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Parametric optimisation of microhardness on heat-treated electroless Ni-YSZ cermet coating

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ABSTRACT

Purpose: The paper discusses the parametric optimisation of the electroless Ni-YSZ cermet coating microhardness upon heat treatment. Heat treatment is a process to increase the mechanical properties of the electroless nickel coating and it can be enhanced by manipulating its parameters. Parametric optimisation is conducted by the design of experiment full factorial 3x3 with 27 runs. Treating temperature, treating time and ceramic particle size parameters at 3-level are evaluated using statistical tool ANOVA in Minitab20.

Design/methodology/approach: Ni-YSZ cermet coating is deposited onto a high-speed steel substrate using the electroless nickel co-deposition method. The temperature and time were varied in a range of 300-400oC and 0-2 hours respectively. The microhardness measurements were carried out using a Vickers microhardness tester (Shimadzu) according to ISO 6507-4. The surface characterisation was analysed using Cambridge Stereoscan 90 Scanning Electron Microscope (SEM) coupled with Energy Dispersive X-ray Analysis (EDXA).

Findings: The optimum condition in obtaining high microhardness on Ni-YSZ cermet coating is evaluated by statistical tool ANOVA in Minitab20 software. It is found that the most significant parameter for high microhardness is at the treating temperature of 400oC followed by treating time at 2 hours using nano-sized YSZ particles. The ceramic particle size is found not a significant parameter in obtaining a high microhardness, however it has effect on interaction between treating temperature and treating time.

Research limitations/implications: The paper only limits to the optimisation condition of microhardness on Ni-YSZ cermet coating hardness property by varying heat treatment parameters.

Practical implications: The optimisation condition obtained might only applicable to the electroless Ni-YSZ cermet coating with similar electroless nickel solution and treatments.

Originality/value: The value of this work is the heat treatment parametric optimisation to obtain high microhardness on electroless Ni-YSZ cermet coating by using the design of experiment 3-level full factorial.

Keywords: Full factorial, Electroless, Ni-YSZ, Cermet, Coating, Microhardness, Treating temperature, Treating time

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ANALYSIS AND MODELLING

1. Introduction

Electroless deposition is a chemical reduction process of metallic ions onto a substrate of any materials, either metals, polymers or ceramics. Electroless deposition processes are available for metal matrix aluminium, nickel, copper, silver and iron [1]. The most common electroless deposition is nickel due to its domineering superalloy properties such as excellent corrosion, wear and abrasion resistance and it can be improved by incorporating hard ceramic particles such as diamonds, carbides and nitrides [2].

Electroless nickel deposition chemical process is illustrated in chemical equation (1)-(4) below. The reduction of nickel ions into metallic nickel is given in equation (2) and it is an in-situ process. Electroless deposits usually contained 2-14% of phosphorus due to the hypophosphate ions reduction as in equation (3). These chemical processes are affected by several parameters such as bath pH, bath temperature, bath concentration, bath loading, soaking time and deposition rate [3,4].

$$(H_2PO_2)^- + H_2O \rightarrow H^+ + (HPO_3)^{2-} + 2H_{abs} \quad (1)$$

$$Ni^{2+} + 2H_{abs} \rightarrow Ni + 2H^+ \quad (2)$$

$$(H_2PO_2)^- + H_{abs} \rightarrow H_2O + OH^- + P \quad (3)$$

$$(H_2PO_2)^- + H_2O \rightarrow H^+ + (HPO_3)^{2-} + H_2 \quad (4)$$

The incorporation of inert ceramic particles into the electroless nickel-phosphorus upon deposition, produce cermet coating. The cermet gives better mechanical properties as the hard and inert ceramic particles enhanced the electroless nickel-phosphorus overall performance [5-7]. The parameters that influence the incorporation of the ceramic particle are dispersion stability of the particle in electroless solution, particle conductivity as well as the particles' hydrophobic or hydrophilic properties [8]. Besides that, particle loading and particle sizes also give influence to the particle incorporation in the cermet [9].

Heat treatment is a method where the coating is heated to a certain temperature and hold at that temperature for a period of time in order to improve its mechanical properties. The effect of heat treatment on the electroless nickel-phosphorus deposit is dependent on the amount of phosphorus that is it decreases as the phosphorus content increases due to the structure change from crystalline to amorphous [10] and the finding is reported in electroless nickel-titanium nanocomposite coating [11]. Upon heating, the electroless nickel coating between 300-400°C caused phase transformation of the coating from amorphous to crystalline structure [12].

In another study, heat-treated electroless nickel-activated carbon increased nickel composition in the coating as the treating time increases from 1 to 2 hours [13]. The effect of heat treatment was also found in electroless nickel boron nitride where it was found 40% increment of the coating hardness after being heat-treated at 300°C however, it reduced the wear rates [14]. The nickel-phosphorus coating heated between 400-700°C followed by water-quenched increased the hardness of the coating as well as the surface adhesion [15]. The microhardness and wear of electroless nickel-phosphorus increase as the temperature increases from 300 to 600°C, however above 600°C for 4 hours show no effect on both due to the formation of Ni_3P crystalline [16,17].

An optimisation study conducted by Ahmadkhaniha et. al. on heat treatment for electroplated Ni/P and NiP/SiC reported that the temperature is the main role for hardness and corrosion resistance [18]. Optimisation on surface roughness of electroless nickel-phosphorus coating using statistical model Box-Behnken design obtaining lowest surface roughness at 0.32 µm [19]. The mathematical model also can be used as a method of optimisation for surface hardening technologies [20].

In this paper, the heat treatment parameters such as treating temperature, treating time and particle size of yttria-stabilised zirconia (YSZ) is analysed to give optimum condition in gaining good microhardness response. The optimisation is done by using the design of experiment adopting the full factorial method that will give higher accuracy compared to the Taguchi method.

Table 1.
Chemical composition of HSS substrate

Element	C	Si	Mn	P	S	Cr	Mo	V	W
Composition, wt.%	0.890	0.200	0.280	0.025	0.0008	3.930	4.720	1.700	6.130

2. Materials and methodology

2.1. Materials

The substrate is a base material for cermet coating to be deposited on it. The substrate used is a high-speed steel (HSS) from Bohler-Bleche GmbH manufacturer with composition in Table 1.

The substrate was cut using a wire-cut electrical discharge machine into dimensions with a diameter of 25 mm and thickness of 1.25 mm (Fig. 1). Reinforcement particles of yttria-stabilised zirconia (YSZ) with 8 mol% by Tosoh Japan are used. There are two types of 8YSZ with different sizes that are nano-sized and micro-sized of nominal 2 μm. The optimum particle loading is in the range of 5-10 g/L [6]. The particle loading for both sizes used is 10 g/L and the mixed particle size is by a ratio of 1:1. Chemicals used for substrate activation and electroless nickel deposition processes are prepared by AR grade chemicals and high purity ionised water as tabulated in Table 2.

Fig. 1. Sample HSS after coating

2.2. Electroless nickel coating

The electroless nickel process requires substrate preparation to ensure the substrate is cleaned and activated. This includes soaking the substrate into 4 different chemicals as in Table 2 followed by electroless nickel solution as described in the previous study [21] and the overall process is illustrated in Figure 2.

Table 2.
AR grade chemical

Trade Name	Soaking time, minute	Temperature, °C
Circudep 3500AB	15	60
Uniphase PHP Pre-catalyst	15	20
Uniphase PHP Catalyst	15	40
Niplast AT78	15	40
Electroless Nickel Slotonip 1850	30	89

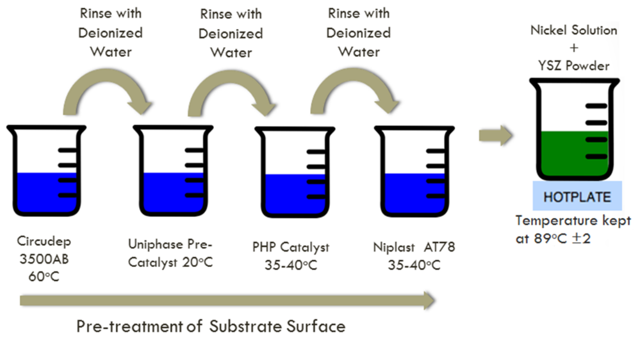

Fig. 2. Illustration of the electroless nickel coating process

2.3. Heat treatment, microhardness and characterisation

The cermet coating was heated in an electric furnace Protherm under a controlled environment of nitrogen gas flow at a constant pressure of 1 atm. The temperature and time were varied in a range of 300-400°C and 0-2 hours respectively. The microhardness measurements were carried out using a Vickers microhardness tester (Shimadzu) under microhardness range; HV0.025 (25 gf) at a 0.245 N force for 10 s according to ISO 6507-4. The surface characterisation was analysed using Cambridge Stereoscan 90 Scanning Electron Microscope (SEM) coupled with Energy Dispersive X-ray Analysis (EDXA).

2.4. Experimental parameters and design

Parametric optimisation is conducted using the design of experiment full factorial of 3x3 method with 27 runs. Three heat treatment parameters are used, namely, treating temperature, treating time and ceramic particle size with a cermet hardness as a response. Treating temperature is varied from 300°C-400°C; treating time between 0-2 hours and ceramic particle size from nano (N), micro (M) and mixed (NM) with 1:1 ratio. The optimization target is aimed to have a high yield of microhardness on the cermet coating. The full factorial 3x3 with three independent parameters is tabulated in Table 3.

Table 3.
Design of experiment full factorial 3^3

Parameters	Level		
	L1	L2	L3
Treating Temperature, °C	300	350	400
Treating Time, hr	0	1	2
Particle size	N	NM	M

The statistical tool used to analyse the data is analysis of variance (ANOVA) in Minitab20 software. The upper and lower limit is set to 95% confidence level.

3. Results and discussion

The treating temperature, treating time and particle size are categorical variables for heat treatment conditions. These categorical variables are independent of each other. The yield of the optimisation is higher the better microhardness HV0.025 of the Ni-YSZ cermet coating deposited onto HSS substrate for full factorial 3x3 with 27 runs are tabulated in Table 4.

3.1. Effect of treating temperature and time

Statistical analysis ANOVA for treating temperature (HTemp) and treating time (HTime) effect is shown in Table 5. Since the interval of upper and lower limit was set to 95% confidence level, the significant differences between the parameters are indicated by the P-value < 0.05. Therefore, both treating temperature and treating time are significant parameters in giving a high microhardness response. However, there is no significant difference for the interaction parameter of treating temperature and treating time (HTemp*HTime). It can be concluded that the interaction between these two parameters is insignificant.

The effect of treating temperature on the microhardness of Ni-YSZ cermet coating is greater compared to the effect of treating time as shown in Figure 3a. High microhardness can be obtained at a temperature of 400°C and a time of 2 hours. The interaction between treating temperature and treating time is almost none. The interaction plot in Figure 3b shows no cross-linked between them. Thus, the interaction between treating temperature and treating time parameters have no effect towards gaining a high micro-hardness of the cermet coating.

Table 4.
Microhardness HV yield for full factorial 3x3

Treating Temperature	Treating Time	Particle Size	Micro-hardness HV
300	0	N	110.3
300	0	N	133.2
300	0	N	118.1
300	1	NM	119.1
300	1	NM	133.0
300	1	NM	104.8
300	2	M	133.4
300	2	M	136.9
300	2	M	143.1
350	0	NM	160.2
350	0	NM	162.1
350	0	NM	158.0
350	1	M	173.8
350	1	M	172.0
350	1	M	148.3
350	2	N	176.1
350	2	N	168.0
350	2	N	166.4
400	0	M	170.0
400	0	M	172.1
400	0	M	173.7
400	1	N	187.1
400	1	N	189.2
400	1	N	187.0
400	2	NM	209.1
400	2	NM	211.9
400	2	NM	166.1

Table 5.
ANOVA for treating temperature (HTemp) and treating time (HTime)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
HTemp	2	16429.1	8214.54	58.38	<0.0001
HTime	2	1326.7	663.33	4.71	0.0226
HTemp* HTime	4	341.3	85.32	0.61	0.6632
Error	18	2532.8	140.71		
Total	26	20629.9			

a)
b)

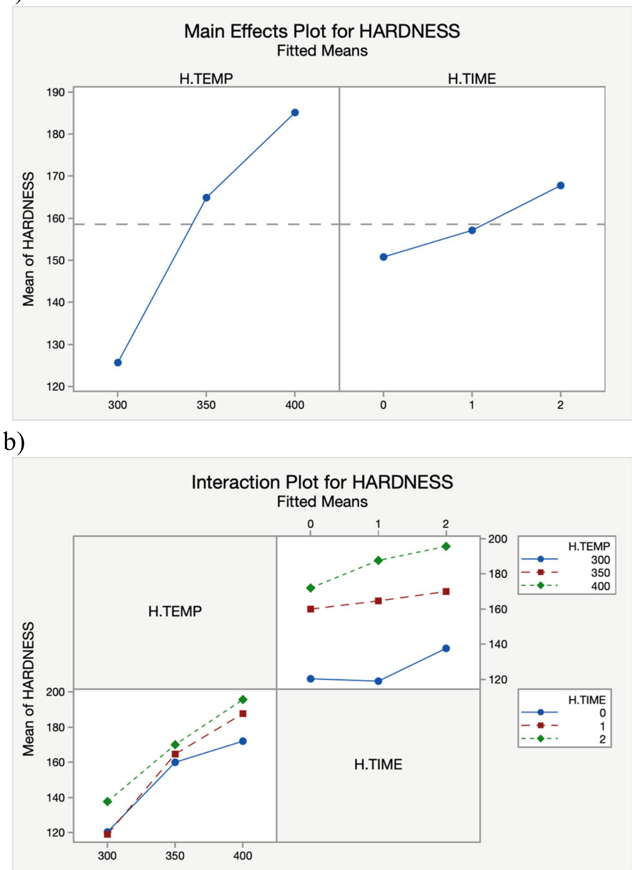

Fig. 3. Treating temperature and treating time effect; a) main effects plot; b) interaction plot

3.2. Effect of treating temperature and particle size

The output of ANOVA for treating temperature and particle size is given in Table 6. The pairwise differences between treating temperature (HTemp) and particle size (PSize) at three levels are represented by the P-value < 0.05. The significant difference is treating temperature and the interaction parameters between temperature and particle size. Particle size parameter is found not to be significant as the P-value is more than 0.05.

The effect of treating temperature and particle size on microhardness is shown in Figure 4a. Treating temperature shows great effect whereas particle size almost has no effect (flat line). High microhardness can be obtained at the highest treating temperature of 400°C and particle size of nano. In terms of the interaction effect between these two parameters, the interaction plot in Figure 4b shows significant interaction between treating temperature and particle size where the lines are crosslinked mostly for treating temperature. This means that the interaction of treating temperature and particle size helps to obtain high microhardness.

Table 6.
ANOVA for treating temperature (HTemp) and particle size (PSize)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
HTemp	2	16429.1	8214.54	58.38	<0.0001
PSize	2	8.3	4.16	0.03	0.9709
HTemp* PSize	4	1659.6	414.90	2.95	0.0489
Error	18	2532.8	140.71		
Total	26	20629.9			

a)
b)

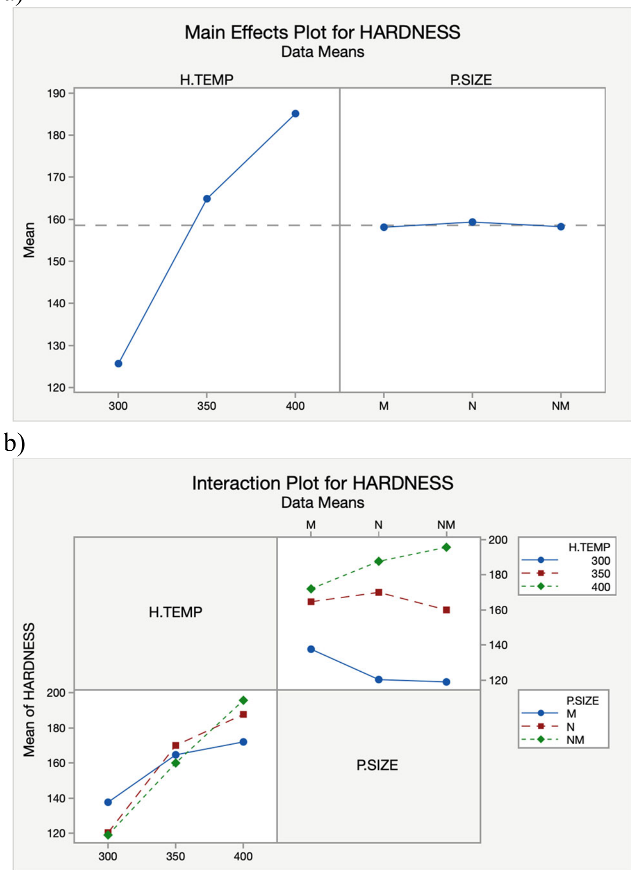

Fig. 4. Treating temperature and particle size effect; a) main effects plot; b) interaction plot

3.3. Effect of treating time and particle size

Table 7 shows the ANOVA for treating time (HTime) and particle size (PSize). It shows that treating time has a significant difference between the upper and lower range of treating temperature. On the other hand, the particle size has no significant difference between nano (N), micro (M) and mixed (NM) ranges. However, the interaction between parameters treating time and particle size has a great significant difference.

Table 7.
ANOVA for treating time (HTime) and particle size (PSize)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
HTime	2	1326.7	663.33	4.71	0.0226
PSize	2	8.3	4.16	0.03	0.9709
HTime* PSize	4	16762.0	4190.51	29.78	<0.0001
Error	18	2532.8	140.71		
Total	26	20629.9			

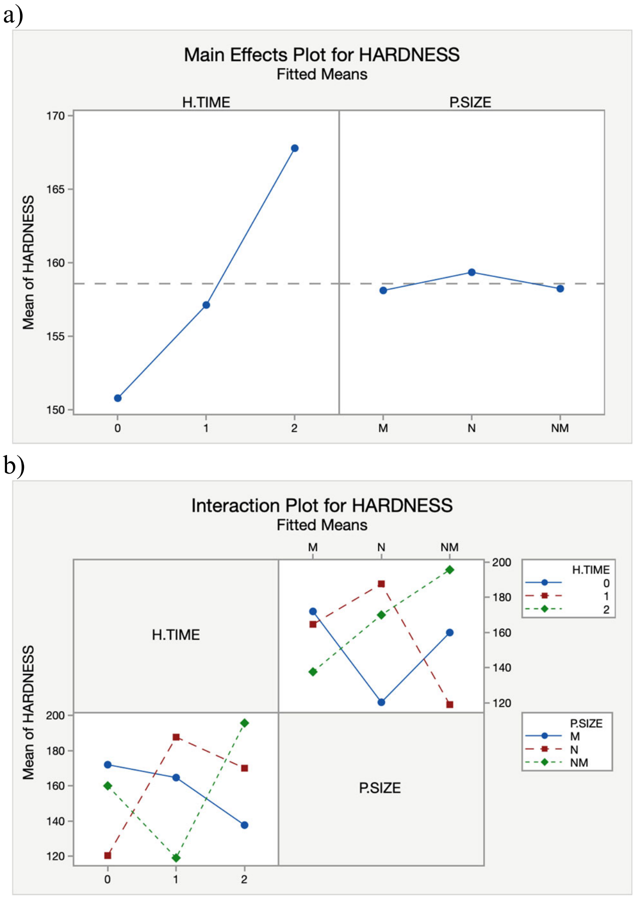

Fig. 5. Treating time and particle size effect; a) main effects plot; b) interaction plot

The effect of treating time and particle size on the microhardness of Ni-YSZ cermet is shown in Figure 5a. Treating time shows a great effect on microhardness whereas particle size has a very minimal effect. High microhardness can be obtained at a high treating time of 2 hours and particle size of nano. The interaction between treating time and particle size is strongly significant. Figure 5b shows crosslinked lines for both parameters plots.

3.4. Optimisation

The optimum conditions to obtain a high microhardness yield of electroless Ni-YSZ cermet coating is at a high temperature of 400°C for 2 hours using nano-sized ceramic particles. Even though particle size shows an insignificant difference as an independent parameter, it has a significant interaction effect on both the treating temperature and treating time.

The optimum treating temperature at 400°C can be explained by the phase transformation within the nickel matrix in the presence of phosphorus as described by Kumar et. al [17]. At a temperature of 300°C and below, the Ni-P structure is amorphous thus it gives low hardness. As temperature increases to 350°C, a precipitation of hard intermediate Ni_3P is crystallised, however, higher temperature and prolong heating caused Ni_3P grain to coarsen [1] which reduce the hardness of the coating. In all, the effect of treating temperature and treating time on the Ni-YSZ cermet coating was mostly influenced by the properties of the nickel-phosphorus matrix rather than the ceramic particle. This is because the ratio composition of the Ni-P matrix to the ceramic YSZ is approximately 70 to 30. Hence, that is the reason why the effect of YSZ particle size, in this case, is not influential. This is supported by Balaraju et. al. findings that the particle size has a small or marginal influence on the microhardness of the cermet coating [22].

Most studies reported that lower particle mass gain obtained as the particle size reduced [23]. However, in this case, the finer particle size shows influence in the interaction between treating temperature and time.

3.5. Surface characterisation

The surface of electroless Ni-YSZ cermet coating with micro-sized particle sample is analysed using SEM-EDXA. The SEM micrograph in Figure 6a shows white dots that are represented the ceramic particle of YSZ surrounded the dark area that is the nickel-phosphorus matrix. Figures 6b-d show the images of selected element mapping of phosphorus, nickel and zirconium respectively. The mapping shows that all elements are distributed uniformly across that cermet coating.

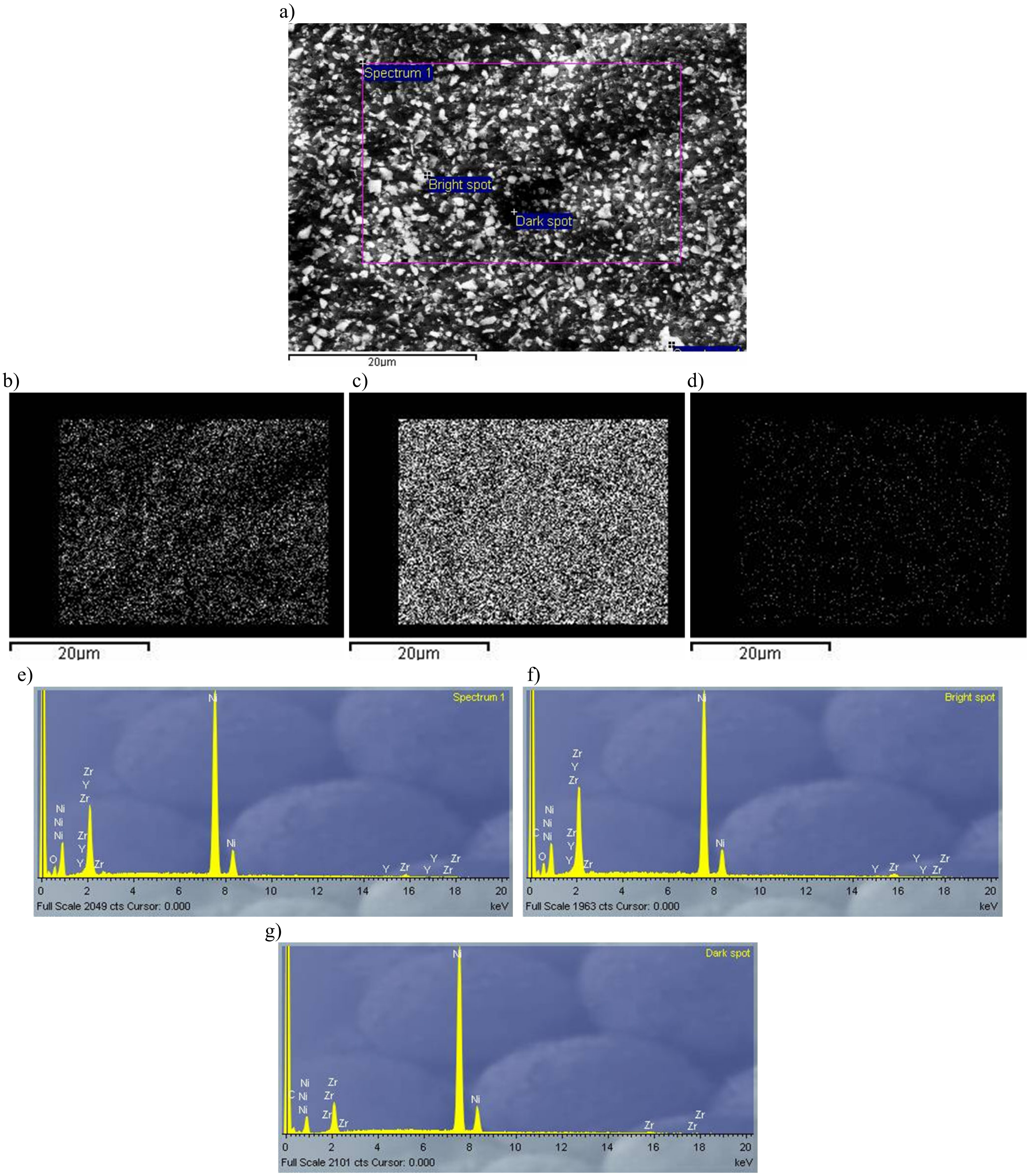

Fig. 6. Characterisation of Ni-YSZ cermet coating: a) SEM micrograph; b) element mapping of phosphorus; c) element mapping of nickel; d) element mapping of zirconium; e) EDXA spectrum 1; f) EDXA spectrum bright spot; and g) EDXA spectrum dark spot

There is no sign of agglomeration or coalescent of ceramic YSZ particles in Figure 6d. The EDXA spectrum of area spectrum 1 in Figure 6e shows the presence of nickel, zirconium and yttria elements. The spectrum bright spot in Figure 6f shows a higher peak of zirconium element compared to spectrum 1. This indicates that the bright spot or white area is the ceramic particle YSZ. The dark spot spectrum in Figure 6g shows the metallic nickel matrix where the zirconium and yttria peaks are at the lowest.

The uniformity of all element mappings clearly indicates that the Ni-YSZ cermet coating is uniformly distributed, which improves the coefficient of thermal expansion of throughout the coating although the coefficient of thermal expansion of these two materials are 16.5 x 10^{-6} K^{-1} and 10.7 x 10^{-6} K^{-1} respectively. This relatively avoids delamination and cracking as it is heated up to 400°C for 2 hours.

4. Conclusions

Parametric optimisation of microhardness on the Ni-YSZ cermet coating is done by the design of experiment full factorial 3x3. Parameters evaluated are treating temperature, treating time and ceramic particle size at three levels.

The outcome of Minitab Analysis of variance (ANOVA) shows that the most significant parameters affecting the high microhardness are the treating temperature followed by the treating time. The particle size parameter is not significant. However, it does show significant interaction with treating temperature and treating time in obtaining high micro-hardness of the Ni-YSZ cermet coating. Overall, the optimum condition in obtaining high microhardness is by having the treating temperature at 400°C for 2 hours using nano-sized YSZ particles.

The characterisation on the Ni-YSZ cermet coating surface indicates that all major elements in the cermet coating are uniformly distributed. This improves the Ni-YSZ cermet coating mechanical stability and integrity.

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