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Effect of heat treatment on microhardness of electroless Ni-YSZ cermet coating

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ABSTRACT

Purpose: The paper discussed the effect of heat treatment on electroless nickel-yttria-stabilised zirconia (Ni-YSZ) cermet coating. Ni-YSZ cermet coating has potential applications such as cutting tools, thermal barriers, solid oxide fuel anode, and various others. The compatibility of ceramic YSZ and metallic nickel in terms of the mechanical properties such as hardness by varying the heating temperature, time and ceramic particle size is highlighted.

Design/methodology/approach: Ni-YSZ cermet coating was deposited onto a high-speed steel substrate using the electroless nickel co-deposition method. The temperature and time were varied in a range of 300-400°C and 1-2 hours, respectively. The microhardness measurements were carried out using a Vickers microhardness tester (Shimadzu) according to ISO 6507-4. The surface characterisation of the cermet coating was carried out 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.

Findings: It was found that the microhardness of Ni-YSZ cermet coating with the ratio of 70:30, respectively, is directly proportional to the heating temperature and time. Heating the Ni-YSZ cermet coating at 300°C from room temperature (rtp) to 1 hour shows a 12% microhardness increment, while from 1 to 2 hours gives a 19% increment. Compared to heating at 350°C and 400°C, the increment is more significant at 33% and 49% for rtp to 1 hour and 8% and 16% for 1 to 2 hours, respectively. In addition, the effect of varying YSZ particle size in the Ni-YSZ cermet gave response differently for heating temperature and heating time.

Research limitations/implications: The paper is only limited to the discussion of the heat treatment effect on Ni-YSZ cermet coating hardness property. The tribological effect will be in future work.

Practical implications: The microhardness data may vary due to the Vickers microhardness force applied and the amount of ceramic particle incorporation and phosphorus content in the nickel matrix.

Originality/value: The value of this work is the compatibility of the ceramic YSZ and metallic nickel matrix in terms of mechanical properties, such as hardness, upon heat treatment.

Keywords: Ni-YSZ, Cermet, Coating, Microhardness, Heating temperature, Heating time, Electroless nickel

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PROPERTIES

1. Introduction

Cermet materials have been state-of-the-art composite materials due to the combination of metals and ceramics. In general, cermet materials have been applied in various industrial applications due to the combined effect of the high operating temperature of metals and high wear and corrosion resistance of ceramics giving to extremely high hardness [1]. The cermet is produced by incorporating ceramic particles in the metal matrix. The particles can be micro- or nano-sized, which will give different effects to the cermet.

There are various ways to produce cermet materials, such as thermal spraying, cold spraying, electrodeposition, or electroless deposition. Thermal spraying is the most common technique used as it gives a very thin and dense coating layer known as a hard coating [2]. However, high temperature causes recrystallisation, evaporation and phase transformation [3]. Common thermal sprayings are physical vapour deposition (PVD) [4], chemical vapour deposition (CVD) [5] and high-velocity oxygen fuel (HVOF) [6,7]. As an alternative, the application of cold spraying was found to give low porosity and surface roughness to the coating [8].

On the other hand, the electrodeposited cermet coating technique using applied current in the electrolytic bath has effects on the current density, electrolyte composition, pH, bath agitation on the physicochemical and mechanical properties of the deposits [9,10], and heat treatment improves the hardness and wear resistance of the cermet [11]. The electroless deposition could be a metal matrix of either aluminium, nickel, copper, silver or iron. The most common electroless technique is electroless nickel deposited or co-deposited since nickel has attractive properties as a superalloy. It can be improved by incorporating carbide [12] or other ceramics.

Electroless nickel deposited gives excellent corrosion, wear and abrasion resistance as well as good ductility, lubricity and electrical conductivity [13]. The deposits usually contained 2-14% of phosphorus, and it does vary the structure of the deposit from amorphous to crystalline. The incorporation of ceramic particles into the Ni-P deposit produces a cermet. The most common ceramic particles incorporated are silicon carbide [14], diamond [15], and cubic boron nitride [16]. The influence of electroless coating parameters significantly affects the deposit quality, and the incorporation of particle quantity can be enhanced by using smaller size particles, stirring agitation and blasting surface treatment [17]. It was also found that different types of complexing agents in the electroless nickel bath also affect the deposit properties [18].

Extensive study has been done to improve the properties of electroless nickel or its cermet by giving heat treatment. It was found that the microhardness of electroless nickel-phosphorus deposit decreases as the amount of phosphorus increases due to the structure change from crystalline to amorphous [19], and a similar finding was found in electroless nickel-titanium nanocomposite coating [20]. In another study, heating the electroless nickel between 300-400°C improves the phase transformation of the deposit from amorphous to crystalline [21].

The amount of nickel composition in electroless nickel-activated carbon was found to increase as the treating time increased from 1 to 2 hours [22]. The hardness of the electroless nickel boron nitride increased by 40% after being heat-treated at 300°C. However, it reduces wear rates [23]. The Ni-P deposit formed under heat treatment between 400-700°C and water-quenched shows an increase in hardness as well as surface adhesion [24]. The microhardness and wear of electroless nickel-phosphorus increased as the temperature increased from 300 to 600°C. However, any temperature above 600°C for 4 hours shows no effect on both due to the formation of Ni_3P crystalline [25].

In this paper, the cermet coating of Ni-YSZ is heated to 300-400°C for 1-2 hours to investigate its effect on the coating's microhardness. The purpose of the cermet coating is to be used as an alternative cutting tool's coating.

2. Materials and methodology

2.1. Substrate

The substrate for the electroless Ni-YSZ cermet coating used is 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 the dimension illustrated in Figure 1 with a thickness of 1.25 mm.

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

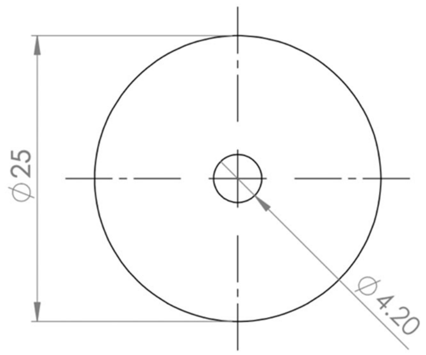

Fig. 1. Illustration of HSS substrate and its dimension

2.2. Reinforcement particle

Reinforcement ceramic particles of yttria-stabilised zirconia (YSZ) with 8 mol% by Tosoh Japan were used. There were two different sizes of 8YSZ used; (1) nano-sized range between 100-500 nm and (2) micro-sized of nominal 2 µm. The optimum particle loading is in the range of 5-10 g/L [14]. Therefore, the particle loading for both sizes used was 10 g/L and the mixed particle size is by a ratio of 1:1.

2.3. Electroless nickel coating

Electroless nickel solutions were prepared with AR grade chemicals and high purity ionised water as described in previous research [26]. First, the HSS substrate was pre-treated in 4 different chemicals, namely cuprolite, pre-catalyst, catalyst and niplast, for 15 minutes each at different temperatures, as illustrated in Figure 2. Then the substrate was placed in Slotonip electroless nickel solution together with 8% YSZ powders for the co-deposition process to occur.

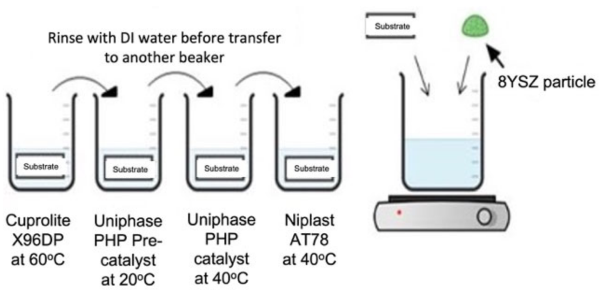

Fig. 2. Illustration of the electroless Ni-YSZ coating process

2.4. 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 1-2 hours, respectively, as conducted by previous studies [19,20,23]. The microhardness measurements were carried out using a Vickers microhardness tester (Shimadzu) under micro-hardness range; HV0.025 (25 gf) at a 0.245 N force for 10 s according to ISO 6507-4. There are a total of 27 samples, and five measurements of microhardness are taken for each sample. The cermet coating surface characterisation was evaluated using JOEL Scanning Electron Microscope (SEM) coupled with Energy Dispersive X-ray (EDX) JSM 7800F. In addition, the crystallographic structure of materials was analysed by X-ray diffraction (XRD) Bruker D8 Advance instrument.

3. Results and discussion

3.1. Ni-YSZ cermet coating

Ni-YSZ cermet coating is deposited onto the HSS substrate, and the elemental composition by EDS is given in Table 2. The composition of metallic nickel to micro-sized 8YSZ ceramic is in a ratio of 70:30 by weight percent obtained by EDS. The average phosphorus content for electroless nickel hypophosphate solution is between 5-11 wt.%, and the detected phosphorus element in the cermet is 7.57 wt.%.

Table 2.
Chemical composition of Ni-YSZ cermet coating by EDS

Element	Composition, wt.%
Ni	67.76
O	5.08
P	7.57
Y	2.16
Zr	17.43

The microstructure of the Ni-YSZ cermet coating in Figure 3 shows SEM images of YSZ ceramic particles (a) nano-sized and (b) micro-sized which are uniformly distributed in the metallic nickel matrix. The white dots are the YSZ ceramic embedded in the grey area of the metallic nickel matrix. The average thickness of all Ni-YSZ cermet coatings is approximately 30 µm by taking the average thickness of the coating specimens using digital Mitutoyo micrometre.

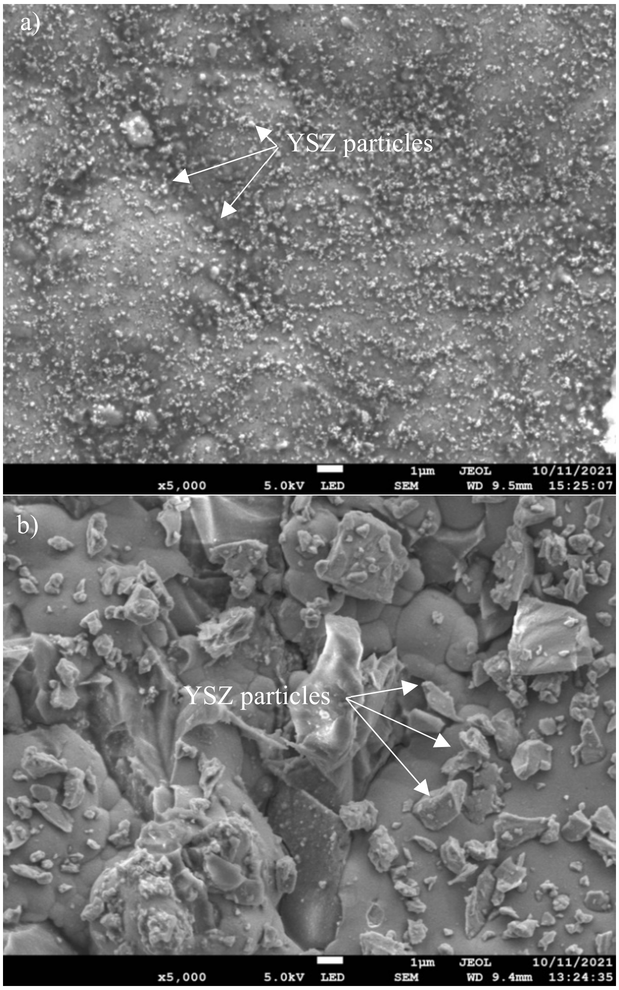

Fig. 3. Microstructure of Ni-YSZ cermet coating for (a) nano-sized (b) micro-sized YSZ particles

3.2. Effect of heating temperature and time

Metallic pure nickel, in general, is very strong and ductile with good corrosion and wear resistance, whereas ceramic yttria-stabilised zirconia is very hard and chemically inert with a high thermal expansion coefficient and ionic conductivity. The combination of these two materials in a cermet gives outstanding properties, especially in terms of its hardenability, wear and corrosion resistance. The properties of in-situ co-deposition of metallic nickel and YSZ particles onto the HSS substrate can be enhanced by applying heat treatment.

The microhardness of Ni-YSZ cermet is found to be directly proportional to the heating temperature and heating time, as shown in Figure 4. For example, heating the Ni-YSZ cermet coating at 300°C from room temperature (rtp) to 1 hour shows a 12% microhardness increment, while heating from 1 to 2 hours gives a 19% increment. Compared to heating at 350°C and 400°C, the increment is more significant at 33% and 49% for rtp or 1 hour, and 8% and 16% for 1 to 2 hours respectively. This indicates that the heating temperature of 350°C to 400°C is optimum as the microhardness increment significantly increases. However, prolonged heating from 1 hour to 2 hours, slightly reduced the microhardness increment for both temperatures.

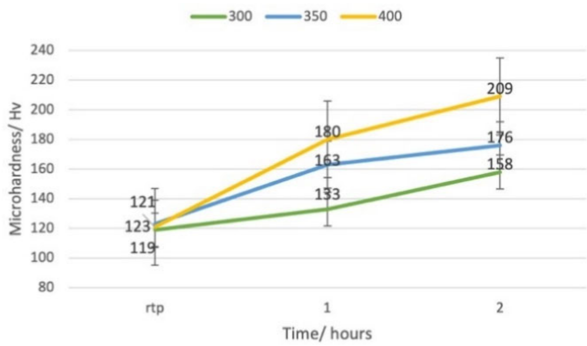

Fig. 4. Effect of heating time and temperature on Ni-YSZ cermet microhardness

This finding can be explained by the phase transformation within the nickel matrix in the presence of phosphorus as described by Kumar et al. [3] which, in this study, is approximately seven wt.% phosphorus content. At a temperature as low as 300°C, the Ni-P structure is amorphous. Thus, it gives low hardness. Figure 5a shows the XRD analysis for Ni-YSZ cermet coating at various temperatures. The peaks of Ni-YSZ cermet can be compared to the standard Ni-peaks and Ni_3P peaks in Figure 5b and Figure 5c, respectively. The EN as-deposited, Ni-YSZ as-deposited and Ni-YSZ heated at 300°C show similar XRD patterns except that the Ni-YSZ coatings show a pronounced peak at 30°, indicating the presence of yttrium element. The XRD spectrum for these coatings is broader compared to the Ni-YSZ at 350°C and 400°C, indicating they are amorphous. The spectrum peaks of Ni-YSZ coatings at 350°C and 400°C are sharp and high, indicating the presence of hard intermediate Ni_3P crystalline phase has precipitated. Prolonged heating up to 2 hours caused Ni_3P grain coarsening [13], that slightly softened the cermet coating.

Thus, the gradient increment of the microhardness showed decrement. Overall, the effect of heating temperature and time on the Ni-YSZ cermet coating was influenced mainly by the properties of the nickel matrix. This is due to the composition of the Ni-P matrix, which is higher than the YSZ ceramic at a ratio of 70:30.

It is also found that Ni-YSZ cermet coating has good compatibility as heating it up to 400ºC for 2 hours had not shown any surface crack or delamination, although there is a difference in coefficient of thermal expansion of these two materials at 16.5 x 10^{-6} K^{-1} and 10.7 x 10^{-6} K^{-1} respectively.

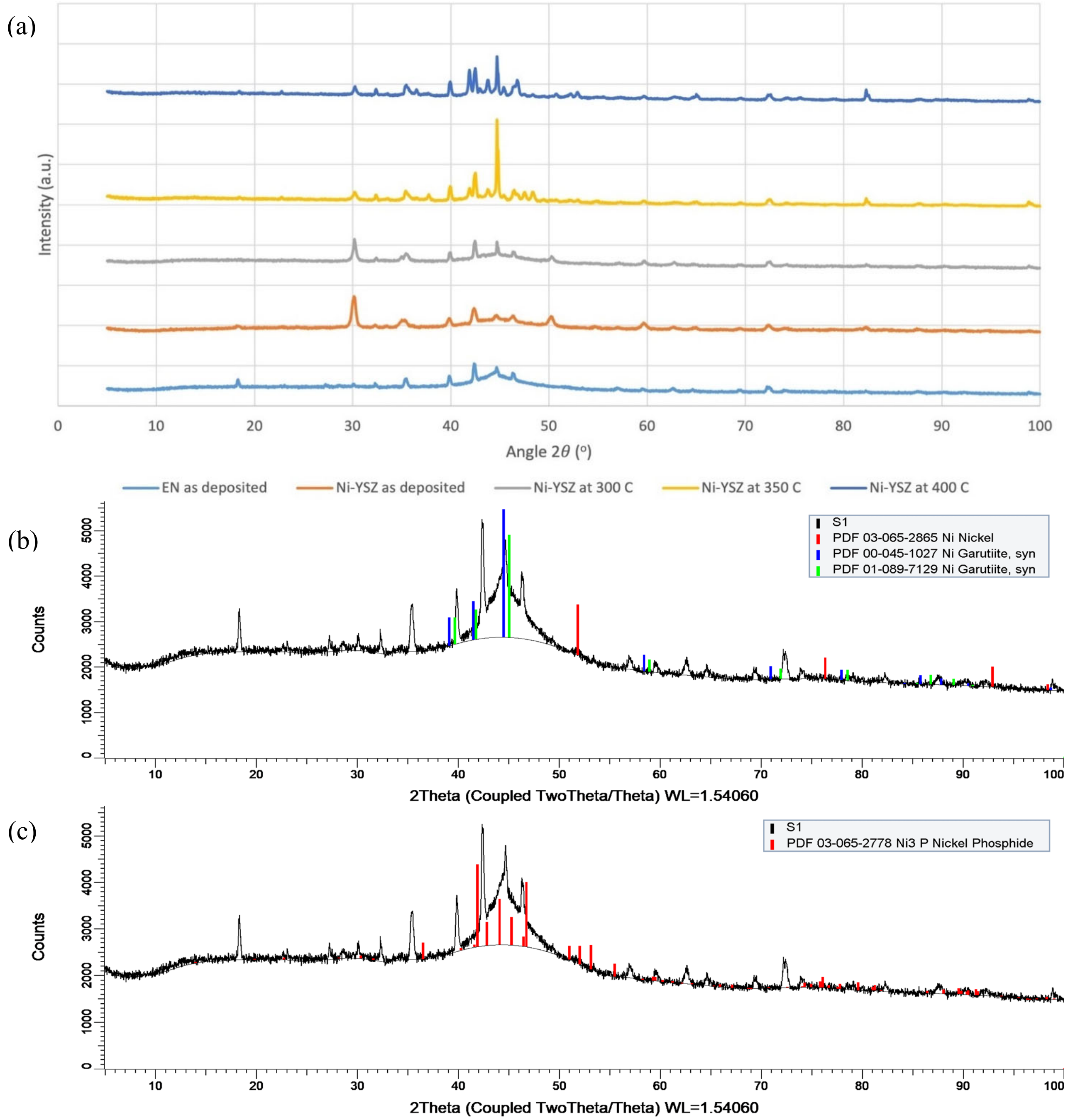

Fig. 5. XRD analysis for (a) electroless Ni and electroless Ni-YSZ coating (b) standard Ni peaks, (c) standard Ni_3P peaks

3.3. Effect of particle size

The YSZ ceramic particles embedded in the nickel matrix of Ni-YSZ cermet coating deposited onto the HSS substrate are varied in terms of their sizes. They are batched into nano (N), micro (M) and mixed if nano and micro (NM) by a ratio of 1:1.

The effect of particle size on Ni-YSZ cermet coating's microhardness for heating temperature between 300-400°C with a heating time of 1 hour is shown in Figure 6. Micro-sized YSZ particle is found to be dominant at all temperature ranges. On the other hand, the nano-sized particles show the lowest microhardness at all temperature ranges. Microhardness increments between nano and micro batches are 17%, 4% and 5% for 300°C, 350°C and 400°C, respectively. The microhardness increment for 350°C and 400°C is not as great as at 300°C.

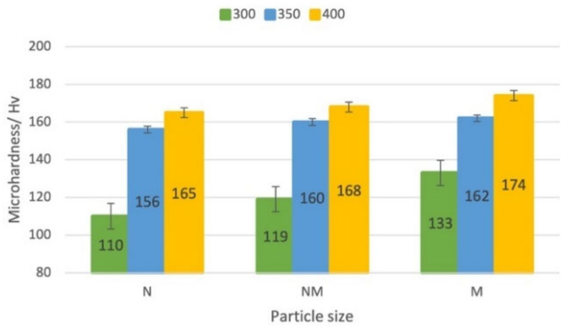

Fig. 6. Effect of particle size at variable heating temperature on cermet microhardness

The micro-sized particles have less surface area for the metallic nickel deposition to be attached or covered compared to the nano-sized ones. The nano-sized particles are too fine to be covered in the nickel matrix, which causes lower particle mass gain, as studied by Khosroshahi [27]. Similar findings were also reported by Baba, and Balaraju, where the larger sized particle gives optimum incorporation and uniform distribution compared to the smaller one regardless of the type of ceramic particle used [17,28]. The particle size has a small influence on the microhardness of the cermet coating [28].

The Ni-YSZ cermet of micro-sized YSZ particle samples heated at 300°C and 400°C for 2 hours are shown in Figures 7a and 7b, respectively. Both SEM images show that the YSZ particles (white colour) are uniformly distributed in the metallic nickel (grey colour), improving the overall surface morphology and coefficient of thermal expansion which reduces delamination and cracking. In addition, Ni-YSZ cermet coating heated at 400°C shows a coarser structure than the one heated at 300°C.

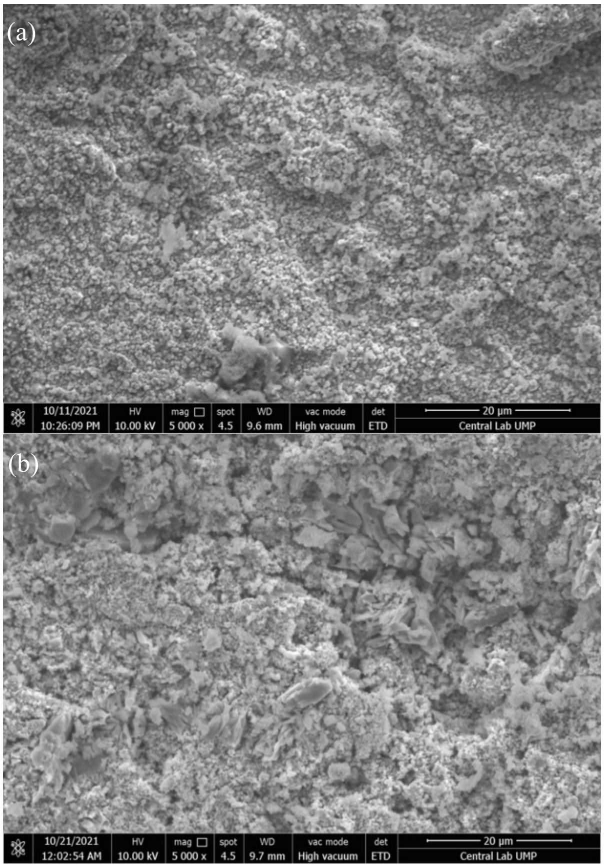

Fig. 7. SEM images of micro-sized YSZ particle heated at (a) 300°C (b) 400°C for 2 hours

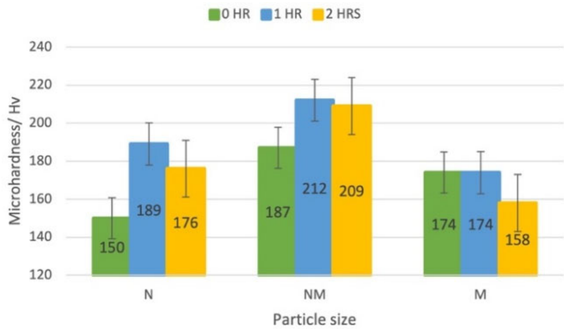

Fig. 8. Effect of particle size on variable heating time on cermet microhardness

The effect of particle size on Ni-YSZ cermet coating microhardness for the heating time between 1-2 hours at a constant temperature of 350°C is shown in Fig. 8. It is found that mixed nano and micro-sized particles showed a dominant influence. Heating time for 2 hours shows low microhardness that might be due to prolonged heating which caused coarsening of the grain size [13].

4. Conclusions

Ni-YSZ cermet coating with 70 to 30 wt.% ratio with an average of 7 wt.% phosphorus content has been deposited onto the HSS substrate via electroless co-deposition with an average thickness of 30 µm. As shown in the SEM images, the cermet has a uniform distribution of YSZ particles within the metallic nickel matrix.

Heating the Ni-YSZ cermet coating at 300ºC from rtp to 1 hour shows a 12% microhardness increment, while from 1 to 2 hours gives a 19% increment. Compared to heating at 350ºC and 400ºC, the increment is more significant at 33% and 49% for rtp or 1 hour and 8% and 16% for 1 to 2 hours, respectively.

The microhardness of Ni-YSZ cermet coating is not much influenced by incorporating the ceramic YSZ as the proportion of the incorporation of the ceramic particles is only 30 wt.%. Hence, the microhardness behaviour of the cermet is represented by the behaviour of the Ni-P, which at temperatures lower than 300ºC, the cermet structure is amorphous. However, at higher temperatures, up to 400ºC, the phase transformation is occurred to produce a hard intermediate Ni_3P crystal.

The larger particle size has a greater influence on microhardness compared to the smaller particle size. The mixed particle size strongly influences the microhardness against heating time.

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