STUDY OF CHARACTERIZATION OF TiN AND TiAlN COATED SS 304 BY PHYSICAL VAPOUR DEPOSITION

Miss. Hiteshri Jadhav[1], Mrs. Pina Bhatt [2], Mr. Parthiv Trivedi[3]

[1] *ME Scholar,Mechanical Department, Shankersinh Vaghela Bapu Institute of Technology, Gujarat, India*
[2] *Phd Scholar,Mechanical Department, R. K University Rajkot, Gujarat, India*
[3] *Assistant professor, Mechanical Department, Shankersinh Vaghela Bapu Institute of Technology, Gujarat, India*

ABSTRACT

Among many coatings being since decades, hard coatings are the one that are engineered and designed for various wear applications. Coatings substantially increase the life of tools with increased cuttings speeds and productivity in various cutting applications. Coating applications have spread in a wide range in industrial applications of which here we have used Physical Vapour Deposition technique.
The material to be used as the substrate is the stainless steel 304 on which coatings of TiN and TiAlN will be carried out by PVD process. The chemical and mechanical tests will also be done to know the percentage of metals present and its initial properties respectively. Microstructural characterization is the key point of focus here and will be done by Field Emission Scanning Electron Microscopy (FESEM), X-Ray Deposition (XRD), Energy Dispersive Spectroscopy (EDS).
The intended conclusion is that the various analysis will lead to disclose the improvements and changes in the microstructural properties of the substrate material on applying coatings.

Keyword: - *coatings, Physical Vapour Deposition, FESEM, EDX, XRD*

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1. INTRODUCTION

Now a days hard coating has found its application in almost every field, and is a basic feature of any mechanical component. On applying hard coatings on the substrate material its performance increases as a component and also improved properties like mechanical, magnetic, optical, tribological etc.[1] Several remarkable developments in the coating equipments and processes resulted in allowing us to produce a wide different ranges of nitridic and oxidic films as monolayer, multilayer or composite coatings. Amongst them nitride based hard coating find increasing use because of their excellent properties like high hardness, wear resistant, thermal stability and corrosion resistant. Development of PVD coating which are of low friction, thermally stable and wear resistant has drastically lowered the excessive use of coolants being used. Due to their superior mechanical and tribological properties hard coatings can substantially enhance the tool’s performance. Cr-based coatings are widely used in industrial cutting tools, friction parts and moulds as they give excellent overall performance. Although Cr-based coatings are just a few microns to the ten of microns, this indeed helps in obtaining the wear resistance, fast superior hardness and other key properties.[2]

Hard coatings prepared by various deposition techniques and conditions exhibit the widest variety of microstructures among materials in terms of grain size, crystallographic orientation, lattice defects, texture, and surface morphology as well as phase composition**.** [3] Engineering components should have superior material properties in the erosive and uncontrolled severe environments. So to extend the properties of the base material coating is a good option these days to be used a protective covering or an shield which improves the properties of the base metal. There a wide variety of commercially available hard coatings in the market for varied applications. Below given is the flow chart of the hard coating techniques:

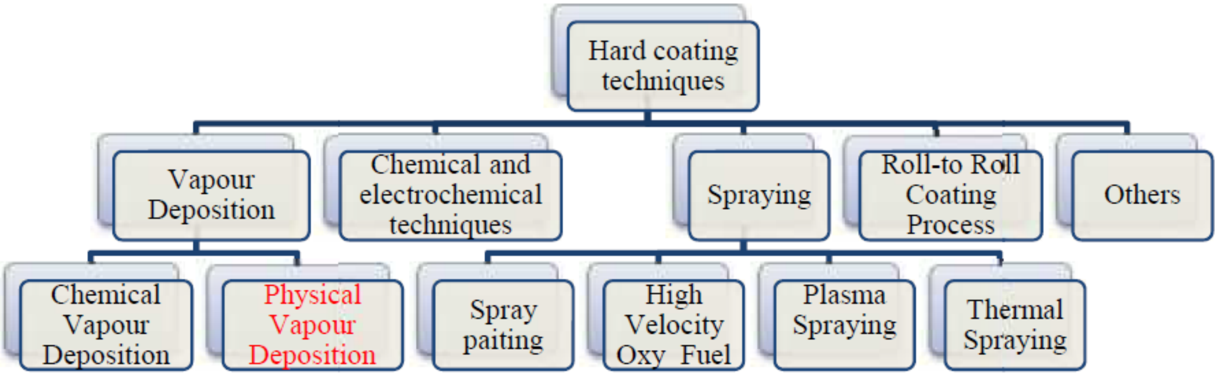


Fig 1 Classifications of Hard Coating Techniques

From the above mentioned coating techniques, we have focused upon the Physical Vapour Deposition.

1.2 PHYSICAL VAPOUR DEOPSITION

Physical Vapour Deposition (PVD) is the variety of some of the vacuum deposition methods by which very thin films were deposited onto various workpiece or material by condensation of the vapourized form of desired film material. Rather than using chemical reaction as in chemical vapour PVD process contains purely physical processes such as plasma sputter bombardment, high temperature vacuum evaporation, combined magnetron and arc process by providing subsequent condensation. These processes are used to form coatings to alter the mechanical, electrical, thermal, optical, corrosion resistance, and wear properties of the substrates. The transition of the metallic components can take place in two ways from solid to vapour phase i.e. either by heating of an evaporation source or sputtering of a target. Cathodic arc and magnetron sputtering techniques allow evaporation of metals with different melting points such as Ti and Al. The PVD arc evaporation process employs higher energy input than the PVD sputtering Process.[4]

1.3 PVD PROCESS

It is basically a vaporization process in which normally mechanism takes place such as atom to atom transfer of materials from the solid form to vapour form then again in solid form which results in gradually building a thin film onto desired workpiece. There are several process taking place in PVD technology i.e ion plating, evaporation and sputtering. Each technology will provide different concentration of film coating according to the desired intensions using unique equipments. The three fundamental steps includes:-

1. Vapour phase generation from coating material stock by -
   - Sputtering
   - Chemical vapours and gases
   - Evaporation
   - Arc Vaporization
2. The transfer of the vapour phase from source to substrate by -
   - Molecular flow
   - Vapour ionization by creating a plasma
   - Line-of-sight
3. Deposition and film growth on the substrate:

Steps can be superimposed or independent on each other depending on the characteristics of desired coating. The final result of the coating is a function of each materials individual property, any process constraints that may exist and interaction of the materials.

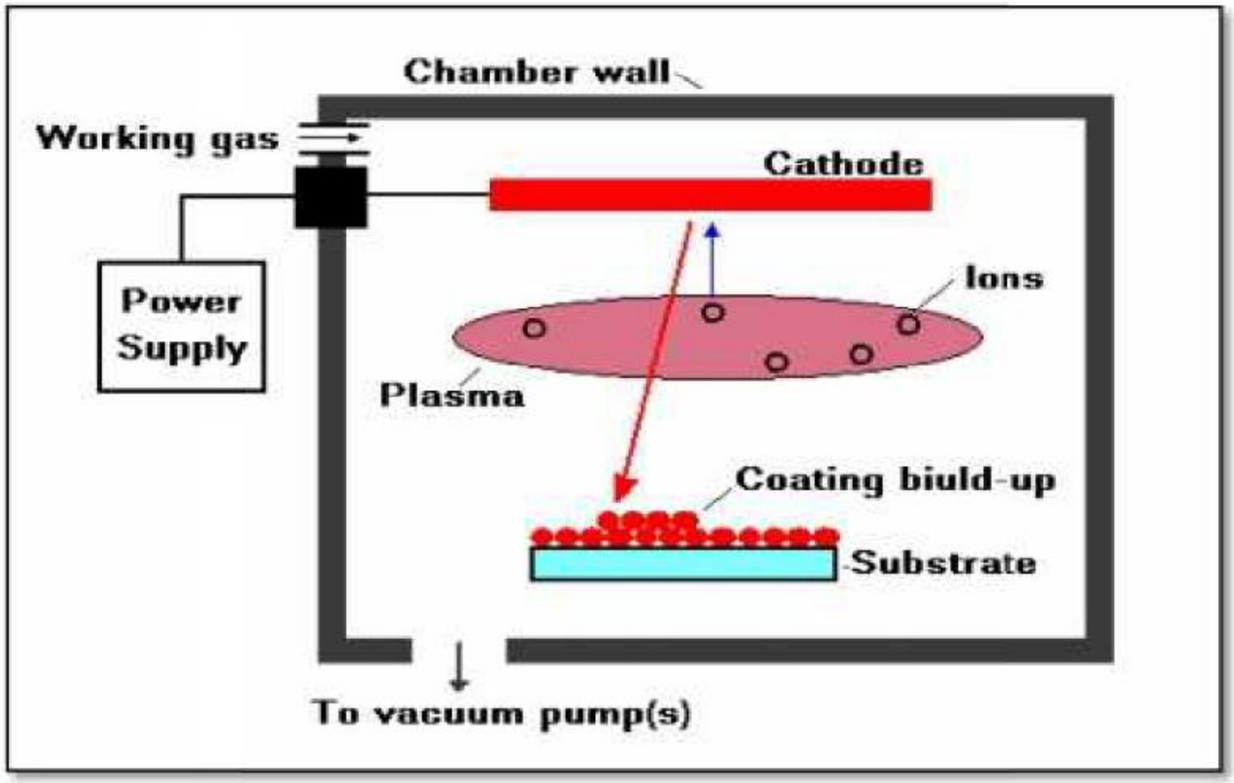


Fig 2 PVD Process[6]

1.4 TYPES OF PVD PROCESS

- Cathodic arc evaporation
- Electron beam physical vapour deposition
- Ion platting
- Sputtering
- Evaporation deposition

1.5CHARACTERISTICS OF PVD PROCESS

 Hardness: Ranging from 2000 to 3000 HV, more than twice the hardness of hard chromium. The intercrystalline compressive stress provides improved resistance against abrasive wear and corrosion.

 Friction Coefficient: The difference between the components is reduced by half when TiN or any such material is applied on to the surface. This is because of the high homogenous and dense crystalline structure.

 Surface / Chemical Affinity: Due to small inter-crystalline and inter-atomic distance, interaction of PVD coatings with substrate materials is immensly low, and as such avoids cold welding.

 Friction Coefficient: Due to the high homogenous and dense crystalline structure, the friction among the steel is reduced by half when a layer such as TiN is applied onto one of the surfaces.

 Thermal Behaviour: PVD coatings can withstand thermal shocks very well. The temperatures may rise from 400°C to 800°C under permanent operational conditions.

 Chemical Resistance: PVD coatings are resistant against most environmental and chemical influences. This provides protection against corrosive wear or deterioration.

2 EXPERIMENTAL PROCEDURE

The procedure can be divided into 3 stages:

1 Selection of substrate material:

The substrate material here selected is SS304 as it has excellent toughness better forming and welding properties. It also has good deep drawing properties and ease of cleaning and fabrication so that sample preparation is easy.

The base material is used as heat exchanger tube boilers, in dying industry, springs, nuts, bolts and screws. SS 304 is also used for heavy gauge components for improved weldability, tubing, storage and hauling tanks, piping valves etc and many more. The substrate material SS 304 has dimensions 10×10mm and 1mm thickness.

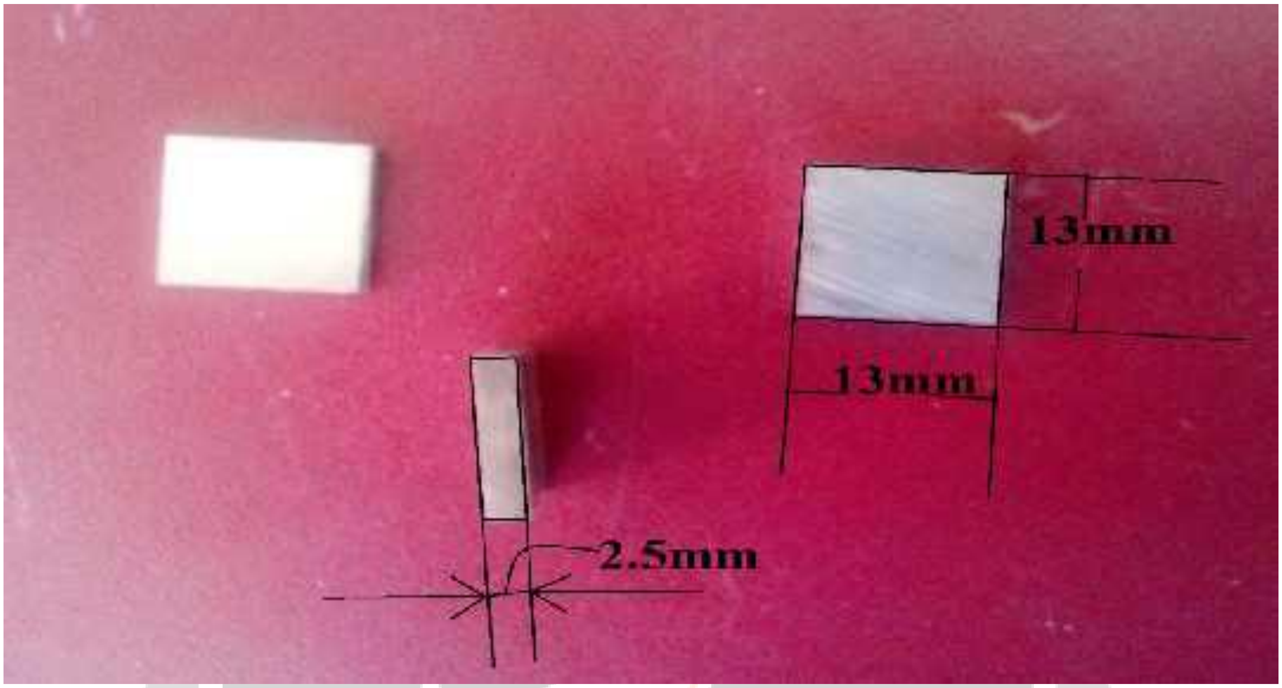


Fig 3 SS 304 material (10mm×10mm×1mm)

The below table gives the chemical compositions of the material, this details were revealed by the chemical test report of the material carried out at Test Well Laboratories Odhav Ahmedabad.

ELEMENTS PRESENT	Chromium	Nickel	Manganese	Carbon	Silicon	Phosphorous	Sulphur
COMPOSITION	18.730%	8.180 %	1.130%	0.054%	0.310%	0.027%	0.10%

Table 1: Chemical composition of SS 304

Mechanical properties of the substrate material were checked at the test well laboratories Odhav Ahmedabad.

Yield load	10.82KN
Ultimate load	21.42KN
Yield stress	284.92MPa min
Ultimate tensile stress	564.04MPa min
Material specification	ASTM, A240-2012, TYPE-304

Table 2 Mechanical properties of substrate material

2 Substrate pretreatment:

The pretreatment process used for cleaning of the substrate material is done with the help of Ultrasonic cleaning process. This process uses an aqueous solution for cleaning of precision metal parts which is based on the spraying and immersion in heated bats with ultrasonic vibration. This process is fully automatic and guarantees higher reproducibility with unsupervised operations. Later the samples are then left free after thorough rinsing and drying in hot air.[5] Its basic advantages are it permits lower consumption of energy, water and cleaning agents. It is designed in a way with ease of operation and maintenance.

3 Coating Process CAE-PVD

The coating materials here used are TiN and TiAlN to be coated on the substrate material SS 304. The coating process used is Cathodic Arc Evaporation Physical Vapour Deposition technique. The coating was carried out at Oerlikon balzers ltd India. The coating was carried out with cathodic arc evaporation process as the name suggests, this method uses and arc discharge between two electrodes to melt and evaporate material. The material to be deposited is made as the cathode and the high current, low voltage discharge melts a small spots on the surface. The cathode spot is the small melted region surface on the solid material from which current flows. At the cathode spot solid material from the cathode transforms into plasma, i.e. electrons and positively charged ions. The electrical current that flows between the electrodes is transported by the generated plasma why the arc process is self-sustained.[7] After a short period of time a new cathode spot is ignited. Composition of film can be controlled by using compound cathodes. Here Ti-Al-N and Ti-N compound cathodes were used. A flow of N2 gas is introduced into to deposition chamber. There is a provision for placing several cathodes. [8]

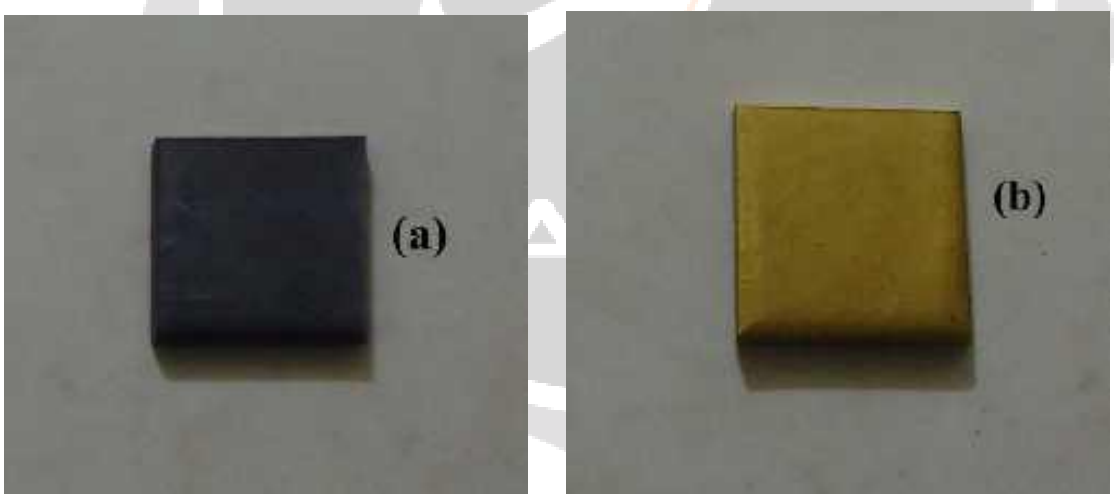


Fig 4 PVD coated TiAlN (a) and TiN (b) on SS 304

2.1 Experimental setup at industry

The machine used for deposition of coating is the front loading Balzers Rapid Coating system. The machine is equipped with eight cathodic arc sources. Then the Nitrogen was supplied to the combustion chamber. The deposition temperature range was from 25°C to 750°C. The nitrogen deposition pressure applied on coating material was 3.5Pa and the substrate bias voltage in between -40v to - 170 v and vacuum pressure was 10^{-6} torr applied such that perfect dispersion of evaporated particle on the substrate surface should be possible. The coating morphology was such that the dense and uniform microstructure should be developed which can bear the higher thermal load.

Machine name	Standard balzers rapid coating machine
Target composition	TiN and TiAlN
Power	3.6KW
Reactive gas	Nitrogen
Deposition pressure	3.5 Pa
Substrate bias voltage	-40V to -70V
Coating thickness	4µ±1µ
Vacuum generated	10^{-6} torr

Table 3 Deposition parameters

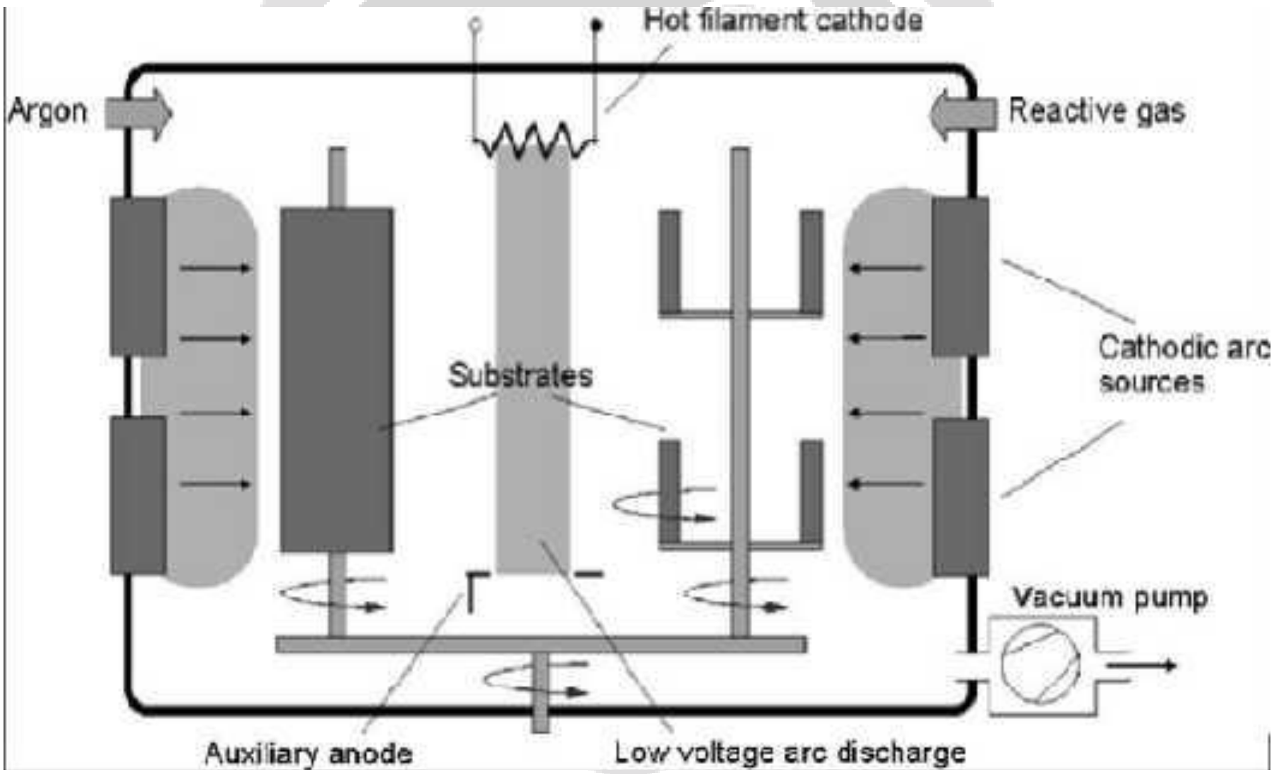


Fig5 Experimental setup of cathodic arc evaporation process

With these parameters and the above discussed processes TiN and TiAlN coatings were successfully coated on to the base material. After carrying out the coating process at the Oerlikon Balzers coating industry various characterization tests were performed to study the various improved properties and the analysis of microstructure. These microstructural characterization techniques with their results are further discussed in the next chapter.

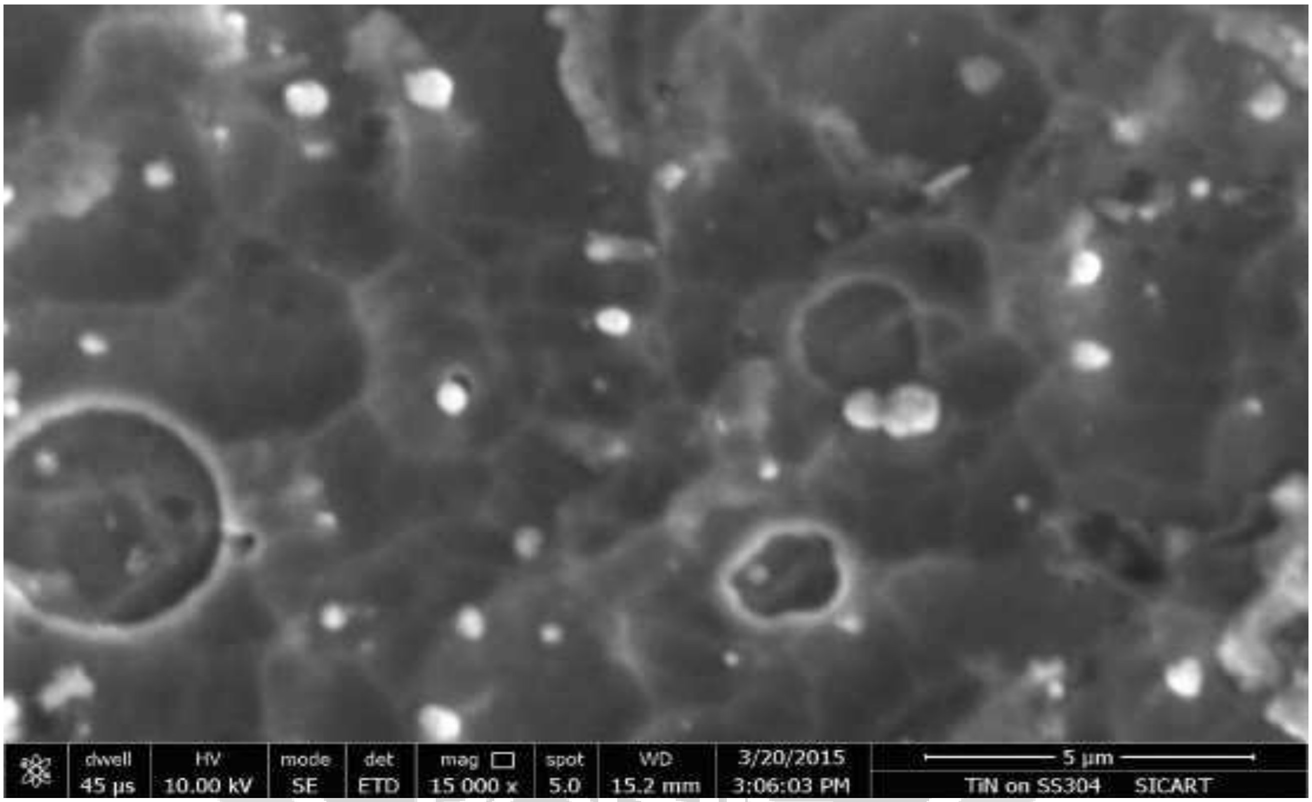
2.2 Analysis of the coated material:

The microstructural analysis of the coated SS 304 by various characterization techniques will be carried out in this stage. The techniques used will be Field Emission Scanning Electron Microscopy (FESEM), Energy-dispersive X-ray Spectroscopy (EDX), Atomic Force Microscopy (AFM) and X-ray Diffraction (XRD).

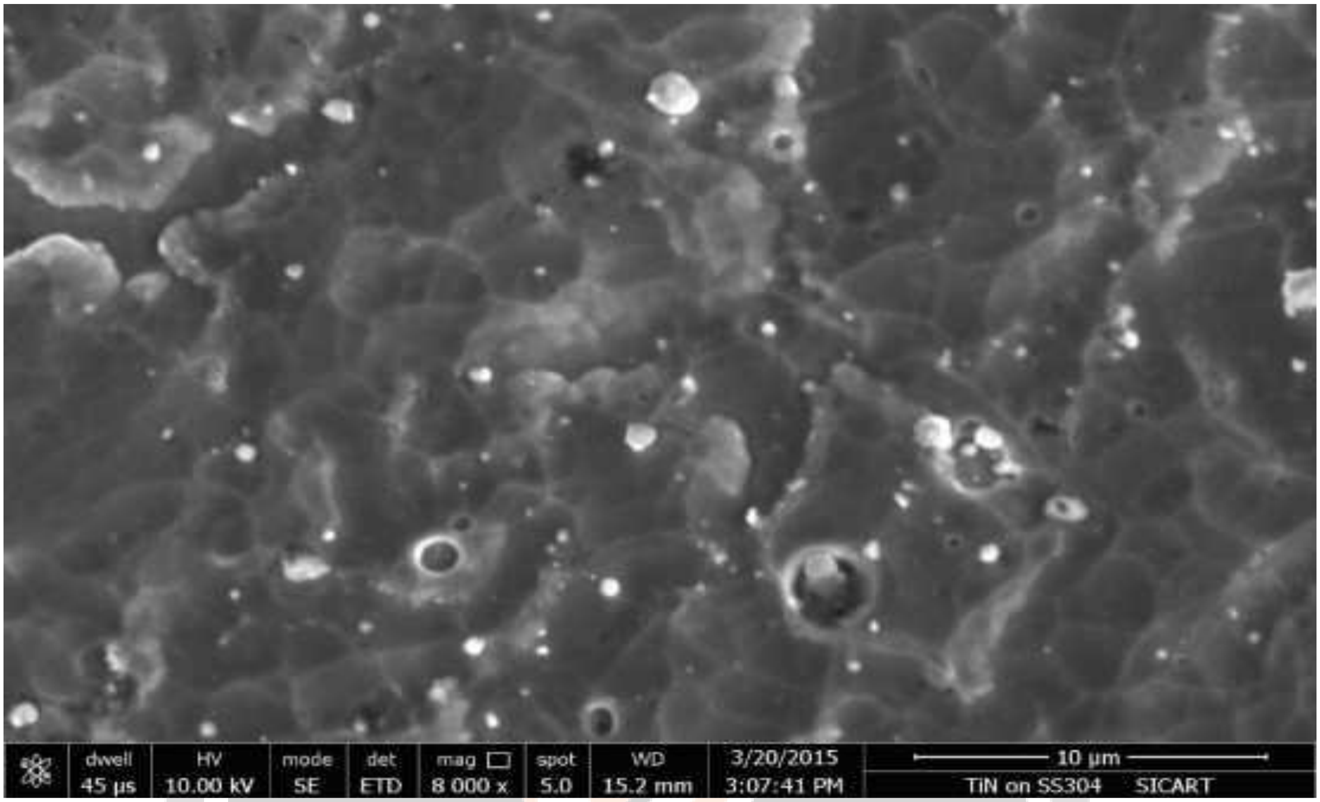
The hardness and thickness tests will also be carried out for the same. These results will lead to disclose the improved properties and characteristics of the substrate metal.

3. RESULTS AND DISCUSSION

3.1 FESEM analysis



(a)



(b)

Fig 6 FESEM image of PVD coated TiN on SS 304 at 5µm (a) and 10 µm(b)

The FESEM result of PVD coated TiN on SS 304 shows the presences of the coating material TiN as the small round white spots in the image. The image of the material was taken at various magnifications but the above image gives a clear idea about the presences of the coating particles and the grain boundaries of the base metal. This image was taken at 15000x magnification.

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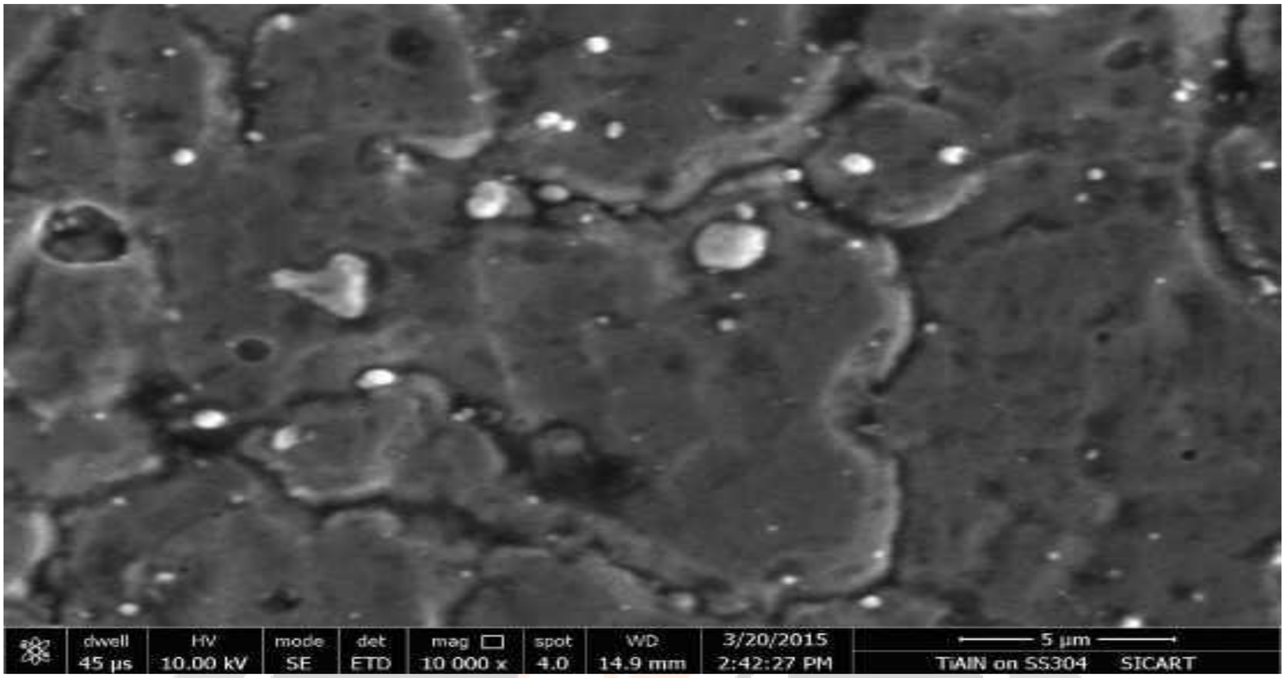
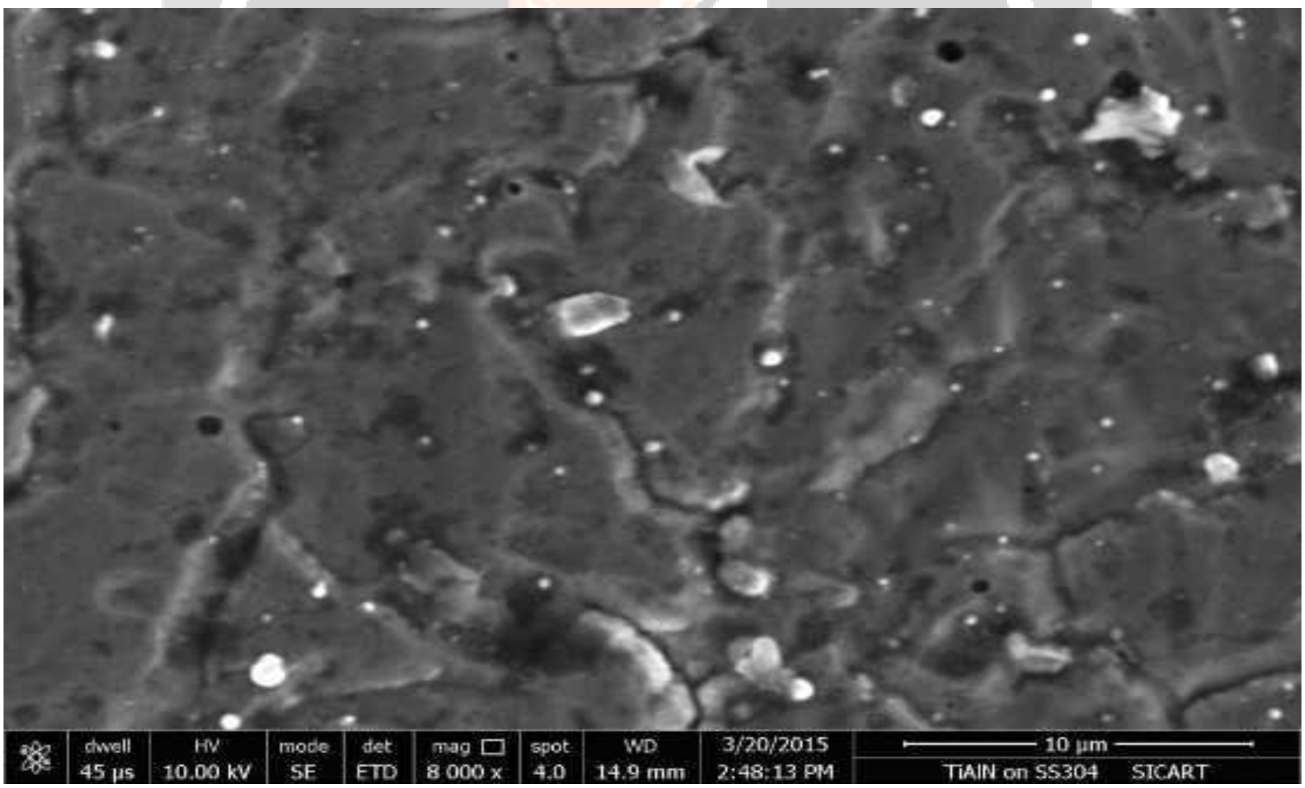


(a)



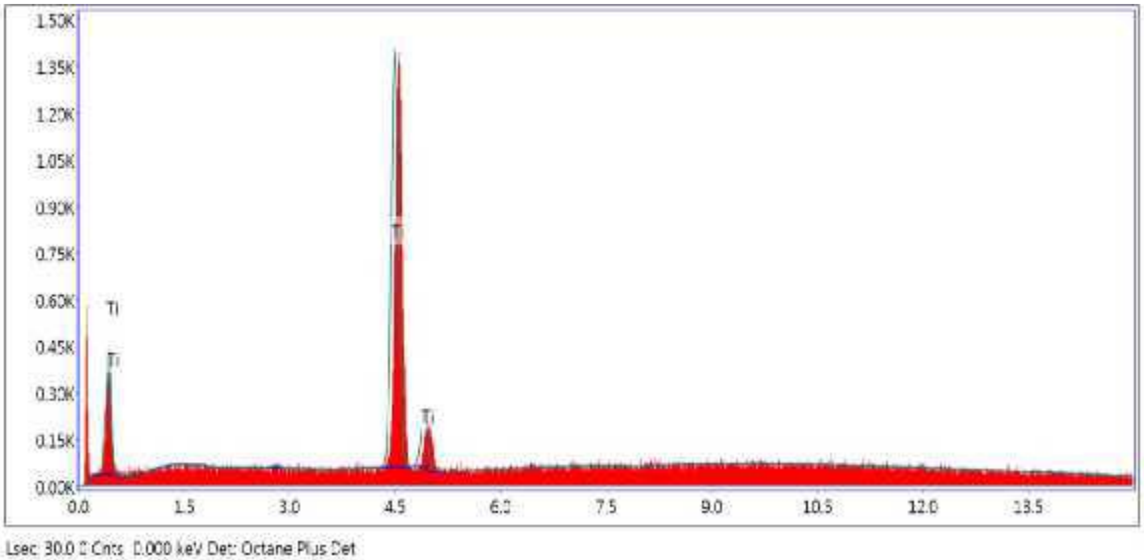
(b)

Fig 7 FESEM image of PVD coated TiAlN on SS 304 at 5µm (a) and 10 µm(b)

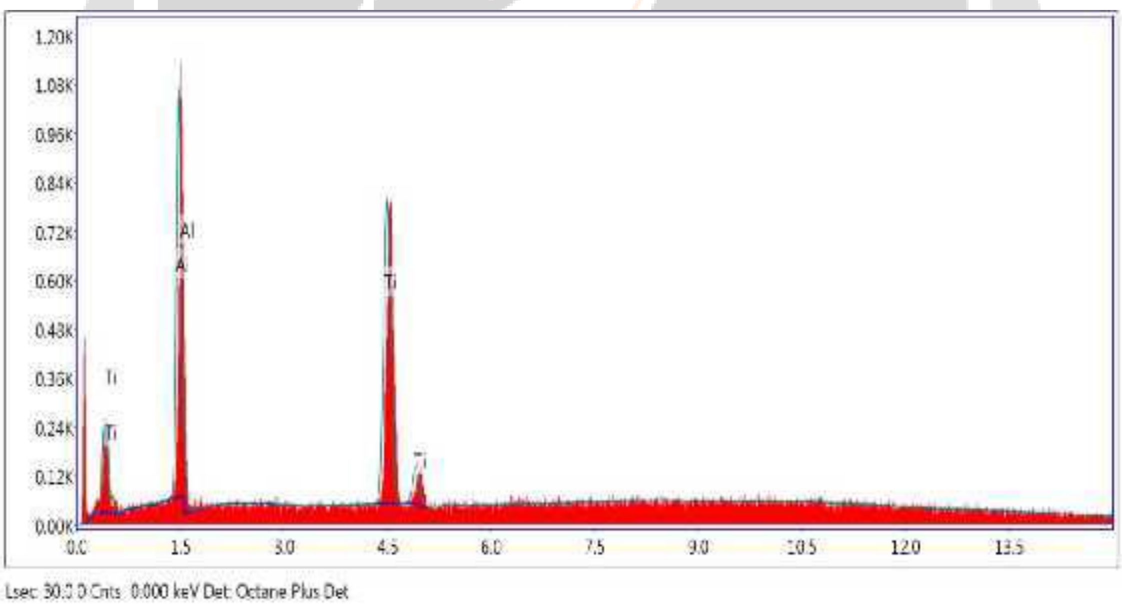
The images of FESEM analysis of PVD coated TiAlN on SS 304 shows a dense structure and as the magnification is increased the images gets more blurred. The coating particles are clearly visible at 800- 15000x magnifications. Fig 7 clearly shows the grain boundaries and the coating particles present.

3.2 EDX analysis

EDX analysis gives the following graph for the elements present in the material.



(a)



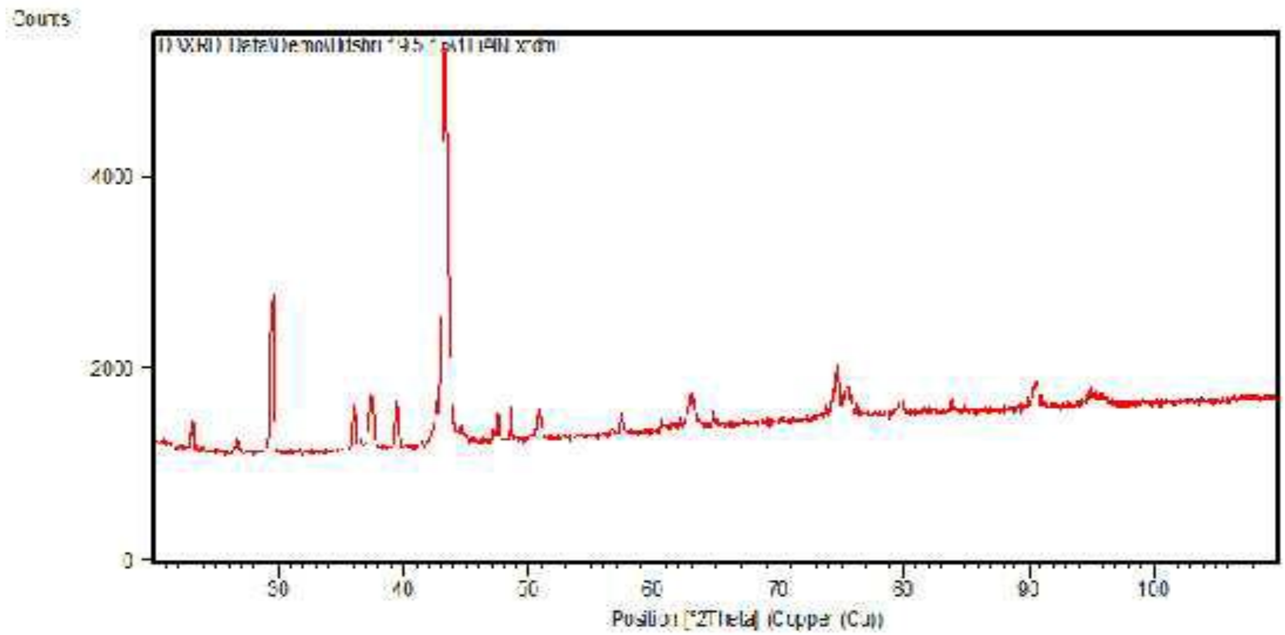
(b)

Graph 3 EDX graph of TiN (a) and TiAlN (b) coatings on SS 304

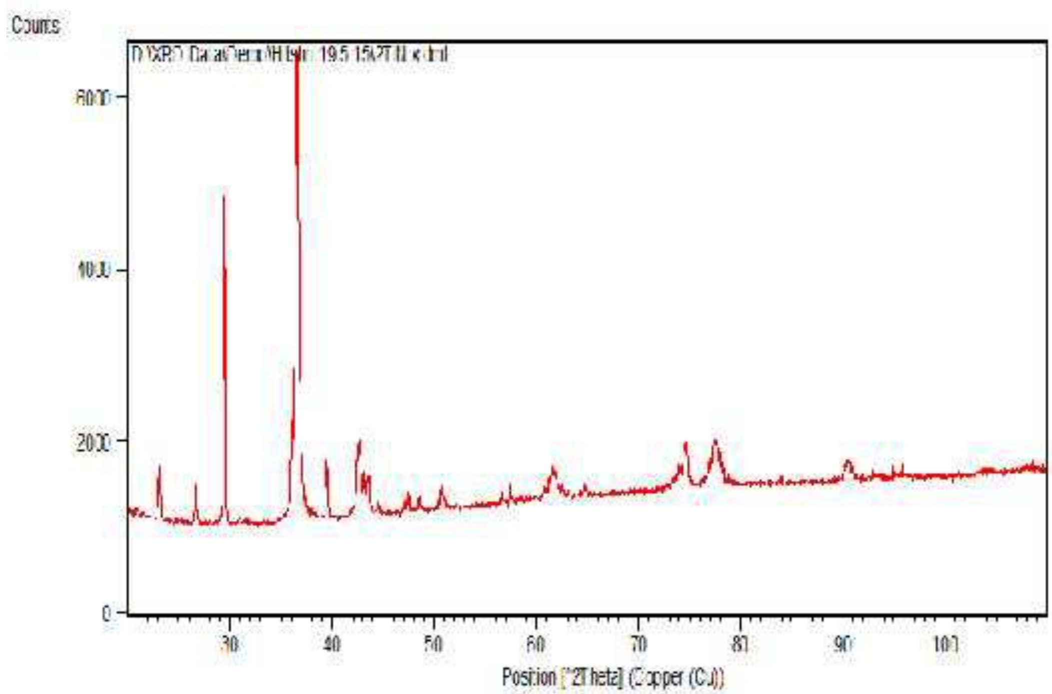
An EDX result for TiAlN coating on SS 304 is as shown in the graphs. The EDX analysis shows the presence of the various elements in the material. The pitch rise in the graph shows the particular element is present in the material.

3.3 XRD analysis

The results generated are presented as peak positions at 2θ and X-ray counts (intensity) in the form of an X-Y plot as shown in the graph below.



(a)



(b)

Graph 4 XRD results of TiAlN (a) and TiN (b) coatings on SS 304

The study carried out by analysing the intensity and the d-spacing values of the graph revealed that the phases present in the coated material were (TI2N)6T i.e. six atoms per unit with a tetragonal structure. Also the (Ti2AlN)8H was present which is a hexagonal structure with 8 atoms per unit. By the study of the peak intensities and the d-spacing the phases present in the sample were Ti2N and TiN both these phases are present in 100% in the sample

4. CONCLUSIONS

- A uniform thin film of TiN and TiAlN coating is deposited on SS 304 by PVDtechnique.
- The FESEM result shows the micrographs of the coated samples at variousmagnifications. The white small spots indicate the presence of coating particles and the grain boundaries are also visible.
- The hardness of the substrate material is increased then it's original.
- The thickness of coatings is 2 ton 4 microns.
- The XRD results revealed the presence of particular phases in the sample.
- The micro hardness of theTiN is 3300 HV and that of TiAlN 2300HV respectively.

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