N}_{0.3}$ phase in the PN–Ti layer.
2. Maximum surface micro-hardness of $1435 \pm 4 \text{ HV}_{0.1}$ was obtained in the layer produced by Ti–PN specimen.
3. Duplex surface treatment dramatically reduced the coefficient of friction and wear factor of the steel and Ti–PN specimens showed the best tribological performance among the other treated specimens.
4. Oxidation and mild fracture wear were the main wear mechanism of Ti–PN coating. The wear mechanism in PN–Ti specimen involved higher oxidative wear followed by plastic deformation wear regimes.

Acknowledgements The authors would like to thank Semnan University for financial support of this research. The first author also thanks Plasma Pajouh Co. and Mr. A. Ziyari for his assistance in the experiments.

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