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Effect of particle size on surface roughness and morphology of heat-treated electroless Ni-YSZ coating

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ABSTRACT

Purpose: The paper discusses the surface characterisation of electroless nickel-yttria-stabilised zirconia (Ni-YSZ) coating with varying YSZ particle sizes and undergoes heat treatment at a temperature between 300-400°C for 1-2 hours for wear resistance purposes. This finding will be helpful to the application of Ni-YSZ as an alternative coating for cutting tools.

Design/methodology/approach: The surface characterisation was analysed using JOEL Scanning Electron Microscope (SEM) coupled with Energy Dispersive X-ray (EDX) JSM 7800F. The crystallographic structure of materials was analysed by X-ray diffraction (XRD) Bruker D8 Advance instrument. The Ni-YSZ coating was deposited using electroless nickel co-deposition of 8YSZ ceramic particles with a nano, mixed and microparticle sizes onto a high-speed steel (HSS) substrate. The coatings were heat treated at temperature 300-400°C and time 1-2 hours. The surface roughness was measured using Mitutoyo surface roughness tester SJ-301.

Findings: The electroless Ni-YSZ coating deposited has an average thickness of 30 µm. It is found that the coating morphology electroless coating without YSZ particle incorporation (EN) and Ni-YSZ nano (N) is smoother compared to the Ni-YSZ mixed (NM) and Ni-YSZ micro (M). The EDS composition analysis shows the YSZ content in the electroless Ni-YSZ coating for N samples is the lowest, whereas NM samples are the highest. This resulted in the surface roughness behaviour where the mixed-size YSZ particle gives the highest roughness at all temperatures. The XRD analysis shows that heating temperatures above 300°C caused the precipitation of Ni_3P crystalline.

Research limitations/implications: Previous studies in the surface characterisation of electroless nickel composite are scarce; thus, the study has limitations in finding supporting data.

Originality/value: The surface characterisation especially related to the surface roughness of the electroless nickel, either the Ni-P or composites or alloys are rarely reported. Thus, this study enlightened the effect of particle size on surface roughness and morphology of heat-treated coatings.

Keywords: Particle size, Electroless coating, Ni-YSZ, Heat treatment, Surface roughness, Surface morphology

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PROPERTIES

1. Introduction

Surface coating for corrosion and wear resistance purposes has been increasingly important and has been applied in many industries [1,2]. The application of surface coating can be divided into the methods of coating and types of materials used. The most common and outstanding thin and dense coating method is thermal spraying. However, this caused deterioration of coating properties due to the effect of high temperatures [3,4]. Another coating method option is cold spraying, which gives the coating low porosity and surface roughness [5]. Electrodeposited and electroless coating are also common methods. The electrodeposited method depends on the electrolyte bath current density, composition, pH and agitation method [6,7]. On the other hand, the electroless method is the in-situ deposition of the metal matrix of either aluminium, nickel, copper, silver or iron. The most common one is electroless nickel due to its high corrosion and wear resistance of nickel [8,9].

The amount of phosphorus content highly influences the properties of electroless nickel coating, either low phosphorus (2 to 5%), medium phosphorus (6 to 9%), and high phosphorus (10 to 13%), where the structure may be microcrystalline, amorphous or a combination of both [10,11]. The advantage of the electroless nickel process that they can be deposited onto almost all surfaces, either metals, polymers or ceramics.

Besides electroless nickel coating, there is electroless composite coating where two or more dissimilar materials are added to the nickel matrix [12]. Commonly, inert ceramic particles such as diamond [13,14], boron nitride [15], alumina [16] and yttria-stabilised zirconia [17] are used to make outstanding properties. The influence of electroless coating parameters plays an important effect on the deposit quality, and the incorporation of particle quantity can be enhanced by using smaller-sized particles, stirring agitation and blasting surface treatment [18,19].

Heat treatment has been used to improve the mechanical properties of the coating, such as its hardness, surface roughness and wear [20]. Electroless nickel coatings have the highest hardness when heated at 300°C to 400°C for 1 to 2 hours [21,22]. The hardness and surface adherence of the Ni-P deposit increased during heat treatment at 400°C to 700°C and water quenching [23]. In another study, heating the electroless nickel between 300-400°C improves the phase transformation of the deposit from amorphous to crystalline [24].

A finding also discovered that the size of nickel crystallites changes as the heat treatment temperature rises [25]. Most of the published works have concentrated on heat treatment and phosphorus concentration effects in Ni-P coatings placed on steels. However, the heat treatment of Ni-P coatings to obtain maximal hardness is done for 1 hour at 400°C. According to Bae [26], the bonding strength was more significant when the specimen was plated with Ni-P than when it was not plated, and it increased with increasing surface roughness. Due to the increased wettability of electroless Ni-P plating with molten solder, an improvement in bonding strength was attributed to the creation of a thicker solder reaction layer beneath the bonding interface.

There is a finding that shows the effect of surface roughness after the coating and concludes that there are higher values of surface roughness after the coatings [27]. The effect of the surface roughness on the nano composition can make the particle concentration the most significant factor in the wear rate, followed by current density and temperature. Furthermore, the surface roughness is affected substantially by the wear behaviour and thickness of the coating [28]. Therefore, this study aims to determine the influence of heat treatment on the surface roughness of electroless Ni-YSZ coating.

The heat-treated electroless Ni-YSZ coating investigation on the surface characterisation by varying particle sizes supports the application of this coating as an alternative coating material for cutting tools.

2. Materials and methodology

2.1. Coating substrate

The substrate is a surface material for the coating to be deposited on is high-speed steel (HSS) from Bohler-Bleche GmbH manufacturer with composition in Table 1. HSS substrate is used to represent the HSS cutting tool. The hardness of the HSS substrate is HBW229.

Table 1.
Chemical composition of HSS substrate

Element	Composition, wt.%
Fe	82.124
C	0.890
Si	0.200
Mn	0.280
P	0.025
S	0.0008
Cr	3.930
Mo	4.720
V	1.700
W	6.130

The substrate was cut to the dimensions shown in Figure 1 using a wire-cut electrical discharge machine with a thickness of 1.25 mm.

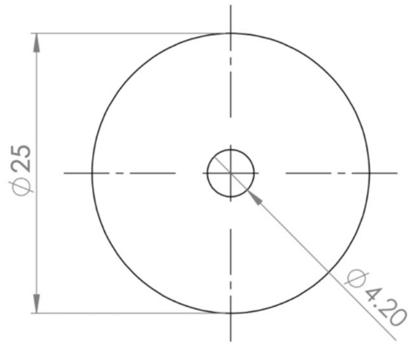

Fig. 1. HSS substrate and its diameter

2.2. Electroless nickel coating

Electroless nickel solutions were prepared by AR grade chemical and high-purity de-ionised water as described in previous research [29]. The HSS substrate was pre-treated in 4 different chemicals namely coprolite, pre-catalyst, catalyst and niplast, for 15 minutes each at different temperatures, as described in Table 2. Then the substrate was placed in Slotonip electroless nickel solution together with 8%YSZ particles for the co-deposition process to complete.

The particles of yttria-stabilised zirconia (YSZ) with 8 mol% by Tosoh Japan were used. There were two different sizes of 8YSZ particle use, (1) nano-sized (N) range between 100-500 nm and (2) micro-sized (M) of nominal 2 μm. The optimum particle loading was in the range of 5-10 g L^{-1} [30]. The particle loading for both sizes was 10 g L^{-1}. The mixed (NM) particle size was added in a ratio of 1:1. The thickness of the coating before and after coating was measured using Mitutoyo digital micrometre.

Table 2.
Operating Parameters for electroless Ni-YSZ Coating

Deposition Parameters	Ni-YSZ coating
Nickel Sulphate	5-6 g L^{-1}
Sodium Hypophosphite	27-33 g L^{-1}
pH	5.7-6.3
Temperature	87± 2°C
Stirring Mechanism	Magnetic stirring (350 rpm)
YSZ Dispersion	5% by volume
Deposition Time	1 hr
Bath Volume	200 ml

2.3. Heat treatment

The electroless Ni-YSZ coating undergoes heat treatment process to improve its mechanical properties. The coating specimens were placed in an enclosed ceramic jar to ensure that the samples are in controlled environment of nitrogen gas flow at a constant pressure of 1 atm. This ceramic jar was then placed in a Protherm electric furnace and heated to the desired temperature. The temperature and time were varied in a range of 300-400 °C and 1-2 hours, respectively as conducted by previous studies [31-33]. The sample coatings were cooled to room temperature in the air.

2.4. Surface roughness

The coating surface was measured using Mitutoyo surface roughness tester SJ-301. The sample was placed on the platform and secured by an anti-slip cloth underneath. The surface roughness stylus was placed just touching the coating surface but not pressing too hard. The best five surface roughness measurements were recorded and averaged. The measurements were taken along the radial direction of the sample at different positions on the surface. The surface roughness, Ra, is the arithmetic average of the surface profile and is the most measured. Ra dimensional unit is micrometre (μm).

2.5. Materials characterisation

The surface characterisation was analysed using JOEL Scanning Electron Microscope (SEM) coupled with Energy Dispersive X-ray (EDX) JSM 7800F. The crystallographic structure of materials was analysed by X-ray diffraction (XRD) Bruker D8 Advance instrument.

3. Results and discussions

3.1. Coating thickness

The electroless Ni-YSZ coating was accomplished by in-situ deposition of metallic nickel incorporated with YSZ particles. The YSZ particles used are in various sizes, nano, micro, and a mixture of nano and micro. The coating thickness of electroless coating without YSZ particle incorporation (EN), Ni-YSZ nano (N), Ni-YSZ mixed (NM), and Ni-YSZ micro (M) are tabulated in Table 3 below. The coating thickness ranges between 0.019-0.039 mm and the overall average thickness of the coating is 30 µm.

Table 3.
Electroless Ni-YSZ coating thickness

Sample	Coating thickness, mm			
	EN	N	NM	M
1	0.039	0.026	0.039	0.032
2	0.035	0.035	0.031	0.032
3	0.028	0.023	0.024	0.032
4	0.031	0.030	0.030	0.028
5	0.029	0.034	0.025	0.029
6	0.032	0.033	0.019	0.028
7	0.029	0.025	0.027	0.021
Average	0.032	0.029	0.028	0.029

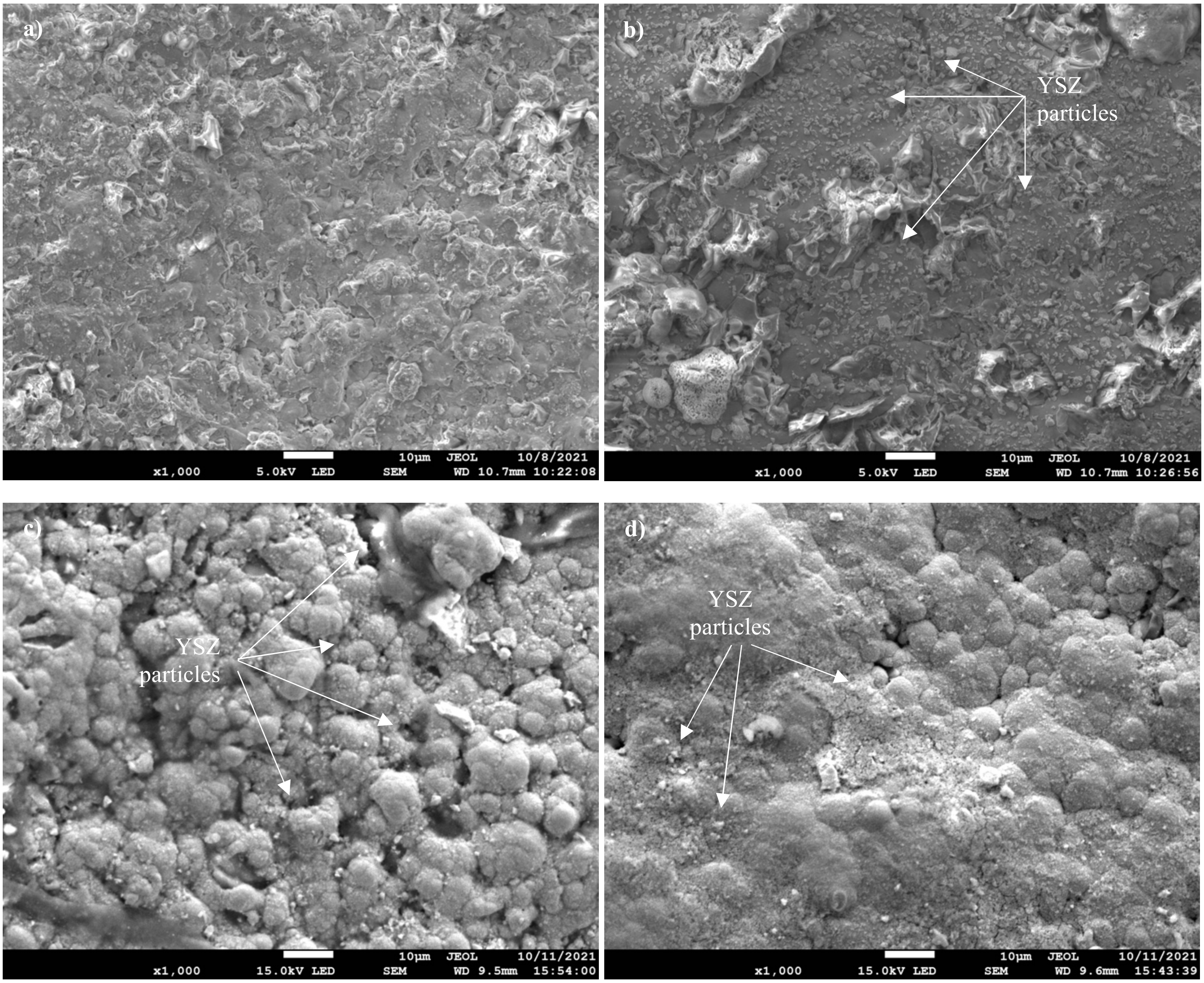

Fig. 2. Morphology of electroless coating for a) EN, b) N, c) NM, d) M

The variation of particle sizes added in the electroless Ni-YSZ coating does not influence the thickness of the coating. In electroless nickel deposition, the coating thickness depends on the coating time [8].

3.2. Coating morphology

FESEM JOEL JSM 7800F characterised the morphology of as-deposited nickel (EN), and Ni-YSZ coatings to observe their morphology. SEM images of coating without YSZ (EN) and electroless Ni-YSZ coatings with varying particle YSZ sizes are shown in Figure 2.

The morphology of electroless coating without YSZ particle (EN) shown in Figure 2a is similar to the N sample in Figure 2b. The different between these images is that in Figure 2b, there are nano-sized YSZ particles uniformly distributed within the grey nickel metallic matrix. The morphology of these coatings is smoother compared to the NM sample in Figure 2c and the M sample in Figure 2d. The NM and M surface morphology are rougher, described as a cauliflower pattern [19]. Thus, varying the particle size does influence the electroless nickel deposition morphology.

3.3. Coating composition

SEM coupled with EDX JOEL JSM 7800F was used to detect the elemental composition within the electroless without YSZ (EN) and Ni-YSZ coating for various particle sizes. The EDX spectrum of the EN, N, NM and M coatings are shown in Figure 3 and their elemental composition in wt.% is tabulated in Table 4.

Table 4.
Elemental composition by EDX

Element	EN, wt.%	Nano (N), wt.%	Mixed (NM), wt.%	Micro (M), wt.%
O K	7.29	7.73	13.75	19.63
P K	10.06	10.26	8.64	8.02
Ni K	82.65	72.59	61.32	58.86
Y L	0.00	0.00	1.57	0.86
Zr L	0.00	9.42	14.72	12.64

Coating as deposited (EN), i.e., without YSZ particle incorporation, shows 83 wt.% nickel and 0 wt.% yttria, as well as zirconium, indicating that there is no YSZ particle in the coating. The amount of YSZ in the electroless Ni-YSZ coating is 9.42 wt.%, 16.29 wt.%, and 13.50 wt.% of N, NM and M samples, respectively. It concludes that nanoparticles give the lowest incorporation of YSZ particle in the coating and the highest is the micro-sized. Another important element in electroless nickel coating is phosphorus. The electroless coating depositions have phosphorus range between 8-10 wt.%. This indicates that the electroless nickel coating is a medium phosphorus content and the deposits have a mixed crystalline and amorphous structure and good corrosion and abrasion resistance properties [8].

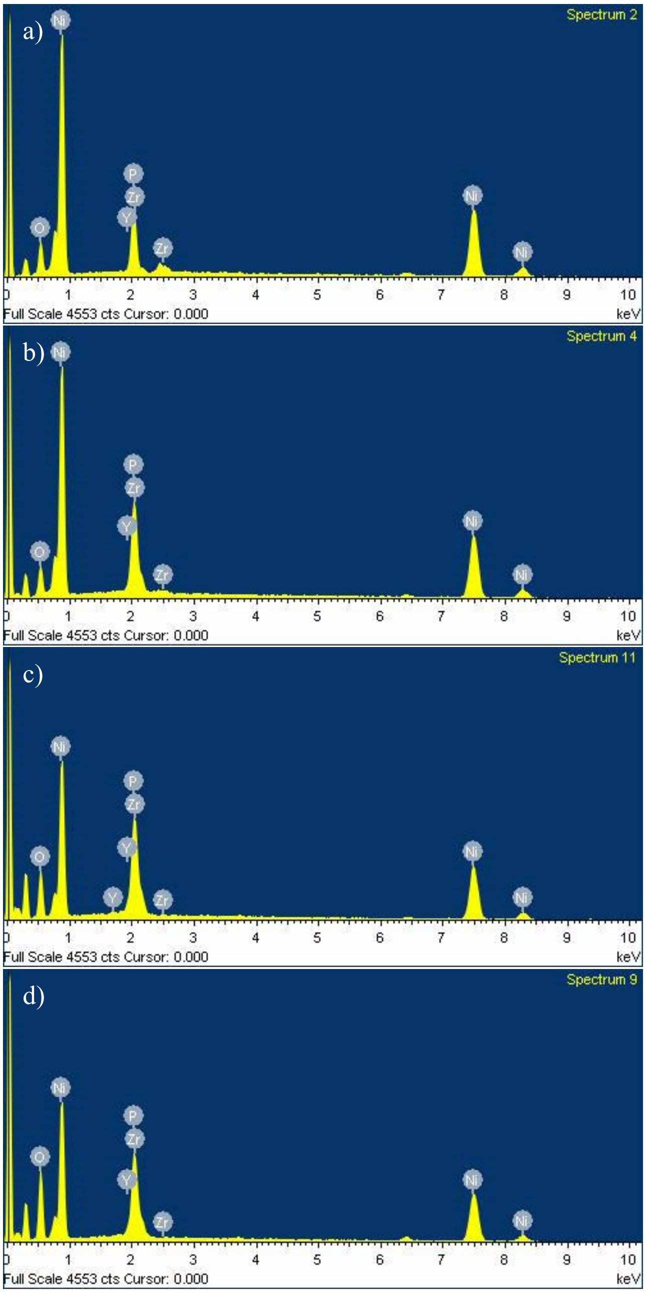

Fig. 3. EDX spectrum of electroless coatings for (a) EN (b) N (c) NM (d) M

3.4. Coating crystallography

Heat treatment was introduced to the coatings at a temperature 300-400°C for 1-2 hours. The crystallographic analysis of electroless Ni-YSZ coating (NM) samples at room temperature (rtp), 300°C, 350°C and 400°C for 1 hour, respectively are shown in Figure 4.

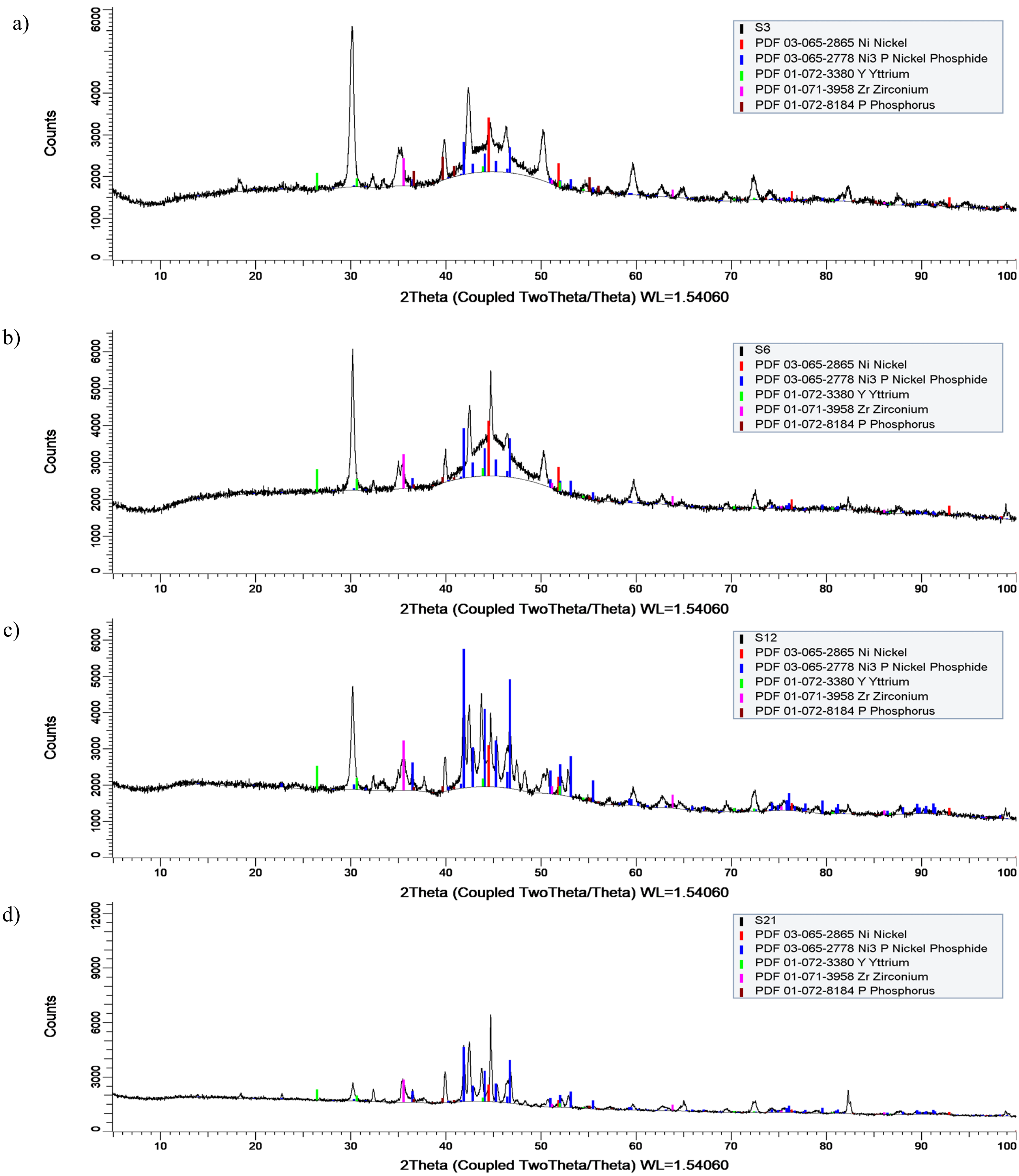

Fig. 4. XRD crystallographic analysis for NM samples at a) rtp, b) 300°C, c) 350°C, d) 400°C

In general, as the temperature increases, the nickel peaks (red) get lower, and the hard nickel phosphide peaks (blue) get higher and sharper. This indicates that the hard intermediate Ni_3P is present at the temperature increase, and it is more pronounced at temperature 350°C and above. The XRD spectrum of electroless Ni-YSZ coating at rtp and 300°C are a broader at 2Theta ranges 40-50° compared to the spectrum of 350°C and 400°C samples that are sharper peaks. These observations conclude that at rtp and 300°C, they have amorphous structure and the sharper peaks for 350°C and 400°C samples indicate crystalline structure. The precipitation of nickel phosphide (Ni_3P) is detected at 300°C, where the phase transformation begins [24]. At a higher temperature of 350°C, more Ni_3P is detected.

3.5. Coating surface roughness

The electroless Ni-YSZ coating is uniformly coated onto HSS substrate with an average thickness of 30 µm. The YSZ particle size of electroless Ni-YSZ coating is varied to investigate the effect on the surface roughness as the coating is heated between 300-400°C for 1-2 hours. The surface roughness measurements of electroless coatings were collected and compared to observe their behaviour as the YSZ particle sizes are varied at various heating temperature for 1 hour and 2 hours.

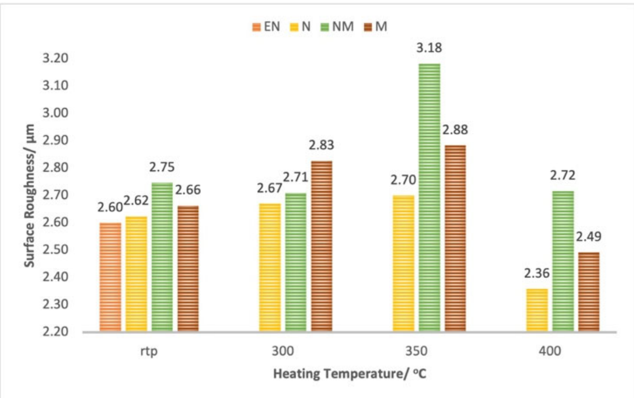

Fig. 5. Surface roughness against heating temperature at 1 hour

The comparison of surface roughness, Ra of electroless Ni-YSZ at various particle sizes against heating temperature for 1 hour is shown in Figure 5. The Ra at rtp for N, NM and M range between 2.60-2.75 µm. The lowest value of Ra for EN is 2.60 µm followed by N at 2.62 µm, and the highest is 2.75 µm for NM. EN and N samples have very small differences in Ra; which the SEM images verify this in Figure 2a and 2b, where they have smoother morphology compared to M and NM samples. This observation also supported by the EDX composition data of YSZ particles tabulated in Table 4 which N sample is the lowest and NM is the highest. Thus, the higher the YSZ particle content in the coating, the higher Ra value is obtained.

In general, most NM samples against heating temperature for 1 hour are the highest and N samples are the lowest. On the other hand, the electroless Ni-YSZ coating with N and M particle sizes is not very much affected by the temperature changes.

Figure 6 shows the graph of the surface roughness, Ra against temperature upon heating for 2 hours. Increasing the heating time from 1 to 2 hours has a small effect on the coating surface roughness. The observation is similar to the 1 hour which the highest Ra is obtained by NM samples and the lowest is the N samples at various temperatures.

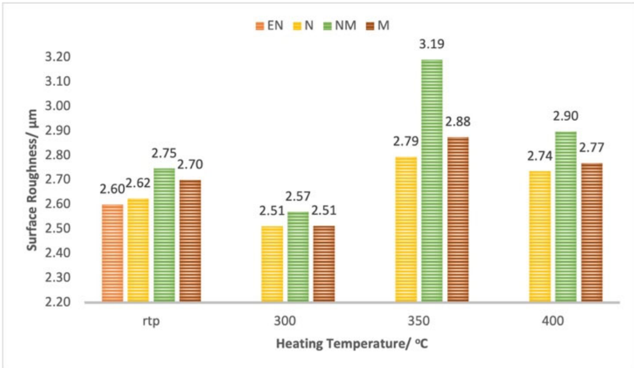

Fig. 6. Surface roughness against heating temperature at 2 hours

The effect of varying YSZ particle sizes together with heat treatment affected surface roughness, Ra, as shown in Table 5. The Ra values at 350°C are highest for all ranges of YSZ particle sizes due to the precipitation of intermediate hard Ni_3P crystalline as supported by the crystallography XRD spectrum in Figure 4. Heating the electroless Ni-P coating to 300-400°C initiates the formation of Ni_3P which increases the hardness and wear resistance of the coating [31]. According to Bae, the effect of surface roughness on the nano composition might cause particle concentration to be the most important determinant of the wear rate, followed by current density and temperature [26].

The SEM morphology for electroless Ni-YSZ coating at 300°C and 400°C are compared in Figure 7. The morphology of electroless Ni-YSZ (M) at 300°C is smoother, with uniformly distributed YSZ particles embedded in the nickel matrix. At 400°C, the morphology is rougher, with a chunk of Ni_3P crystalline phases in a few areas. This supports XRD analysis in which the Ni_3P crystalline phases precipitated at a higher temperature.

Table 5.
Effect of particle size time and temperature on surface roughness and coating structure after heating for 2 hours

Temperature, °C	Ra, µm			Coating structure
	N	NM	M	
rtp	2.62	2.75	2.70	Amorphous
300	2.51	2.57	2.51	Amorphous
350	2.79	3.19	2.88	Crystalline
400	2.74	2.90	2.77	Crystalline

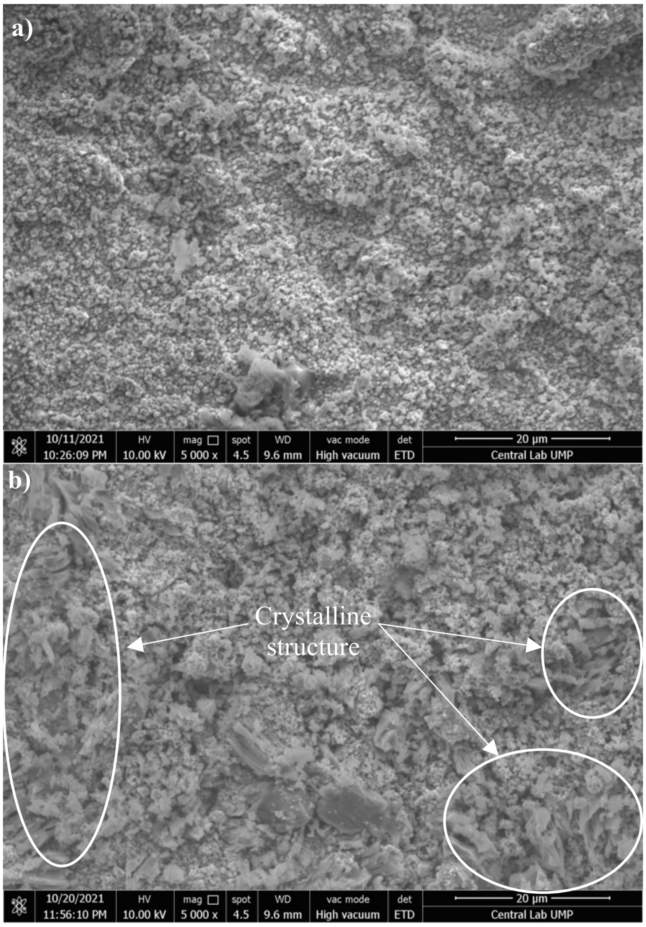

Fig. 7. Comparison of SEM morphology for electroless Ni-YSZ coating (M) heated for 2 hours at a) 300°C, b) 400°C

4. Conclusions

Electroless Ni-YSZ coating is a composite coating deposited onto an HSS substrate. The coating thickness measurements after coating were taken and averaged, giving 30 µm. It was found that nano-sized particle has the lowest incorporation of YSZ particle in the coating. The amount of YSZ in the electroless Ni-YSZ coating is 9.5 wt.%, 16.3 wt.%, and 13.5 wt.% of particle size nano, mixed and micro, respectively.

The surface roughness at rtp is varied by varying YSZ particle sizes ranging between 2.60-2.75 µm. The lowest surface roughness without YSZ particle at 2.60 µm followed by nano at 2.62 µm and the highest is 2.75 µm for mixed sizes.

The coating morphology by SEM images of EN and Ni-YSZ N is smoother compared to the Ni-YSZ NM and M YSZ. This clarifies the observation of the surface roughness with micro-sized YSZ particles.

The EDS composition analysis shows the YSZ content in the electroless Ni-YSZ coating for N samples are the lowest, whereas NM samples are the highest. This resulted in the surface roughness behaviour where the mixed size YSZ particle gives the highest roughness at all temperatures. The XRD analysis shows that heating temperatures above 300°C caused the precipitation of Ni_3P crystalline.

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Additional information

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