Microstructure and thermoelectric properties of CrN and CrN/Cr$_2$N thin films

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Abstract

CrN thin films with an N/Cr ratio of 95% were deposited by reactive magnetron sputtering onto (0001) sapphire substrates. X-ray diffraction and pole figure texture analysis show CrN (111) epitaxial growth in a twinned domain fashion. By changing the nitrogen versus argon gas flow mixture and the deposition temperature, thin films with different surface morphologies ranging from grainy rough textures to flat and smooth films were prepared. These parameters can also affect the CrN$_x$ system, with the film compound changing between semiconducting CrN and metallic Cr$_2$N through the regulation of the nitrogen content of the gas flow and the deposition temperature at a constant deposition pressure. Thermoelectric measurements (electrical resistivity and Seebeck coefficient), scanning electron microscopy, and transmission electron microscopy imaging confirm the changing electrical resistivity between 0.75 and 300 m$\Omega$cm, the changing Seebeck coefficient values between 140 and 230 $\mu$VK$^{-1}$, and the differences in surface morphology and microstructure as higher temperatures result in lower electrical resistivity while gas flow mixtures with higher nitrogen content result in single phase cubic CrN.

Keywords: magnetron sputtering, thermoelectrics, chromium nitride, seebeck coefficient, thin films

Supplementary material for this article is available online

(Some figures may appear in colour only in the online journal)
1. Introduction

Chromium nitride (CrN) is a hard and corrosion resistant compound that has gained interest for various applications such as medical implants [1], silver luster decorative coatings [2], and wear-resistant coatings for cutting tools especially when hot corrosion resistance is needed [3, 4]. Chromium nitride is an interstitial compound, in which nitrogen atoms reside in the octahedral spaces between the chromium atoms in an fcc lattice (i.e. rock-salt cubic), making the compound susceptible to stoichiometry deficiency. There is also a second interstitial compound, hexagonal Cr2N, which can be formed in CrN films as a secondary phase [5, 6]. In addition to the more traditional applications and properties of CrN, studies on the thermoelectric properties of CrN have shown that CrN has a moderate electrical resistivity, low thermal conductivity, and high Seebeck coefficient values. Quintela et al [7] reported on 1:1 stoichiometric CrN thin films, showing a Seebeck coefficient of approximately $-120 \mu V K^{-1}$ and a dimensionless figure of merit ($\zeta T$) of 0.12 at room temperature (compared to 0.1 for PbTe:Tl). Their work emphasizes the fact that annealed samples have better electrical and thermal conductivity, resulting in films with enhanced thermoelectric properties. Kerdsongpanya et al [8] reported a Seebeck coefficient of $-230 \mu V K^{-1}$ and a power factor of $1.7 \times 10^{-3} \text{Wm}^{-1} \text{K}^{-2}$ near 400 °C for CrN thin films deposited on sapphire by magnetron sputtering. The high electrical resistivity of CrN stems from the localized 3d orbitals which give the electrons large effective masses and consequently high electrical resistivity and low thermal conductivity (between 1.7 and 4.0 $\text{Wm}^{-1} \text{K}^{-1}$) [9–11]. With CrN oxidizing at 700 °C and the thermal decomposition (in an inert atmosphere) starting at 1000 °C, CrN has the potential to be a suitable n-type semiconductor for mid-to-high-temperature thermoelectrics. As for semiconducting properties, previous reports have announced different band gaps for CrN. Constantin et al [12] prepared 1:1 stoichiometric CrN thin films deposited by molecular beam epitaxy, and reported a resistivity-derived 71 meV band gap at room temperature, by using the lin$(\rho/\rho_0) = (E_g/2k_B)(1/T)$ equation which relates the resistivity of a semiconductor to ambient temperature and the energy band gap where $\rho$ is the electrical resistivity, $\rho_0$ is the resistivity pre-factor, $E_g$ is the semiconductor band gap, $k_B$ is the Boltzmann constant and $T$ is temperature. Zhang et al [13] measured the optical properties of CrN and concluded that the band gap is 0.19 eV with an error bar of 0.46 eV, demonstrating difficulties in this regard. On the other hand, at low temperatures the resistivity of CrN indicates metallic properties [14]. This is explained by a magnetic phase transformation at the Néel temperature (approximately 280 K), which causes antiferromagnetic orthorhombic CrN to transform into paramagnetic rock-salt cubic CrN with temperature increase. Adding N vacancies and/or other defects coupled with a narrow band gap will increase the charge carrier density resulting in metallic conducting properties [15–17]. Gall et al [18] measured an optical band gap of approximately 0.7 eV and conclude that the semiconducting properties stem from the existence of magnetic moments and the Hubbard energy rather than the crystal structure, indicating a Mott insulator. They also noted that depending on crystal quality, the temperature dependence of electrical resistivity ($d\rho/dT$) may either be positive or negative for the low-temperature orthorhombic phase.

In general, these studies have led researchers to propose chromium nitride as a possible thermoelectric material in addition to other traditional thermoelectric materials which are limited to niche applications [19]. For example, products based on bismuth telluride (Bi$_2$Te$_3$), which has one of the highest $\zeta T$ values at room temperature (approximately 1), and lead telluride (PbTe), which operates with a significant $\zeta T$ of 1.4 at 500 °C, are both restricted because of the low global production of tellurium [20, 21]. As a result, the chemical stability and mechanical properties of CrN could address this and other limiting factors [22–24].

In order to optimize semiconductors for thermoelectric properties and expand their use in a wider range of high temperature applications, cheaper and more stable materials like CrN should be explored. Nitride thin films (and other thermoelectric material) can be optimized by adding dopants, synthesizing semiconductor/metal multilayers and nanostructuring, leading to the simultaneous decrease of the electrical resistivity and the thermal conductivity [25, 26] and resulting in an improved thermoelectric performance (or figure-of-merit). The interest in using thermoelectrics based on early transition-metal nitrides is further underlined by the results on ScN-based materials, both pure ScN [27–31], ScN-based solid solutions and alloys [32–34], and superlattices [35–37].

The thermoelectric properties of CrN (which can be further enhanced by alloying [38]) present this material as a promising semiconductor for use in mid-temperature thermoelectric applications. However, the effects of the microstructure and the CrN, system on the thermoelectric properties are not well understood as both properties can affect the electronic band structure (hence the Seebeck coefficient), electrical resistivity and thermal conductivity (by phonon scattering). Here, we study the effects of film microstructure and phase purity on the Seebeck coefficient, electrical resistivity and thermal conductivity (by phonon scattering).

The deposition process was carried out by reactive dc magnetron sputtering in a high vacuum chamber base pressure ($3.0 \times 10^{-7}$ mbar at deposition temperature). The films were deposited onto (0001) sapphire substrates ($10 \times 10 \times 0.5 \text{mm}^3$, ultrasonically cleaned in acetone and isopropanol 5 min each and blown dry using N$_2$) that were heated at 600, 650 and 700 °C for 1 h prior to the deposition process. These temperatures were also maintained during the deposition process. The 300 nm thick films were deposited using a 7.5 cm Cr (Lesker, 99.95% pure) target and an argon/nitrogen gas mixture (99.9997% pure for both gases). The target power for
The electrical resistivity of all samples was obtained by measuring the sheet resistance of the films with a four-point probe Jandel RM3000 station and multiplying the sheet resistance with the film thickness (obtained from cross sectional SEM imaging). In-plane electrical resistivity and Seebeck coefficient value measurements of CrN and CrN/Cr2N samples were simultaneously performed from room temperature up to 800 K by an Ulvac-Riko Zem 3 system with a special setup for thin film measurements in a low-pressure helium atmosphere. The measurement error is no more than 7% for this setup as mentioned in [8]. Temperature-dependent Hall carrier concentrations were determined on 5 × 5 mm² substrates using the IPM-HT Hall-900 K system (developed and constructed by Fraunhofer IPM in Freiburg, Germany) and includes a 10% error bar. Thermal conductivity of CrN and Cr2N was determined by modulated thermorelectance microscopy (MTRM). In this setup, a pump beam at 532 nm (delivered by a Cobolt Samba MLD laser and with the intensity modulated by an acousto-optical modulator at a frequency f) is focused on the surface of the sample with an objective lens (N.A. = 0.5). The samples were coated by a 100 nm gold layer, ensuring that the heat source is located at the surface. Thermal waves are excited in the sample and monitored by the reflectivity surface change recorded around the pump location by another focused laser beam. A 488 nm Oxius laser is used to maximize the probe sensitivity towards the thermal field in the gold cap layer. A photodiode and a lock-in amplifier recorded the AC reflectivity component, in a frequency range between 1 kHz and 1 MHz. Finally, the experimental profiles of the amplitude and the phase of the reflected probe beam were fitted according to a standard Fourier diffusion law to extract the thermal conductivity of the films and includes a measurement error of 15% [42–48].

3. Results

3.1. Initial experiments

The main growth parameters for the reactive sputter-deposition of chromium nitride were the substrate deposition temperature (kept constant for 1 h before initiating the growth process) and the argon/nitrogen gas flow mixture. In an initial study of deposition temperature, we observed that low temperature deposition at 550 °C or lower will lead to the formation of polycrystalline films with high electrical resistivity, while deposition temperatures higher than 700 °C typically results in predominant growth of Cr₂N over cubic CrN consistent with earlier observations [49]. Thus, we limit the presentation of experiments below to the most important temperature ranges between 600 °C and 700 °C.

The gas flow mixture of argon and nitrogen also affected the films. A 60% argon and 40% nitrogen gas flow mixture resulted in sharp CrN (1 1 1) XRD peaks. However, an 80%
argon and 20% nitrogen gas flow mixture led to the formation of the Cr$_2$N phase. Thus, the 20% nitrogen gas mixture (11 sccm N$_2$, 43 sccm Ar) is used to study phase mixtures of CrN and Cr$_2$N while as the 40% nitrogen gas mixture (21 sccm N$_2$, 33 sccm Ar) results in single phase CrN. The aim of this research is to study the thermoelectric properties of chromium nitride showing this phase variation: CrN, Cr$_2$N and CrN/Cr$_2$N.

3.2. Effects of process parameters on CrN film growth

Figure 1(a) shows $\theta - 2\theta$ diffractograms for a set of CrN thin films deposited at 600 °C, 650 °C, and 700 °C under the conditions of a 40% nitrogen gas mixture and a grounded substrate. In addition, XPS measurements show that all the studied CrN films are sub-stoichiometric, with N = 45% and Cr = 55% (considering a 3% error bar). XPS spectra for all samples are available in the supplementary material. These values match the elastic recoil detection analysis (ERDA) values obtained by Kerdsongpanya et al [8] for similar samples deposited in the same PVD system. As for the single phase Cr$_2$N films, the approximate stoichiometry is N = 69% and Cr = 30%. Oxygen contamination of all films was measured to be less than 2%.

The diffraction pattern shows the CrN (1 1 1) peak located at $2\theta = 37.49^\circ$ corresponding to a 0.23 nm lattice spacing. The peak located at $2\theta = 41.69^\circ$ is the sapphire (0 0 0 6) substrate peak. The inset shows a zoomed image of the CrN (1 1 1) peak. The fringes are so called Laue oscillations (layer thickness fringes) which appear only for very smooth films, and the separation of these correspond to the film thickness.
Figure 1(b) shows $\theta - 2\theta$ diffractograms for a similar set of samples when deposited with 20% nitrogen gas mixture. At 600°C, a Cr$_2$N (0 0 0 2) phase impurity peak (with a smaller intensity compared to the CrN peak) is seen at approximately $2\theta \approx 40^\circ$. Increasing the deposition temperature to 650°C decreases the Cr$_2$N (0 0 0 2) peak intensity, while an increase to 700°C will result in single-phase Cr$_2$N.

Figures 2(a) and (b) shows rocking curve measurements of single phase CrN (1 1 1) deposited at 700°C comparing the full width at half maximum (FWHM) of the (1 1 1) and (2 2 2) peaks. The FWHM for CrN (1 1 1) is 0.015° compared to the 0.041° for CrN (2 2 2), indicating the existence of mosaicity.

Figure 3 is a pole figure of the CrN {1 1 1} peaks (single phase CrN, deposited at 700°C) and includes six equally distanced and discrete poles surrounding the central pole at $\psi = 70.5^\circ$ which is the angle between {1 1 1} crystal planes in a cubic crystal structure. A single crystal fcc structures deposited in the [1 1 1] direction should only show three surrounding poles, indicating a twin domain epitaxial growth (where the domains are rotated 60° with respect to each other) [50], a well-known feature for cubic material deposited on sapphire.

Table 1 lists sample features according to their processing parameters, compound phase, roughness analysis, electrical resistivity and charge carrier concentration measurements for samples shown in figures 1 and 2.

Table 1. Deposition temperature, nitrogen gas flow mixture, roughness measurement, electrical resistivity and charge carrier concentration measurements for samples shown in figures 1 and 2.

| Sample name | N$_2$ gas (% | Deposition temp. °C | Phase and thickness | Rq (nm) | Peak to peak (nm) | $\rho$ at RT ($\mu\Omega \cdot cm$) | $k$ at RT (W m$^{-1}$ K$^{-1}$) | $n$ ($\times 10^{18}$ cm$^{-3}$) |
|-------------|---------------|----------------------|----------------------|---------|-------------------|-------------------------------|----------------|------------------|
| Cr40-700    | 40            | 700                  | Cubic CrN 280 nm     | 3       | 18                | 750                           | 4.0            | 39               |
| Cr40-650    | 650           | CrN 280 nm           | 8                   | 50      | 300000            | 50                            | 12.0           | 81700            |
| Cr40-600    | 600           | CrN 280 nm           | 5                   | 29      | 300000            | 300000                       | 310            | 1190             |
| Cr20-700    | 20            | 700                  | Hex. Cr$_2$N 280 nm  | —       | —                 | 830                           | 12.0           | 81700            |
| Cr20-650    | 650           | CrN/Cr$_2$N 310 nm   | —                   | —       | 830               | 12.0                          | 42             | 193              |
| Cr20-600    | 600           | CrN/Cr$_2$N 320 nm   | —                   | —       | 1300              | 12.0                          | 81700          | 1190             |

Figure 3 shows the SEM images of the samples corresponding to the diffraction patterns in figure 1. These images show a temperature-activated change in the surface morphology. The insets in images (a)–(c) belong to AFM measurements performed on the single phase CrN samples.
show a large variation in the electrical resistivity depending on the deposition temperature. Films deposited at 600 °C (sample Cr40-600) show a resistivity of 300 mΩ cm, while as films deposited at 700 °C (sample Cr40-700) show a resistivity of 0.75 mΩ cm (400 times lower). Samples Cr20-600, Cr20-650 and Cr20-700 (deposited with a 20% N₂ gas mixture) show a competing relationship between film deposition temperature and electrical resistivity, as the low nitrogen mixture during the deposition process leads to the formation of the metallic secondary phase Cr₂N. Although single phase Cr₂N has a relatively low electrical resistivity of 0.05 mΩ cm, the mixture of CrN and Cr₂N does not decrease the electrical resistivity below that of sample Cr40-700 with a resistivity of 0.75 mΩ cm.

In order to study the secondary phase Cr₂N inside the film, TEM images of single phase CrN (sample Cr40-700) are compared with the CrN/Cr₂N mixture films (sample Cr20-650 and Cr20-600). Figure 4(a) shows a bright field TEM image for CrN films deposited at 700 °C with a 40% nitrogen gas flow mixture (Cr40-700). Figure 4(b) shows the HRTEM image and the selected area electron diffraction (SAED) pattern of the same film, exhibiting epitaxial growth. The observed lattice spacing of 0.23 nm corresponds to the CrN d(111) lattice spacing in the XRD diffraction pattern. Figure 4(c) shows a TEM image of sample Cr20-650 deposited at 650 °C with a 20% nitrogen gas flow mixture. The HRTEM image in figure 4(d) shows that these deposition parameters lead to the formation of 10 × 15 nm nanoinclusions which can be seen residing in the CrN matrix.

Figure 4(e) shows a TEM image of a CrN/Cr₂N film deposited at a lower temperature (sample Cr20-600). The corresponding HRTEM image (figure 4(f)) shows that a decrease in the deposition temperature has caused an increase in the Cr₂N grain size in the film and results in 20 × 50 nm Cr₂N nanoinclusions randomly dispersed in the CrN matrix. These changes also include crystal defects and grain boundaries.

3.3. Thermoelectric properties

Thermoelectric measurements (i.e. the Seebeck coefficient and the electrical resistivity) as a function of temperature were measured to study the effect of the secondary phase Cr₂N when mixed with CrN films which includes Cr40-700, Cr20-600, and Cr20-650. In addition, the Seebeck coefficient and electrical resistivity of single phase CrN was also measured as a reference (sample Cr20-700). Figure 5(a) shows the temperature dependent electrical resistivity of all measured samples. For single phase CrN and CrN with small-sized nanoinclusions (Cr40-700 and Cr20-650) the resistivity increases with increasing temperature (metallic-like behavior) and peaks near 700 K before showing a decrease. This metal-like behavior is unexpected for semiconductors. For the CrN/Cr₂N films with 20 nm× 50 nm nanoinclusions (Cr20-600), the resistivity peaks at temperatures near 550 K before showing the decrease.

Figure 5(b) presents the Seebeck coefficients as a function of temperature for the investigated samples. The results show that small-sized (10 nm × 15 nm particles) CrN phase impurities does not have a large effect on the Seebeck coefficient and the values are approximately the same as single phase CrN with a $-230 \mu VK^{-1}$ Seebeck coefficient at 600 K. The Seebeck coefficient of samples with large-sized (20 nm ×
50 nm particles) Cr$_2$N impurities is measured to be approximately $-160 \mu$VK$^{-1}$ and is almost constant with temperature. All measurements show negative Seebeck coefficients characteristic of n-type semiconducting behavior.

Figure 5(c) shows the power factor for all of the mentioned samples. The results show power factors as high as $5.0 \times 10^{-3}$ Wm$^{-1}$K$^{-2}$ at room temperature for single phase and low impurity CrN films and $1.5 \times 10^{-3}$ Wm$^{-1}$K$^{-2}$ for the film with large amounts of Cr$_2$N. The power factor decreases with temperature (because of the electrical resistivity increase) and drops to $2.5 \times 10^{-3}$ Wm$^{-1}$K$^{-2}$ near 800 K. Additional measurements on single phase CrN do not show any major changes in either the thermoelectric properties or the XRD pattern.

Table 1 also includes the temperature-dependent Hall carrier concentration results for the mentioned samples at room temperature and 800 K. As expected, there is a steady increase in the samples carrier concentration depending on the phase, starting from $0.39 \times 10^{19}$ cm$^{-3}$ for CrN and increasing up to $8.17 \times 10^{22}$ cm$^{-3}$ for Cr$_2$N (room temperature measurements). These results stem from the respective semiconducting and metallic properties.

Finally, modulated thermoreflectance microscopy was used to measure the thermal conductivity of single phase CrN and single phase Cr$_2$N at room temperature. The results show that epitaxial and semiconducting CrN (with a reduced number of defects and grain boundaries) has a thermal conductivity of approximately 4.0 Wm$^{-1}$K$^{-1}$ while as metallic Cr$_2$N showed a value of approximately 12.0 Wm$^{-1}$K$^{-1}$.

4. Discussion

4.1. Film growth and structure

It is well known that higher deposition temperatures lead to enhanced adatom mobility and grain boundary diffusion resulting in grain coalescence and coarsening [51]. As a result, higher deposition temperatures could lead to a decrease in the defect density and grain boundaries (which affect electron mobility by scattering) leading to better electrical conductivity. This could explain the large power factor of sample Cr40-700 compared to previously reported power factors of polycrystalline bulk samples [52]. It should be noted that the CrN (1 1 1) peak belonging to CrN is smaller than the Cr$_2$N (1 1 1) peak. This indicates that a rise in the temperature will scatter the charge carriers and increase the electrical resistivity. However, the most intriguing result is the anomalous electrical resistivity of CrN and CrN/Cr$_2$N (figure 5(a)). Unlike normal semiconducting behavior where resistivity decreases with temperature, the electrical resistivity of under-stoichiometric CrN and CrN/Cr$_2$N films increases with temperature. This behavior contrasts with the Seebeck coefficient values which matches that of n-type semiconducting. A likely explanation is that the nitrogen vacancies are acting as donors by greatly increase the charge carrier density, creating non-zero quasi-metallic density of states at the Fermi level for the sample [57, 58]. In this case, a rise in the temperature will probably enhance electron–electron scattering resulting in a small decrease in the conductivity values.

Thermal conductivity measurements show that sample Cr$_2$N has a room temperature thermal conductivity of 4.0 Wm$^{-1}$K$^{-1}$. This is the upper value in CrN thermal conductivity where electron and phonon scattering are minimized. Thus, an estimated $\zeta T$ value at room temperature for single phase CrN thin films would be less than 0.4.

As for the Cr$_2$N film (sample Cr20-700), its metallic properties result in a near-zero Seebeck coefficient which is not suitable for thermoelectrics. However, CrN also shows a relatively low thermal conductivity value with good electrical conductivity and can be further studied for other applications such as diffusion barriers [59] or hard coatings [60].
5. Conclusions

In summary, chromium nitride thin film synthesis and characterization is reported. Deposition temperatures and gas flow mixtures can be used to change the properties of the film (i.e. Seebeck coefficient, electrical conductivity) by altering the crystal microstructure and/or adding secondary phases. Low nitrogen gas flow mixture will result in the formation of a secondary phase—hexagonal Cr$_2$N—which takes shape in the form of randomly dispersed nanoinclusions and embedded in a CrN matrix.

Single phase CrN and CrN/Cr$_2$N films (with small amounts of Cr$_2$N phase impurities) show thermoelectric properties with the power factor reaching a maximum value of $5.0 \times 10^{-3}$ Wm$^{-1}$K$^{-2}$ at 300 K and $2.5 \times 10^{-3}$ Wm$^{-1}$K$^{-2}$ near 800 K. The maximum Seebeck coefficient is measured to be $-230 \mu$VK$^{-1}$ at 600 K. The room temperature thermal conductivity for the single phase CrN film is measured to be 4.0 Wm$^{-1}$K$^{-1}$. CrN thin films show an interesting behavior of increasing electrical resistivity with temperature (which is the opposite behavior for semiconductors). Also, adding metallic Cr$_2$N nanoinclusions to the CrN matrix increases the electrical resistivity of the film due to the increased number of grain boundaries and crystal defects. Thus, for future research, it would be advisable to alloy CrN with a metallic compound that shares the same rock-salt cubic crystal structure in order to prevent the formation of such defects. This could lead to an enhancement of the power factor and thermoelectric figure of merit.

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