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Characterizing the atomic and electronic structure of the defects in CrN films by advanced TEM

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Transition metal nitrides (TMN) have found wide-spread applications in the cutting- and machining-tool industry due to their extreme hardness, thermal stability and resistance to corrosion. The increasing demand of these nitrides requires an in-depth understanding of their structures at the atomic scale. This has led to numerous experimental and theoretical researches. Among TMNs, CrN exhibits fascinating structural, magnetic, and electronic properties that are of fundamental importance to condensed-matter physics [1-6]. Detailed studies of the structure of CrN will definitely reveal its unique property. On the other hand, it is inevitable to generate structural disorders during film deposition, where various defects can be introduced. Apparently, these defects involved will influence the properties of CrN films. Recently available aberration corrected TEM enables quantitative characterization and evaluation of materials structure. In this study, we apply C_S-corrected HRTEM, quantitative electron diffraction, and EELS analysis to understand the intrinsic structure of CrN films. The films used in this study were deposited by reactive direct current magnetron sputtering of a Cr target in an $Ar+N_2$ atmosphere at a constant total pressure of 1 Pa, a target power of 6 kW, and a temperature of 350°C.A TEM/STEM JEOL 2100F operated at 200 kV and equipped with an image-side C_S-corrector and a Gatan imaging filter (Tridiem) was used to characterize the film structure.

A comparative study is carried out. The first part of this paper will be ordered defects, i.e. nitrogen (N) vacancies, which were found at the Cr/CrN interface during growth of CrN on a Cr interlayer, used to provide sufficient adhesion to the Si(100) substrate. These ordered N vacancies result in formation of numerous stacking faults, which are accompanied by strong distortions in the crystal structure and a reduced lattice constant. The strain measurement indicates an isotropic distribution within the N-deficient layer (Figure 1). By a combination of HRTEM, EELS and ab-*initio* calculations, atomic and electronic structures are clarified. Moreover, a generalized relationship between the lattice constant and N vacancy concentration in CrN is established [5].

The second part is about randomly distributed defects in the films introduced during film deposition which are frequently encountered. To explore the defects in a quantitative way, a specimen consisting of three sublayers deposited under different substrate bias voltages, i.e. -40, -80 and -120 V, and having each 1 µm thickness, was chosen for visualizing the structural inhomogeneity. The cross-sectional bright field image shows no clear interface between the layers. Apart from C_S-corrected HRTEM image analysis, quantitative electron diffraction analysis was also performed. It reveals that the intensity ratios of (111), and (200) reflections (I_{111}/I_{200}) sensibly varies with the defect density. It is seen that the ratio noticeably increase from the layer grown at -40 V to the -80 V layer and to the -120 V layer as Figure 2 shows.

A high defect density also triggers a variation of electronic structure as observed by EELS measurements of different layers grown at different bias voltages. It is noted that a higher randomly distributed defect density in the film can also give a detectable change in the near edge fine structure, particularly reflected in the change of the N-K edge, as compared with 'perfect' bulk CrN.

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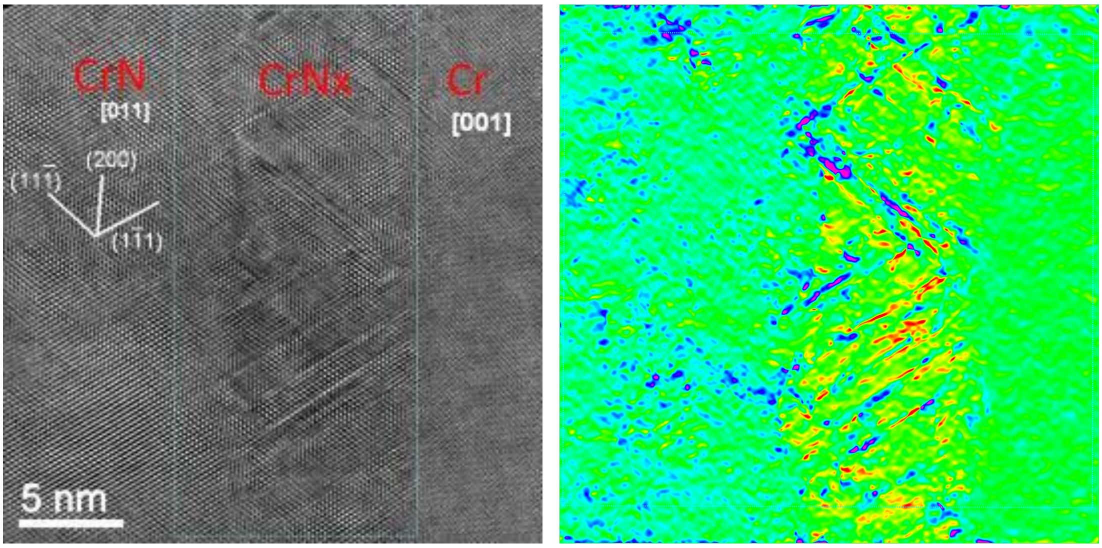

Figure 1. Left: a typical HRTEM image of the CrN/Cr interface along CrN [011] direction (Wien filtered), a defect layer between Cr and CrN originates from the ordered N-vacancies on {111} planes. Right: a strain map (e_{yy}) obtained by geometrical phase analysis, demonstrating the anisotropic distribution of strain within the defective CrN_x layer.

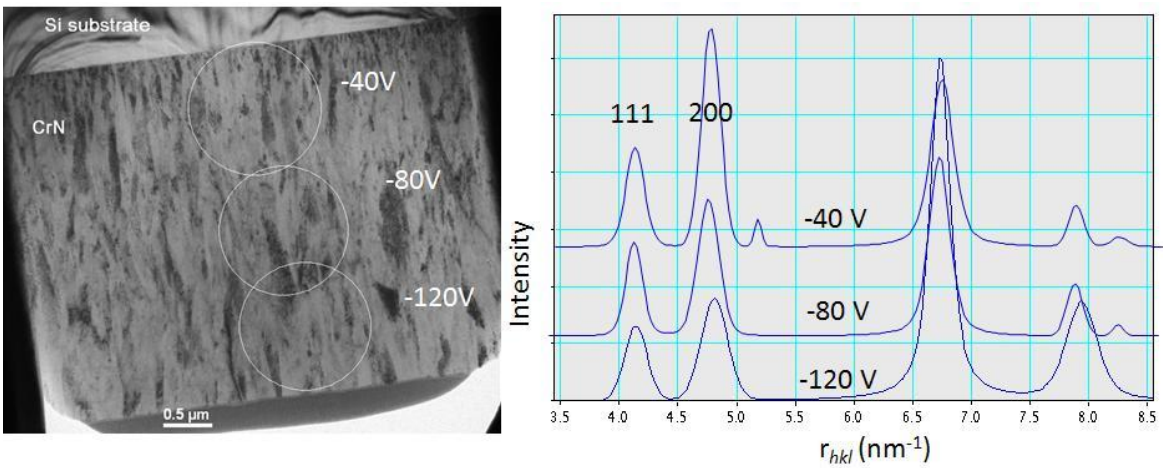

Figure 2. Left: A bright field image of a CrN layer on Si substrate prepared by focused ion beam method. The CrN layer is about 3 µm in thickness, deposited under different bias voltage, -40, -80 and -120 V. Right: The radial intensity distribution of ring pattern recorded at the different positions in the bright field image as schematically labeled. The plot indicates that the intensity ratio of (111) and (200) refections varies with the bias voltage. The detailed analysis gives a different ratio (I_{111}/I_{200}), which is 0.49, 0.57 and 0.6 at the respective layer deposited at a bias voltage of -40, -80 and -120 V.