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Physicochemical and tribological properties of Si_3N_4 thin films deposited on Si by D.C. reactive magnetron sputtering

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Abstract – Silicon nitride thin films have many applications in microelectronics and tribological applications. In the present work, these coatings were deposits by D.C. reactive magnetron sputtering on single crystal Si(001) substrates as a function of temperature, time and N_2 partial pressure. The physico-chemical and tribological properties were investigated as a function of deposition parameters (temperature, time and N_2 partial pressure), as well as after a post-deposition annealing in $^{18}O_2$ ambient in the 250-1000ºC temperature range. Films hardness were not affected after a 1000ºC annealing, evidencing an outstanding oxidation resistance.

Amorphous silicon nitride (a-Si_3N_4) films have recently drawn immense interest [1] due to its excellent mechanical and electrical properties, which allows application in microelectronics devices, as well as in extreme tribological conditions. Moreover, when it is used in synergistic combination with c-TiN, it can produce a superhard material [2]. Si_3N_4 films are conventionally deposited at high temperatures (700-900ºC) by chemical vapor deposition (CVD), but in most of the above mentioned applications, low temperature deposition is preferred. Between the many existing deposition processes, physical vapour deposition presents many advantages, since it can be performed at lower temperature and presents high deposition rates. Nevertheless, in both applications, silicon nitride should present high temperature oxidation resistance. In the present work, Si_3N_4 thin films were deposited on single crystal Si(001) substrates by reactive magnetron sputtering using nitrogen as the reactive gas. The structural and tribological properties of the films were investigated as a function of nitrogen partial pressure (0.3-1x10^{-2} mbar) and substrate temperature (25-500 ºC). Oxidation resistance was evaluated after high temperature (250-1000ºC) $^{18}O_2$ annealings. Rutherford backscattering spectrometry (RBS) were used to evaluate the films composition (stoichiometry) and deposition rates, where fully stoichiometric Si_3N_4 were observed for all deposition temperatures at a N_2 partial pressure of 3x10^{-3} mbar. X-ray diffraction (XRD) analyses presented in Fig1. shows that the films are amorphous for low temperature deposition, becoming crystalline at temperatures above 400ºC. Figure 2 shows nanohardness measurements as a function of deposition temperature, where one can see that the higher hardness figure (22 GPa) is obtained at 300ºC. Since oxidation resistance at high temperatures is crucial for extreme tribological operation conditions, the samples deposited at 300ºC was submitted to thermal annealings in $^{18}O_2$ ambient (Fig3.). We observed a small decrease in the hardness at 500ºC, while at 1000ºC no decrease is observed, indicating a high temperature oxidation resistance. This fact may be related to changes in the crystalline structure of the films, as well as to $^{18}O_2$ incorporation during annealing. Nuclear reaction analyses, XRD and x-ray photoelectron spectroscopy (XPS) results will be presented in order to give a deeper understanding on this issue.

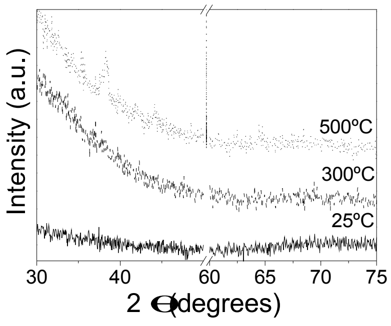

Figure 1: Diffractograms of Si_3N_4/Si samples deposited at different temperatures.

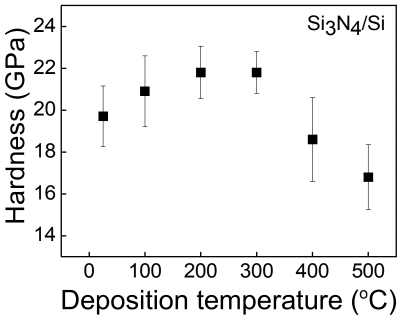

Figure 2: Nanohardness of Si_3N_4/Si samples as a function of the deposition temperature.

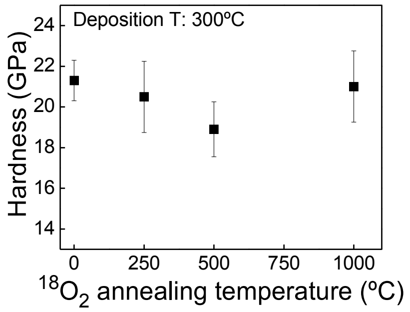

Figure 3: Nanohardness of Si_3N_4/Si sample deposited at 300ºC as a function of $^{18}O_2$ annealing temperature.

References
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