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A comparative study of transport properties in polycrystalline and epitaxial chromium nitride films

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Polycrystalline CrN_x films on Si(100) and glass substrates and epitaxial CrN_x films on MgO(100) substrates were fabricated by reactive sputtering with different nitrogen gas flow rates (f_{N_2}). With the increase of f_{N_2} , a lattice phase transformation from metallic Cr_2N to semiconducting CrN appears in both polycrystalline and epitaxial CrN_x films. At $f_{\text{N}_2} = 100$ sccm, the low-temperature conductance mechanism is dominated by both Mott and Efros-Shklovskii variable-range hopping in either polycrystalline or epitaxial CrN films. In all of the polycrystalline and epitaxial films, only the polycrystalline CrN_x films fabricated at $f_{\text{N}_2} = 30$ and 50 sccm exhibit a discontinuity in $\rho(T)$ curves at 260–280 K, indicating that both the N-vacancy concentration and grain boundaries play important roles in the metal-insulator transition. © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4772682>]

I. INTRODUCTION

CrN has a lot of important practical applications in hard and protective coatings due to its high hardness and good corrosion resistance.^{1,2} On the other hand, CrN has also attracted much attention as potential electronic or spintronic materials owing to its magnetic and electronic properties.^{3,4} Considerable experimental and theoretical work have demonstrated that CrN undergoes a magnetic and structural phase transition from a paramagnetic NaCl structure at room temperature to a low-temperature antiferromagnetic orthorhombic P_{nma} phase at Néel temperature of 273–286 K.^{5–7}

A variety of electrical transport properties in CrN have been observed, such as, a semiconducting behavior with $d\rho/dT < 0$,^{8–11} a metallic behavior with $d\rho/dT > 0$,^{12–14} and continuous and discontinuous $\rho(T)$ curves at 260–280 K.^{8–13,15} These differences in electrical transport properties have been attributed to the sensitivity of the transport properties to N stoichiometry.^{12,15} In addition, the discontinuity in $\rho(T)$ curves has usually been observed in CrN powders or polycrystalline CrN films, rather than in epitaxial films, suggesting that the epitaxial constraints could affect the transition.^{14,16} Therefore, it is necessary to carry out a comparative study of the electrical transport properties of polycrystalline and epitaxial CrN films with different N concentrations in order to clarify the conductance mechanism.

In this article, CrN_x films fabricated on Si(100), glass, and MgO(100) substrates at different nitrogen gas flow rates (f_{N_2}) by reactive sputtering have been investigated systematically. With the increase of f_{N_2} , the dominant lattice phase transforms from metallic Cr_2N to semiconducting CrN . The polycrystalline CrN_x films fabricated at $f_{\text{N}_2} = 30$ and 50 sccm exhibit a discontinuity in $\rho(T)$ curves at 260–280 K, indicating

a metal-insulator transition, while the transition is absent in all of CrN_x films on MgO(100) substrates.

II. EXPERIMENTAL DETAILS

CrN_x films were fabricated on Si(100), MgO(100), and glass substrates by dc reactive facing-target sputtering from a pair of pure Cr targets (4N) with base pressure better than 8.0×10^{-6} Pa. The sputtering pressure was kept at 1.0 Pa with a total 100-sccm gas mixture of Ar (5N) and N_2 (5N), in which, the N_2 gas flow rates were set to 10, 20, 30, 50, 70, and 100 sccm. The substrate temperature was kept at 550 °C during film deposition. The film thickness was ~ 140 nm determined by a Dektak 6M surface profiler. The structures of the films were characterized by x-ray diffraction (XRD) and transmission electron microscopy (TEM). The chemical states of each element were analyzed using x-ray photoelectron spectroscopy (XPS). The electrical transport properties were measured by a standard four-terminal method using a Quantum Design physical property measurement system (PPMS-9) in the temperature range of 2–305 K.

III. RESULTS AND DISCUSSION

Figure 1 shows the XRD patterns (θ – 2θ) of CrN_x films on Si(100) substrates, revealing the evolution of various phases with different f_{N_2} . Obviously, there is a transformation from $\text{Cr}_2\text{N} + \text{CrN} \rightarrow \text{CrN}$ with the increase of f_{N_2} . At $f_{\text{N}_2} = 10$ sccm, three peaks from hexagonal $\text{Cr}_2\text{N}(\bar{1}\bar{1}1)$, (302), and $(\bar{2}\bar{2}1)$ and a peak from the face-centered-cubic (fcc) $\text{CrN}(111)$ can be observed. When f_{N_2} increases from 10 to 30 sccm, $\text{CrN}(200)$ and (220) peaks appear and become stronger, accompanied by the weakening of the peaks from hexagonal Cr_2N phase. CrN phase becomes dominated at $f_{\text{N}_2} = 30$ sccm. With further increasing the f_{N_2} (≥ 50 sccm), only the peaks from fcc CrN phase are present. Moreover, the (111) peak of CrN shifts to lower diffraction angle with

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FIG. 1. XRD patterns of CrN_x films deposited on Si(100) substrates at different f_{N_2} .

the increase of f_{N_2} because the increased nitrogen atoms make lattice constant become larger.¹⁵

Figure 2(a) gives the XRD patterns (θ – 2θ) of CrN_x films on MgO(100) substrates. The diffraction peaks from $\text{CrN}(h00)$ or the overlap of $\text{CrN}(h00)$ and $\text{MgO}(h00)$ can be observed, indicating that the films grow along the (100) orientation. However, tilting 2θ scans need to be carried out

FIG. 2. (a) XRD patterns of CrN_x films deposited on MgO(100) substrates at different f_{N_2} . (b) The tilting 2θ scans of CrN_x film fabricated at $f_{\text{N}_2} = 30$ sccm. (c) The tilting 2θ scans of CrN_x film fabricated at $f_{\text{N}_2} = 50$ sccm. In (111) lattice, $\alpha = 35.3^\circ$, $\beta = 45^\circ$; in (110) lattice, $\alpha = 45^\circ$, $\beta = 0^\circ$.

through rotating the sample by a certain degree to further clarify the structure, since the film fabricated at $f_{\text{N}_2} = 30$ sccm on Si(100) substrate consists of two phases and the orientation does not necessarily mean epitaxy. In tilting 2θ scans, (111) plane ($\alpha = 35.3^\circ$, $\beta = 45^\circ$) and (110) plane ($\alpha = 45^\circ$, $\beta = 0^\circ$) are chosen as the scanned planes. The tilting scans in Fig. 2(b) show that the $\text{Cr}_2\text{N}(002)$, $(\bar{2}\bar{2}1)$, and (300) peaks are visible, revealing that two phases exist in the film fabricated at $f_{\text{N}_2} = 30$ sccm on MgO(100) substrate. For the tilting scans in Fig. 2(c), only (111), (222), and (220) diffraction peaks from the overlap of CrN and MgO are visible, suggesting that the film fabricated at $f_{\text{N}_2} = 50$ sccm on MgO(100) substrate is epitaxial, as discussed for epitaxial $\text{Ti}_{1-x}\text{Cr}_x\text{N}$ films.¹⁷ Accordingly, the films fabricated at $f_{\text{N}_2} > 50$ sccm on MgO(100) substrates are epitaxial.

In order to further investigate the microstructure of CrN_x films, the high-resolution TEM analysis was carried out at CrN_x /substrate interface fabricated at $f_{\text{N}_2} = 30$ sccm, as shown in Fig. 3. The low-magnification image of CrN_x /glass interface is presented in Fig. 3(a), which shows a sharp interface and a film thickness of ~ 140 nm. Figure 3(b) gives the high-resolution TEM image at CrN_x /glass interface, from which different oriented grains with blurred grain boundaries can be observed. The d spacings indexed in Fig. 3(b) are from dominant $\text{CrN}(200)$ and minor $\text{Cr}_2\text{N}(102)$ planes. The corresponding selection area electron diffraction (SAED) in the inset of Fig. 3(b) displays rings, indicating a polycrystalline structure. Similarly, a film thickness of ~ 140 nm can be observed in Fig. 3(c), though the contrast of the CrN_x /MgO interface is blurred. The high-resolution TEM image in Fig. 3(d) shows the quasi-epitaxial growth of the film. The inset of Fig. 3(d) is the corresponding SAED, where the diffraction spots correspond to fcc structure. Actually, the spots are from the overlap of dominant fcc CrN and Cr_2N , such as the overlap of $\text{CrN}(220)$ and $\text{Cr}_2\text{N}(300)$, which is consistent with the tilting 2θ scans in Fig. 2(b), confirming the existence of two phases in the film fabricated at $f_{\text{N}_2} = 30$ sccm on MgO(100) substrate.

For analyzing the valance states of the elements in the films fabricated at different f_{N_2} , XPS measurements were performed to analyze the binding energy of N_{1s} and Cr_{2p} , as shown in Fig. 4. In Fig. 4(a), the N_{1s} peak positions of the films fabricated at $f_{\text{N}_2} = 30$ sccm are 396.8 and 397.0 eV, for MgO and Si substrate, respectively, while the Cr $2p$ peak positions in Fig. 4(b) are identical for MgO and Si substrate. With the increase of f_{N_2} on Si substrate, there is a negative chemical shift of N_{1s} peak from 397.0 to 396.8 eV, as shown in Fig. 4(a). Meanwhile, in Fig. 4(b), Cr $2p_{3/2}$ peak shifts from 574.1 to 574.4 eV and Cr $2p_{1/2}$ peak shifts from 583.4 to 583.8 eV with the increase of f_{N_2} on Si substrate. The simultaneous occurrence of negative chemical shift of N_{1s} peak and positive chemical shift of Cr $2p$ peak with the increase of f_{N_2} can be interpreted as increasing charge transfer from Cr to N with increasing nitrogen,¹⁸ indicating a decrease of N-vacancy concentration with the increase of f_{N_2} .

Figure 5(a) shows the temperature dependence of the normalized resistivity $\rho(T)/\rho(305\text{ K})$ of polycrystalline CrN_x films deposited on glass substrates under different f_{N_2} . With the increase of f_{N_2} , the conductance characteristic transforms from metallic to semiconducting. The films fabricated at

FIG. 3. (a) Cross-section TEM image and (b) cross-section high-resolution TEM image at the $\text{CrN}_x/\text{glass}$ interface fabricated at $f_{\text{N}_2}=30$ sccm. (c) Cross-section TEM image and (d) cross-section high-resolution TEM image at the CrN_x/MgO interface fabricated at $f_{\text{N}_2}=30$ sccm. The inset of (b) or (d) is the corresponding SAED.

FIG. 4. (a) N_{1s} and (b) Cr_{2p} XPS spectra of CrN_x films fabricated at different f_{N_2} .

$f_{\text{N}_2}=10$ and 20 sccm are metallic, i.e., the resistivity decreases as temperature decreases. At $f_{\text{N}_2}=30$ sccm, the film exhibits a metal-insulator-like transition around 263 K [Fig. 5(b)]. When f_{N_2} reaches 50 sccm, the resistivity shows a steep decrease with decreasing temperature in the range of 260 – 280 K, and the resistivity increases with decreasing temperature for temperature less than 145 K. The resistivity of the films fabricated at $f_{\text{N}_2}=70$ and 100 sccm increases with decreasing temperature, exhibiting semiconducting behavior [Fig. 5(c)]. As shown in the inset of Fig. 5(a), the room-temperature

FIG. 5. (a) Normalized resistivity versus temperature of CrN_x films deposited on glass substrates. The top inset shows the dependence of $\rho(305\text{ K})$ on f_{N_2} . (b) The magnified ρ - T plot of the films fabricated at $f_{\text{N}_2}=30$ and 50 sccm. (c) $\log \rho$ - T^{-1} curves for the films fabricated at $f_{\text{N}_2}=70$ and 100 sccm.

resistivity increases from 1.44×10^{-4} to $0.25 \Omega \text{ cm}$ with the increase of f_{N_2} , which is consistent with nitrogen-dependent resistivity of other transition metal nitrides such as ZrN ,^{19,20} TiN ,¹⁹⁻²¹ or NbN ,²² and should be attributed to the increase of nitrogen composition.¹⁹⁻²² Figure 5(c) is a semilog plot of the normalized resistivity vs T^{-1} , for polycrystalline CrN films fabricated at $f_{\text{N}_2}=70$ and 100 sccm. The plotted curves are nonlinear over the measured temperature range, indicating that the temperature dependence of resistivity cannot simply be explained by thermal activation.

Figure 6(a) shows the temperature dependence of the normalized resistivity $\rho(T)/\rho(305 \text{ K})$ of CrN_x films on $\text{MgO}(100)$ substrates fabricated at different f_{N_2} . The films fabricated at $f_{\text{N}_2} \leq 30$ sccm are metallic, while the films fabricated at $f_{\text{N}_2} \geq 50$ sccm are semiconducting. It should be noted that the films fabricated at $f_{\text{N}_2}=30$ and 50 sccm show no anomaly on temperature dependence of resistivity, as shown in Fig. 6(b). The room-temperature resistivity of the films in the inset of Fig. 6(a) increases from 1.74×10^{-4} to $1.24 \Omega \text{ cm}$, as f_{N_2} increases from 10 to 100 sccm. Similarly, $\rho(T)$ behaviors of the semiconducting epitaxial CrN films cannot also be described by thermally activated model, as shown in Fig. 6(c).

The variable-range hopping (VRH) resistivity of disordered systems was shown by Mott to behave as $\ln \rho \propto T^{-1/4}$,^{23,24} while Efros and Shklovskii (ES) argued that Coulomb interactions create a gap that leads to $\ln \rho \propto T^{-1/2}$ at low temperatures.^{25,26} Here, we use a universal scaling relation considering both Mott VRH and ES hopping, which has previously been reported for epitaxial $\text{CrN}(001)$ films by Zhang *et al.*¹¹ The expression first proposed by Aharony *et al.*²⁷ is

$$\ln(\rho/\rho_0) = Af(T/T_x), \quad (1)$$

where the scaling factors ρ_0 , A , and T_x depend on individual material properties, but the function $f(x)$ is universal, given by²⁷

$$f(x) = \frac{1 + [(1+x)^{1/2} - 1]/x}{[(1+x)^{1/2} - 1]^{1/2}}. \quad (2)$$

FIG. 6. (a) Normalized resistivity versus temperature of CrN_x films deposited on $\text{MgO}(100)$ substrates. The top inset shows the dependence of $\rho(305 \text{ K})$ on f_{N_2} . (b) The magnified ρ - T plot for the films fabricated at $f_{\text{N}_2}=30$ and 50 sccm. (c) $\log \rho$ - T^{-1} curves for the films fabricated at $f_{\text{N}_2}=50$, 70, and 100 sccm.

To investigate whether our data can be described by the scaling function or not, the measured resistivity data are fitted using Eqs. (1) and (2) for the semiconducting CrN films. The resulting curves are plotted as red solid lines in Fig. 7. The scaling function could only well describe the temperature-dependent resistivity for polycrystalline CrN film fabricated at $f_{\text{N}_2}=100$ sccm for $15 \text{ K} < T < 100 \text{ K}$ in Fig. 7(a) and the corresponding epitaxial film for $63 \text{ K} < T < 160 \text{ K}$ in Fig. 7(b). These results indicate that the low-temperature electrical transport of CrN with the least N vacancy is dominated by both Mott VRH and ES VRH.

Finally, we focus on the discontinuity in $\rho(T)$ curves that has been reported to be associated with a structure transition of CrN from cubic to orthorhombic. At $f_{\text{N}_2}=30$ and 50 sccm, polycrystalline CrN_x films show a transition of resistivity at 260–280 K. Here, the N-vacancy concentration may be just appropriate for the structure transition. The structure of cubic CrN without nitrogen vacancies is shown in Fig. 8(a), where the perfect lattice makes the transition difficultly appear, such as the CrN films fabricated at high f_{N_2} . However, when there are nitrogen atoms lost, it is easy for the structure transition to happen near T_N accompanying the relaxation of lattice strain, such as the films fabricated at low f_{N_2} . Figures 8(b) and 8(c) show the view of a $2 \times 2 \times 2$ CrN supercell with two nitrogen atoms lost along $[110]$ direction, resulting in the lattice contraction along $[110]$ direction and $\alpha < 90^\circ$, as the temperature decreases and the lattice strain is relaxed. Thus, the structure transforms from cubic to orthorhombic.

With further increasing f_{N_2} (> 50 sccm), N-vacancy concentration is small and the crowded nitrogen atoms go against the shear deformation of CrN grains into the orthorhombic structure. At lower f_{N_2} (10 and 20 sccm), N-vacancy concentration should be large, but the transition of resistivity cannot be observed because the dominant phase is metallic Cr_2N at lower f_{N_2} , as shown in XRD results of Fig. 1, and the transition from CrN phase could be neglected, if any.

In contrast, for $f_{\text{N}_2}=30$ and 50 sccm, CrN_x films on MgO substrates, do not show a transition of resistivity. As shown in Figs. 8(d) and 8(e), the presence and absence of phase transition may be explained as following: Polycrystalline CrN film

FIG. 7. Normalized resistivity versus temperature for (a) polycrystalline and (b) epitaxial CrN films. The lines are fitting data by Eqs. (1) and (2), and the symbols are the experimental data.

FIG. 8. Perspective view of (a) the cubic CrN cell and (b) the cubic CrN cell with two N atoms lost. (c) Perspective view and top view of the orthorhombic CrN cell with two N atoms lost. The red balls represent the nitrogen vacancies. Schematic diagram of (d) polycrystalline CrN film with several grains and (e) epitaxial CrN film.

should exhibit the phase transition since the transition is likely facilitated by underdense grain boundaries that allow grain slide and independent shear deformation for each grain. However, it is very hard for epitaxial CrN film to distort into orthorhombic structure, as two of its three crystal axes are fixed by the substrate.

IV. CONCLUSION

Both polycrystalline and epitaxial CrN_x films fabricated by reactive sputtering show a phase change from $\text{Cr}_2\text{N} + \text{CrN}$ to CrN with increasing f_{N_2} . Meanwhile, there is a transformation of conductance from metallic to semiconducting, and at $f_{\text{N}_2} = 100$ sccm, the electrical transport at low temperatures is described by both Mott and ES VRH. The polycrystalline CrN_x films fabricated at $f_{\text{N}_2} = 30$ and 50 sccm exhibit a discontinuity in $\rho(T)$ curves at 260–280 K, while the transition is absent in all of CrN_x films on $\text{MgO}(100)$ substrates. The metal-insulator transition of CrN is strongly affected by N-vacancy concentration as well as the substrate and associated grain boundaries.

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