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Effect of rare earth on microstructure and wear behaviour of Ni based microwave clad

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In the present investigation microwave energy has been utilized to develop wear resistant Ni-based clad without Nd_2O_3 (unmodified coating) and with addition of Nd_2O_3 (modified coatings) in varying wt. % of 1-3. The clads have been developed using domestic microwave oven at frequency of 2.45 GHz and power 900 W for duration of 360 s. The unmodified and modified clads have been characterized in terms of microstructure, XRD, microhardness and two body abrasive wear. The average thickness of developed clads has been measured as 1.0 mm approximately. A two body abrasive wear behaviour of unmodified and modified coatings at sliding speed of 36 m/min & 72 m/min with abrasive grit size of 220 & 600 has been investigated. The results revealed a better abrasive wear resistance at lower sliding speed and fine grit size. Further, the best results of microstructure, formation of hard phases, improved microhardness and wear resistance have been shown by the samples having coating composition as 3 wt. % of Nd_2O_3.

Keywords: Rare earth, Cladding, Coating, Ni alloy, Neodymium oxide, Microwave, Microstructure, Micro-hardness

1 Introduction

Rare earth elements (REE) consist of lanthanum, cerium, praseodymium, neodymium, promethium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium and lutetium [1]. Efforts have been made by many researchers to study the effect of rare earth elements in several applications of surface engineering [2-7]. Several authors [2-3] reported that the optimum addition of rare earths like CeO_2 in Ni based alloy refined microstructure, improve hardness which revealed excellent resistance to corrosive wear and abrasive wear. It was also found that an optimum addition of rare earth elements in metal alloy will improve its mechanical and metallurgical properties of steels. Wang *et al.*[7] investigate the effect of rare earth La_2O_3 addition in various ratios (wt. % = 0.5, 1.0, 2.0, 4.0, 6.0) in Fe based powder particles and compared the results and reported that addition of La_2O_3 refines the microstructure, decreases the dendrite arm spacing, purifies the grain boundaries and improves the coefficient of friction of laser clad coatings. Prasad *et al.*[8] investigated the effect of La_2O_3 addition in Ni based coating powder and reported refined microstructure, enhanced microhardness and wear properties of the developed coatings. Wang *et al.*[9,10] and Zhang *et al.* [11] studied the microstructure and wear properties with addition rare earth oxide CeO_2 and La_2O_3 and successfully decreased the friction coefficient of coating and wear resistance of the coating enhanced significantly, improved microhardness and refine microstructure. Quan Xu *et al.*[12] reported reduction in friction coefficient and wear resistance increases with addition of 4 wt% Y_2O_3 in Ni-based alloy coatings on 6061 aluminium alloy developed by laser cladding. With increase of Y_2O_3 addition, the corrosion rate of deposited metal decreased and micro-hardness increased accordingly. Rahman *et al.*[13] reported that the CeO_2 addition in coating powder improved oxidation resistance. Chen *et al.*[14] studied the effect of Nd_2O_3 (varying % from 0 to 8) additive on microstructure and tribological properties of plasma-sprayed $NiCr-Cr_2O_3$ composite coatings. The results showed that Nd_2O_3 could refine microstructure of $NiCr-Cr_2O_3$ composite coating and make Cr_2O_3 distribution more uniform in the coating, which leads to the increase of average microhardness and reported improved wear resistance and lowest porosity. However still there is no literature published to investigate the application of rare earth Nd_2O_3 addition in hard coatings developed by microwave heating. Cladding developed by microwave heating

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Table 1 — Chemical composition of substrate and Ni based powder.

Element	C	Si	Fe	Cr	Ni	Mn	B	Mo	Cu
Substrate Wt. (%)	0.28-0.4	0.2-0.8	Bal.	1.4-2	-	1.29	-	0.22	0.03
Powder (1045 Ni based) Wt. (%)	0.35	3.7	2.6	8.9	Bal.	-	1.8	-	-

methods possess many advantages such as metallurgical bonding between coating and substrate, volumetric and uniform heating, less dilution of clad with substrate, reduce thermal gradient and refined microstructure of coating.

In present investigation P20 tool steel has been selected as substrate material. P20 tool steel is most commonly used by many automotive, electrical and electronic industries for making moulds / dies for production of various plastic components. The plastic parts are manufactured through injection molding process in which a melted reinforced polymer is forced through a nozzle into a mold, where its solidifies and cools. Finally, cooled plastic part is ejected and cycle is repeated. Now a day in competitive environment most of the industries are replacing their metal parts into fiber-reinforced plastic due to its less weight and save cost. Generally, polymer reinforced with abrasive glass fibre, which are abrasive in nature and thus deteriorate the core/cavity surface of the mold which affects the quality of the produced components [15-17]. To overcome this problem, hard coatings on the core/cavity surfaces of mold have to be provided. These coatings are generally developed by different processes such as PVD, CVD and HVOF etc. Thus in view of these facts, in the present investigation an attempt has been to made develop a clad of Ni-based cladding with (RE modified coating) and without (unmodified coating) addition of Nd_2O_3 on P20 tool steel through microwave hybrid heating method and study the effects of Nd_2O_3 addition on microstructure, hardness and abrasive wear behaviour of developed clad.

2 Materials and Methods

2.1 Substrate and Coating Powder

Tool steel (P20) was used as a substrate material for coating deposition. The substrate having dimension of 8mm x 8mm x 6mm (L x B x H) were used for coating development. Before coating the substrates were grind against 220 abrasive grit size so as to get artificial texture. Then the substrates were dipped for 5 minutes and cleaned with acetone before clad/ coating development.

Table 2 — Chemical composition of various coating developed.

Cladding materials	Coating designations
1045 + 0 wt. %Nd_2O_3	Unmodified
1045 + 1.0 wt. % Nd_2O_3	Modified 1% Nd_2O_3
1045 + 2.0 wt. % Nd_2O_3	Modified 2% Nd_2O_3
1045 + 3.0 wt. % Nd_2O_3	Modified 3% Nd_2O_3

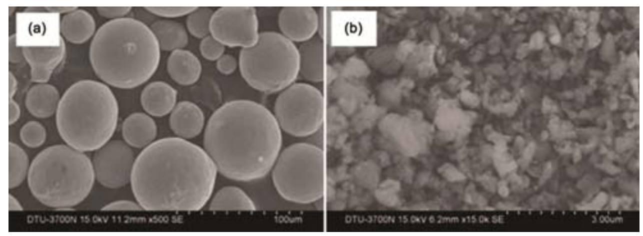

Fig. 1 — Morphology of raw powder used for deposition (a) Ni based alloy and (b) RE Nd_2O_3.

The compositions of substrate material and Ni-based clad powder (1045 Höganäs) supplied by M/s Höganäs India Pvt. Ltd. is shown in the Table 1. The nickel based alloy is most commonly used in industrial applications due to its excellent corrosion and wear resistant properties. In present investigation Ni-based powder was further modified with the addition of Nd_2O_3 compound in varying wt. % (1, 2 & 3 wt. %) to study its effects on microstructure and wear behaviour of clad coatings. The chemical composition and designation of clad/ coatings are illustrated in Table 2. The rare earth oxide Nd_2O_3 was supplied by M/s Central Drug House (p) ltd Delhi, India. The typical morphology of raw Ni based and Nd_2O_3 powder are shown in Fig.1. It was clearly seen that spherical shaped Ni-based powder and irregular polygonal shaped Nd_2O_3 compound having particle sizes ranges from 20-106 µm and 2-10 µm respectively was used for development of clad coatings.

2.2 Development of Unmodified and RE Modified Microwave Coatings

In present study clad coatings with all compositions were developed using domestic microwave oven for 360 s at 900 W by microwave hybrid heating method as shown in Fig. 2. The unmodified (Ni-based) and rare earth modified (Nd_2O_3) composite powders in varying wt. % (1, 2 & 3 wt. %) were pre-heated at 100 °C for 24 hours to remove moisture in powder. The coating powder was manually preplaced on the substrate which was accommodated in the graphite mold. The height of mold was 1 mm more than the height of the substrate to ensure uniform thickness of the coating powder. As the coating powder and substrate was not able to interact directly with microwave at room temperature. To overcome this problem microwave hybrid heating technique was used for development of all coatings with suitable suscepter particles [18-22]. Charcoal/ suscepter powder was used initially to couple with microwave at room temperature. The graphite sheet was placed between the coating and charcoal powder, so that carbon from charcoal can't diffuse with coating powder during microwave heating. When microwave starts initially many molecules of charcoal starts absorbing microwave having positive charge at one end and negative charge at the other end, and therefore, rotates as they try to align them self with the alternative electric field [23-24]. Thus, this to and fro motion exceeds 2.45 billion times a second. Because of this fast motion heat is generated due to friction between the molecules. This heat is then transferred to the coating powder through conduction and radiation mode of heat transfer. Once, Ni-powder reaches to elevated temperature it's directly start absorbing microwaves [23-24]. One important advantage of this process is that when the metallic powder reaches to melting stage, it start behave like opaque body, by this way the dilution of coating material with substrate is very less [25].

2.3 Characterization of Unmodified and RE Modified Microwave Coatings

The developed coatings/ clads were sectioned transversely at the centre, polished and washed properly with acetone prior to proceeding for investigations. The samples for microstructure were prepared using standard metallograhy procedure. The microstructures of the cladding were analysed by using Hitachi S-3700N scanning electron microscope. Abrasive wear analysis of worn out samples of all coatings were analyzed using SEM micrographs. The XRD pattern using Cu Kα radiations of all coatings was obtained to identify various phases. The scan rate was 1.5°/ minute and scan range from 30° to 100°. The average microhardness of transverse section of clad coatings was evaluated at a load of 50 g for 30 s using vicker's microhardness tester.

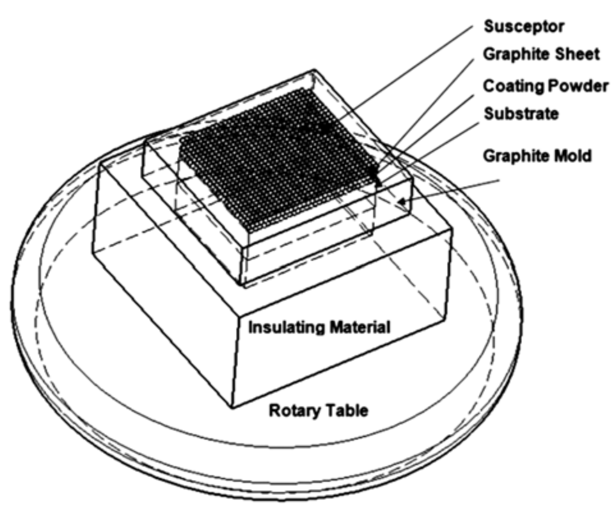

Fig. 2 — Schematic representation of microwave hybrid heating process for development of coatings.

2.4 Abrasive Wear Characterization

DUCOM wear testing machine was used to study abrasive wear of unmodified and modified microwave clads. Wear pin of size 8 x 8 x 6 mm^3 was clamped in clamping vice and held against the abrasive medium. The abrasive wear study of unmodified and modified coating was divided into four cases depending upon the combination of abrasive grit sizes (220 & 600) and sliding speeds (36 & 72 m/min.) as described in Table 3. To attain constant rotating speed, the disc was allowed to run idle for 2 min afterward load of 10 N was applied. The average of abrasive wear weight loss was calculated on the basis of two samples of each coating composition. The test samples were cleaned in acetone before and after testing. To measure the weight loss an electronic micro-balance was used with an accuracy of 0.0001 g.

3 Results and Discussion

3.1 Microstructure Analysis of Unmodified and RE Modified Microwave Coatings

SEM micrographs (1000X magnification) of unmodified and RE modified cladding are shown in

Table 3 — Details of abrasive wear test.

	Parameter	Descriptions
1.	Test setup	Pin on disc. (Ducom)
2.	Counter disc	Material: MS Dia. 120mm
3.	Wear pin	Substrate size 8x8x6 mm
4.	Abrasive Paper (grit size)	
a.	Case A & Case B	220
b.	Case C & Case D	600
5.	Sliding Speed (m/min.):	
a.	Case A & Case C	36
b.	Case C & Case D	72
6.	Normal load (kg)	1.0
7.	Track Dia. (mm)	55
8.	Temperature	Room temperature

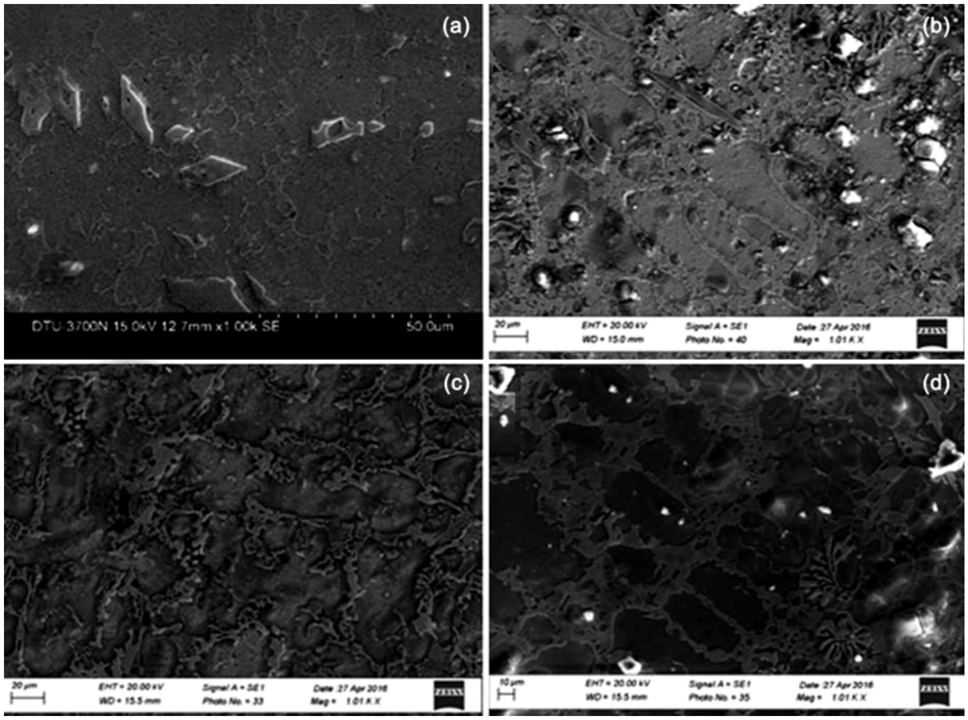

Fig. 3 — Microstructure of (a) Unmodified, (b) 1wt.% Nd_2O_3 modified, (c) 2 wt.% Nd_2O_3 modified and (d) 3 wt.% Nd_2O_3 modified coatings.

Fig. 3 (a, b, c, & d) respectively. The average thickness of cross section of unmodified and RE modified clad coating was measured in the range of 0.95 to 1.10 mm with the help of SEM micrographs shown in Fig. 4. Microstructure of RE modified clads observed dense microstructure and low porosity. RE modified coating are more homogeneous in chemical composition distribution and refined microstructure than Ni based (unmodified) coatings. In RE modified coating Nd_2O_3 expedite the formation of crystal nuclei, which restrict the formation of large grains thus the results in refined microstructure [26]. It was further noticed that morphology of modified coatings became refined than unmodified (Fig. 3a) coating with increase in wt. % of rare earth oxide Nd_2O_3 from 1- 3wt.% (Fig. 3 (b-d)). The microstructure of the coating is more refined at 3 wt.% Nd_2O_3. Rare earth elements also improve wettability of two materials which tends to form new phases due to its high chemical activity [4]. The coating with 1wt.% Nd_2O_3 leads the formation of cracks which is visible in Fig. 3b. It is clear from Fig. 3d that the microstructure of the coating with 3wt.% Nd_2O_3 observed skeleton structure reinforced with hard phases such as NdC_2, Nd_2NiB_{10}, Cr_3Si, $NdFeSi_2$ and Cr_2B. Moreover coating with 3wt.% Nd_2O_3, an intermetallic

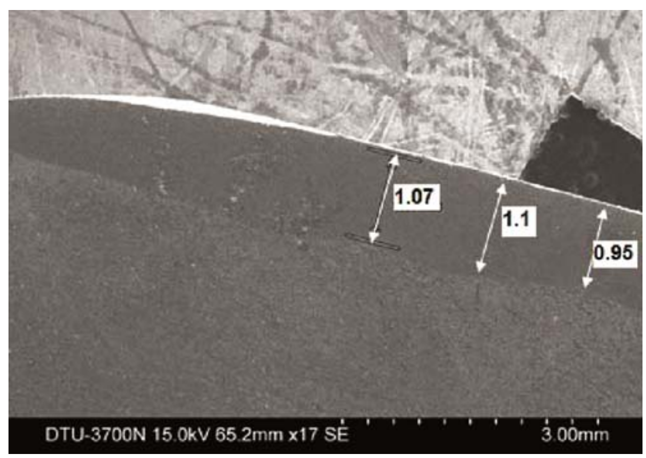

Fig. 4 — SEM micrograph of cross section of clad.

compound Cr_3Si was present which has higher elastic modulus and hardness, due to the generation of chromium alloyed with silicon related with unalloyed chromium [18]. The elemental analysis of unmodified coating confirm the presence of C, Si, Fe, Cr, B, and Ni shown in Fig. 5a and for modified coatings with 1, 2 & 3 wt. % of Nd_2O_3 confirms the presence of various elements of Ni based alloy and neodymium represented in Fig. 5 (b-d). Further it was noticed from elemental analysis that all the claddings shows good metallurgical bonding with substrate through

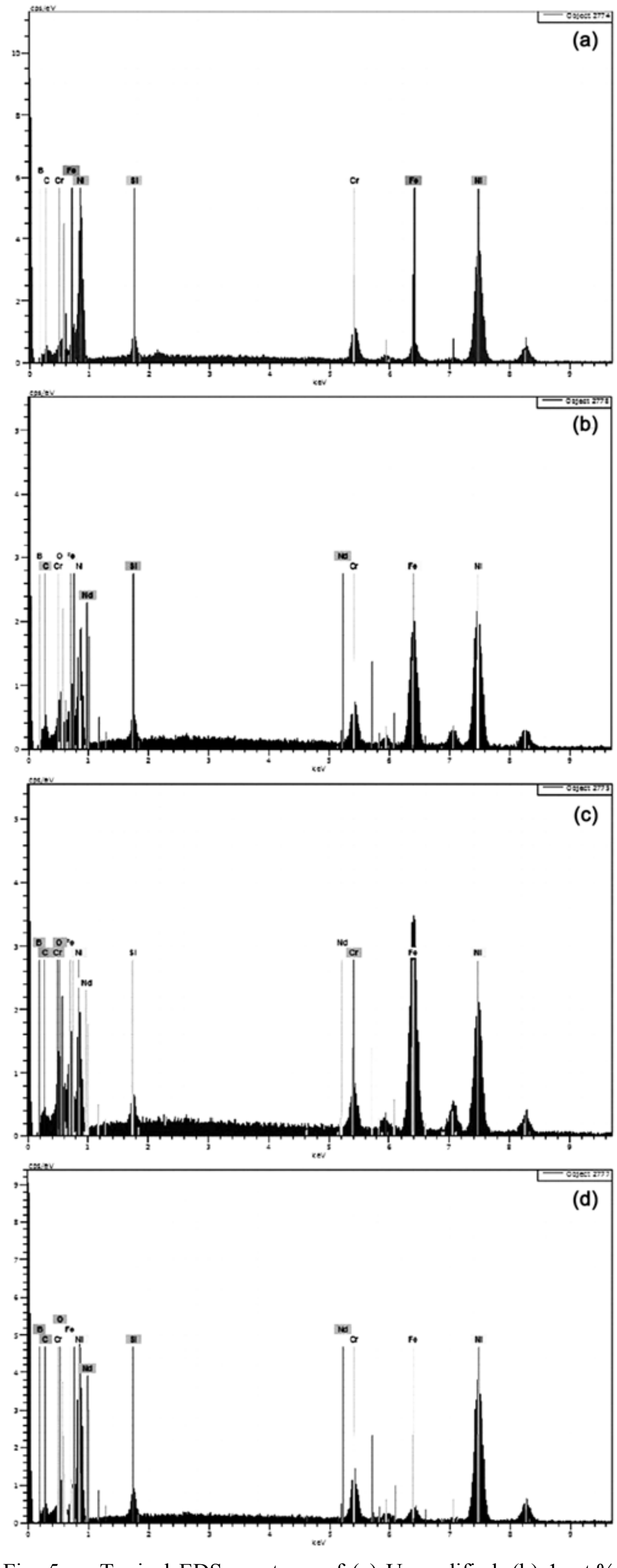

Fig. 5 — Typical EDS spectrum of (a) Unmodified, (b) 1 wt.% Nd_2O_3 modified, (c) 2 wt.% Nd_2O_3 modified and (d) 3 wt.% Nd_2O_3 modified coatings.

partial mutual diffusion of elements like iron from the substrate to clad. The distribution of all elements such as C, Fe, Si, Cr, B, Ni, and Nd, present in all four coatings were detected uniformly distributed by comparing the elemental maps of unmodified (Ni-based) and Nd_2O_3 modified coatings with (1, 2 & 3 wt. %) were presented in Fig. 6.

3.2 XRD Analysis

The various phases such as Fe_5C_2, Ni_3Si and Ni_6Si_2B were identified by XRD analysis of unmodified coating. The XRD pattern of the coating with 3% Nd_2O_3 and various phases such as Ni_3Si, Fe_5C_2, Ni_4B_3, NdC_2, Nd_2NiB_{10}, Cr_3Si, $NdFeSi_2$ and Cr_2B are shown in Fig. 7. The Nd_2O_3 addition can be confirmed by the presence of NdC_2, Nd_2NiB_{10} and $NdFeSi_2$.

3.3 Microhardness Analysis

Microhardness was carried out to evaluate the effect of increasing content of neodymium oxide compound on hardness. The average of 12 readings at three sections per sample was studied. The average microhardness value of unmodified coatings was 721 ± 21 and RE modified coatings at 1, 2 & 3 wt. % of Nd_2O_3 was evaluated to be 806 ± 29, 858 ± 26 and 993 ± 14 HV. It was noticed that average microhardness value increases with increase in weight fraction of Nd_2O_3 [14]. The microhardness at 1wt.% Nd_2O_3 and 2 wt.% Nd_2O_3 modified claddings was found to be 10% and 15% higher than unmodified coating. Similarly, at 3wt.% Nd_2O_3 the microhardness was improved by 27 % as compared to unmodified cladding as shown in Fig. 8. The higher hardness of 3% Nd_2O_3 is due to the existence of refined microstructure (Fig. 3d) and presence of hard phases such as Ni_4B_3, NdC_2, Nd_2NiB_{10}, Cr_3Si, $NdFeSi_2$ and Cr_2B respectively as compared to unmodified coatings.

3.4 Abrasive Wear Analysis

Abrasive wear weight loss of all coatings at different parameters are shown in Fig. 9 and various parameters/cases for abrasive wear analysis of unmodified and RE modified coatings are described in Table 3. It was observed that wear resistance of RE modified coatings increased with increase in wt. % of rare earth oxide Nd_2O_3 in Ni-based alloy as compared with unmodified coating. It was analysed that weight loss of unmodified coatings during abrasive wear test were more in all four coatings in all cases A, B, C and D. Further it was noticed that abrasive wear weight loss of all coatings increases with increase in sliding speed from 36 to 72 m/min (Case A, C (36) & Case

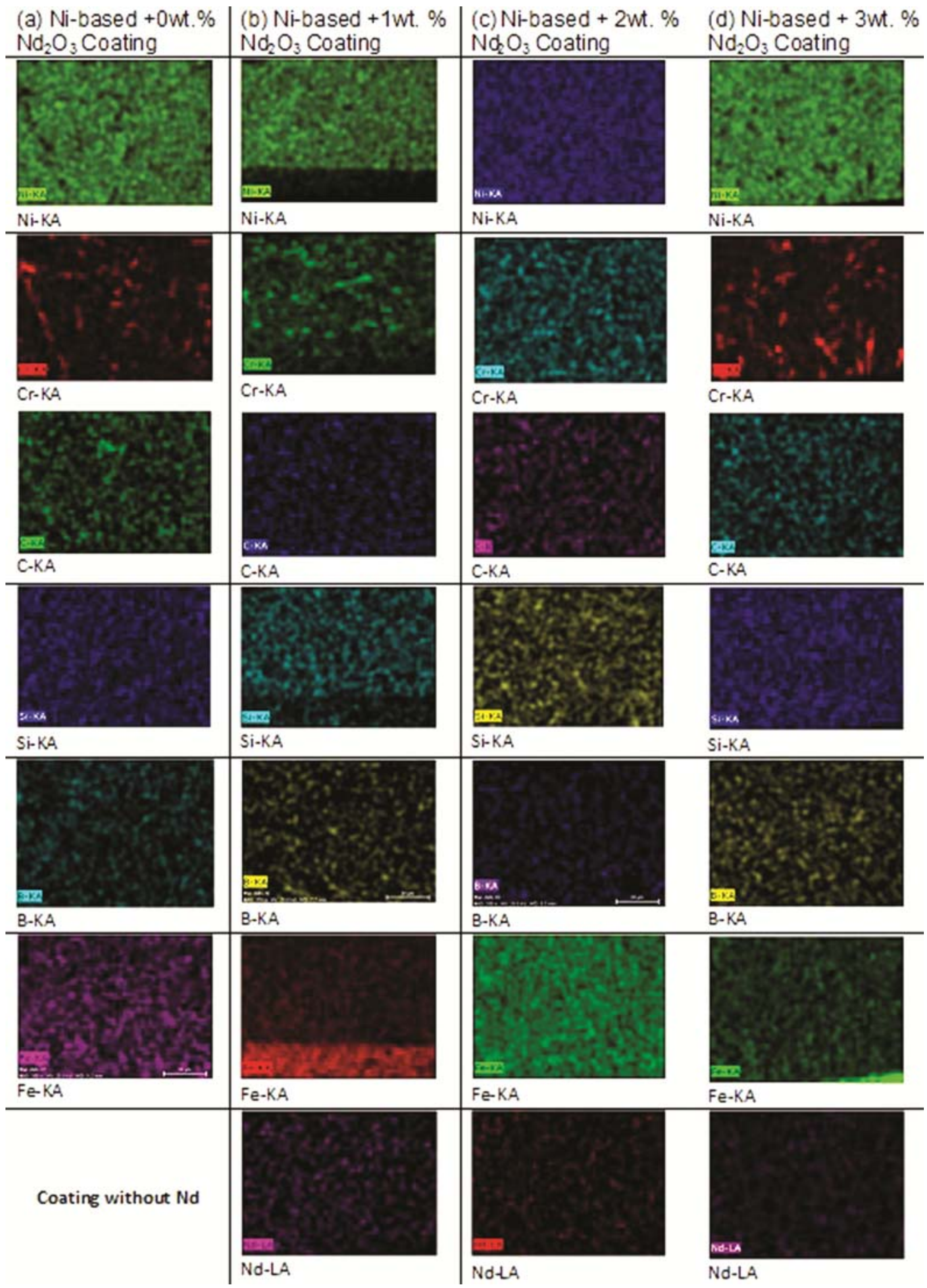

Fig. 6 — Comparison distribution of various elements of (a) Unmodified (1st column), (b) 1 wt.% Nd_2O_3 modified (2nd column), (c) 2 wt.%Nd_2O_3 modified (3rd column) and (d) 3 wt.% Nd_2O_3 modified (4th column) coatings.

B, D (72)) using 220 or 600 grit size abrasive. Similarly abrasive wear weight loss of all coatings decreases with increase in grit size from 220 to 600 grit size (Case A, B (220) & Case C, D (600)) [2-5,14].

To study the abrasive wear behaviour of unmodified and rare earth modified coatings for various cases (Case A-D), SEM micrographs of the worn out surfaces of unmodified and rare earth modified coatings with 1wt.% Nd_2O_3, 2wt.% Nd_2O_3 and 3wt.% Nd_2O_3 are shown in Fig. 10 were analyzed. The worn surfaces of unmodified coatings (Fig.10a (i), (ii), (iii) & (iv)) shows wider wear tracks due to

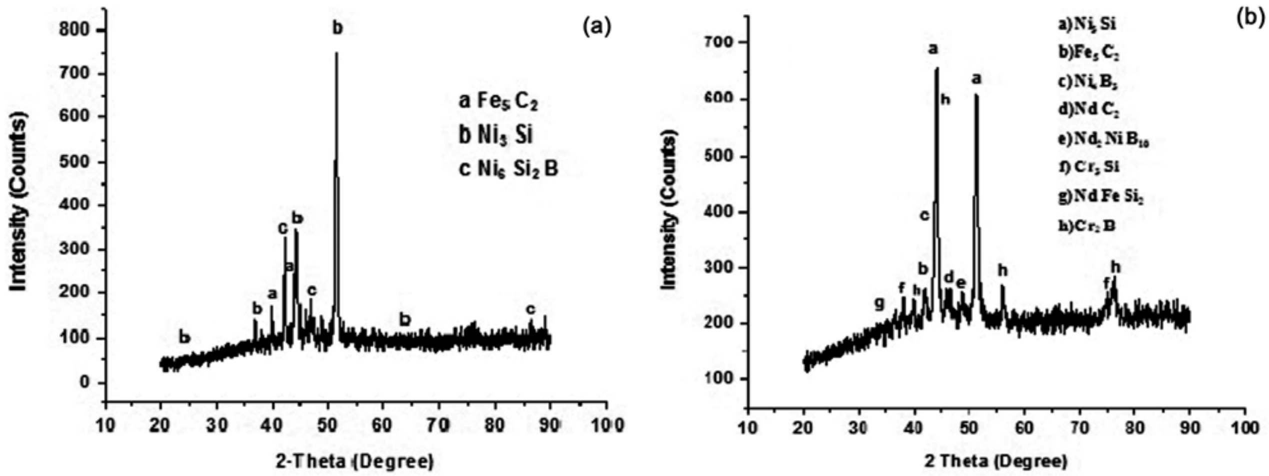

Fig. 7 — XRD pattern of (a) Unmodified and (b) 3wt. % Nd_2O_3 modified coatings.

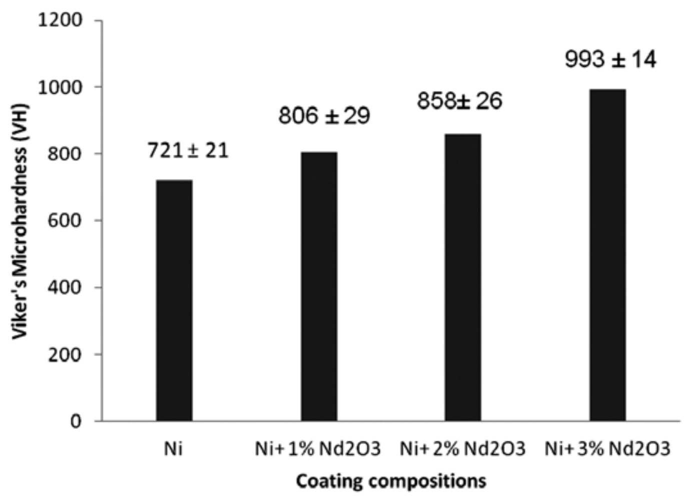

Fig. 8 — Average Vickers microhardness of 1045 (Unmodified) and Nd_2O_3 modified coatings.

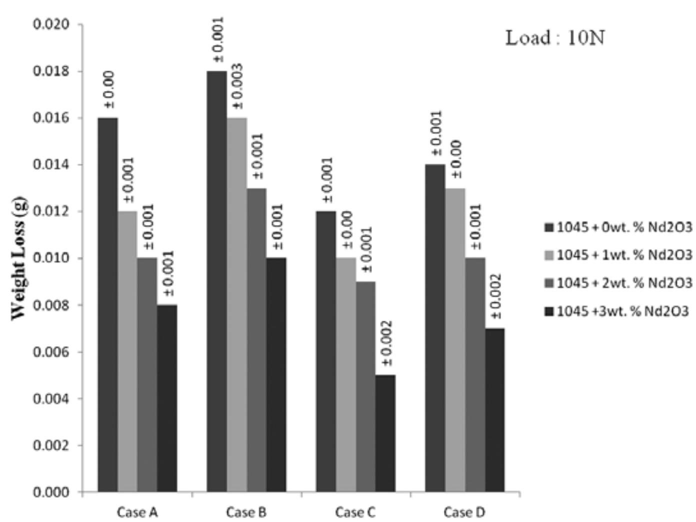

Fig. 9 — Abrasive wear weight loss of 1045 (Unmodified) and Nd_2O_3 modified coatings (Case A – 220 grit size and 36 m/min sliding speed, Case B – 220 grit size and 72 m/min sliding speed, Case C – 600 grit size and 36 m/min sliding speed, Case D – 600 grit size and 72 m/min sliding speed).

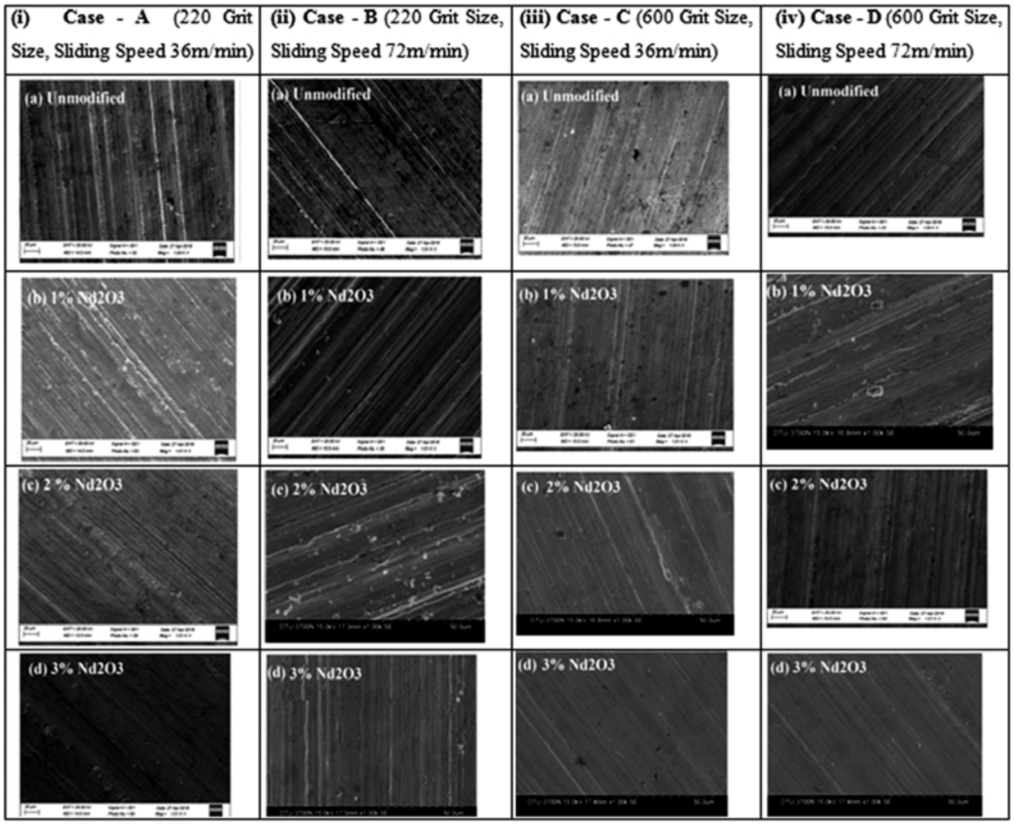

Fig. 10 — SEM Images of abrasive worn out surface of (a) 1045 + 0wt. % Nd_2O_3 (Unmodified) (b) 1045 + 1wt. % Nd_2O_3, (c) 1045 + 2wt. % Nd_2O_3 and (d) 1045 +3wt. % Nd_2O_3 coatings as mentioned column-wise.

cutting, craters and ploughing mechanisms due to less hardness as compared to RE modified coatings (Fig.10b,c,d (i), (ii), (iii) & (iv)). It was also noticed from Fig. 10 that abrasive wear grooves are wider, at higher sliding speed (Case B &D) and reduced grit size (Case A & B).

Further, overall comparison of SEM images confirm that at 3 wt.% Nd_2O_3 (Fig.10 d (i), (ii), (iii) & (iv)) coating results in lower abrasive wear due to high hardness and refined microstructure[14] as compared to other coatings (unmodified Fig.10 a (i), (ii), (iii) & (iv), 1 wt.% Nd_2O_3 (Fig.10 b (i), (ii),(iii) & (iv) and 2 wt.% Nd_2O_3 (Fig.10 c (i), (ii),(iii)&(iv)).

4 Conclusions

The following conclusions can be drawn from this work:

(i) In this investigation, unmodified and modified coatings on P20 tool steel substrate were successfully developed in 360s using Samsung domestic microwave oven of 900W power and 2.45GHz frequency.

(ii) It was noticed that the coating with increase in wt.% of rare earth Nd_2O_3 exhibit refined microstructure, higher microhardness and increased abrasive wear resistance.

(iii) The coating with addition of 3wt.% of Nd_2O_3 was effectively refined microstructure and improved 27% higher hardness due to the presence of hard phases such as Ni_4B_3, NdC_2, Nd_2NiB_{10}, Cr_3Si, $NdFeSi_2$ and Cr_2B respectively as compared to Ni-based coating.

(iv) Cutting, ploughing and crater were the main wear mechanisms in these coatings. Further, it was also found that weight loss of coatings during test was higher when coarse grit size and higher sliding speed was applied.

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