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Research Article

# Development and characterization of Ti-Nb-N coatings on stainless steel using reactive DC magnetron sputtering

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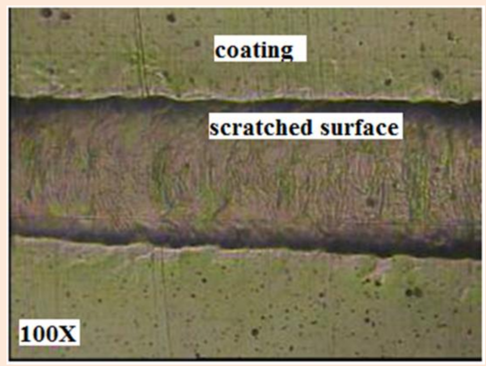
## Abstract
Thin film ceramic coatings are extensively used to improve the surface properties of the components especially in aerospace applications, surgical, tool industry, artificial implants etc. To enhance the surface properties of stainless steel, the substrate was coated with a 1μm thick coating of Ti-Nb-N by reactive DC magnetron sputtering at different $N_2$ flow rates, substrate biasing and Nb-Ti ratio. The coated samples were evaluated by the following techniques: hardness by Knoop micro-hardness tester, phase analysis by X-ray Diffraction (XRD), compositional analysis by Energy Dispersive X-ray Spectroscopy (EDS) and adhesion by scratch test. The micro-hardness test yielded appreciable enhancement in the surface hardness with the highest value being1450 HK. Presence of three prominent phases namely NbN, $Nb_2N_3$ and TiN resulted from the XRD analysis. EDS analysis revealed the presence of Ti, Nb and Nitrogen. Adhesion was evaluated on the basis of critical loads for cohesive ($Lc_1$) and adhesive ($Lc_2$) failures with values varying between 7–12N and 16–25 N respectively, during scratch test for coatings on SS substrates.

**Keywords:** Coating, Sputtering, Microhardness, Energy Dispersive X-ray Spectroscopy, X-ray Diffraction, Scratch

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## Introduction

Functional properties of a material can be enhanced by protective coatings. Protective layer impart corrosion resistance, oxidation resistance, wear resistance, low friction coefficient and high surface hardness. Protective layer modifies the surface properties and maintains its stability in severe service conditions thereby increasing the life of the components [1-3].

Transition metal nitrides of binary systems are widely used as protective coatings [4]. Two such coatings namely TiN and NbN are widely used because of their favorable mechanical properties and high corrosion resistance [4]. TiN coated component combines high load bearing capacity, high wear resistance and good fatigue strength [5]. Ti-based hard nitride coatings are being used for various applications [6, 7]. NbN exhibit good mechanical properties coupled with high wear resistance, high temperature stability, chemical inertness, high melting point and good electrical conductivity [8, 9] and therefore used for field emission cathode [10], protective surface coating [11] and diffusion barrier in microelectronic devices [12]. Few studies have been performed on the ternary Ti-Nb-N ceramic coating deposited by reactive magnetron sputtering on various aspects such as texture, structure, properties [13-15]. To this date, the properties of Ti-Nb-N coatings, deposited by reactive D.C. magnetron sputtering, have not been evaluated on SS substrate. Thus the objective of this paper is to report the results of characterization of these coatings on SS substrate and compare them with the results from previous studies so as to provide the conditions for a founded choice of their applications.

Coatings are deposited by various techniques which include physical vapor deposition (PVD) [16], chemical vapor deposition (CVD) [17], thermal spray coating [18], vacuum plasma coating [19], laser surface alloying [20] etc. Commonly used physical vapor deposition techniques are reactive magnetron sputtering [21, 22], cathodic arc deposition [23], ion beam assisted deposition [24, 25] and pulsed laser deposition [26].

In the reactive DC magnetron sputtering target metal is sputtered in the presence of a gas or mixture of gases that will react with the sputtered metal to form a protective layer [27-29]. Application of magnetron sputtering is increasing in the areas including hard, wear-resistant coatings, low friction coatings, corrosion-resistant coatings, decorative coatings and coatings with specific optical or electrical properties [30].

In the present study, hard ceramic Ti-Nb-N coatings were deposited on stainless steel (SS) substrates using reactive DC magnetron sputtering by varying the process parameters. Thin film coatings formed on the surface of SS has been characterized by its micro-hardness, scratch-adhesion, phases present and composition. Surface hardness was determined by Knoop micro-hardness tester. Knoop hardness number (HK) is the ratio of applied load (kg) and unrecovered projected area of indentation ($mm^2$) [31]. Phase identification was done by X-ray diffraction (XRD) which is based on the Bragg's law [32]. Scratch test was carried out with a Rockwell diamond indenter at various loads. The effect of loading was studied and critical loads for cohesive and adhesive failures were observed. Depth of penetration was also studied during scratch test. Scanning Electron Microscope (SEM) image was produced by scanning the surface with a focused beam of electrons. Various signals were detected by the interaction of electrons with atoms in the coated samples which contain information about the sample's composition. Secondary electron imaging was used due to its very high resolution [33]. Elemental analysis was carried out by Energy Dispersive X-Ray Spectroscopy (EDS). As the energy of the X-rays is characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they were emitted, this allows the elemental composition of the specimen to be measured [34].

## Experimental

AISI SS-304 substrate was used for the deposition of Ti-Nb-N ceramic coatings. The substrate of dimensions 40 mm ×25 mm ×2 mm and 100 mm ×100 mm ×0.2 mm were used. Deposition of Ti-Nb-N film was carried out by reactive DC magnetron sputtering using Ti-Nb (with varying surface area ratio of Nb-Ti mentioned in Table 1) target of 160 mm diameter and 4 mm thickness. The distance between target and substrate was kept constant at 60mm.Chamber was evacuated to a base pressure of $2\times10^{-6}$ mbar. Pressure during deposition was kept at $5\times10^{-3}$ mbar by admitting high purity argon (Ar) and nitrogen ($N_2$) gases into the chamber. Flow of Ar gas was kept constant at 20 cc/min and $N_2$flow was varied between 7.0-16.5 cc/min. Power to the target was supplied through a stabilized D.C. power supply of 0-1000V (6A maximum). Substrate bias was kept at 100V for four samples(A,B,C,D) and 50V for one sample(E) (keeping the $N_2$/Ar flow ratio constant at 25%) by means of a stabilized D.C. power supply of variable voltage (0-300V) and current (0-700mA). Five samples were prepared by varying the process parameters: ratio of Nb-Ti target (surface area), biasing (Volts), nitrogen flow (cc/min)and sputtering current (mA). Each sample was coated with a 1 µm thick Ti-Nb-N coating. The samples prepared with their process parameters can be viewed in **Table 1**.

**Table 1** Samples prepared at different process parameters.

| Sample | Target Nb-Ti ratio | Surface Biasing Volts(V) | Nitrogen flow cc/min | Current mA |
|---|---|---|---|---|
| **A** | 50:50 | -100 | 7 | 700 |
| **B** | 40:60 | -100 | 10.5 | 350 |
| **C** | 40:60 | -100 | 14 | 350 |
| **D** | 40:60 | -100 | 16.5 | 400 |
| **E** | 40:60 | -50 | 16 | 350 |

After the successful deposition of the coating by DC magnetron sputtering, the samples were characterized to determine the hardness by Knoop hardness tester, elemental composition by EDS-SEM, phases present by XRD, and adhesion by scratch test.

### *Micro-hardness measurement*

Knoop Micro-hardness tester (Future Tech FM-7 model) was used to evaluate the surface hardness of the coating. Knoop-diamond indenter was used with a load of 25gm for a dwell time of 5 seconds as per ASTM standard. The hardness measurements were performed on both the coated and the non-coated substrate. The readings were reported in HK (Knoop hardness number). Five readings on each sample were taken and the mean of these values was used.

### *Elemental Composition*

The compositional analysis of the coating was performed using scanning electron microscopy (SEM - AIS 2100 Seron Tech) at 20kV in conjugation with EDS Analysis (INCA E350). For effective analysis of the coating, the samples were cut into small dimensions (10mm×10mm) and the cross sections were cold mounted in a direction such that the coating and the substrate are distinctly visible. The mounted samples were polished.

### *Phase analysis*

XRD analysis was performed on the surface of the coated substrates to observe and analyze the phases present. XRD (make-Diano) was carried out at 20 mA and 35 kV in combination with $Cuk_{\alpha}$ radiations in routine Bragg–Brentano $\theta$–$2\theta$ geometry.

### *Scratch-adhesion Testing*

Scratch-adhesion tester (CSEM, Revetest) was used to evaluate the adhesive strength of the coatings. The scratch test was performed on the surface of the coated substrate. The scratch was carried out in a progressive mode with linearly increasing load from 0.9 N to 30 N with a loading rate of 30 N/min. The scratch indenter used was a Rockwell diamond indenter with a spherical tip of radius of 200µm.The coating was scratched for a length of 3mm with a scratch speed of 3.09 mm/min.

## Results and Discussions

### *Micro-hardness*

The hardness results obtained were as shown in **Table 2**.

**Table 2** Representing the hardness results obtained along with the process parameters

| Sample | Nitrogen flow Cc/min | Hardness (HK) |
|---|---|---|
| **A** | 7 | 920 ±2 |
| **B** | 10.5 | 1125 ±3 |
| **C** | 14 | 1280 ±1 |
| **D** | 16.5 | 1450 ±2 |
| **E** | 16 | 1406 ±1 |

The hardness for the non-coated stainless steel substrate was 290HK.The average value of the HK for each of the samples is seen from the table 2. It was observed that the hardness increased with increase in the nitrogen flow as can be seen in the **Figure 1**. The maximum hardness observed was 1450HK at a $N_2$ flow rate of 16.5cc/min. The changes in hardness values are attributed to changes in grain size, phase, stoichiometry, residual stress or appearance of texture [35]. There was appreciable increase in the hardness of the substrate after its coating with Ti-Nb-N. There has been no previous hardness evaluation of Ti-Nb-N coatings specifically. Although there have been studies with respect to surface hardness of ternary systems like Ti-Al-N by one of the current authors [36]. In addition to this, one of the current authors has worked on hardness evaluation of Magnetron Sputtered NbN Films with Nb Interlayer on SS as well as mild steel [37].There was an appreciable increase in hardness with NbN films with Nb interlayer [37] which broadens the scope of research in Ti-Nb-N coatings with a metal interlayer.

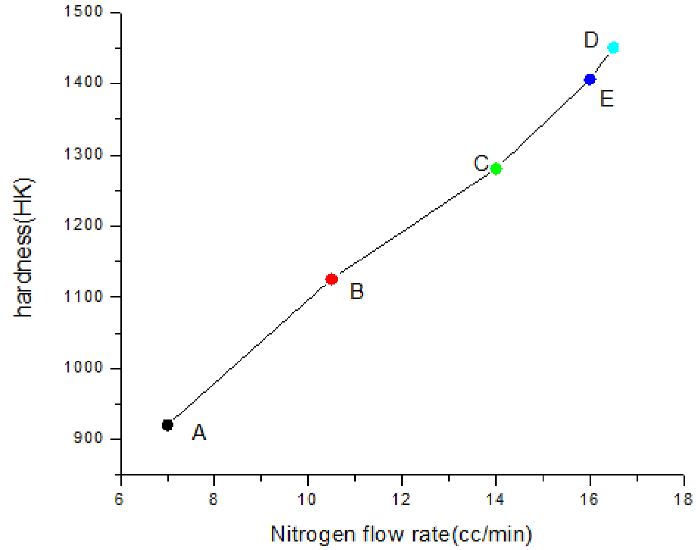

**Figure 1** Variation of hardness with the increasing nitrogen flow

***SEM-EDS (compositional analysis)***

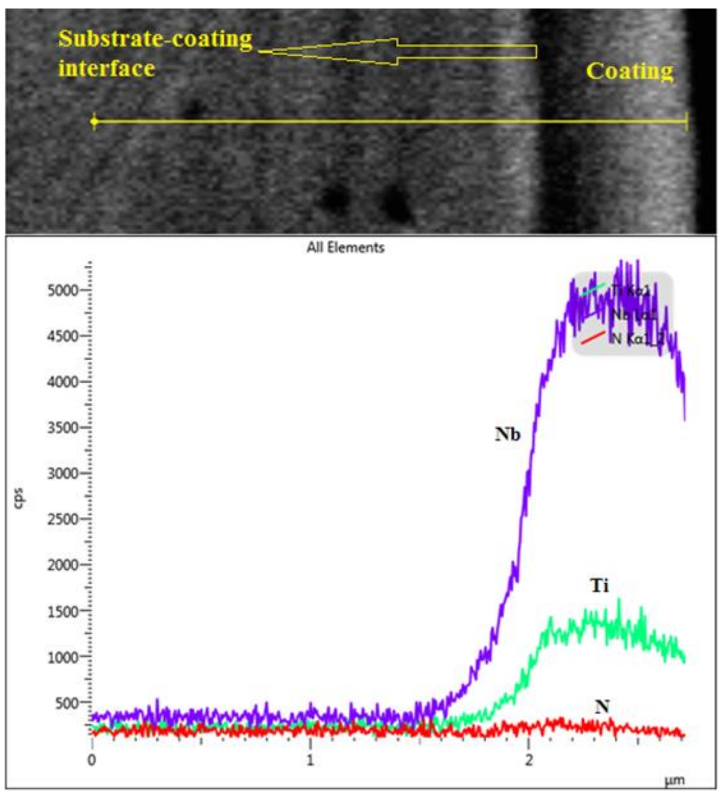

**Figure 2** SEM micrographs at 1000X

Line scan of the laterally cold mounted samples was performed individually. However, the results of only representative samples are shown. The line scan shown in the **Figures 2** and **3** are of sample (C). The scans were carried out at 1000X and 1500X magnification respectively. The coating is very well distinguished from the substrate and the mold material in the SEM images presented in the Figures 2 and 3. There is a significant rise in the composition of Ti and Nb as the coating is scanned which is clearly depicted in the SEM micrographs in the Figures 2 and 3. At the same time it can be observed that the iron content decreases steeply from the substrate end at the interface of the substrate and coating. The composition of Ti, Nb, N decreases steeply at the coating mold interface as depicted in Figure 2.The micrographs shown in the Figures-2 and 3 depict that the coating is composed of Ti, Nb and N. The qualitative analysis of elemental composition showed the presence of Ti, Nb and N for all the samples irrespective of the variation in nitrogen flow rate.

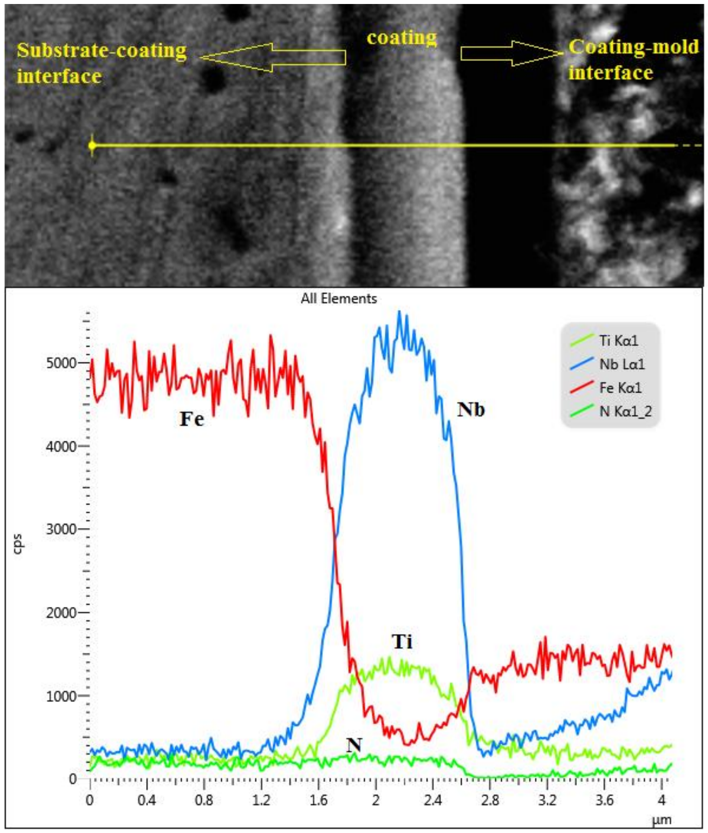


**Figure 3** SEM micrographs at 1500X

***XRD***

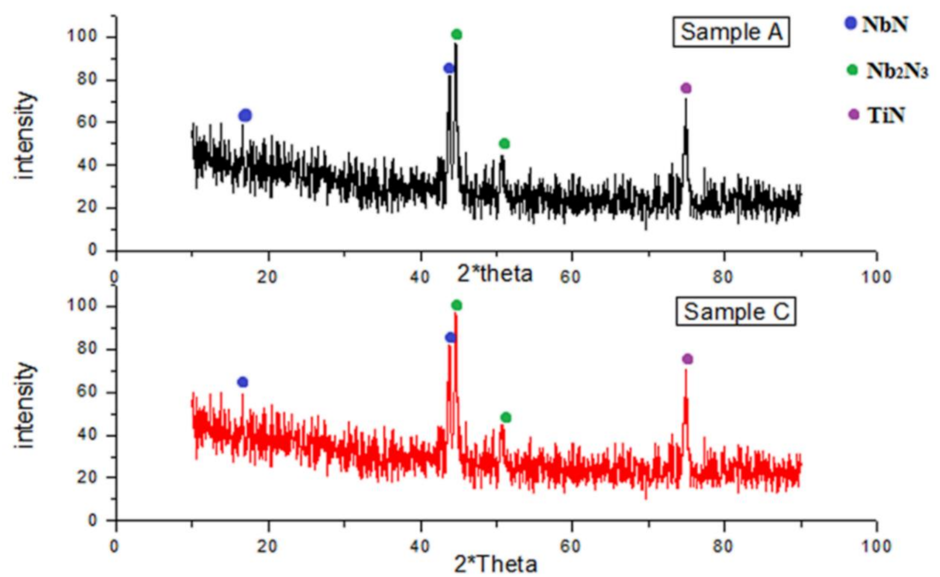


**Figure 4** The XRD pattern for samples depicting phases present

X-ray diffraction was carried out on the surface of the coated SS substrates to find the phases present in the coating. Mainly three phases were found in significant amount: NbN, $Nb_2N_3$ and TiN. A representative analysis is depicted in **Figure 4** for samples A and C. It was observed that all the peaks produced by the phases lie between the references of NbN and TiN which is coherent with the results of previous experiments with Ti-Nb-N coatings [38]. Also previous studies on Ti-Nb-N coatings have mentioned significant presence of NbN and TiN in the XRD evaluation which is again coherent with the results obtained in this study [38, 39].

***Scratch-adhesion testing***

Friction force and depth of indentation for all the scratch tests were recorded online along with the indenter movement to check the critical loads for cracks, chipping, delamination, coating failure, and any other phenomena. Tests performed at different loading rates were observed to give almost similar results, and variation in loading rate had little impact; therefore, loading rate for scratch tests was kept at 30 N/min. The above mentioned observations were revealed as the scratch progressed, such as upper layer removal, pile-up on the sides, visibility of small cracks to long wide cracks within the coatings, pores, chipping, and partial or complete delamination of the coating. **Figure 5** exhibits the scratch patterns for Ti-Nb-N coating on SS (sample **C**) taken at various loads. Start of the scratch is shown at 1N load; at 10.5 N load, segregation, cracks within coating and pores were visible though coating was still intact; at 19N load, delamination at few places was observed along with pores, chipping and pile up; at 29.3N load, coating delamination was observed at many places.

There are two critical loads stated for the failure of the coatings namely, $Lc_1$ and $Lc_2$.The first critical load i.e. $Lc_1$ signifies initial cohesive failure of the coating which can be attributed to the appearance of initial small cracks within the coating. At this point of time the coating remains intact without any delamination. The second critical load $Lc_2$, corresponds to primary adhesive failure of the coating, which can be attributed to the first observation of adhesive failure such as chipping, partial delamination, pores, or some phenomena, where substrate beneath coating is exposed. For coatings deposited on SS substrates $Lc_1$ varied between 7–12N and $Lc_2$ between 16–25 N.

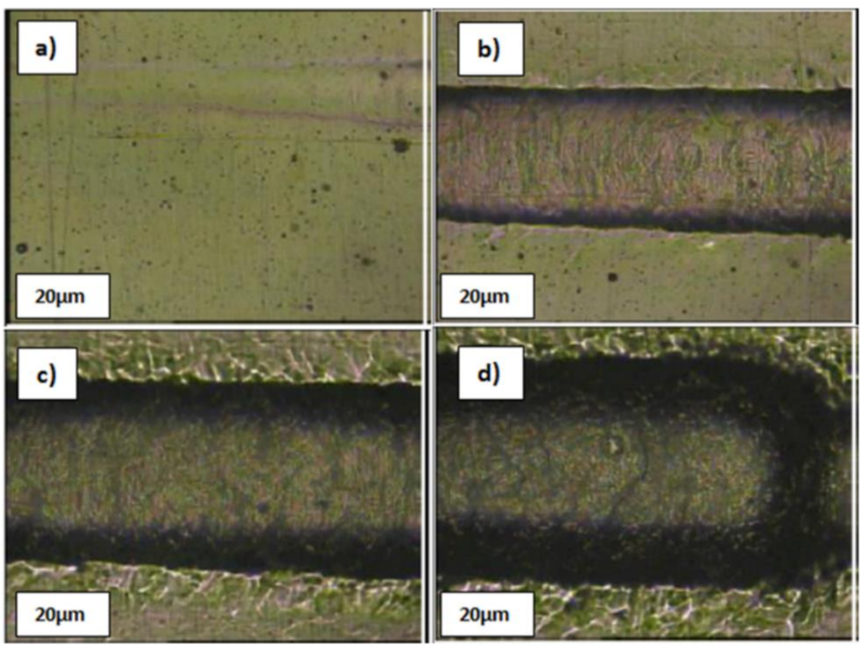


**Figure 5** Scratch test for Ti-Nb-N coating on SS (sample C) at (a) 1N showing initiation of scratch, (b) at 10.5N depicting segregation, cracks and pores, (c) 19N revealing chipping, cracks, pores, pile-up, and (d) 29.3N, showing chipping, cracks, pores, pile-up, and delamination

Coefficient of friction ($\mu$) was observed to increase with the increase in scratch load. $\mu$ represents the value for the combination of coatings and substrate. This increase in $\mu$ is attributed to the increasing contribution from the substrate. For the coated substrates, the value was found to vary within a narrow range of 0.23–0.26 at 30N (as can be seen from the **Figure 6** for sample C) load irrespective of deposition at different $N_2$ flow rates.

Depth of penetration increased with the increase in applied load as can be seen from **Figure 7** (Sample C). At 30N load, on an average, the coated SS samples had 10-12$\mu$m depth of penetration for different nitrogen flow rates. The change in nitrogen flow rates had no significant effect on the penetration depth. These results can be compared with the results obtained for SS substrates coated with NbN so as to know the relatively better coating in terms of abrasion resistance. The variation in depth of penetration is much less in Ti-Nb-N coating as compared to NbN coating with Nb interlayer which was found to be 12-25$\mu$m [37]. However, the scope of error in such testing may rise from factors such as natural slopes of the samples (thickness variation in sample) and mounting errors.

Acoustic emission signals depicted small peaks wherever cracks occurred and big peaks where coating delaminated. For brittle coatings the height of the peaks increased. Acoustic emission signals (shown in **Figure 8**) confirmed the critical loads for various failure events as observed in the scratch micrographs in Figure 5.

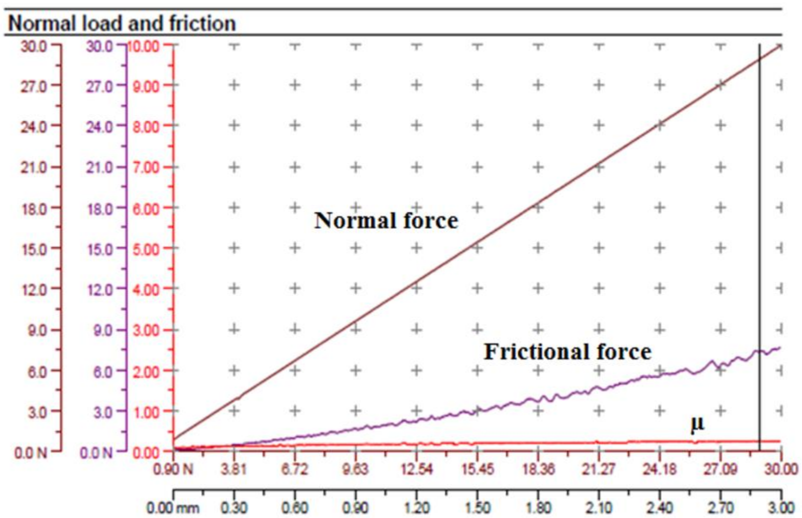


**Figure 6** presenting coefficient of friction ($\mu$) vs. load (scratch length), normal force vs. load (scratch length) and frictional force vs. load (scratch length) for sample C

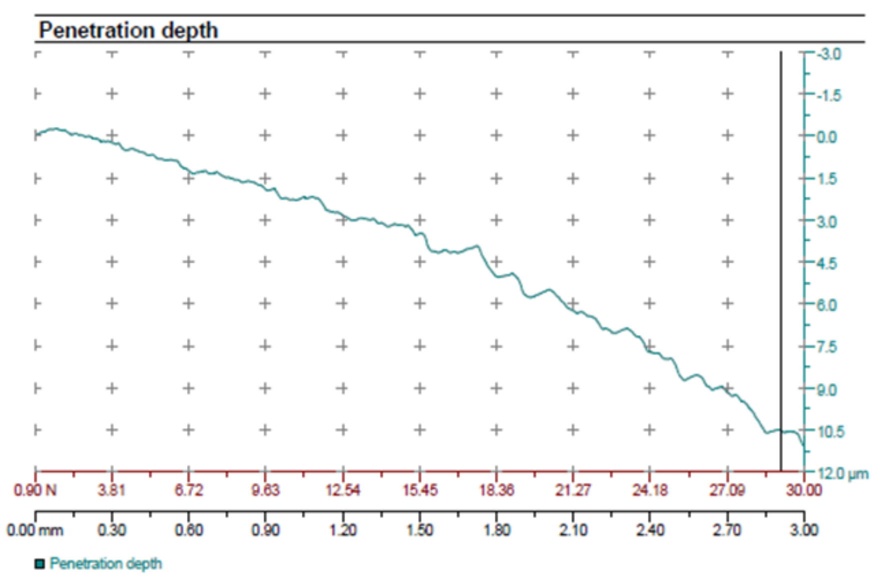


**Figure 7** representing the variation in depth of penetration with increase in load (scratch length) for sample C

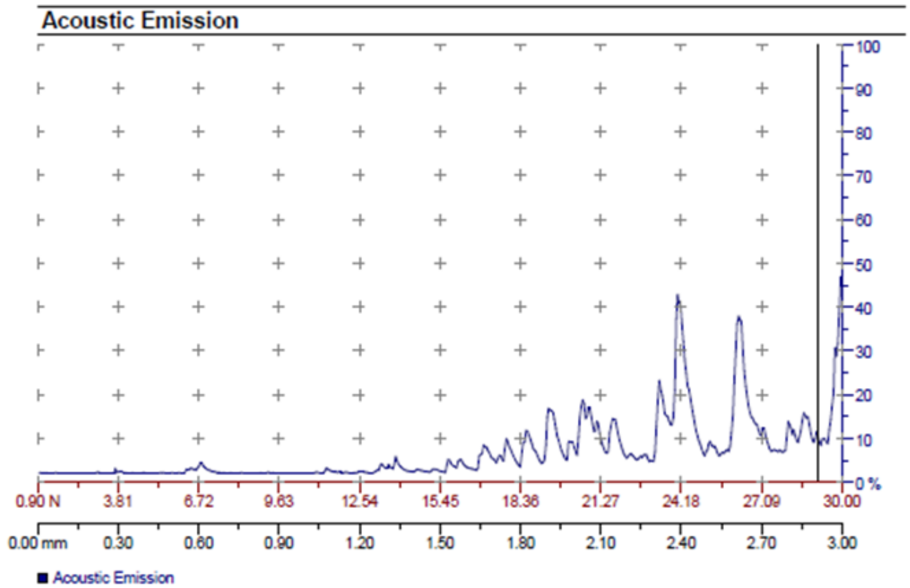


**Figure 8** representing the acoustic emission graph for the scratch test of sample C

## Conclusion

Ti-Nb-N coatings were deposited on SS substrates by reactive DC magnetron sputtering. Nitrogen flow rate was varied from 7cc/min to 16.5cc/min. Coatings were characterized for their hardness by Knoop micro-hardness tester, phase analysis by X-ray diffraction technique, and adhesion by scratch tester. The effect of $N_2$ flow was evaluated. Effect of Ti-Nb-N coating on SS substrate was studied for the improvements in surface hardness by Knoop micro-indentation and adhesion by scratch test. The following results were inferred:

- Surface hardness for the coated substrates increased with increase in nitrogen flow. Surface hardness on SS reached a maximum of 1450HK25 at a $N_2$ flow rate of 16.5cc/min.
- SEM-EDS analysis shows the presence of Ti, Nb and Nitrogen in the coating in a uniformly distributed fashion over a range of 1$\mu$m.
- XRD analysis reveals the presence of three prominent phases namely NbN, $Nb_2N_3$ and TiN.
- Critical loads for cohesive ($Lc_1$) and adhesive ($Lc_2$) failures during scratch test for coatings on SS substrates were between 7–12N and 16– 25 N, respectively.
- Coefficient of friction ($\mu$) during scratch test was 0.23–0.26 at 30 N load for coatings deposited on SS samples. Increase in loading rate had little impact on critical loads.
- Further Tribological studies can be done to measure the wear rate and wear volume.
- There is a scope for high temperature testing of such coatings on SS.

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