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Full Length Article

Investigation of percolation thickness of sputter coated thin NiCr films on clear float glass

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1. Introduction

The structure and the optical properties of thin metal films are of considerable interest due to their potential use in optical coatings. Thin metallic films are widely used in low emissivity coatings as buffer layers and/or interlayers [1]. The microstructure and morphology of thin metallic films deposited on glass substrates depend on growth conditions. Layer-layer, three-dimensional (3D) island, layer-island are three main growth mechanisms [2,3]. The key target of this study is to understand the kinetic growth mechanism and investigate the percolation thickness of thin NiCr films deposited by magnetron sputtering. Percolation phenomena occurs in the early stages of film growth. Percolation thickness of a thin film can be defined as the first point in the growth process at which connected clusters exist across the coating [4]. In this work, critical percolation thickness of sputter coated thin NiCr films on clear float glass was investigated by spectroscopic ellipsometry. The results were also supported by four point probe measurements. Analysis of the optical and electrical properties of NiCr films was performed by spectroscopic ellipsometry to demonstrate the island-percolation transition of the films. NiCr films are not expected to be conductive for the thicknesses below the percolation thickness. Film growth is expected to start with seperate islands and a continuous film is formed after a certain film thickness. We used Drude-Lorentz

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ABSTRACT

Percolation thickness of reactively sputtered nickel chromium (NiCr) thin films is reported in this study. Nickel-chromium films with the thicknesses in between 1 and 10 nm were deposited on 4 mm clear glass substrate by dc magnetron sputtering. Optical properties such as refractive index, extinction coefficient and also sheet resistance, carrier concentration and mobility of NiCr films were determined by a combination of variable-angle spectroscopic ellipsometry and four point probe measurements. We show both the percolation phenomena in atmosphere and critical percolation thickness for thin NiCr films by both electrical and optical techniques. The two techniques gave consistent results with each other.

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Model in order to fit ellipsometric data of NiCr films. The model showed that NiCr thin films of different thickness have different structural behavior [5].

2. Sample preparation

NiCr films were deposited on commercially available 4 mm clear soda-lime-silicate (SLS) float glass (SISECAM, Turkey) with the dimensions of 10×10 cm. Before deposition, 4 step cleaning process (tap water, basic solution, acidic solution, deionized water) was performed on the samples. The films were deposited in large scale industrial in-line horizontal coater HISS 300S (Von Ardenne, Germany) by DC magnetron sputtering with the thicknesses in between 1 and 5 nm in 0.5 nm steps. As an addition to this set of thin films, two more NiCr thin films were coated with the thicknesses of 7.5 nm and 10 nm. All film thicknesses were confirmed by TEM (Tranmission Electron Microscopy) cross-section analyses. In order to control the oxidation of thin metallic films, reflectance and transmittance (RT) measurements of NiCr films were performed right after deposition. The samples were stored in vacuum bags and RT measurements were repeated in the 7th, 14th and the 21th days of deposition. The measurements showed no difference between in the deposition day and those specified days. This indicates that the vacuum bags can be used as effective oxidation prevention for thin NiCr films. The samples' dynamic deposition rate is 10.90 nm·m/Kw·min. Coating parameters were set fixed namely, with a base pressure of 2.0E-7 mbar and working pressure of 2.2E-3 mbar. Sputter power was 1.2 kW, Ar flow was 220 sccm giving a dynamic

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Fig. 1. Sheet resistance with respect to nominal film thickness.

deposition rate (DDR) of 10.90 nm·m/Kw·min. Thickness of the films were adjusted via changing the speed of the carrier according to the given DDR.

3. Methods

3.1. Four point probe method

Sheet Resistance –Rs values- were determined for each thin NiCr film by using four point probe technique [6] by Four Point Probe (Jandel, England).

3.2. Spectroscopic ellipsometry

In order to obtain the sheet resistance values and to make a correlation with the four point probe results, the films were measured by spectroscopic ellipsometry via using the following SENTECH Instruments ellipsometers: SENresearch 4.0–SER 850 DUV was used for the spectral range from 190 to 2500 nm at two different angles of incidence (AOI) of 50° and 70° . SENDIRA was used for the MIR spectral range from 1700 nm to 25,000 nm at an AOI of 70° . Backside of the glass substrates were roughened to prevent backside reflections. The results were fitted by Drude-Lorentz oscillator model and the complementary equations of carrier concentration, resistivity and mobility given in Eqs. (1)–(5) [6–8].

4. Results

4.1. Influence of thin film thickness on sheet resistance

In order to investigate the influence of thickness, sheet resistance of thin NiCr films with different thicknesses were measured by four-point-probe method. In Fig. 1, sheet resistance R_s is plotted with respect to nominal film thickness. Fig. 1 shows that there are two thickness ranges in between 1.5–3.5 nm and 4–10 nm with different descending slopes of R_s depicted by the trendlines. This is an indication for an electrical/structural change of the NiCr layer between 3.5 - 4.0 nm which can be the possible percolation thickness.

4.2. Spectroscopic ellipsometry measurements: sheet resistance investigation

Sheet resistance of each NiCr film was investigated also by spectroscopic ellipsometry measurements. Studied films were Table 1

Optical model description of the single layer thin NiCr film on clear float glass.

Material	Dispersion type	
Air		
NiCr	Drude-Lorentz osc.	
Glass	Brendel oscillator	

Table 2

Drude-Lorentz oscillator parameters for 1 nm NiCr film.

Oscillator type	ω_0/cm^{-1}	$\omega_{\rm p}/cm^{-1}$	ω_τ/cm^{-1}
Drude oscillator	-	20662.99	35385.75
Lorentz-oscillator (1)	126270.10	193899.80	351937.41
Lorentz-oscillator (2)	22444.26	79525.75	76874.82
Lorentz-oscillator (3)	53531.70	80137.76	28565.76
Lorentz-oscillator (4)	31019.93	29553.63	39467.30

Table 3

Drude-Lorentz oscillator parameters for 10 nm NiCr film.

Oscillator type	ω_0/cm^{-1}	ω_p/cm^{-1}	ω_τ/cm^{-1}
Drude oscillator	-	74804.14	13855.74
Lorentz-oscillator (1)	114865.70	150311.86	130278.82
Lorentz-oscillator (2)	723.60	35745.93	62056.38
Lorentz-oscillator (3)	40841.52	55964.06	27530.36
Lorentz-oscillator (4)	17899.42	36135.98	19275.91

measured using two ellipsometers with a combined spectral range of 190 nm to 25000 nm. Single layer thin NiCr film on 4 mm clear float glass was assumed as the optical model as shown schematically in Table 1. NiCr film was modeled using a Drude Lorentz oscillator model. The Drude term and four Lorentz oscillators were used to describe the NiCr dispersion of n, k (Tables 2 and 3). Thin strongly absorbing films are prone to an ambiguity in the simultaneous determination of thickness and optical constants. In this work, techniques such as the Combination of Ellipsometric and Transmission (CET) measurements were not necessary due to the accurate film thickness determination by TEM cross-section analyses. Confirmed thickness values obtained by TEM were used as fixed preset parameters in the optical modeling. Float glass was modeled using the Brendel oscillator model [11]. A total number of eight Brendel oscillators were necessary to describe the complex dispersion of float glass from the DUV to MIR spectral range (Fig. 2 and Table 4).

As it can be seen from Tables 2 and 3, Lorentz oscillators located in VIS to UV spectral range are not affecting the Drude parameter which is used for sheet resistance determination. Only Lorentz



Fig. 2. Dispersion of float glass.

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