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Article Type:	Full Length Article
Keywords:	Nano-composite coating, self-lubrication, Nano-indentation, Nano-wear.
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Nano-mechanical and nano-tribological characterisation of Ni-Co-BN nano-composite coating for bearing applications.

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Abstract

This paper presents the development of novel nano-composite coating of Nickel-Cobalt-Boron Nitride (Ni-Co-BN) on an Aluminium-Silicon (Al-Si) substrate using the physical vapor deposition (PVD) technique. The multi-layered nano-composite coating was fabricated from nickel, cobalt, and boron nitride targets, in a radio frequency magnetron sputtering system, at 250^{o}C with a thickness of 500 nm. The composition and morphology of Ni-Co-BN coatings were investigated using field emission scanning electron microscopy (FESEM), energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD). Mechanical studies on Ni-Co–BN coatings were performed at low loads varying from 500 to 1250µN to investigate the impact of load on reduced modulus and hardness. Besides this, nano-wear studies were carried out with loads varying from 0.5N to 1N to investigate the distortion and cracking efficiency of the coating. The results show that the reduced modulus and hardness of the Ni-Co-BN coating decrease as the load increases. The wear rate of the Ni-Co–BN coating increases with the rise in load from 4.08 x 10^{-5} to 1.608 x 10^{-4}mm^{3}/m. The Ni-Co–BN coating displayed smooth wear scars on the sample surface with no fractures or debris, suggesting that the coated material flowed in a plastic way near the wear scar. The behavior of the coating suggests its strong suitability for bearing applications.

KEYWORDS: Nano-composite coating, self-lubrication, Nano-indentation, Nano-wear.

INTRODUCTION

Aluminum alloys are receiving a lot of research observation these days. The main factors that limit the use of aluminum and its alloys, in industry, are poor wear resistance and inferior hardness. This is despite the fact that aluminum alloys have high strength-to-weight ratios. Nevertheless, there is a rising need for mechanical parts to eliminate the uses of harmful lubricants in many food and textile industries [1]. Aerts et al. have suggested the use of self-lubricating to tackle the problems of severe wear [2]. Furthermore, it was revealed by Banday et al. that tribological properties can be enhanced by using self-lubricating multi-layered nano-coatings [3]. Takaya et al. studied the wear characteristics of an anodic oxide coating of aluminum, imbued with an iodine component, which has a low COF and functions as a solid lubricant, under extreme conditions/hazardous environments [4]. However, to solve the problems caused by wear and friction, machine parts are coated with solid lubricant coatings as standard lubricants do not deliver the appropriate degree of performance. Furthermore, solid lubricants are free from contaminants and can be used on machine components where liquid lubricants cannot be used, due to accessibility issues [5]. Because of environmental concerns, the use of common lubricants in industrial applications has decreased, while the use of coatings has increased significantly [6]. Various types of solid lubricants used are PTFE, waxes, graphite, boron nitride, and tungsten disulfide, etc, [7].

Boron nitride (BN) is primarily utilized as a solid lubricant for applications requiring low friction, because of the weak inter-atomic interaction between their multi-layer structures. BN possesses a lamellar shape with strong bonding between the atoms. However, the weak inter-atomic connection between the layers leads to poor strength. The lamellar shape of BN, and the ineffective Van der Waals force among each layers are responsible for the low friction [8]. Researchers have also found that the inclusion of various metals to ceramics leads to the formation of BN/metal multi-layer coatings (Co, Ni, Au, TiN, or TiB_2 or mixed metals). The addition of boron nitride to various metals (Li et. al.) results in increased hardness and reduced wear coefficient [9]. To improve the corrosion performance compared with pure BN, another alternative is to use an element belonging to the Fe group, such as Ni, Fe, Co, Sn [10-15]. Felipe et al. observed that self-lubricating Ni-P coatings have better wear and low friction than uncoated ones [16]. Subramanian et al. observed that the inclusion of BN provided better corrosion resistance. It has also been discovered that various deposition methods, such as physical vapor deposition technique, low pressure, atomic layer deposition, magnetron sputtering, plasma chemical vapor deposition, pulsed laser technique, and thermal spraying, can be used to deposit solid lubricants on a substrate (PLD) [17]. Buranawong et al. deposited nano-crystalline $AlTi_3N$

coating, by using the magnetron sputtering process, and found that surface roughness and thickness, of the coatings, increased with the rise in titanium current and the deposition period [18]. Furthermore, Subramanian et al. observed that the co-deposition improves the various properties of coatings, however, the inclusion of any self-lubricating material into the metals matrix can drastically increase the matrix's hardness and wear resistance [19]. Amico and d'Oliveira studied Aluminium coatings, coated on the specimen of varying roughness, utilizing high-velocity, electric arc deposition, and other deposition procedures. The findings revealed that the roughness of coatings placed on warmed substrates is lower as compared to substrates placed at room temperature [20]. The wear and corrosion, characteristics of Ni-Co coating may be greatly increased by a composite coating made by incorporating new particles into it [21-24]. The wear and corrosion properties of the coating were created by the addition of BN to Ni. Ni-Co alloy coatings are extensively employed in automobiles as well as aerospace industries, and other disciplines, owing to their superior physical, chemical, and mechanical qualities [25-29].

Researchers have investigated the tribological characteristics of Ni-Co coating and found that the addition of Ni percentage decreased the friction coefficient of the paired parts, however, coating wear rises as Ni concentration increases in the coating [30]. The single layer of BN, cannot provide the desired degree of performance in many engineering applications. However, it has been discovered that layers with diverse mechanical qualities, such as BN/MoS2 improve coating features such as wear resistance, hardness as well as chemical inertness. The tribological behavior of multi-layer coatings was investigated by Ma et al [31]. The lubricating layer on hard TiN coatings was found to reduce friction and develop low stress at the substrate/coating contact [32]. The review of the result of the literature reveals that adding metal layers improves properties like hardness, and wear resistance as well as load-bearing capacity. Furthermore, no literature has yet described the deposition of the self-lubricating coating of Ni-Co-BN on an Al-Si substrate.

In this study, a 500 nm multi-layer nano-composite coating was applied on an Al-Si alloy using a magnetron sputtering system. Nano-indentation was done to find out the effect of mechanical characteristics on the coating-system substrate. Tribological investigations were performed at different loads to evaluate the wear and load-withstanding ability of the coating. The objective of the research is to look at how Ni-Co and BN synergistically affect the mechanical as well as tribological characteristics of the Ni-Co-BN coating applied to an Al-Si alloy.

EXPERIMENTAL PROCEDURES

2.1 Coating parameters

Prior to the process of the deposition, all the specimens were polished at constant load on an automatic polishing machine using wet emery papers of grit size 800, 1000,1200, 2000, and 2500, sequentially. Following this, the surface was again polished using diamond paste with particle sizes of 0.5μm and 0.25μm on velvet cloth till the mirror surface was attained. Following the polishing procedure, ultrasonic cleaning was done followed by acetone for five minutes to remove impurities, and then it was dried in an oven at 50°C for five minutes. A 3D profilometer was used to evaluate the surface roughness of the samples. The 3D surface topographical image and the surface roughness of samples as represented in Fig. 1(a) and (b), respectively., and the surface roughness, R_a of the samples was measured to be 15 nm.

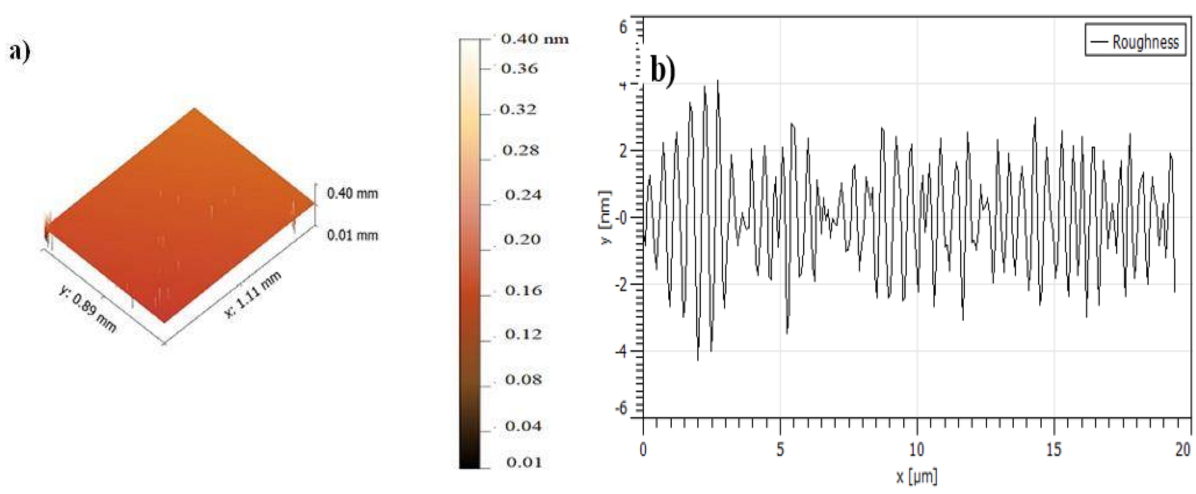


Fig. 1 a) 3d Surface topographical image of Ni-Co-BN coating and b) Surface roughness of Ni-Co-BN surface.

2.2 Coating design

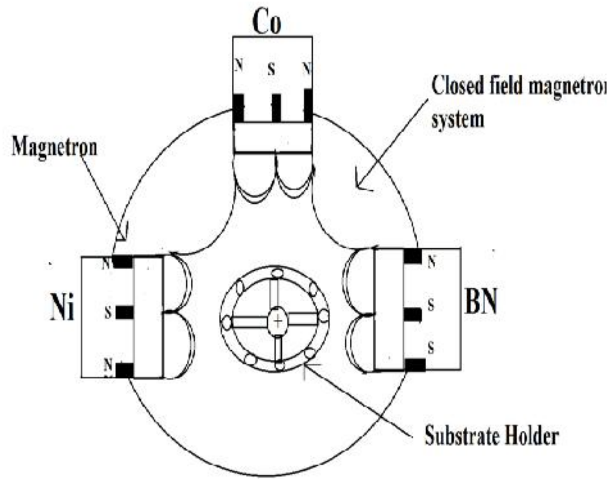


Fig.2 Schematic Diagram of Magnetron sputtering system

The coating was deposited using a PVD magnetron sputtering process. The sputtering process was carried out using mini lab 060 by Moorfield Nano technology UK. Before the coating deposition, the substrate was pre-etched by argon for 10 minutes at 45-watt RF power. Discs of BN (99.99 % purity), Co (99.99 % purity), and Ni (99.99 % Purity), each having a 2-inch diameter, were used as sputtering targets. Low pressure of $4x10^5$ pa was maintained in the chamber using tribo-molecular pump and only argon was allowed to enter the chamber during the deposition. The details of the coating deposition are illustrated in Table 1.

Table 1: shows the various coating parameters

Characteristics	**Results**
Deposition temperature	Room Temperature
Pressure	20×10^{-3} mbar
Power	Ni 0.5 W (DC), Co 70 W(RF1),and BN 70 W(RF2).
Air flow	20.7sccm
Deposition rate	Ni 0.12(A/s), Co 0.08(A/s) and for BN 0.12 (A/s)
Target to substrate distance	16cm (approx.)
Deposition time	2 hours
Coating thickness	500 nm

`

2.3 Coating Thickness

The thickness of coating was measured using a field emission scanning electron microscope (FESEM) ZEISS Gemini SEM 500. The coating layer of Ni-Co-BN is clearly shown from the cross-section figure. The Ni-Co-BN coating had a final thickness of 500 nm.

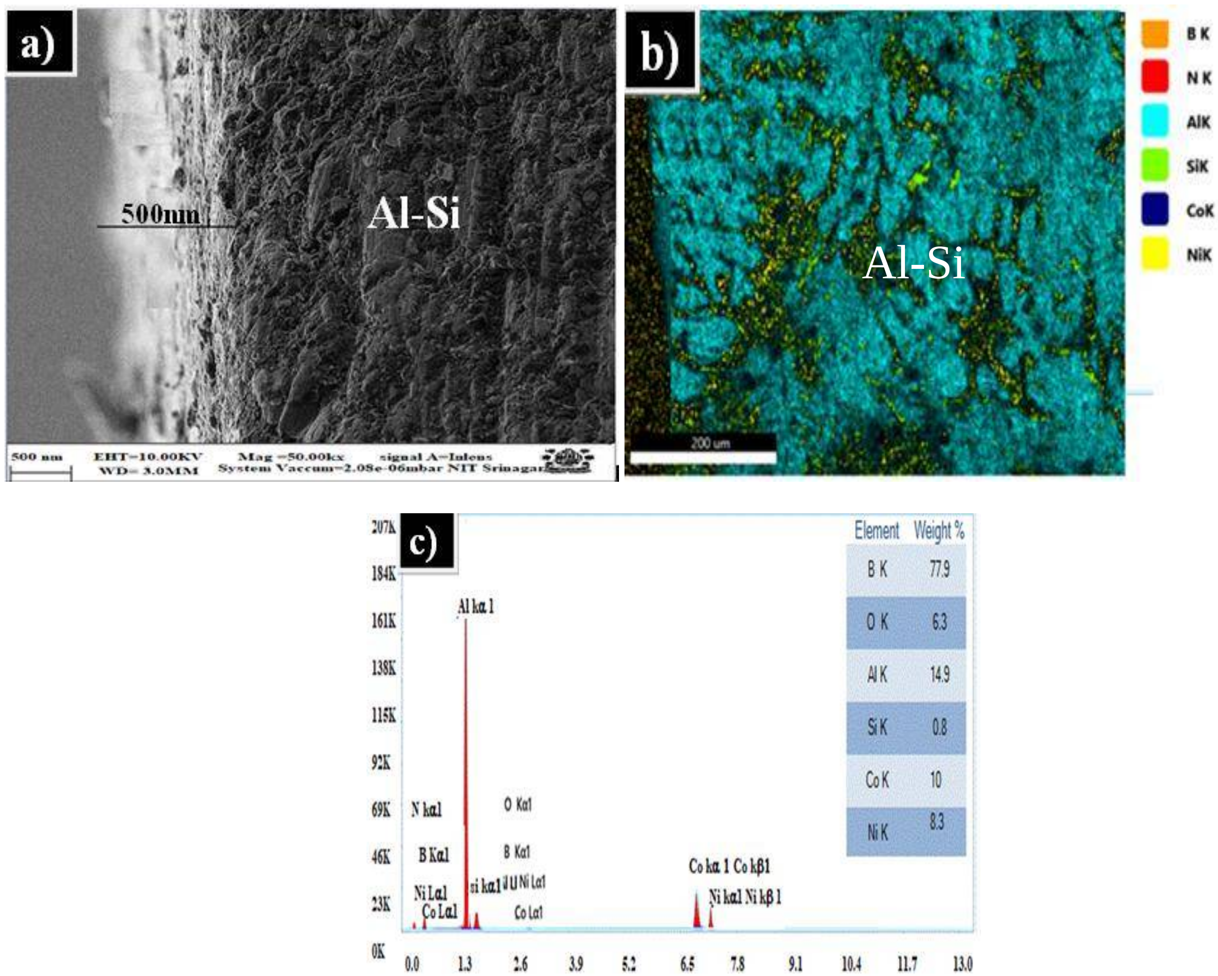


Fig. 3 Cross-sectional images a) FESEM showing thickness b, c) cross-sectional EDS images showing the elemental composition of the coating.

2.4 Surface analysis and testing

The surface morphology and elemental composition of Ni-Co-BN coating were analyzed by field emission scanning electron microscope and Energy dispersive spectroscopy. The phase constituents of samples were obtained by using a Rigaku Smart Lab X-ray diffractometer (Rigaku Smartlab). Fig.4 shows the structural analysis of the deposited Ni-Co-BN coating at various magnifications. The Ni-Co-BN particles are dispersed uniformly in the matrix, the grains are sharp and the BN fragments are surrounded by them. The developed coating showed a uniform and dense structure. No cracks are visible in the coating.

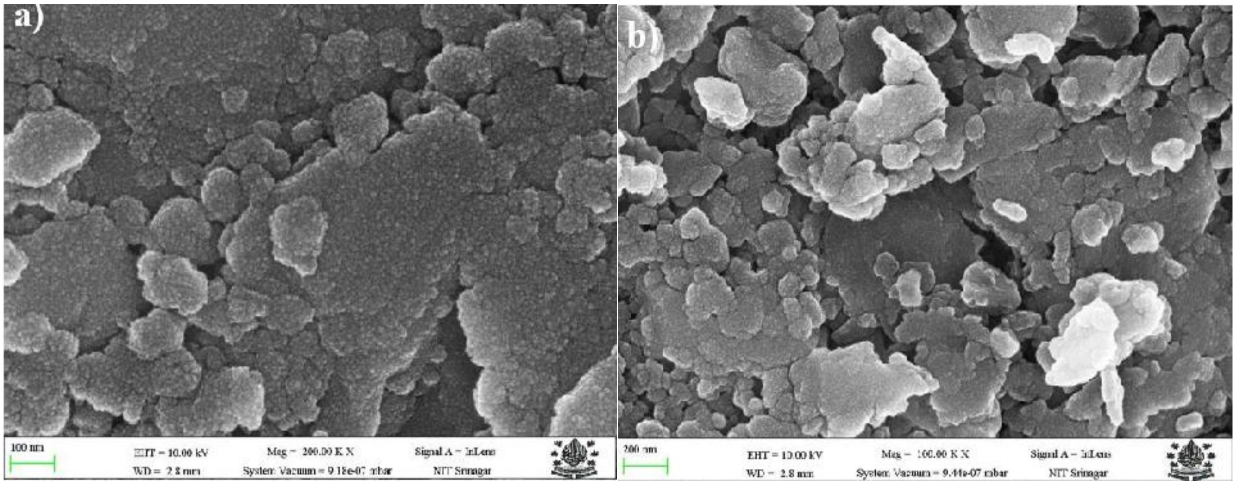
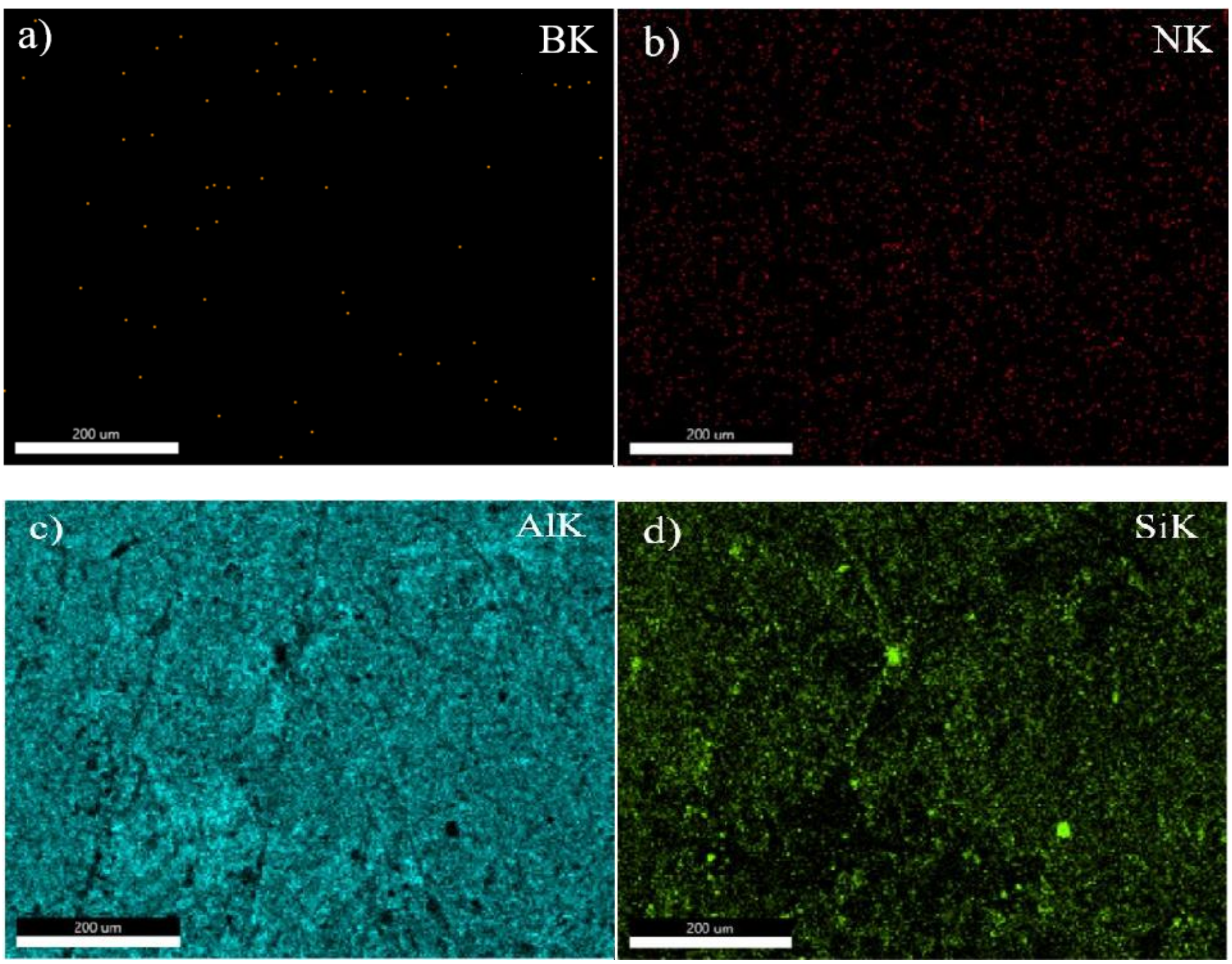


Fig.4 Ni-Co-BN coating images obtained by FESEM



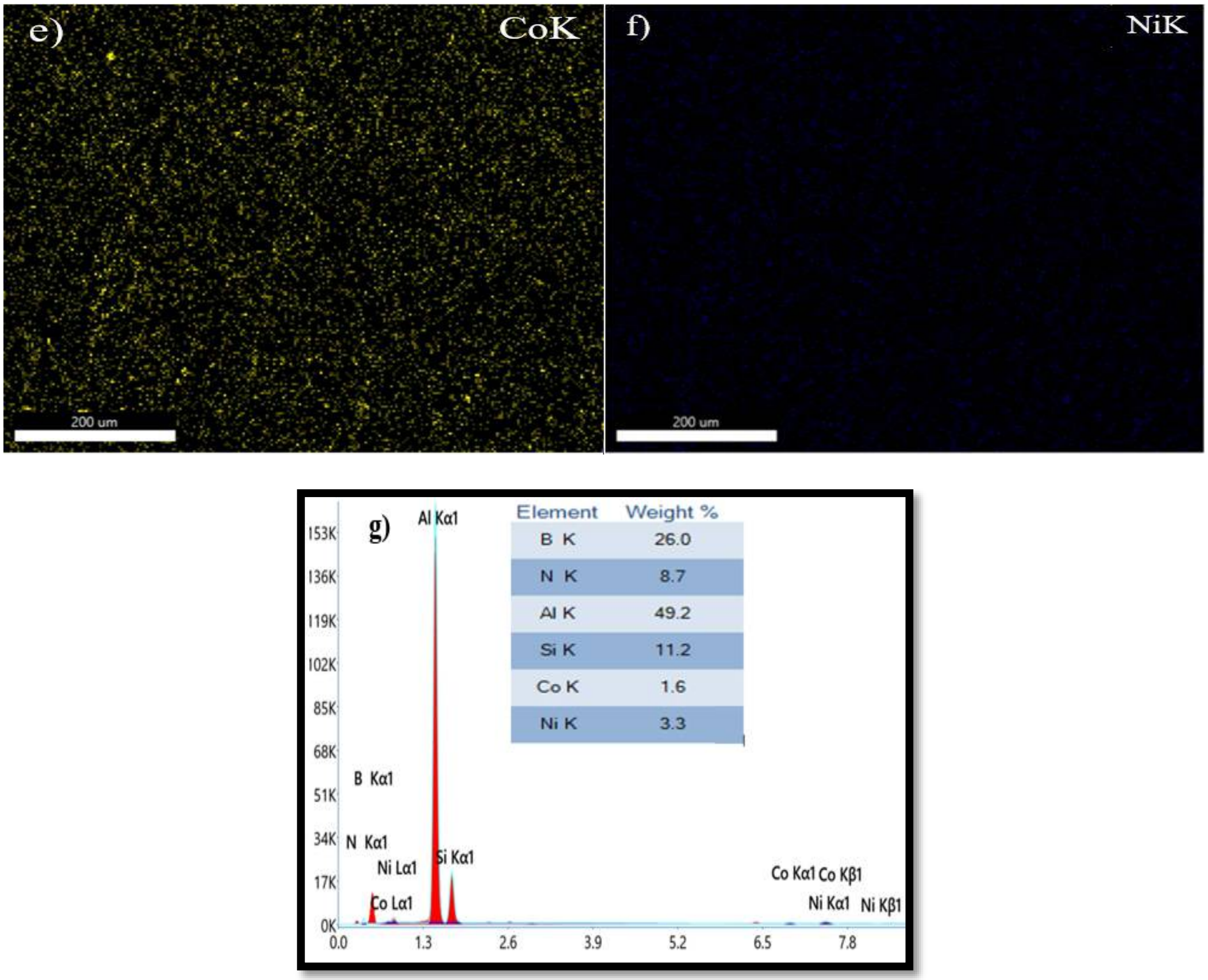


Fig. 5 (a-f), shows the Energy dispersive spectroscopy (EDS) spectrum of Ni-Co-BN coating and g shows the Elemental composition of Coating

EDS mapping, as illustrated in Fig 5 (a-g), indicates that the contents of Ni-Co-BN elements are present. It is also evident from the images the that the amount of Nickel, Cobalt, and Boron Nitride is uniformly distributed throughout the surface of the substrate. The XRD studies were performed at a scan speed of 5.0985 deg./min and a scan range of 5^o-90^o. Fig. 6 represents the X-ray diffraction of Ni-Co-BN coatings and base material. The ICDD database card numbers for mentioned phase (Ni, Co, and BN) are 01-078-7536, 01-071-4238, and 00-026-0773 respectively, the structure of Ni is confirmed by XRD with α=90 and β=90 and γ = 90 m and the lattice constants obtained for Ni, for a 3.537and b = 3.537 and c =3.537, for Co α= β = γ =90 a=b= c= 3.632 and similarly for BN α= β = γ =90 a=b= 2.5870 c= 4.315. The crystallinity and purity are confirmed by the XRD spectrum.

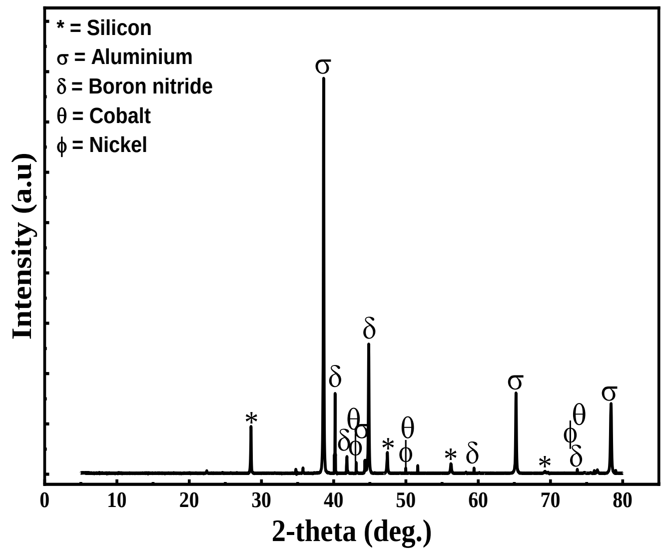


Fig. 6 x-ray diffraction of Ni-Co-BN coating on Al-Si substrate

Nano-indentation tests were carried out on a clean surface to determine Young's modulus and hardness values using a Hysitron TriboScan Nano-indenter with Berkovich indenter. The mechanical properties were evaluated by Oliver and Pharr's technique [33–34].

3 Results and discussion

The mechanical properties of a Ni-Co-BN coated sample were investigated at the nanoscale level using low stresses, and the following are the results:

3.1 Hardness and Young's modulus

Indentation tests on Ni-Co-BN coating were performed at a load of 500 µN to 1250 µN. Fig 7 shows various load-displacement slopes of the developed coating through a load range of 500 µN -1250 µN. The depth of the indent varies from 22 nm to 50 nm, as shown in Fig 6. The greatest depth of 50 nm has been observed at a peak load of 1250 µN, which is clear from load-displacement slopes, indicating that the indent depth is smaller than the thickness of the applied coating of 500 nm. The greatest depth reveals the coating's elastic-plastic properties, whereas, the contact depth indicates the plastic deformation caused by indentation load. The figure also shows that on increasing the normal load during indentation, the indent, as well as the contact depth, increases. The non-linear, grooved unloading curves, at all loads, show that the coated substrate is similar to the Oliver and Pharr model.

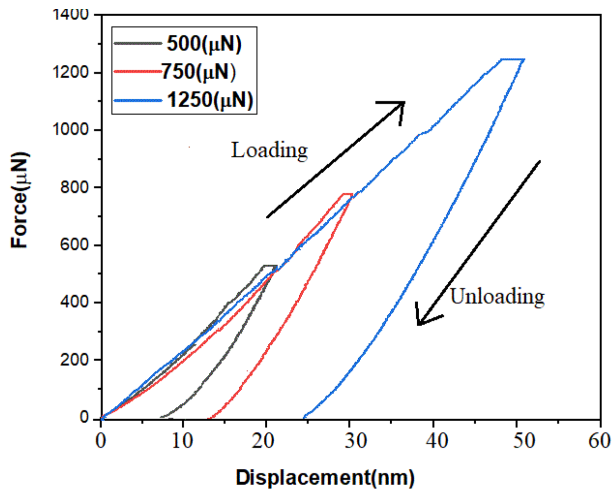


Fig. 7 Multiple loads vs displacement curves produced by indentation at various load.

Fig.8 shows 2D and 3D indentation pictures taken with a scanning probe microscope (SPM) from 500 to 1,250 μN. Fig.8(a)–8(b) show that the Ni-Co-BN coating is stacked around the indent marks during indentation studies, indicating that the deposited coating is soft. Fig.8(c)–8(d) show deposited coating piling up between 500 to 1,250 μN. The effect of indentation stress on reduced modulus of a Ni-Co-BN coated sample is shown in Fig 8. With increasing applied stress from 500 μN to 1,500 μN, Young's modulus of Ni-Co-BN coating falls from 182.92 GPa to 148.96 GPa, indicating the coating has a non-linear elastic property. The surface pores present in Ni-Co-BN coating were the primary causes for decreases of reduced modulus with growing load. Banday et al. were the first to report the oxygen presence in the surface pores. Furthermore, the cause for the decrease of young's modulus, with a growing normal load, is the 10% of the coating-substrate penetration effect. An improved penetration depth will incorporate the substrate effect into the measurement of coating properties. On the other hand, in comparison to the reduced modulus (27 MPa–100 MPa) achieved from different research studies (Hui et al., 2011), in this research study, a greater value of reduced modulus (182.92 GPa) of Ni-Co-BN coating was achieved. This increase in reduced modulus value is linked to the process of PVD. When using the PVD technique, the coating is developed from the solid targets to the surface of the specimen with a similar stoichiometry as the targets and at higher rates than other coating techniques employed by many researchers. The effect of hardness and reduced modulus on the load is explained in table 2.

Table 2 Nano-mechanical properties of Ni-Co-BN coating on Aluminium-silicon substrate

S. No.	Load(μN)	Loading type	Hardness H(GPa)	Reduced Modulus E_r (GPa)	Contact Depth (nm)	Max. Depth(nm)
1	500	Basic QS Trapezoid	21.22	182.92	15.7	22.08
2	750	Basic QS Trapezoid	20.30	165.59	20.3	30.66
3	1250	Basic QS Trapezoid	16.26	148.96	28.6	50.3

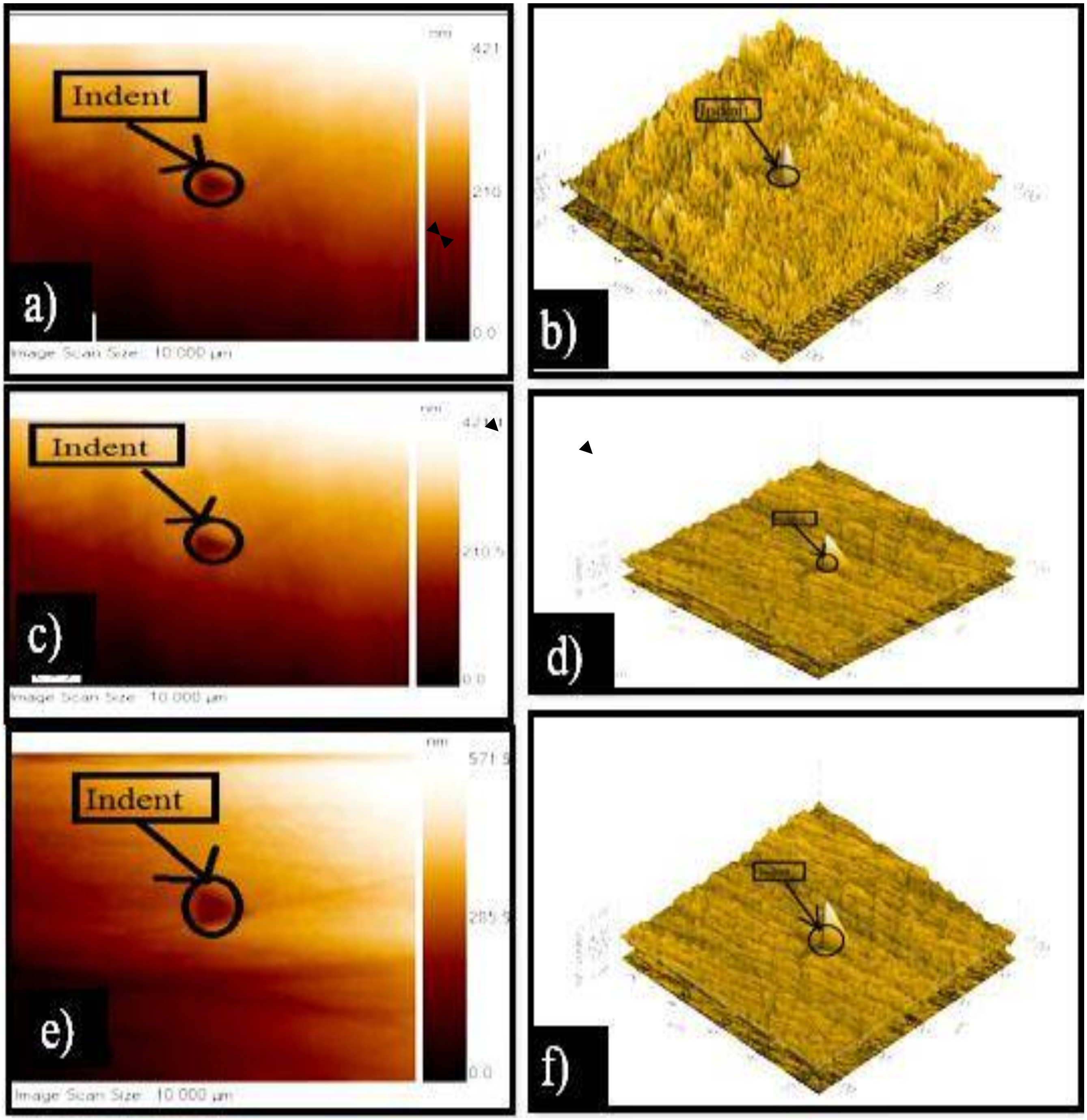


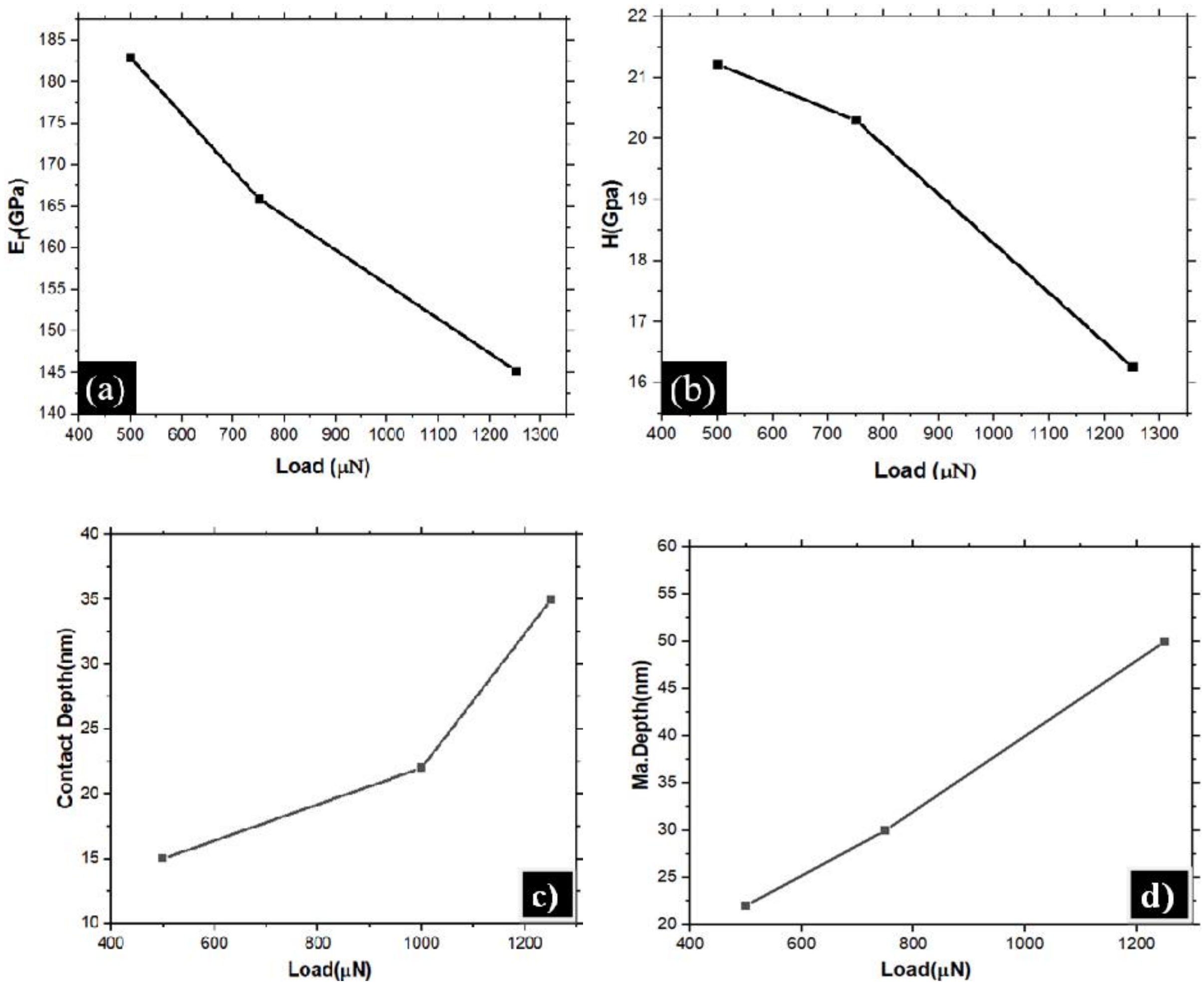
Fig. 8 2D and 3D images of indentation at 500µN (a,b) 750µN (c,d) 1250µN (e,f).

The effect of nano-indentation load on reduced modulus and hardness is shown in Fig.9 (a and b) and the impact of nano-indentation on load vs contact depth and the maximum depth is shown in Fig. 9 (c and d). As the indentation load is increased from 500 µN to 1250 µN, the nano-hardness of Ni-Co-BN drops from 21.22 GPa to 16.26 GPa. The decreasing value of nano-hardness of Ni-

Co-BN coating, with rising load, is the impact of indentation size. As a result, it is determined that, as the indentation load increases, the nano-hardness decreases. Li and Bradt (1993) reported the Indentation size effect, which is provided in equation (1).

$$P = Ad^{n} \ldots (1)$$

Where P = load during indentation and d = size of the indentation. From the curve-fitting of test results, the values of A and n are determined. The exponent n has a value between 1 and 2. (Jang, 2006). When compared to other coatings developed by other researchers, it was discovered that Ni-Co-BN coating on Al-Si substrate had a greater value of nano-hardness (21.22 GPa) (Hui et al., 2011, S. Banday and M.F. Wani).



9 (a) Load vs reduced modulus, (b) Load vs Hardness, (c) load vs Contact depth, (d) load vs Maximum depth of Ni-Co-BN coating of load range from 500μN to 1250μ.

Researchers have carried out nano-indentation on Ti/MoS_2 during a load of 750 to 1500 μN. The end results show a maximum hardness of 16.10 (GPA) under the load of 750 μN and a reduced modulus of 169.729 (GPA) [35]. The value of hardness and reduced modulus, obtained in this research, study, is quite high than that obtained by other researchers. Hence Ni-Co-BN coating has improved the mechanical properties of the Al-Si -substrate.

3.2 Nano-wear Properties of Ni-Co-BN Coating

The tribological characteristics of Ni-Co-BN nano-coatings were determined by wear tests using a computer-integrated MFT-2000 nanotribometer. The pin-on-disc experiments were performed as per ASTMG99-17 standards.

A steel ball (EN8) of diameter 1.6 mm diameter was used as a counter body. The coated sample was attached to the drive and it rotates constantly in a predetermined way at a certain rpm, while as the ball was fixed against the coated sample under normal load. The sliding distance was kept constant and the load was varied. Before the test, the ball was cleaned in acetone for 5 minutes, followed by drying in the oven at 50^oC.

The tests were performed out in ambient as well as in humid conditions to investigate material loss (i.e. wear) and plastic deformation. All these tests were performed at a constant speed of 30 rpm in the counter-clockwise direction at different sample radii.

Nano-wear testing of Ni-Co-BN coating was performed using a nano-tribometer to determine the material loss and load-bearing ability of the developed coating. The wear testing was carried out on 4 different loads 0.5N, 0.7N, 0.8N, and 1 N. To confirm the reliability and accuracy of the tests, various, sets of tests were carried out for each test requirement, and the average value of these results was presented. Furthermore, the worn surface of the samples were scanned using FESEM, after each test, to analyze the process of wear.

The COF was calculated simultaneously during wear testing. The coating provided outstanding lubricating properties with COF ranging from 0.8-0.4 during wear testing. The developed Ni-Co-

BN coating effectively proved no metal-to-metal contact was made and ensured lubrication all over the system.

The wear rates of the developed coating were also determined to be quite good as, at the low load of 0.5N, the wear rate was 4.08x 10^{-5}mm^3/Nm and for the loads varying from 0.7 N to 1 N. The wear rate ranges from 8.08x10^{-5} to 1.6808x10^{-4} mm^3/Nm. The wear property rises as the load is increased, the wear rate was highest at a load of 1N. The rapid Wear rate increases with load is related to Archard's law [36-37]. Fig.11 (a to d) displays the FESEM images of the worn surfaces It is a clear indication from the images, the wear appears to be primarily abrasive and as the load increases, the wear gets worse and is illustrated in Fig 11. The pile-ups of BN coating were detected in and around the wear tracks even at a low load of 0.5N, as seen in Fig 11 (a). There is a gradual increase in wear as the load was increased for the load of 0.7 N, 0.8 N, and 1 N, as shown in fig 11 (b to d). It was determined that when the load was increased, the wear worsens, and even more material has been stacked close to the worn tracks. The results of the nano-wear test coincide well with those of the measurement and mechanical tests. The hardness reduces with increasing load and the wear rate increases. Additionally, it was found that the inclusion of BN into the coating enhances the tribological properties of Ni-Co-BN coating.

The wear height is determined as follows:

$$H = h_1 - h_2 \ldots .(2)$$

The wear volume is determined by equation 3:

$$V = (W_s) \times H \ldots (3)$$

Archard's equation was used to determine the wear rate as follows:

$$K = \frac{V}{LS} \ldots .(4)$$

Where H = wear depth; h1 = height outside the wear track; h2 = height within the wear track;

K = wear rate; Ws = wear scan size; V = wear volume; S = sliding distance; and L = load.

EDS evaluation of the worn tracks was performed to determine coating failure and its load-bearing capacity. Fig 11 (b and d) shows the EDS analysis of the Marked Region up to a load of 0.5 to 0.7 N and reveals the high intensity of the elements Ni, Co, and BN and does not show any coating failure. Fig 11 (f) demonstrates how the elements of Ni, Co, and BN become less intense as the load is increased to 0.8N, and non-failure of coating and smooth wear track. Fig 11 (h) shows the EDS studies were performed in the marked region, and it is obvious from the debris that the coating has been stripped from the surface, and high-intensity peaks of iron, carbon, and oxygen indicate the existence of EN steel ball. This demonstrates that the oxidative wear of the EN steel ball was quite noticeable during the wear process.

Results obtained from this research study are quite good compared with the results obtained by earlier researchers [38-41] Hence, it is evident that the Ni-Co-BN coating decreases the COF and increases wear rate, compared with the Al-Si substrate. The Nano-wear characteristics of the Ni-Co-BN coating are shown in Table 3

Table 3 Nano-wear properties of Ni-Co-BN Coating on aluminum-silicon (Al-Si) substrate

S.NO	**Load (N)**	**COF**	**Wear rate (mm^3/Nm)**
1	0.5	0.8	$4.08x10^{-5}$
2	0.7	0.7	$8.08x10^{-5}$
3	0.8	0.5	$1.20x10^{-4}$
4	1	0.4	$1.608x10^{-4}$

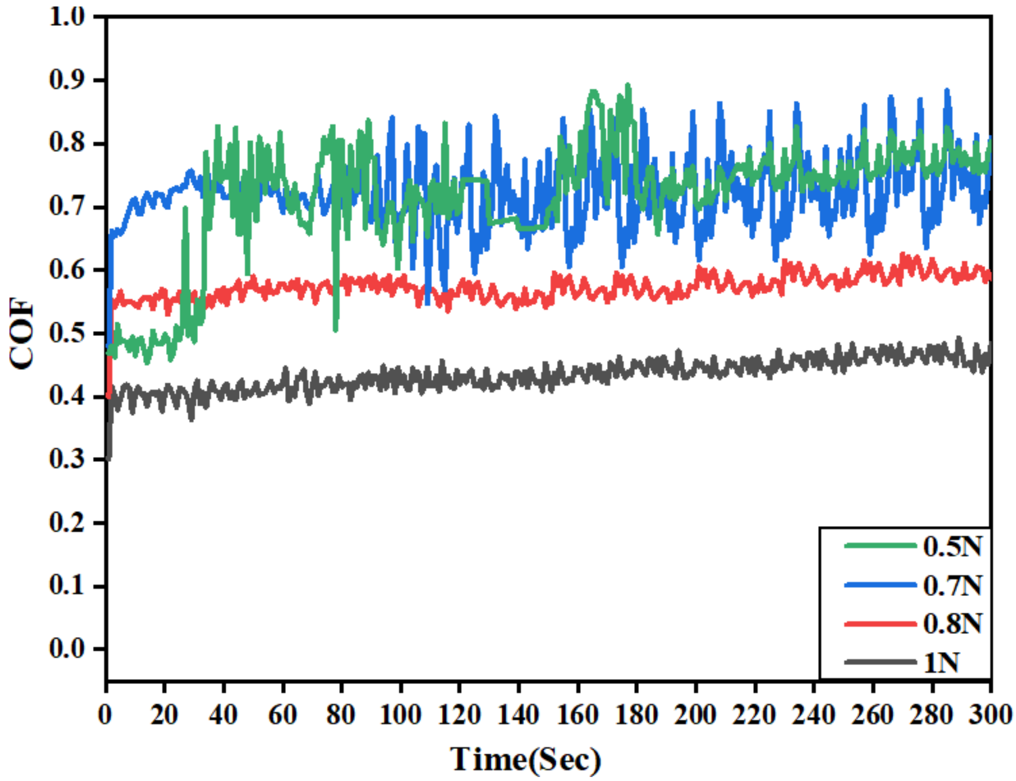
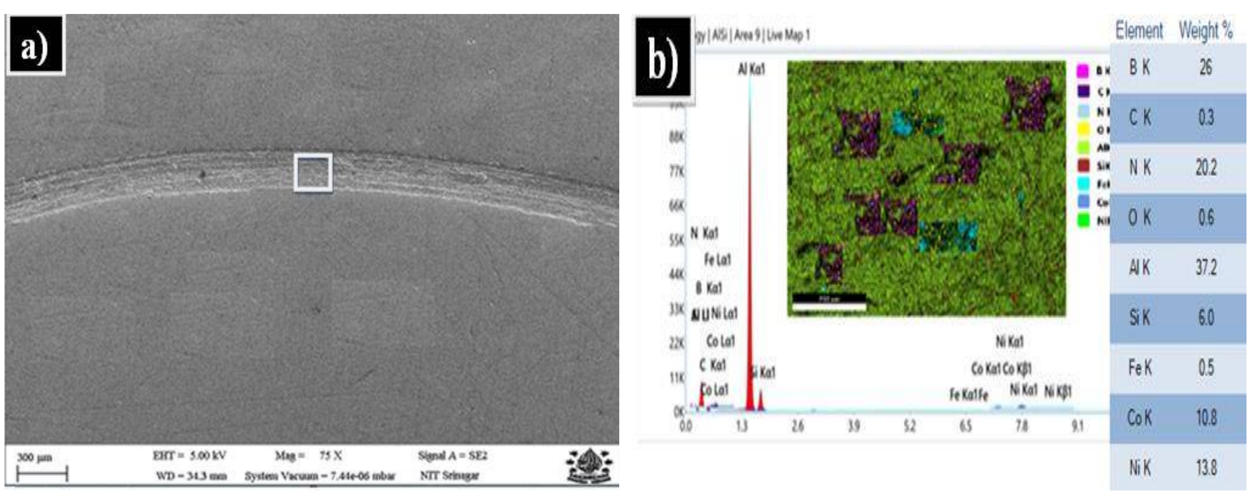


Fig. 10 Variation of coefficient of friction with time for Ni-Co-BN coating on Al-Si



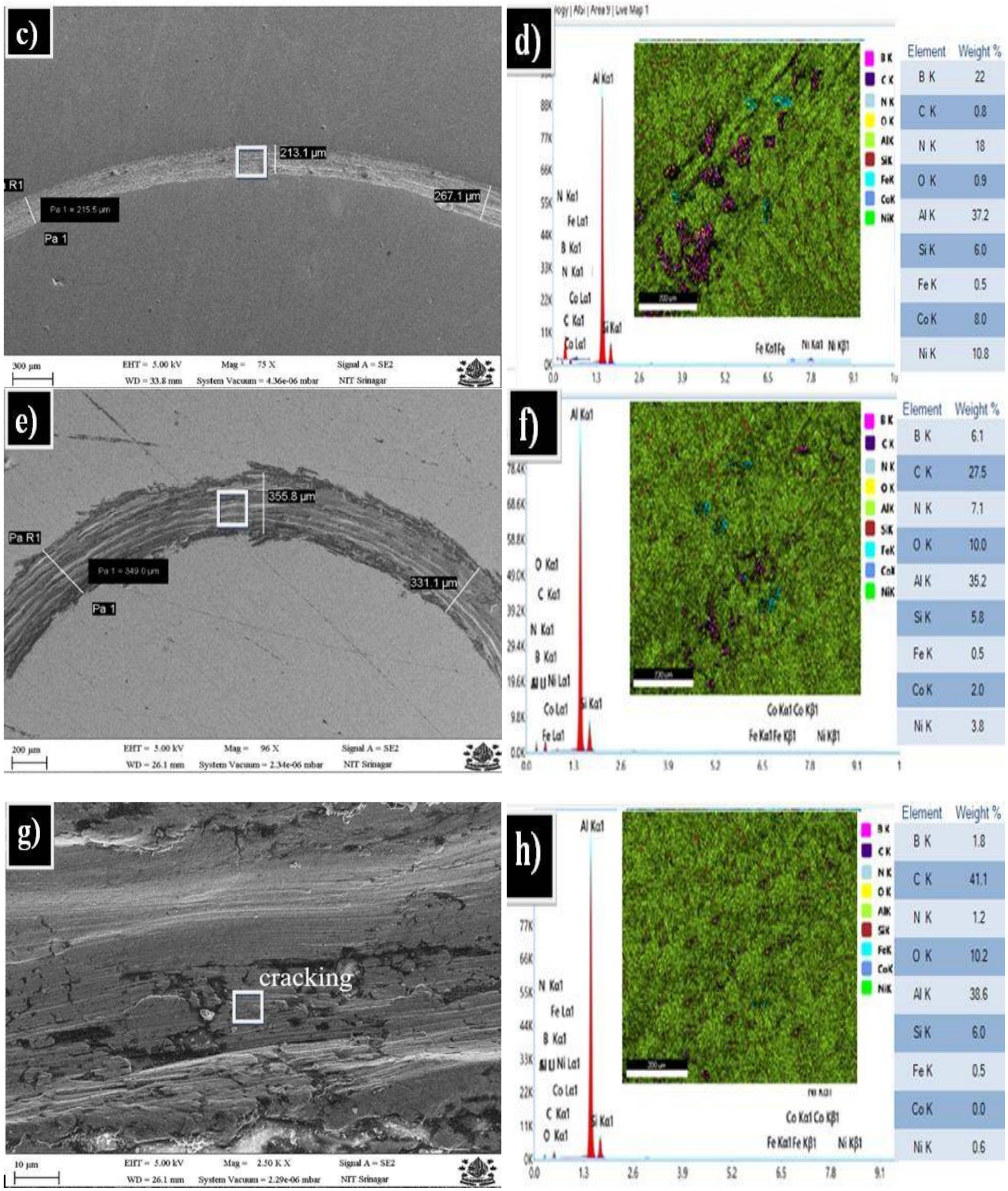


Fig.11 FESEM and EDS images of the wear tracks at (a,b) 0.5N (c,d) 0.7N (e,f) 0.8 (g,h) and 1N

CONCLUSION

In this study paper, mechanical and nano-tribological characteristics of a nano-composite Ni-Co-BN coating, deposited over an aluminum-silicon substrate, was determined. The magnetic sputtering technique was used to deposit the coating.. Ni-Co-BN has been properly deposited on Al-Si substrate at a temperature of 250^{o}C. The coating characterization was done by using FESEM, EDS, and XRD. The pin-on-disc tribometer was used to determine the coefficient of friction and the wear rate of the coating was calculated against steel balls at various loads. Furthermore, nano-hardness tests were performed to determine the hardness and reduced modulus at different loads.The following are the major conclusions:

1. During nano-hardness testing, a maximum hardness of 21.22 GPa was determined which resembles to a load of 500 µn at a contact depth of 26.7 nm. Furthermore, on increasing the load, the hardness was decreased.
2. The maximum value of reduced modulus of 148.96 GPa was calculated at a load of 1250 µN.
3. The coating showed the lowest wear rate of $4.08x10^{-5}$ mm^3/Nm at a load of 0.5 N. Furthermore wear rate of Ni-Co-BN coating increases from 4.08×10^{-5} to $1.6 \times 10^{-4} mm^3$/Nm with increasing load.
4. During the wear test, the coefficient of friction was 0.8 to 0.4 which indicates the self-lubricating property of the coating. The Ni-CO-BN coating successfully prohibited material-to-material contact and lubrication all over the tests without facing any failure. The reduction in friction coefficient with increasing load can be explained by using the Hertzian contact model load. The model states that the coefficient of friction (CoF) depends on the contact pressure for two solids in an elastic state. [42-46]

As a result, Ni-Co-BN coatings formed using the magnetic sputtering approach have improved the nano-mechanical and nano-tribological capabilities of the substrate and can be employed as a self-lubricating coating.

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Declaration of interests

- The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Nano-mechanical and nano-tribological characterization of Ni-Co-BN nano-composite coating for bearing applications

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ABSTRACT

In this study, BN with the addition of Ni and Co have both been deposited on Aluminium -Silicon using the PVD technique. The composition and morphology of Ni-Co-BN coatings were investigated using field emission scanning electron microscopy (FESEM), energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD). Mechanical studies on Ni-Co–BN coatings at low loads ranging from 500 to 1250 μN were conducted to investigate the effect of load on reduced modulus and hardness. Besides this, nano-wear studies were carried out with loads varying from 0.5 N to 1 N to investigate the distortion and cracking behavior of the coating. The results show that the reduced modulus and hardness of the Ni-Co-BN coating decreases as the load increases. The wear rate of the Ni-Co–BN coating increases with an increase in load from 4.08×10^{-5} to 1.608×10^{-4} mm^3/m. The Ni-Co–BN coating displayed smooth wear scars on the sample surface with no fractures or debris, suggesting that the coated material flowed in a plastic way near the wear scar. The behaviour of the coating suggests its strong suitability for bearing applications.

1. Introduction

The primary goal of designers in automobiles and aerospace applications is to reduce the weight of cylinder liners, bearings, pistons, and piston rings. The weight reduction of these tribo elements is important not only for increasing efficiency but also for energy conservation and environmental preservation. For achieving this, the most promising for designers and engineers is the use of aluminum alloys of various series. Aluminum alloys are receiving a lot of research observation these days. But many factors limit the use of aluminum and its alloys, in industry, as they have poor wear resistance and low hardness. However, to solve these problems various types of tribological solutions are applied (lubricants, coating, and special structure designs). Nevertheless, there is a rising need for mechanical parts to eliminate the uses of harmful lubricants in many foods and textile industries. Surface engineering like coatings is one the best and most effective methods of increasing surface properties. Coatings modify tribological and mechanical properties by decreasing the friction coefficient and increasing the surface hardness. However, the inclusion of metals, ceramics, or mixed metals in the single and multilayer coatings resulted in a more sophisticated coating with outstanding multifunctional properties. The tribological properties can be enhanced by using self-lubricating single or multilayered nano-coatings. as standard lubricants do not deliver the appropriate degree of performance. Furthermore, solid lubricants are free from contaminants and can be used on machine components where liquid lubricants cannot be used, due to accessibility [1–5]. Aerts et al. have suggested the use of self-lubricating to tackle the problems of severe wear [6]. Takaya et al. investigated the wear characteristics of an anodic oxide coating of aluminum, imbued with an iodine component, which has a low coefficient of friction and functions as a solid lubricant, under extreme conditions/hazardous environments [7]. Various types of solid lubricants used are graphite, BN and transition metal dichalcogenides, organic polymers like PTFE, soft metals like Au, Ag, Ni and metal oxides like PbO, and Mos_2 which have excellent lubricant properties [8,9].

Boron nitride (BN) is primarily utilized as a lubricant for applications requiring low friction, because of the weak inter-atomic interaction between their multi-layer structures. BN possesses a lamellar shape with strong bonding between the atoms. However, a weak inter-atomic connection between the layers leads to poor strenth.The lamellar shape of BN and the ineffective Van der Waals force among each layer are responsible for the low friction. Researchers have also found that the inclusion of various metals to ceramics leads to the formation of BN/metal multi-layer coatings (Co, Ni, Au, Fe, Ni, or TiN, or TiB_2 or mixed metals). The addition of boron nitride to various metals results in

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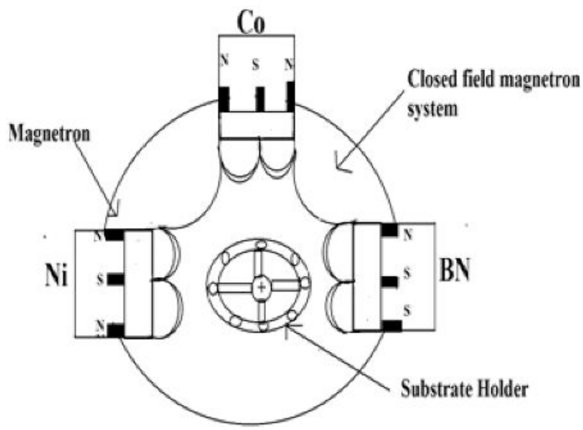

Fig. 1. Schematic view of magnetron sputtering system.

Table 1
Coating parameters.

Characteristics	Results
Deposition temperature	Room Temperature
Deposition rate	Ni 0.12(A/s), Co 0.08(A/s) and for BN 0.12 (A/s)
Target to substrate distance	16 cm (approx.)
Deposition time	2 h
Coating thickness	500 nm

increased hardness and reduced wear coefficient. The nanocomposite materials may also exhibit new functional properties. Ni-Co alloys are extremely hard and have excellent wear and corrosion resistance. They are frequently used as protective coatings on the surfaces of parts [10–18]. Buranawong et al. deposited nano-crystalline $AlTi_3N$ coating, by using the magnetron sputtering process, and found that the thickness and surface roughness, of the coatings, increased with the titanium current, and the deposition duration rises [19]. Furthermore, Davod et alfound that the creation of compact covering with tortuous grain boundaries, and Ni-Co-P-SiO_2 composite coatings demonstrated higher corrosion resistance compared to Ni-P and Ni-Co-P coatings [20]. Amico and d'Oliveira studied aluminum coatings, coated on specimens of varying roughness, utilising high-velocity, electric arc deposition, and other deposition procedures. The findings revealed that the roughness of coatings placed on warmed substrates is lower as compared to substrates placed in room temperature [21]. The corrosion and wear characteristics of Ni-Co coating may be greatly increased by a composite coating made by incorporating new particles to it. The corrosion and wear properties of the coating, are created by the addition of BN to Ni. Its benefits in electrochemical catalysis, fuel cells, hydrogen storage materials, microelectronics, biomedical applications, composite materials, and fuel cells Ni-Co alloy coatings are extensively employed in automobiles as well as aerospace industries, and other disciplines, owing to their superior physical, chemical, and mechanical qualities [22–28].

Researchers have investigated the tribological characteristics of Ni-Co coating and found that Ni content decreased the wear and friction coefficient of the paired parts, despite the fact that coating wear rises as Ni concentration increases in the coating and improved to various benefits by employing dopants such as Ti, Cr, Ni, Au, PTFE, PbO, and others. The single metal cannot provide the desired degree of performance in many engineering applications. However, it has been discovered that layers with diverse mechanical qualities, such as BN/MoS_2 improve coating features such as wear resistance, hardness as well as chemical inertness.The lubricating layer on hard Ni coatings was found to reduce friction and develop low stress at the substrate/coating contact [29–32]. The review of the result of the above literature reveals that adding metal layers improves properties like hardness, and wear resistance as well as load-bearing capacity. Furthermore, no literature has yet described the deposition of the self-lubricating coating of Ni-Co-BN on an Al-Si substrate.

The main objective of this research was to fabricate a single-layer composite coating with exceptional mechanical and tribological properties at the nano-scale level by alternating different metals that can not be obtained using single pure metal. In this study, a nanocomposite coating of 500 nm was deposited on Al-Si alloy as an alternate lubricant where liquid lubricant fail. The aim of this study is to look at how Ni-Co and BN synergistically affect the mechanical as well as tribological characteristics of the Ni-Co-BN coating applied to an Al-Si alloy [33–35].

2. Experimental procedures

2.1. Coating deposition

The coating was deposited using a PVD magnetron sputtering process. Prior to coating deposition, all the specimens were polished at constant load on an automatic polishing machine using wet emery papers of grit size 800, 1000,1200, 2000, and 2500, sequentially. Following this, the surface was again polished using diamond paste with

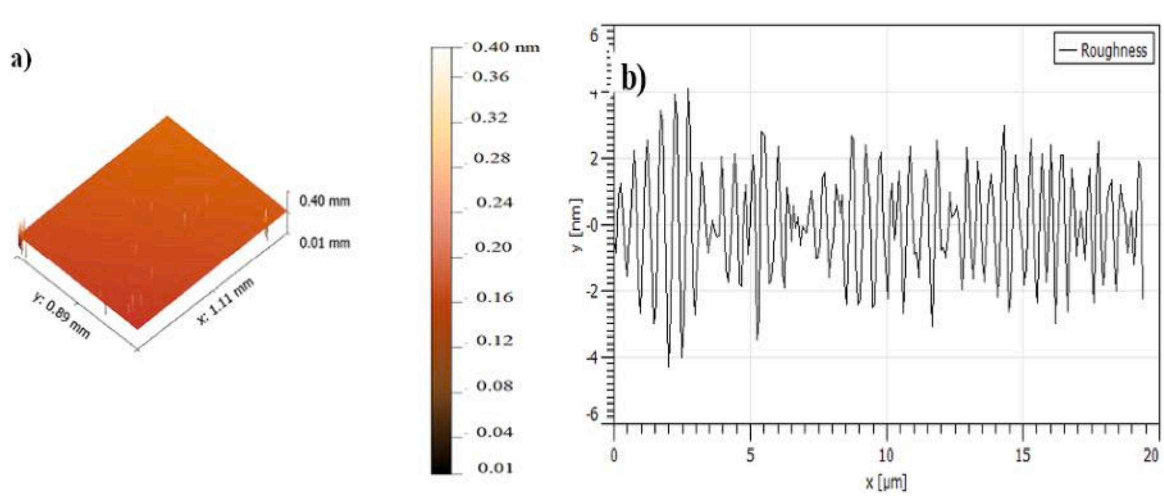

Fig. 2. (a) 3d Surface topographical image of Ni-Co-BN coating and (b) Surface roughness of Ni-Co-BN coating surface.

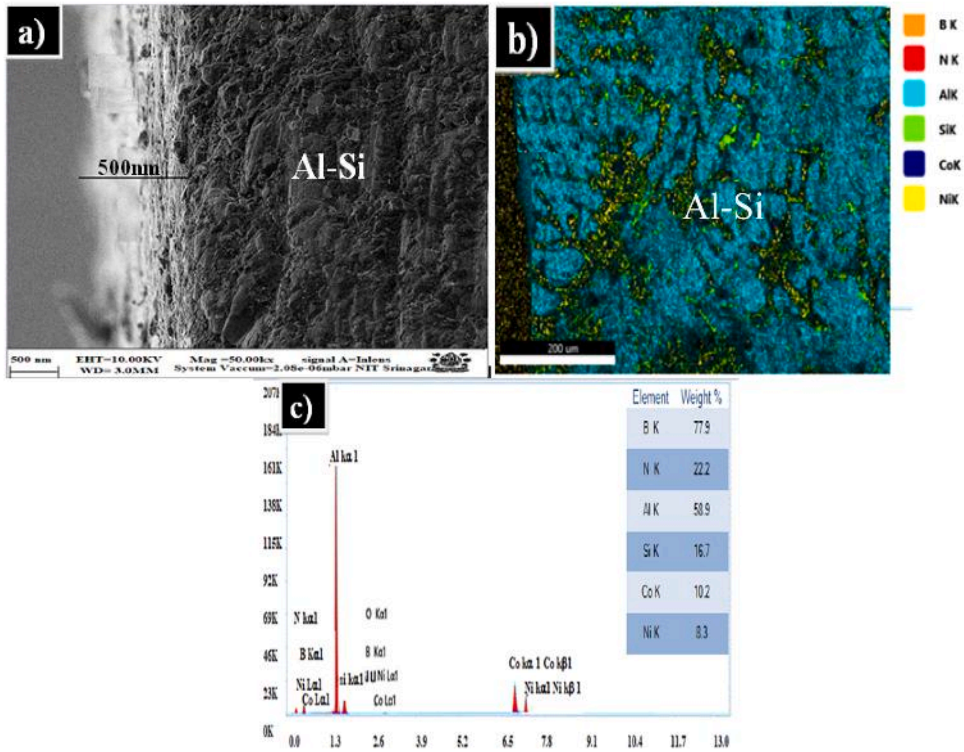

Fig. 3. Cross-sectional images of Ni-Co-BN shows the thickness and EDS (a) FESEM and (b, c) EDS.

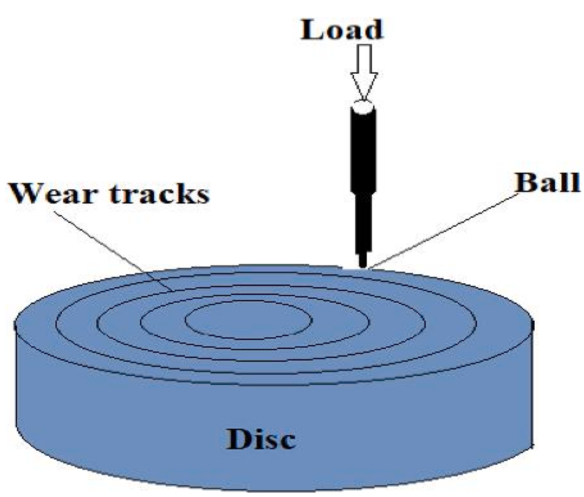

Fig. 4. Schematic representation of tribological tests.

particle sizes of 0.5 μm and 0.25 μm on velvet cloth till the mirror surface was attained. Following the polishing procedure, ultrasonic cleaning was done followed by acetone for five minutes to remove impurities, and then dried in an oven at 50 °C for five minutes.

The schematic representation of the multitarget RF sputtering apparatus, utilised for coating deposition, is shown in Fig. 1. The equipment consists of three independent target holders and RF power is applied to two of them. The separation between the substrate and the target is around 16 cm. Discs of BN (99.99% purity), Co (99.99% purity), and Ni (99.99% Purity), each having a 2-inch diameter, were used as sputtering targets as shown in Fig. 1. The deposition process takes place in a chamber that has been evacuated to low pressure of 4×10^{-5} pa with power of Ni 05 W (DC), Co 70 W (RF1), and BN 70 W (RF2) and airflow rate of 20.7 sccm, using the turbomolecular pump. Only argon was allowed to enter the chamber during the deposition. The details of the coating deposition are listed in Table 1.

A 3D profilometer was used to evaluate the surface roughness of the samples. The 3D surface topographical image and the surface roughness of samples are shown in Fig. 2(a) and (b) respectively, and the surface roughness, R_a of the samples was measured to be 15 nm.

2.2. Coating thickness

The coating thickness was measured using a field emission scanning electron microscope (FESEM), ZEISS Gemini SEM 500. The samples were cut using wire EDM and were polished using different grades of emery papers. Furthermore, the cross-sectional surface was again polished using diamond paste on a velvet cloth till the mirror surface was attained. Following the polishing procedure, the samples were cleaned using acetone to remove impurities and dried in an oven. Fig. 3(a) shows FESEM image of the Ni-Co-BN coating demonstrating that the coating has grown in a uniform layering structure. The Ni-Co-BN coating had a final thickness of 500 nm. Fig. 3(b) shows the image of EDS image which confirms the presence of Ni, Co, and BN particles in the coating. Fig. 3(c) Shows the elemental composition indicating that Ni-Co-BN elements are present.

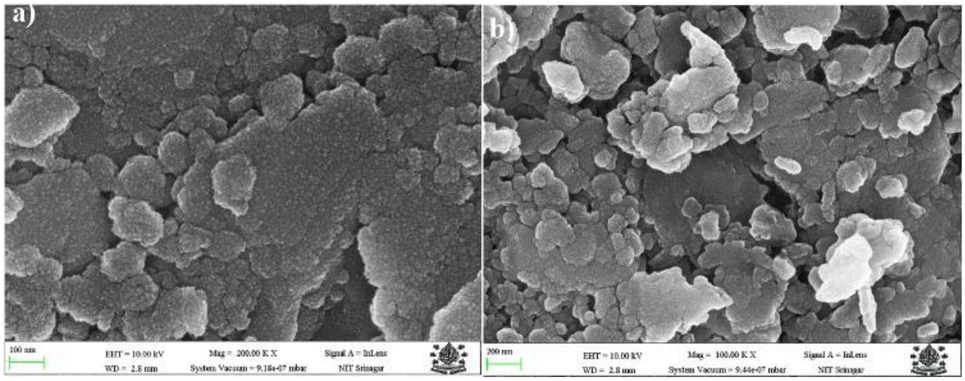

Fig. 5. FESEM images of Ni-Co –BN coating at different magnifications.

2.3. Surface testing

Nano-indentation tests were carried out on the coated samples to determine reduced modulus and hardness values using a Hysitron TriboScan Nano-indenter with Berkovich indenter tip (radius of 50 nm) which enables real-time indentation depth monitoring and in-situ SPM imaging. The tests were performed at three different loads to determine the coating's hardness and reduced modulus as a function of the applied indent load. The mechanical properties (hardness, reduced modulus) were evaluated in accordance with Oliver and Pharr's technique [36, 37].

The schematic view of the tribological tests is shown in Fig. 4. The tribological characteristics of Ni-Co-BN nano-coatings were determined by wear tests using a computer-integrated MFT-2000 nano-tribometer. The pin-on-disc tests were carried out as per ASTM G99–17 standards. A steel ball (EN8) of diameter 1.6 mm was used as a counter body on the coated disc samples. The ball was held stationary against the sample under normal load. The sample holder is connected to the drive which rotates continuously in a predetermined direction at a fixed radius and rpm for each test. The sliding distance was kept constant and the load was varied. Before the test, the ball was ultrasonically cleaned in an acetone bath for 5 min and then dried in the oven for 5 min at 50 ^{o}C.

2.4. Surface analysis

The microstructure and elemental composition of Ni-Co-BN coating was analyzed by field emission scanning electron microscope and Energy dispersive spectroscopy (EDS). The phase constituents of samples were recorded using a Rigaku Smart Lab X-ray diffractometer (Rigaku Smartlab). Fig. 5 shows the surface morphology of the deposited Ni-Co-BN coating at various magnifications. The Ni-Co-BN particles are dispersed uniformly in the matrix, the grains are sharp and the BN fragments are surrounded in them. The developed coating showed a uniform and dense structure. No cracks are visible in the coating.

EDS mapping, as shown in Fig. 6(a-g), indicates that the contents of Ni-Co-BN elements are present. It is also evident from the images the that the amount of Ni, Co, and BN is uniformly distributed throughout the surface of the substrate. The XRD studies were performed at a scan speed of 5.0985 deg./min and scan range of 5^{o}-90^{o}. Fig. 7 represents the X-ray diffraction of Ni-Co-BN coatings and base material. The ICDD database card numbers for mentioned phase (Ni, Co, and BN) are 01–078–7536, 01–071–4238, and 00–026–0773 respectively, the structure of Ni is confirmed by XRD with $\alpha = 90$ and $\beta = 90$ and $\gamma = 90$ m and the lattice constants obtained for Ni, for a 3.537and b = 3.537 and c = 3.537, for Co $\alpha = \beta = \gamma = 90$ a=b= c= 3.632 and similarly for BN $\alpha = \beta = \gamma = 90$ a=b= 2.5870c= 4.315. The crystallinity and purity are confirmed by the XRD spectrum.

3. Results and discussion

The mechanical properties of a Ni-Co-BN coated sample were investigated at the nanoscale level using low stresses, and the following are the results:

3.1. Hardness and Young's modulus

Indentation tests on Ni-Co-BN coating were performed at a load of 500 μN to 1250 μN. Fig. 8. shows various load-displacement slopes of the developed coating through a load range of 500 μN – 1250 μN. The depth of the indent varies from 22 nm to 50 nm, as shown in Fig. 8. The greatest depth of 50 nm has been observed at a peak load of 1250 μN, which is clear from load-displacement slopes, indicating that the indent depth is less than the thickness of the applied coating of 500 nm. The maximum depth reveals the coating's elastic-plastic properties, whereas, the contact depth indicates the plastic deformation caused by indentation under load. The figure also shows that on increasing the normal load during indentation, the indent, as well as the contact depth, increases. The non-linear, grooved unloading curves, at all loads, show that the coated substrate is similar to the Oliver and Pharr model.

Fig. 9 shows 2D and 3D indentation pictures taken with a scanning probe microscope (SPM) from 500 to 1250 μN. Fig. 9(a- b) show that the Ni-Co-BN coating is stacked around the indent marks during indentation studies, indicating that the deposited coating is soft. Fig. 9(c-d) shows deposited coating piling up between 500 and 1250 μN. The effect of the indentation stress on Young's modulus of a Ni-Co-BN coated sample is shown in Fig. 8. With increasing applied stress from 500 μN to 1500 μN, Young's modulus of Ni-Co-BN coating falls from 182.92 GPa to 148.96 GPa, indicating the coating has a non-linear elastic property. The surface pores present in Ni-Co-BN coating are the main causes for decreases of Young's modulus with growing load. Banday et al. were the first to report the existence of oxygen in subsurface pores. Furthermore, the cause for the decrease of young's modulus, with a growing normal load, is the 10% of the coating-substrate penetration effect. An improved penetration depth will incorporate the substrate effect into the

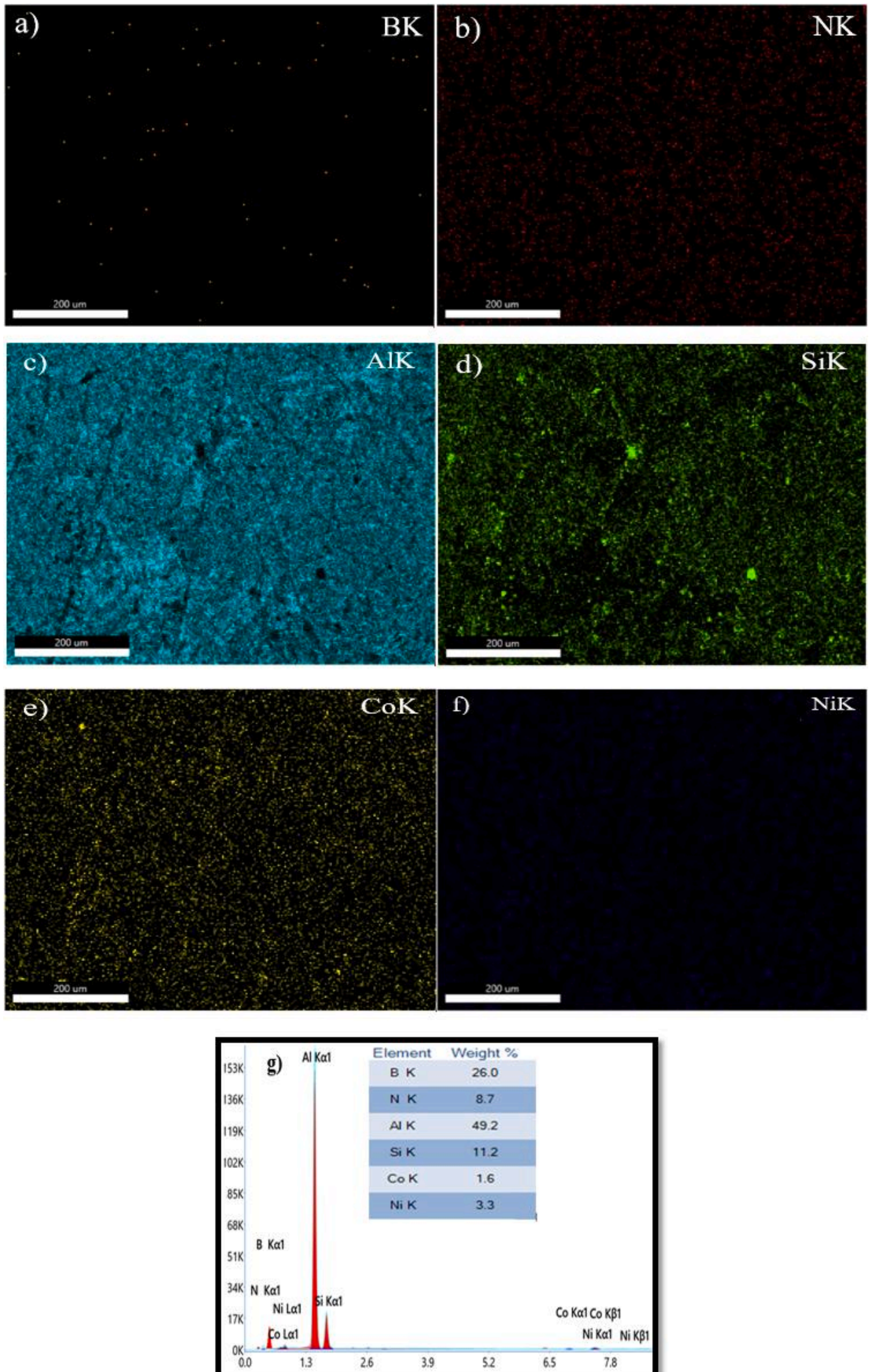

Fig. 6. EDS spectrum of Ni-Co-BN coating deposited on Al-Si substrate (a-f), and (g) Elemental composition of Coating.

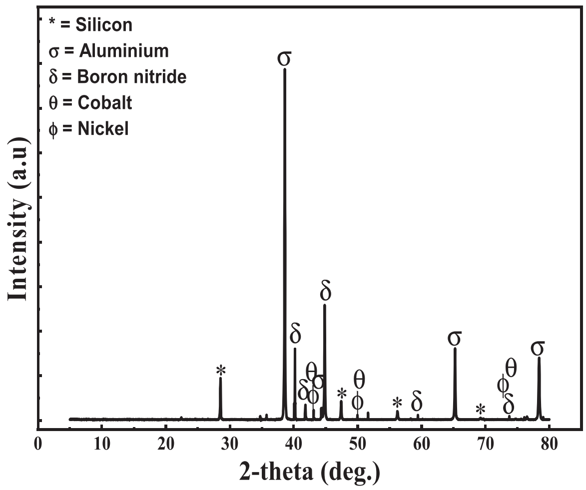

Fig. 7. X-Ray diffraction of Ni-Co-BN coating on Al-Si substrate.

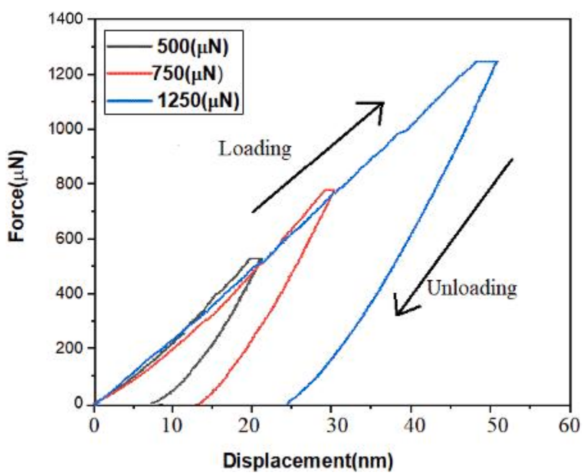

Fig. 8. Multiple load vs displacement curves obtained by indentation at different load.

measurement of coating properties. On the other hand, in comparison to Young's modulus (27–100 MPa) achieved by other researchers (Hui et al., 2011), in this research study, a greater value of Young's modulus (182.92 GPa) of Ni-Co-BN coating was achieved. This increase in the value of young's modulus is attributed to the process of PVD. When using the PVD technique, the coating is developed from the solid targets to the surface of the specimen with a similar stoichiometry as the targets and at higher rates than other coating techniques employed by many researchers. The effect of hardness and reduced modulus on the load is shown in Table 2.

The effect of nano-indentation load on hardness and reduced modulus is shown in Fig. 10 (a and b) and the effect of nano-indentation load vs contact depth and a maximum depth is shown in Fig. 10 (c and d). As the indentation load is increased from 500 μN to 1250 μN, the nano-hardness of Ni-Co-BN drops from 21.22 GPa to 16.26 GPa. The decreasing value of nano-hardness of Ni-Co-BN coating, with rising load, is the impact of the indentation size effect (ISE). As a result, it is determined that, as the indentation load increases, the nano-hardness decreases. Classical power law (Li and Bradt 1993) also reported the ISE, which is provided in Eq. (1).

$$P = Ad^n \ldots \quad (1)$$

Where P = indentation load and d = indentation size. From the curve-fitting of test results, the values of A and n are determined. The exponent n has a value between 1 and 2. (Jang, 2006). When compared to other coatings developed by other researchers, it was discovered that Ni-Co-BN coating on Al-Si substrate had a greater value of nano-hardness (21.22 GPa) (Hui et al., 2011, S. Banday and M.F. Wani).

Researchers have carried out nano-indentation on Ti/MoS_2 at a load of 750–1500 μN. The results show a maximum hardness of 16.10 (GPA) under the load of 750 μN and a reduced modulus of 169.729 (GPA) [38, 39]. The value of hardness and reduced modulus, obtained in this research, study, is quite high than that obtained by other researchers. Hence Ni-Co-BN coating has improved the mechanical properties of the Al-Si -substrate.

3.2. Nano-wear properties of Ni-Co-BN coating

The tests were carried out in ambient and humid conditions to investigate material loss (i.e. wear) and plastic deformation. All these tests were performed at a constant speed of 30 rpm in the counter-clockwise direction at different sample radii.

Nano-wear testing of Ni-Co-BN coating was performed on the nano-tribometer to determine the wear loss and load-bearing capacity of the developed coating. The wear testing was carried out on 4 different loads 0.5 N, 0.7 N, 0.8 N, and 1 N. To confirm the reliability and accuracy of the tests, various, sets of tests were performed for each test condition, and the average value of these results was presented. Furthermore, the worn surface of the tested samples were scanned using FESEM, after each test, to analyze the wear process.

Fig. 11 shows the frictional curves of the tests that were conducted. The COF was calculated simultaneously during wear testing. The coating provided outstanding lubricating properties with COF ranging from 0.8 to 0.4 during wear testing. The developed Ni-Co-BN coating successfully prevented metal-to-metal contact and lubrication throughout the tests without experiencing any failure. The decrease in the coefficient of friction with the increase in load can be explained using the Hertzian contact model load. The model states that the COF is a function of the contact pressure for a pair of solids in an elastic state [40,41].

The wear rates of the developed coating were also determined to be quite good as, at the low load of 0.5 N, the wear rate was 4.08×10^{-5} mm^3/Nm, and for the loads ranging from 0.7 N to 1 N, the wear rate ranges from 8.08×10^{-5} to 1.6808×10^{-4} mm^3/Nm. The wear rate rises as the load increases; the wear rate was highest at a load of 1 N. The rapid increase in wear rate with normal load is related to Archard's wear law [42]. The FESEM images of the wear tracks is shown in Fig. 11 (a to d). It is clear from the figure that the wear mechanism is mostly abrasive as the load increases the wear becomes severe, as shown in Fig. 12. The pile-ups of BN coating were detected in and around the wear tracks even at a low load of 0.5 N, as seen in Fig. 12 (a). There is a gradual increase in wear as a load was increased for the load of 0.7 N, 0.8 N and 1 N, as shown in the figure. Fig. 12 (b to d). It was determined that when the load increases, the wear becomes severe and more material has been stacked near the wear tracks. The results of the nano-wear test coincide well with those of the mechanical test measurement. As load increases,

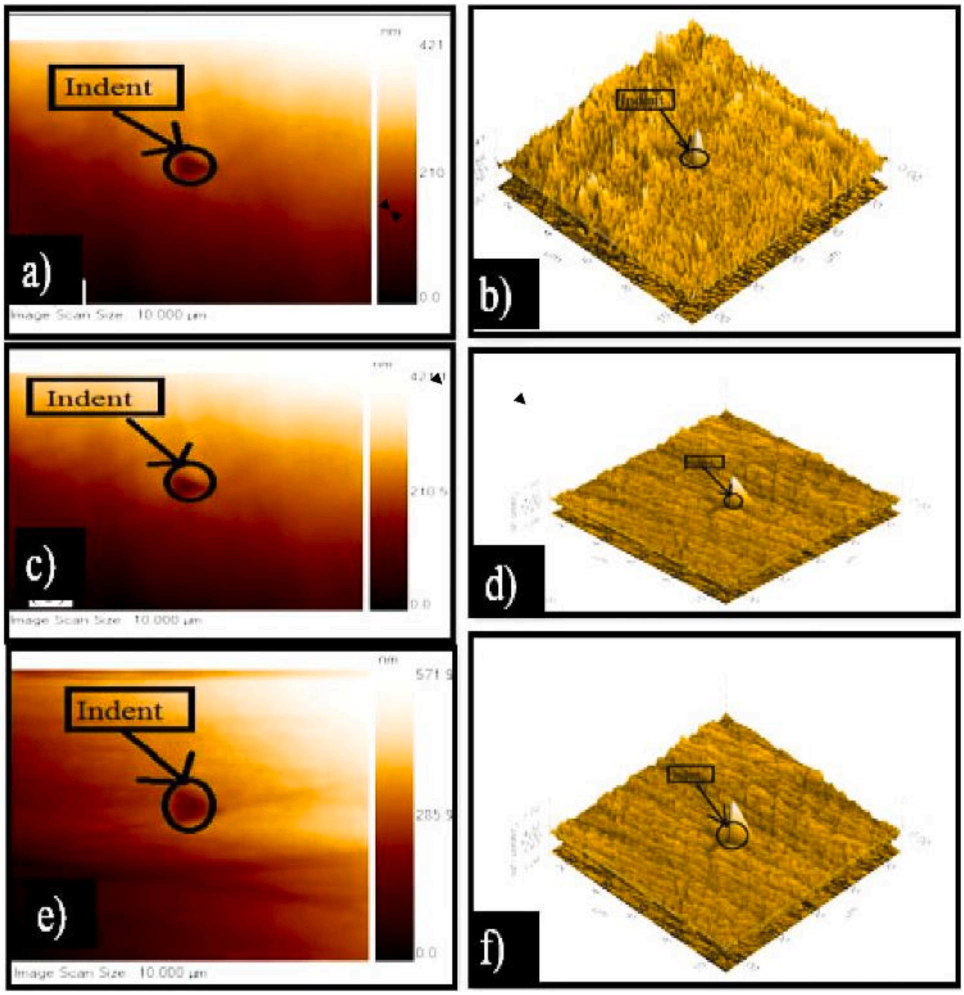

Fig. 9. 2D and 3D images of indentation of (a,b) 500μN (c,d) 750μN and (e,f) 1250μN.

Table 2
Nano-mechanical properties of Ni-Co-BN coating on Aluminium-silicon substrate.

S. No.	Load (μN)	Loading type	Hardness H (GPa)	Reduced Modulus E_r (GPa)	Contact Depth (nm)	Max. Depth (nm)
1	500	Basic QS Trapezoid	21.22	182.92	15.7	22.08
2	750	Basic QS Trapezoid	20.30	165.59	20.3	30.66
3	1250	Basic QS Trapezoid	16.26	148.96	28.6	50.3

the hardness decreases, and the wear rate increases. Additionally, it was found that the inclusion of BN into the coating enhances the tribological properties of Ni-Co-BN coating.

The wear height is calculated as

$$D = d_1 - d_2 \ldots \tag{2}$$

The wear volume is calculated by Eq. 3:

$$V_W = (s_w)^2 \times D \ldots \tag{3}$$

The wear rate is calculated by Archard's Eq. 4.

$$W = \frac{V_w}{PL} \ldots \tag{4}$$

Where, D = height of wear; d_1= height outside wear track; d_2 = height inside wear track;

V_W = volume of wear; S_W = wear scan size; W = rate of wear; P = load; and L= sliding distance.

EDS evaluation of the worn tracks was performed to determine coating failure and its load-bearing capacity. Fig. 12 (b and d) shows the EDS analysis of the Marked Region up to a load of 0.5–0.7 N and reveals the high intensity of the elements Ni, Co, and BN and does not show any coating failure. Fig. 12 (f) demonstrates how the elements of Ni, Co, and BN become less intense as the load is increased to 0.8 N, and non-failure of coating and smooth wear track. Fig. 12 (h) shows the EDS studies were performed in the marked region, and it is obvious from the debris that the coating has been stripped from the surface, and high-intensity

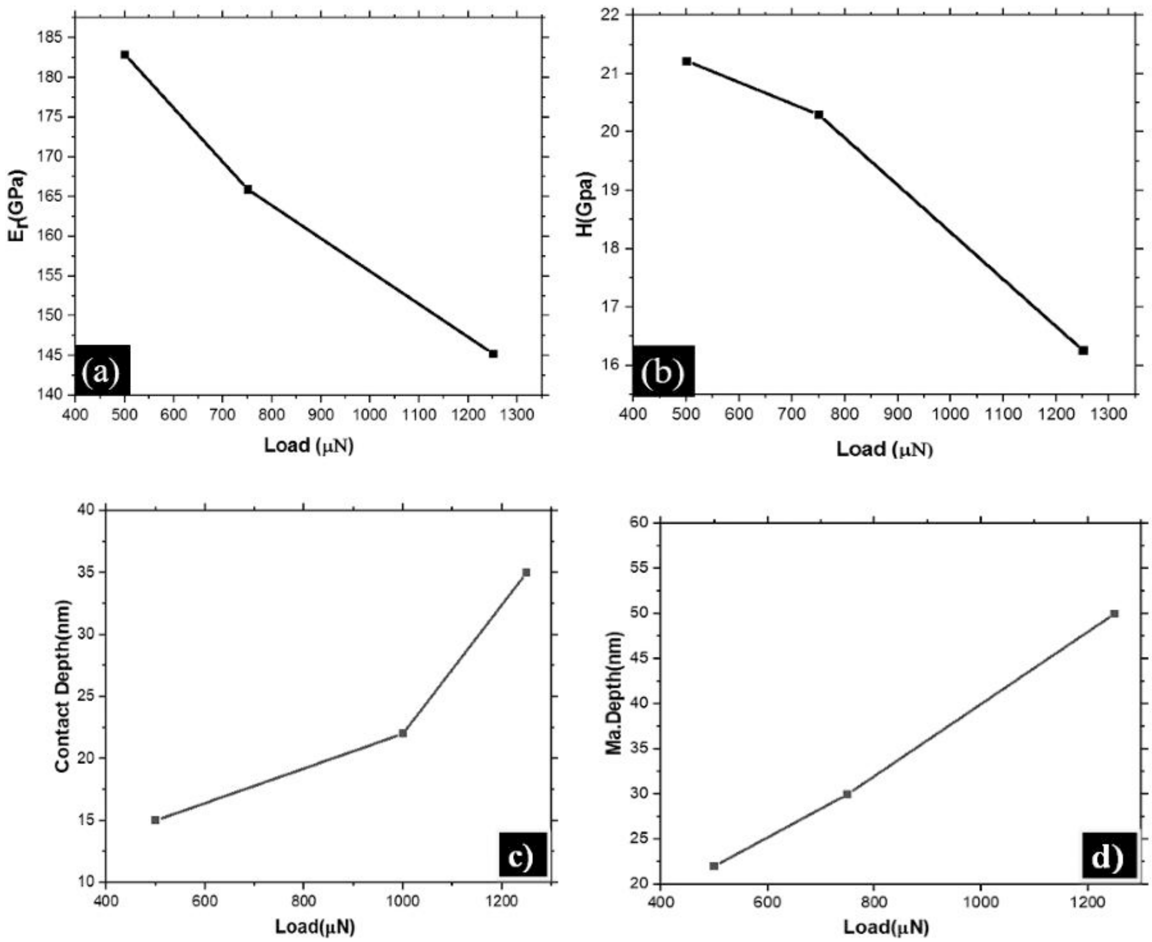

Fig. 10. Load vs Young's modulus (a), Load vs Hardness (b), load vs Contact depth (c), load vs Maximum depth (d) of Ni-Co-BN coating of load range from 500 μN to 1250μ.

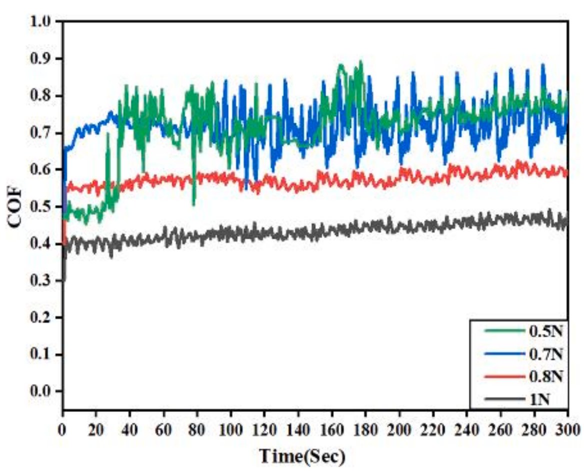

Fig. 11. Variation of coefficient of friction with time for Ni-Co-BN coating

peaks of iron, carbon, and oxygen indicate the existence of EN steel ball. This demonstrates that the oxidative wear of the EN steel ball was quite noticeable during the wear process.

The results obtained from this research study are quite good compared with the results obtained by earlier researchers [43–45]. Hence, it is evident that the Ni-Co-BN coating decreases the COF and increases wear resistance, compared with the Al-Si substrate. The Nano-wear properties of the Ni-Co-BN coating are shown in Table 3.

4. Conclusions

In this research paper, the nano-mechanical and nano-tribological properties of a nano-composite Ni-Co-BN coating, deposited on an aluminum-silicon, were determined. The conclusions drawn from the results of these research studies are given below:

- The coating hardness depends upon the wt% of BN The hardness of coating increased with an increase in the wt% of BN and a maximum hardness of 21.22 GPa was obtained with 20 wt% BN.
- The reduced modulus of coating increased with increase in wt% of BN and the maximum value of reduced modulus of 148.96 GPa was obtained with 20 wt% BN.
- The COF value decreased as the BN content in the coating increased. The lowest COF 0.4 was obtained at 20 wt% BN.
- The wear rate increased with an increase in of wt% of BN. The lowest wear rate of 4.08×10^{-5} mm^3/Nm was obtained at 20 wt% of BN. Further, the wear rate increases from 4.08×10^{-5} to 1.6×10^{-4} mm^3/Nm with decrease wt% percentage of BN.

As a result, Ni-Co-BN coatings formed using the magnetic sputtering approach have improved the nano-mechanical and nano-tribological capabilities of the substrate and can be employed as a self-lubricating coating.

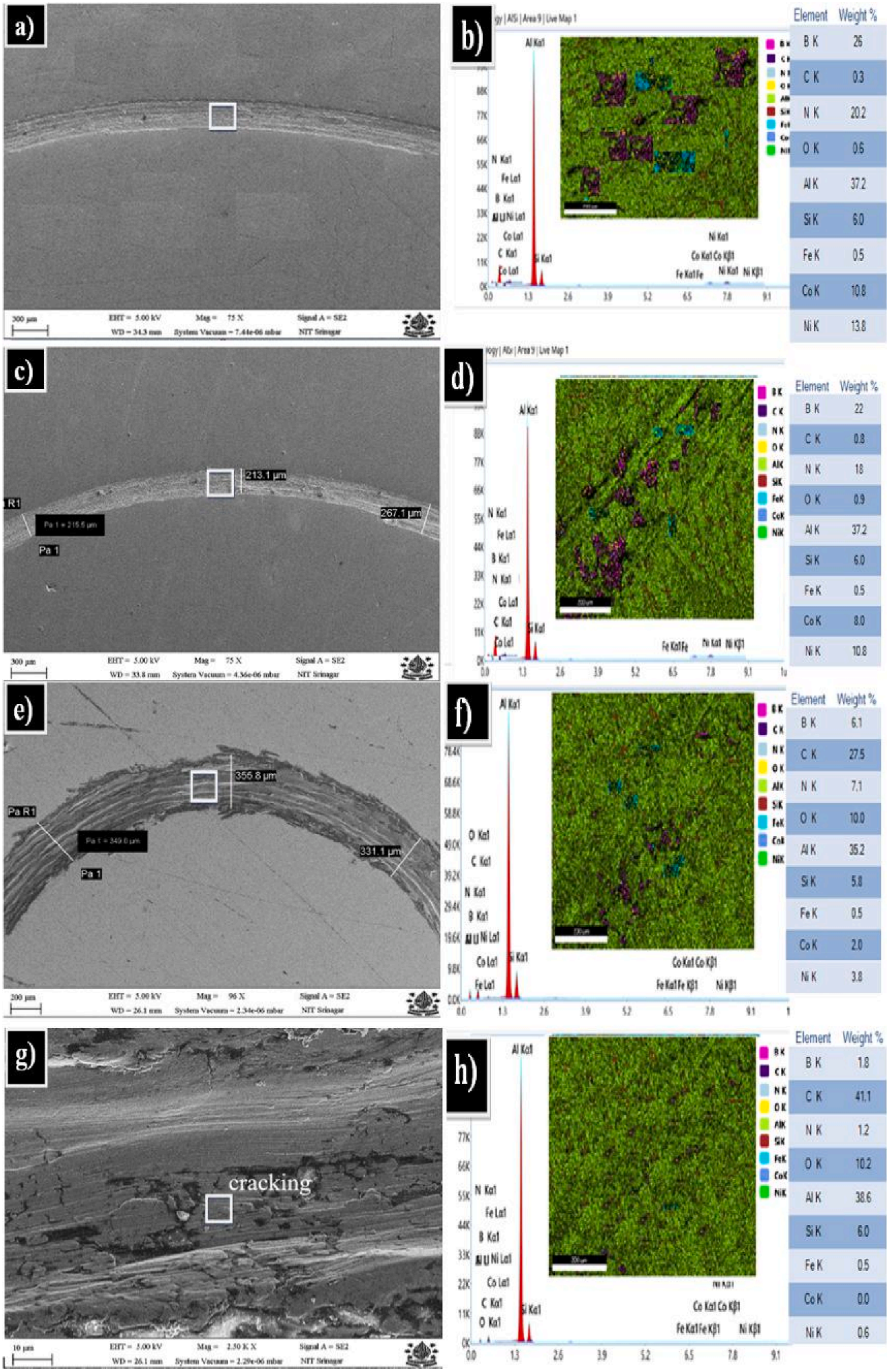

Fig. 12. FESEM and EDS images of the wear tracks at (a,b) 0.5 N (c,d) 0.7 N (e,f) 0.8 (g,h) and 1 N.

Table 3
Nano-wear properties of Ni-Co-BN Coating on aluminum-silicon (Al-Si) substrate.

S.NO	Load (N)	COF	Wear rate (mm^3/Nm)
1	0.5	0.8	4.08×10^{-5}
2	0.7	0.7	8.08×10^{-5}
3	0.8	0.5	1.20×10^{-4}
4	1	0.4	1.608×10^{-4}

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

No data was used for the research described in the article.

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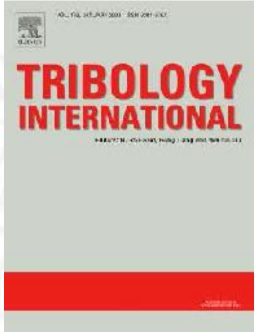

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