

# Influence of Deposition on the Mechanical Property Correlation in Al-O Thin Films with Complex Microstructures by RF Magnetron Sputtering

R.Hariharan , R.Raja , A.S .Anish , S.Balaji , M.Aswin , J.Roshan

**Abstract:** *AL-O films were ready on unwarmed 4140 steel abstract by RF magnetron sputtering technique. Post-deposition hardening of AL-O films in vacuum was found to enhance film structure and electrical characteristics like dense structure, swish surface stress relief and increase electrical resistance appropriate hardening temperature conjointly reduced loss issue. The coordination between hardening conditions and also the body of the films (crystalline structure and microstructure) was examined by X-ray diffraction (XRD) scanning electron microscopy (SEM) atomic force microscopy (AFM) and FESEM with EDAX. The mechanical behaviour of nano crystalline CNC metals has attracted widespread interest through the bulk of efforts have centred on economically pure metals.*

**Keywords:** EDAX; XRD; AFM; FESEM

## 1. INTRODUCTION

### 1.1 Aluminium Oxide:

Alumina or aluminium oxide Can be a compound of metal and finished gas Al<sub>2</sub>O<sub>3</sub>. This mainly occurs from the oxides of many metallic elements and is especially known as aluminium (III) compounds.. it's usually known as corundum, and should even be known as aloxide, aloxite, or aluminium oxide betting on explicit structures or applications. It happens normally in its crystalline polymorphic area  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> due to the mineral corundum, sorts of that kind the important gemstones ruby and sapphire. Al<sub>2</sub>O<sub>3</sub> is imperative in its utilization to give metallic component metal, as partner rough because of its hardness, and as a recalcitrant material because of its high the point of solidification.

### 1.2 ALUMINIUM OXIDE STRUCTURE

The most common form of crystalline corundum is understood to be a mineral, ie a thermodynamically stable substance. Octahedron void element. Each center of Al<sup>3+</sup> is octahedral. In its natural philosophy, minerals use a symmetrical spatial lattice with a set of regions R-3c (the number in the international table

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is 167). The original unit contains 2 corundum formulation units.

Aluminium oxide is present in different phases, as well as cubic  $\gamma$ - and  $\eta$ -phases, a monoclinic  $\theta$ -section, a hexangular cross section  $\chi$ , a rhombic  $\kappa$ -section and, hence, a  $\delta$ -section that will be polygonal or orthorhombic. Each includes a distinctive crystalline structure and properties. Cube-shaped  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> has the necessary technical applications. The presumed  $\beta$ -Al<sub>2</sub>O<sub>3</sub> was NaAl<sub>11</sub>O<sub>17</sub> [11].

Molten corundum, close to the melting point, is about 2/3 of the tetrahedron (ie, 2/3 of the square measure of Al, enclosed in four elementary neighbours) and 1/3 5-coordinated, little or no.

### 1.3 APPLICATION OF ALUMINA

More than 90% of the alumina, often referred to as fused alumina (SGA), is used to assemble metal components, sometimes using the Hall-Elo method. The rest is often referred to as special alumina and is used to reflect its immobility, heat resistance and resistance in an extremely wide range of applications

- Filler
- Glass
- catalytic
- Purification
- Abrasive
- Paint
- Composite fiber

### 1.4 HIGH SPEED STEEL

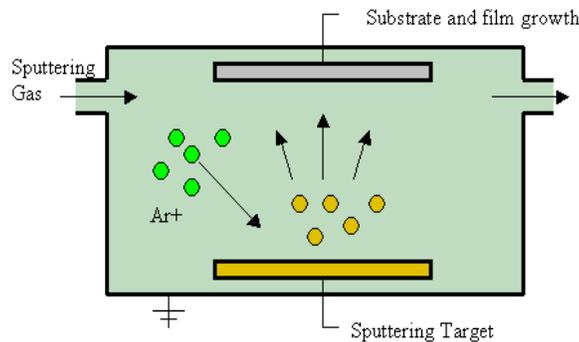
AISI 4140 Chrome - Moly High Tensile Steel, typically prepared solidified and tempered to Condition "T" in segments up to 100mm, with an enduringness of 850 – a 1000 MPa and going for this quality place bigger segments. It offers a terribly sensible adjust of quality, strength and wear-protection.

### 2.1 SPUTTERING:

Target utilized as a part of sputtering process is tantalum. Measurements of the target are 2inch dia and 3mm thickness. Coating of the substrates were done under different parameters like Base pressure 3.7x10<sup>-6</sup>bar and Working pressure 11.5x10<sup>-3</sup>m.bar.



Temperatures for coating the substrates are 2000c and 4000c. Time taken for covering one substrate is 1 hr at 100w power. Voltage and amperes utilized for covering are 503v and 203A individually. Argon and nitrogen are embedded amid covering at the proportion of 15:2.8sccm..



**Fig.2.1 SPUTTERING PROCESS**

The sputtering gas is usually associate in nursing chemical element like noble gas. For economical momentum transfer, the mass of the sputtering gas ought to be near the mass of the target, thus for sputtering lightweight components, Ne is desirable, whereas for significant components Kr or atomic number 54 are used. Receptive gases can even be wont to sputter mixes. The compound might be molded on the objective surface, in-flight or on the substrate waging on the technique parameters.



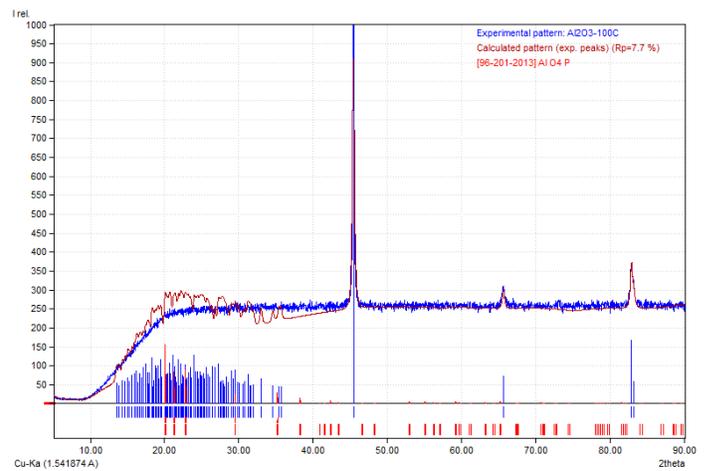
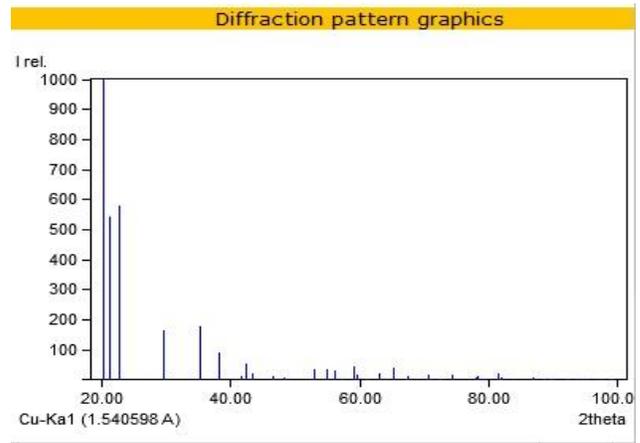
**Fig.2.2 after coating RF sputtering in various temperature levels**

**RESULT AND DISCUSSION**

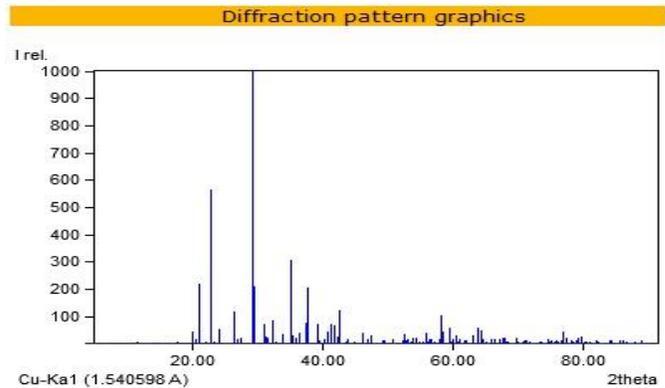
**3.1 X-RAY DIFFRACTION**

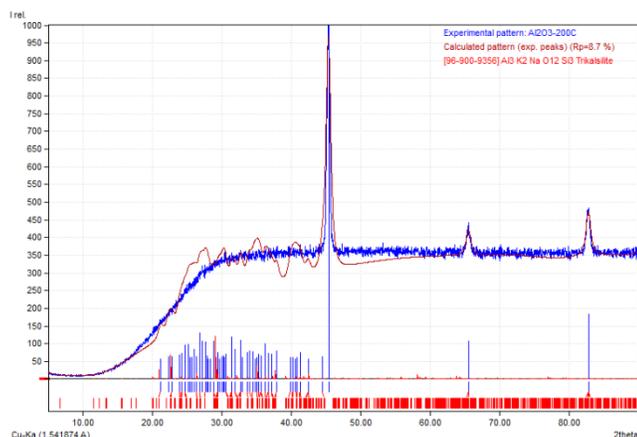
The stage arrangement, and in addition the structure of the film, were inspected by X-beam diffraction examination. The XRD examples of AlO thin films appear in Figure 3.1 The brilliant pinnacles (h-AlO 0002), (C-AlO 002), and (h-AlO) were gotten in the X-beam diffraction thinks about. The uncoated pinnacles of 100 ° C, 200 ° C and 300 ° C were utilized with Origin 8 and Match! Looked at. Optical wonder designs from the distinguished pinnacles reminiscent of the development of the hexagonal segment of AlO were recorded as per the polygonal shape structure. Knowing the wavelength ( $\lambda$ ), the full width at 0.5 forces (FWHM) of the pinnacles ( $\beta$ ) and the optical marvels point ( $\Theta$ ), the molecule estimate (D) was ascertained utilizing the Scherrer equation.  $D = 0.9 \lambda / \beta \text{ Cos}\theta$ .

For 100 °C :

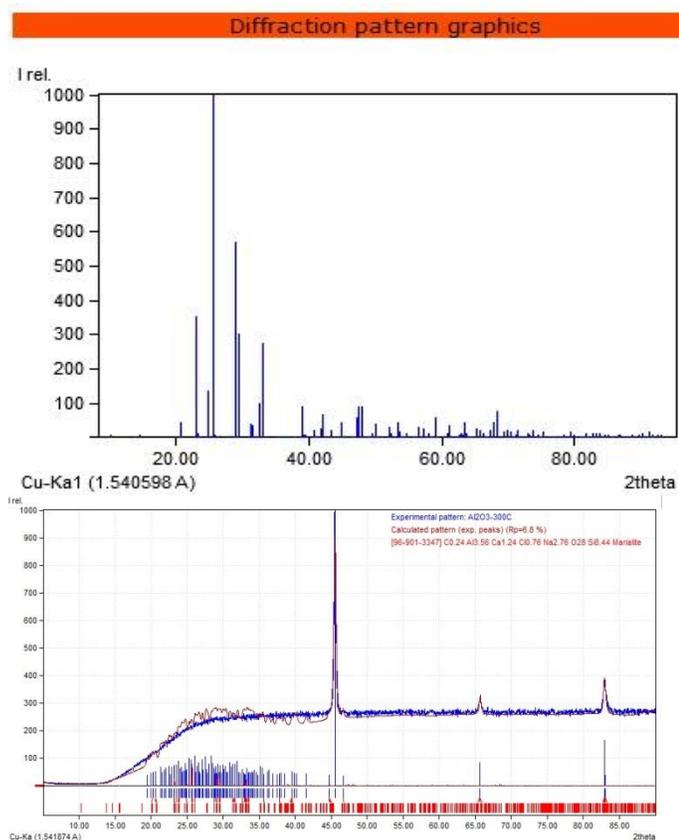


**FIG.3.1 XRD pattern of the AlO thin films deposited on High speed substrates at temperature 100 ° c For 200 ° C :**





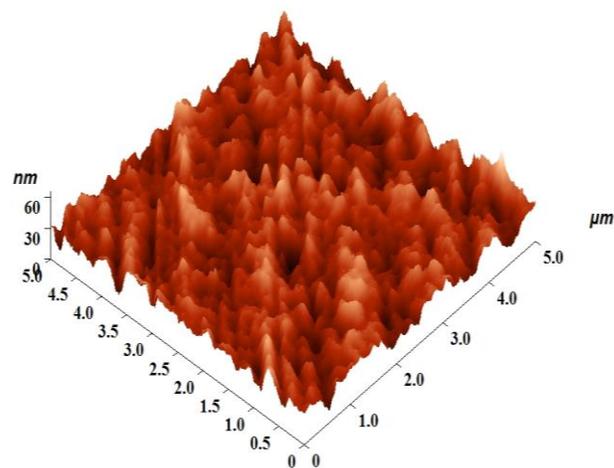
**FIG.3.2 XRD pattern of the AIO thin films deposited on High speed substrates at temperature 200 °C For 300 °C :**



**FIG.3.3 XRD pattern of the AIO thin films deposited on High speed substrates at temperature 300 °C**

### 3.2 Atomic force microscopy studies (AFM)

Surface topographical characterization was done by Atomic Force research. The AFM scan was carried on 3 samples coated with AlN at temperature, 200°C for 60nm severally. The scan was carried with semi-contact mode on sputtered AIO for a scan space of 5µm × 5µm on the surface. From the AFM pictures (refer Fig.3.3 ) aluminium surface have average roughness of 6.78922 nm, 5.59142 nm and 16.6085 nm for room temperature, 200°C temperature coatings respectively. From the results found it can be determined that due to the low average roughness, there will be low friction co-efficient decreasing the wear on the worm gear.



**Fig 3.4 5µm × 5µm 3D IMAGE OF COATED SAMPLE AT 200°C**

### 3.3 Micro Hardness

Characterization of mechanical properties of the films was performed using vickers hardness. The hardness  $H_v$  100 gms was carried for temperature 100 °c (310,315,314) & 200 °c (358,369,363) & 300 °c(270,285,277 respectively). The film connection pins and sleeves in the Convair 580 aircraft were hardened to a Vickers hardness specification of 390 HV5 by the process manufacturer and '5' was 5 kg. However, on an aircraft flying Paternaer flight 394, it was later found that these pins had been replaced by substandard components, resulting in rapid wear and eventually causing loss of the aircraft. The accident investigators found during the inspection that the hardness of the pins below the standard was only 200-230 HV5. Currently, ome watch factories increase the area of the crystal glass by testing the Vickers hardness test. As they increasingly create higher watches, they use it as a joint victim of the buyer's sales data. Due to the Vickers hardness check, every day customers are currently creating a sober watch crystal strength. Some of the manufacturer's regional departments claim that they need to watch glass at 6000 Vickers, and different watches score 700.

### 3.4 Scanning Electron Microscopy (SEM)

Scanning electron microscopy produces images by searching the specimen for a rectangular space (raster scan) on the central beam. Once the pillar communicates with the example, it loses vitality through a progression of components. The lost vitality again ends up different structures, for example, warm, low-vitality auxiliary electrons and high vitality backscattered electrons, light discharge or



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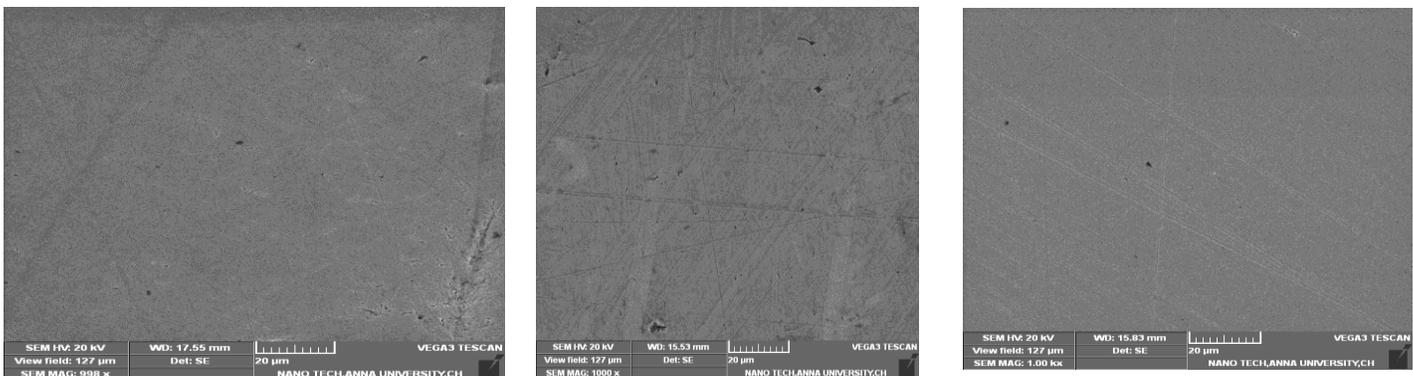
X-beam emanation, every one of these structures are given to convey about the surface of the example, for example, its geology and Composition. Once the signal is generated, the image displayed by the degree of association SEM maps the variable intensity of any of these signals to the image at a position that is comparable to the beam position on the sample. In the SEM image of the sub-closed aircraft shown below, and where appropriate, the image was created from signals generated by secondary negative ion detectors in most SEMs, standard or conventional imaging modes. By and large, the picture determination of partner degree SEM is at least partner degree request of size poorer than that of a TEM. As a result of the SEM picture, it depends on surface procedures instead of

transport however is prepared to scale the gathered examples to a couple of centimeters (according to the settings and settings) that includes a good depth of field, then will turn out pictures that square measure smart portrayals of the three-dimensional type of the example. Another favourable position of SEM is its selection referred to as environmental scanning microscope (ESEM) will turn out pictures of adequate Quality and spine, models are wet or vacuum or gas.

*For 100 °C :*

*For 200 °C :*

*For 300 °C:*



**Fig.3.5 SEM of the Al<sub>2</sub>O<sub>3</sub> thin films deposited on High speed substrates at temperature 100 °c, 200 °C, 300 °c respectively**

### CONCLUSION

Aluminum Oxide (Al<sub>2</sub>O<sub>3</sub>) thin films were set up on low carbon steel substrates at an alternate temperature (RT,200,400,500,600) using RF magnetron sputtering method. The prepared films were characterized by XRD, AFM and Nano indentation techniques to study the microstructural and mechanical properties of the films respectively.

The XRD analysis showed that the film prepared at RT and 100 C showed the amorphous nature of the films. The films prepared at 200 -300 c showed the peaks corresponding to hexagonal (0002) and cubic (002) structure, preferential orientation along (002) for the sputtered Al<sub>2</sub>O<sub>3</sub> films with cubic structure and also hexagonal phases are present at low substrate temperature.

Al<sub>2</sub>O<sub>3</sub> coatings were with successfully prepared RF magnetron sputtering on low steel substrate. Al<sub>2</sub>O<sub>3</sub> coatings would possibly attain higher erosion polarization protection and moderately stable consumption potential among the SBF surroundings than the uncoated low steel. Thusly, the covered examples would have lower erosion and therefore the substrate coated at 100°C, 200°C exhibited the most effective corrosion resistance for the coating investigated

within the studies. The damage resistance and therefore the corrosion resistance of low steel was multiplied by Al<sub>2</sub>O<sub>3</sub> coating.

Aluminium Oxide (Al<sub>2</sub>O<sub>3</sub>) provides good protection for corrosion for the metal substrates. The surface morphology of the films was characterized by Scanning Electron microscopy (SEM) studies. It shows the graceful morphology of the films with uniform distribution of the crystallites and most of the grains have the same grain size. The roughness and thickness of the films are 126 nm, -454 nm and -257.57 nm at temperatures of respectively. In the Al<sub>2</sub>O<sub>3</sub> thin films, the surface geology comprises of groups of same sizes with unpredictable shapes. The unpredictable type of grains recommends that at low substrate temperatures the mechanical vitality isn't sufficient for the association of the grains which can be light-emitting diode to the presence of further phases as evident from the XRD analysis. it's evident from the AFM micrographs that the form of the grains changes with increasing substrate temperature additionally to the grain size



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