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# Cr-N THIN FILMS PREPARED BY REACTIVE RF MAGNETRON SPUTTERING

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**ABSTRACT:** Chromium Nitride thin films Cr-N have been deposited on carbon steel substrates by r.f. magnetron sputtering using a pure Cr target. In order to generate nitriding process of Cr, Ar and $N_2$ gas mixing in different ratios of Ar/ $N_2$ was used. To characterize Cr-N thin films, composition and structural properties were determined respectively by EDS (Energy Dispersive Spectroscopy) analysis and X ray diffraction (XRD). Hardness and Young modulus were also measured. SEM images were used to determine surface morphology of produced Cr-N thin films.

**KEYWORDS:** plasma, chromium nitride, thin film, annealing treatment, steel

## Extended abstract

Reactive sputtering is widely used for the thin-film growth of some metal compounds in plasma of Ar and reactive gases (eg Nitrogen). Cr-N films have been of interest for some applications to electrolytic materials, to metallic luster, tribology behavior, corrosion and temperature-resistant materials [1–3]. Cr-N coating may be produced by several techniques such as HCD (hollow cathode deposition), Direct Arc, FCVA (filtered cathodic vacuum arc) and Ion Beam processes [4–10]. In Magnetron Sputtering method, the advantages are good uniformity, high deposition rate and low temperature [11–13].

Chromium nitride coatings were deposited on steel XC100 substrates by r.f. magnetron sputtering varying working gas composition (between 4 and 16%$N_2$). Deposited thin films were then subjected to a heat treatment of high vacuum annealing at the temperature of 700°C for 50 min. X-Ray diffraction and Energy Dispersive Spectroscopy (EDS) were used respectively to study the microstructure and composition. Scanning Electron Microscope (SEM) was used to determine the surface imaging of produced Cr-N thin films. The hardness and Young's modulus are also measured.

Fig. 1 shows XRD patterns of Cr-N films deposited on XC100 steel substrates for 8 %$N_2$ and heated at the temperatures of 700°C. The XRD patterns show that at low percentage of nitrogen (%$N_2 \leq 8$), in the mixture of gases the peaks attributed to steel substrates XC100 constituting the phase Feα are detected. In parallel, we observe a pick of pure chromium. The appearance of other peaks, with different intensities is clearly shown; they are identified as chromium nitrides $Cr_2N$ and CrN lines. The formation of these nitrides was also observed by S. W. Rusel et Al [14] at 650°C. This proves the solubility of nitrogen atoms of gas mixture used in the chromium deposited films at 8%$N_2$ after annealing at 700°C. Chromium nitrides are formed in the early stages of annealing due to the presence of chromium and nitrogen mixtures in the deposited films. This provision permitted to create more stable positions for the rearrangement of atoms and consequently facilitates the diffusion of nitrogen in chromium film which justified the reduction of its atomic concentration shown by EDS analysis. The

diffusion of nitrogen in chromium film is helped by the size effect ($r_N/r_{Cr}$ <0.59) [15] in the process of the energy provided by thermal annealing. From the SEM images, the surface of Cr-N thin films deposited on steel substrate at 8%$N_2$ in the mixture gas and annealed at 700°C is gray highly reflective. This corresponds to the formation of $Cr_2N$ chromium nitride, which is revealed by XRD analysis. The surface contains a scattering of white oxides spots caused by the increasing of oxygen, that is detected by EDS analysis. The hardness increases gradually to 21.81 GPa at 8%$N_2$with the increase in the formation of $Cr_2N$, then decrease slightly with the nitrogen pressure increasing. The hardness recorded 18.54GPa at 12%$N_2$, due to the appearance of CrN which is less hard than $Cr_2N$; this decrease may be due to the presence of oxides which appeared on the surface morphology of the films. It must be noticed that the oxides weaken the mechanical properties of materials. The variation of Young's modulus is similar to the hardness variation according to nitrogen partial pressure parameter. However, the film which contains the $Cr_2N$ has the Young's modulus values higher than those of CrN. we can say that the $Cr_2N$ is harder than CrN although less adherent.
In the present investigation, Cr-N thin films were produced by RF sputter plasma nitriding process. Ar: $N_2$ gas mixing, in different percentages, was used as working gas. Increasing the nitrogen partial pressure in the working atmosphere produces changes from a hexagonal $Cr_2N$ to cubic CrN microstructure. The hardness obtained for a coating with dominant CrN phase produced with highest nitrogen flow is of (17.64 GPa).When the $Cr_2N$ phase is dominant the hardness has a relative maximum (21.81 GPa).

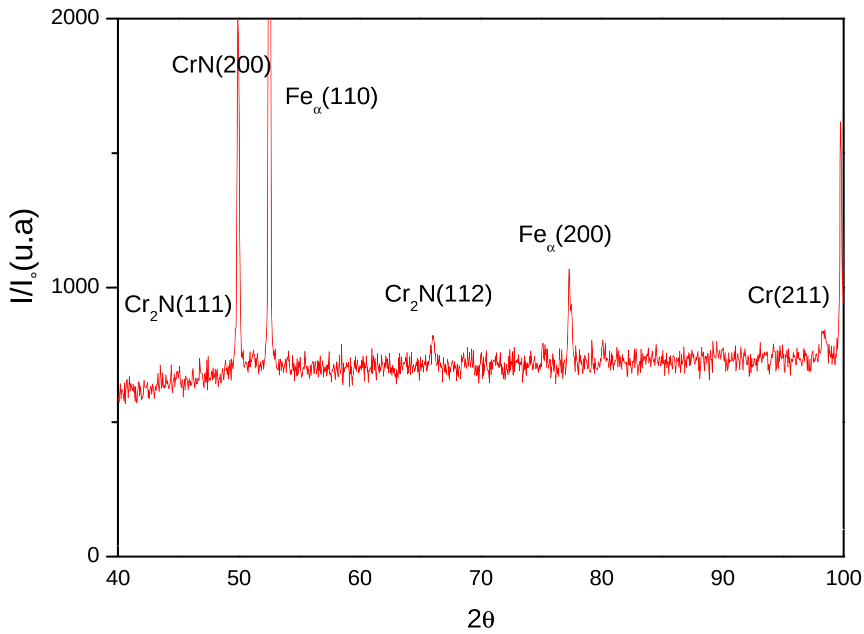


**Figure1: XRD pattern of Cr-N film deposited on XC100 steel at 8% $N_2$ in the mixture gases and annealed at the temperatures of 700°C.**

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