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Analysis of Hardness of Nano-Composite (TiSiBC) Coated SS 304 sheet

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Abstract: *The coating of the Nano-composites on sheet metal enhances its mechanical properties for a product and increases its mean life and the performance. In the present study, TiSiBC Nano-composites coating on SS-304 sheet is deposited by magnetron sputtering. It is evaluated that the, SS-304 sheet deposited with layers of varying thickness, has different mechanical properties. It is further evaluated that as the thickness of the deposited layer increases, the hardness of the sheet metal increases and wear of the material decreases.*
Keywords: *Palletized, PVD Coatings, Ti-Si-B-C coating*

I. Introduction

Since the inception of coating technology, attempts are being made to develop and improve the material hardness. Hard surface coating deposition has now become a processing step in different tooling industries now days [1]. Recent nanostructured coatings are drawing researcher's attention because of the prospects of incorporating material with super hardness, high toughness, wear resistance and thermal stability at excessive temperature [2, 3]. These nanostructured coating are being called as 21st century materials as per their ability to design uniqueness, property combinations and synthesizing capabilities compared to conventional composites [4].Various PVD coating techniques used for deposition of thin hard film on substrate materials. TiC, TiB_2 and TiN are the first generation PVD coating and still being used as hard protective coating [5]. However, because of lower fracture toughness and low oxidation resistance at elevated temperature, it could not be used for advance engineering application.

Nano-composite coatings can have varying hardness by suitable tailoring of microstructure to get favourable combination of Young's Modulus (E) and Hardness (H). Hardness as well as H/E ratio helps to find out the wear resistance of material [6, 7]. The inception of the state of the art Nano-structured coating with enhanced hardness (about 40 GPa) and temperature stability (> 1200 ^{o}C) is one of the significant concern of the modern material science. According to previous experimental results, it can be considered that not only the grain size has strong influence on the properties of the solid but also structural state at the interface. Properties of the material are strongly depend on condition of the grains boundary when the atoms density at grain boundary reaches to 30-50% [8].

There are three basic mechanism responsible for improving the hardness using Nano-composite coating, i) plastic deformation generated with dislocation, ii) material's Nano-structure, iii) cohesive forces acting among atoms [9].

Nano-composites are the distinctive designed and improved property materials. These composites certainly have minimum one phase in Nano-metric dimension range. It is observed that properties of material changes by changing the size of particle to a particular level. The increased interaction between phases at nanometers level is the main driving force for improvement in properties. These materials consist of minimum two phases with Nano-crystalline and/or amorphous structures. The transition region groups in Nano materials are a) Transformation from crystalline to amorphous phase, b) changeover between two phases of different material and c) conversion between two favored orientations of grains from the same material [9, 10].

The main reasons to huge development of Nano-materials are high hardness, increased toughness, high thermal stability, oxidation resistance and wear resistance. The main constituents for such high hardness and high toughness are the amorphous phase incorporated in between the crystallites and the size of the crystallites. Nanocomposite coated films with improved hardness can exist in different nanostructure [11].

Material properties depend on grain boundary conditions, grain distortion and interfaces, defects at the boundary and free volume value. Hardness of the material is affected by grain size [12].

Among different Nano-composite hard coatings, the Ti-Si-B-C, coatings have shown a very favourable hardness, modulus, and H/E ratio and oxidation resistance. Thickness plays a significant role on the properties, structure and interaction with the substrates in the terms of mechanical properties. The present investigation discusses our findings on the hardness of Ti-Si-B-C coating of different thickness ranging from 10µm to 25µm deposited by magnetron sputtering at SS 304 sheet.

II. Present Work

In this study, TiSiBC targets are prepared in National Metallurgical Laboratory, Jamshedpur. TiSiBC target material was developed by mixing TiB_2, SiC, TiC, Si and Ti powders [13]. These powders were milled in FRITCH Vibratory Cup Mill for three times. Speed of the milling kept at 1000 rpm for 2 minute with a dwell time of 10 minute between each cycle. The powdered mixture was added with 2-3 drops of PVA solution as binder and grinded for 15 minutes in a ceramic mortar and pestle.

The mixture then palletized into disc shape of 50 mm diameter using hydraulic pressure of 100 Kg/cm^2. Palletized targets were sintered in a Thermal Technology INC High Temperature Graphite Furnace at various temperature with varying dwell time for each temperature as shown in Fig.2.

Fig.2: Temperature variation during sintering of TiSiBC target

2.1 Sample Preparation

The SS 304 sheet were cut into pieces and were polished by using Silicon Carbide (SiC) paper of Grit Size, 300, 400, 600, 800, 1000 and 1200 subsequent to cloth polishing using Alumina slurry. Sample cleaned with cotton swab soaked in Acetone followed by ultrasonic cleaning in Acetone at 35 ^oC for 3 minute.

2.2 Coating deposition

Ultrasonically cleaned samples coated with TiSiBC deposited layers using Planar Magnetron Sputtering Unit, HINDHIVAC Pvt Ltd. Bengaluru. The base pressure and pressure during deposition in the chamber were maintained at 2 x 10^{-5} and 0.01-0.02 mbar respectively. The substrate heater temperature, operating voltage and current was kept constant at 450^{o}C, 0.3 kV and 0.5 amp respectively throughout the deposition process. The film thickness of 10µm, 15µm and 20µm were obtained.

2.3 Phase and microstructural analysis

Each coating analysed by using X-Ray Diffraction model & make: D8 Discover, Bruker AXS GmbH-Germany in normal mode. The source used as Cu-Kα radiation of wavelength 1.54 Å with 2θ scan in range of 20^o to 80^o. Microstructural and compositional analysis of the deposited samples accomplished using ultra-high resolution emission SEM make & model: FEI Nova Nano SEM 430.

2.4 Hardness measurement

Nano-hardness was measured using XP Nano indenter. Mechanical properties behaviour of materials were also studied using Berkovich indenter. Micro hardness testing was also done using Leica VMHT Auto with Vickers hardness indenter by applying 1 gm to 2000 gm load.

III. Result And Discussions

SS 304 substrates were coated using TiSiBC Nano-composite deposited layer of different thickness. XRD pattern for different coating thickness are shown in Fig.3.

It is seen (Fig.3) that only substrate peaks are forming for all coating thickness confirming that the coated film is X-Ray amorphous. The height of the peaks decreased with increased thickness. This is associated to lesser volume of substrate exposed to X-Ray source due to more thickness of layer.

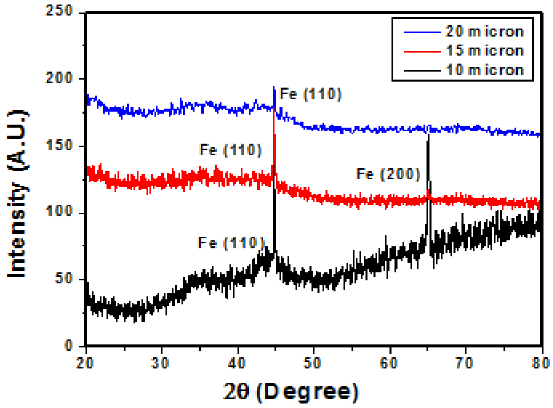

Fig.3: XRD of TiSiBC film for 10µm, 15µm and 20µm coating

The microstructure of the TiSiBC at different thickness is shown in the Fig.4. The size of TiSiBC Nano-grains found increasing with thickness of layer due to increased diffusion between grains in growing and lower layers during deposition. The mean grain size found 115 nm for 10µm, 124 nm for 15µm and 142 nm for 20µm. layer thickness.

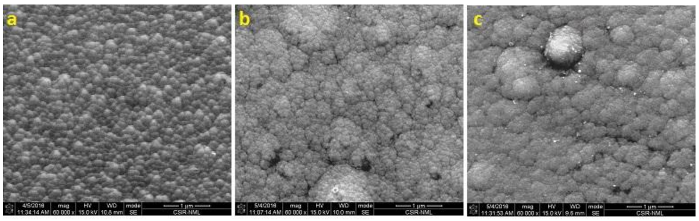

Fig.4: SEM micrographs of as deposited TiSiBC film for a) 10µm, b) 15µm and c) 20µm coating thickness

Fig. 5 shows EDS analysis, the peaks of Ti, Si, B, and C and the wt. percentage of the content of the coating shown in Table 1.

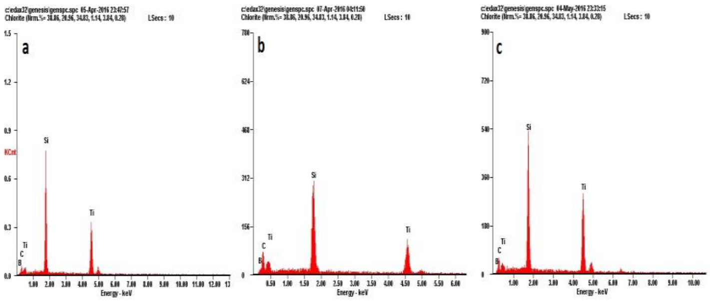

Fig.5: EDS analysis of as deposited Ti-Si-B-C coating for a) 10µm, b) 15µm and c) 20µm coating thickness

Table 1: E D S analysis of as deposited Ti-Si-B-C coating for all thickness

Thickness (µm)	Chemical Composition (Wt. %)			
	B	C	Si	Ti
10	13.62	13.38	28.24	44.76
15	14.17	13.83	24.77	47.24
20	14.69	13.55	24.91	46.85

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The compositional analysis shows intense peak for Si and Ti followed by C and B. It is found that the composition was more or similar with thickness.

3.1 Mechanical Properties:

Micro-hardness of the TiSiBC deposited substrate measured by using Leica VMHT Auto for each coating thickness (Table 2).Average hardness of 3000 HV found for 10µm, followed by 3429 HV for 15µm and 3473 HV for 20µm respectively.

Thickness (µm)	Hardness (HV)
10	3000
15	3429
20	3473

Table 2: Hardness and elastic modulus of the different coating thickness of Ti-Si-B-C Nano-composite

The least hardness for 10µm appeared due to substrate interference whereas for 15µm and 20µm coating thickness the substrate interference was less due to which hardness found increased.

The hardness of TiSiBC film at different thickness with the depth measured by Nano-indentation shown in Fig.6.

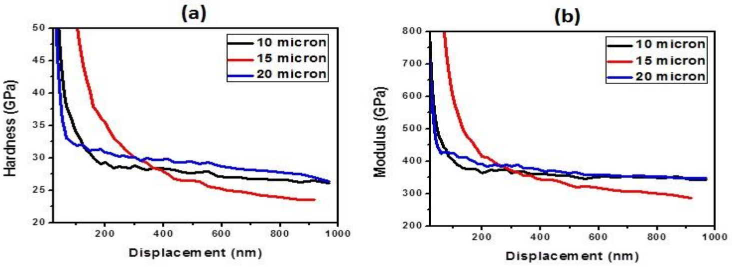

Fig.6: a) Displacement Vs Hardness and b) Displacement Vs Modulus graph for TiSiBC coated samples at different thickness

The average hardness and elastic are modulus tabulated in Table.3. The Nano-indenter results almost similar hardness value of 31 GPa for all thickness, however modulus for 20 µm thick film increased.

Thickness (µm)	Hardness (GPa)	Modulus (GPa)
10	31.08	381.7
15	31.48	384.5
20	31.55	409.2

Table 3: Hardness and modulus of the different coating thickness of Ti-Si-B-C Nano-composite

IV. Conclusion

Quaternary Ti-Si-B-C Nanocomposite thin film were successfully deposited by using magnetron sputtering unit to achieve film thickness of 10µm, 15µm and 20µm on the SS304 substrate material.

All as deposited coating, layers were X-ray amorphous. The EDS analysis affirmed the existence of Ti, Si, B and C. The composition analysis were found as Ti:Si:B:C :: 3.5:2:1:1. Grain size of the coated film increased with thickness. The micro-hardness of the film enhanced with thickness of layer. Nano-indentation hardness and modulus were of similar range. Slight increase in hardness was observed. With increase of thickness due to lessening substrate contribution becoming lesser in thicker film.

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