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Microstructural and mechanical properties of magnetron sputtered SiO_xN_y thin films

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Abstract

Silicon oxynitride (SiO_xN_y) films were deposited over the Ni-plated steel by reactive radio frequency (RF) magnetron sputtering technique by varying the oxygen partial pressure. As-deposited films were characterized using the X-ray diffractometer (XRD) and field-emission scanning electron microscopy (FE-SEM) to study the structural and morphological properties. The grain size obtained from the XRD pattern and the FE-SEM were in good agreement. The hardness of the film increases with increase in the oxygen partial pressure. The sample deposited at high oxygen partial pressure showed the decreased hydrophobic nature.

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Keywords: Silicon oxynitride; XRD; FE-SEM; Contact Angle; Hardness.

1. Introduction

Materials like DLC [1, 2], TiN [3, 4] and WC are used to improve the mechanical properties such as hardness, wear in auto mobile components. In addition to above materials, SiOxNy is a good industrial material which exhibits good mechanical properties such as hardness, Young's modulus, high flexural strength and also low thermal expansion co-efficient, high oxidation resistance and high thermodynamic stability [5,6]. Nickel alloyed and nickel

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plated components are used in various parts of auto mobile components such as Turbocharger (turbine vane), exhaust valve, drive shafts and brake fluid lines hence Ni-plated steel was chosen has substrate. In the past SiOxNy film were deposited by PECVD technique leads to various disadvantages incorporation of hydrogen [7]. RF magnetron sputtering is a most versatile technique and it eliminates the hydrogen incorporation in SiOxNy film. In the present study, SiOxNy films have been deposited over the Ni-plated steel by RF magnetron sputtering technique and the influence of oxygen partial pressure on structural properties, surface morphology and hardness were studied. Also the surface changes due to the oxygen changes were investigated by contact angle measurement.

2. Experimental Details

SiO_xN_y films were deposited over Ni-plated steel by RF magnetron sputtering technique by varying the oxygen partial pressure. Ni-plated steel substrates were initially treated with soap solution (Triton X-100), after that ultrasonication with distilled water followed by sodium hydroxide (10 % of NaOH solution), gently washed with acetone and dried using air blower. The deposition equipment consists of three magnetron arranged in such a way that each subtends to 20° angle of inclination to the substrate table. The substrate table which holding the substrates are placed at a distance of 5 cm approximately from the magnetron. The deposition chamber is evacuated to the base pressure of 3.7×10^{-5} mbar with the help of rotary and diffusion pump combination. .A power supply operated at rf-frequency of 13.56 MHz, with the help of auto tuner power of 70 W applied to the magnetron. Highly pure Ar gas used as sputter gas, nitrogen and oxygen gases were used as reactive gases. The pre-sputtering was done for 5-10 min to remove the surface impurity on the target. The crystalline structure of the films were investigated using XRD (XPert PRO) in the 2θ range of 10 to 80° using CuK_α radiation of wavelength λ = 1.5406 Å at room temperature. The surface morphology of the films was studied using the FE-SEM (JEOL JSM 170F) operated at 3.0 kV. The hardness of the films was measured with the help of microhardness tester (SHIMZADU). The contact angle of the different samples was measured with help of sessile-drop method.

Table. 1.Deposition Parameter

Parameters	**Values**
Target	Silicon (99.99 % purity)
	2.5 inch dia,3 mm thick
Base pressure	3.7×10^{-5} mbar
Ar pressure	0.7×10^{-2} mbar
N_2 pressure	1.5×10^{-2} mbar
O_2 pressure	2, 2.6, 3 $\times 10^{-2}$ mbar
Substrate	Ni-plated steel
Power	70 W

3. Results and Discussions

3.1. Structural Properties

Fig. 1. shows the XRD patterns of SiO_xN_y films. XRD pattern confirmed polycrystalline nature of the films with the multiple number of peaks in the 2θ angles of 44.5, 52.1 and 76.8° corresponding to the reflections of plane (4 0 1), (3 1 2) and (4 1 3) respectively (JCPDS card no. 01-083-2149). The film exhibits orthorhombic structure with (4 0 1) reflecting plane as a preferential orientation plane. The crystallite size (D) was calculated from the XRD using the Scherrer's equation,

$$D = 0.9\lambda / \beta \cos\theta \qquad (1)$$

where, λ is wavelength of X-ray, β is the full width at half maxima and θ is the Bragg's diffraction angle and the lattice strain (ε) in the film was calculated by the formula,

$$\varepsilon = \beta / \tan\theta \qquad (2)$$

The crystallize size varies from 18 to 102.2 nm and the lattice strain decreases with from 0.485 to 0.089 %.