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Real-time *in-situ* investigation of copper ultrathin films growth

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ABSTRACT

The results of real-time *in-situ* spectroscopic investigation of copper films growth are presented. The films were deposited on fused silica substrates by magnetron sputtering. Three growing stages of copper films were *in-situ, ex-situ* analysed and compared with modelled values. The minimum continuous film thickness of about 10 nm was determined by such comparison. This new empirical method might be applied for multilayer coatings deposition.

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1. Introduction

Ultrathin copper films properties differ from bulk and depend on film thickness. Previously, initial copper growth stages on α-Al_2O_3(0001) surface by Auger, electron energy-loss spectroscopies and by low-energy electron diffraction were studied in details [1]. Thickness dependence of the electrical parameters [2–5], microwave reflection [5] and reflection in the IR range [2] for deposited ultrathin copper films were investigated. It was calculated the specific features of reflection, transmission and absorption of ultrathin copper films under the oblique incidence of two counter-propagating coherent waves of the microwave frequency range [6]. *In-situ* ultrathin copper film growth was investigated by the change of electrical resistivity [7–10], stress measurement [11] and with the help of scanning tunnelling microscopy [12], low-energy electron diffraction, electron spectroscopies [13]. It was reported [14] about the attempt of *in-situ* optical transmittance spectroscopy investigation of the copper films growth, but authors had difficulties with copper oxidation from the residual oxygen (base pressure values were 3–5 $\times$ 10^{-5} Torr). To the best of our knowledge, up to now, no successful effort has been made to investigate in details real-time *in-situ* transmittance measurements of ultrathin copper film formation.

In this work, the real-time *in-situ* optical transmittance spectroscopy was implemented to investigate the growth of ultrathin copper. Copper was deposited on fused silica substrate by magnetron sputtering. Change of optical and structural properties were investigated. Moreover, proposed method allows following growth processes and estimate the minimum continuous film thickness. The method idea is in comparison of optical spectra with modelled spectra. This is of high interest, for example, for preparation of multilayer optical coatings [15,16].

2. Experimental details

Copper was sputtered using an unbalanced magnetron source (Torus™ sputter gun) with a planar target (Cu, 99.95% purity, diameter of 101.6 mm and thickness of 6 mm) in a Kurt J. Lesker sputtering system (PVD225). The system was initially pumped down to a base pressure below 3 $\times$ 10^{-7} Torr. The magnetron was driven by a pulsed DC power supply (Advanced Energy Pinnacle Plus). In this work, the repetition frequency was 100 kHz, the duty cycle was 80% and the fixed power was 200 W. The argon (Ar, >99.999% pure) flow rate was 20 sccm and the pumping speed was adjusted to attain the argon pressure at the same value of 2.2 mTorr. The settings of the Ar flow rate and the pumping speed were not changed during the experiments. Prior every experiment, the target was pre-sputtered for 5 min to remove any surface oxides. The target-to-substrate distance was 230 mm. The system was equipped with magnetron and substrate shutters. The experiments were done at room temperature. Film thickness during deposition was controlled by quartz crystal and optical broadband monitoring (BBM). The thick film thickness after

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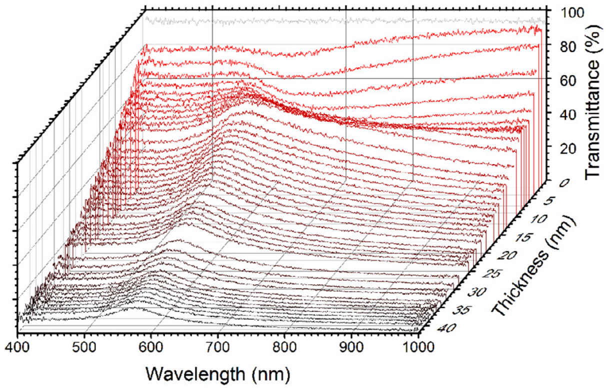

Fig. 1. Transmittance spectra evolution during copper film growth on fused silica substrate. Recorded real-time *in-situ*.

deposition was measured at the film edge by profilometry (Veeco Dektak 150). The BBM transmittance spectra were recorded in the 400–1000 nm wavelength range through the growing films.

To determine crystal phase composition of the films, X-ray diffraction (XRD) measurements of samples were performed using a Bruker D8 Advance diffractometer using Cu K_{α} (λ = 1.5406 Å). The X-ray source was operated at the voltage of 40 kV and current of 40 mA. The Grazing Incidence (GIXRD) method was used. The angle between the collimated X-ray beam and the specimen surface (θ angle) was adjusted to 0.5.

The morphology of the films was characterized using scanning electron microscope (SEM) workstation Helios Nanolab 650 (USA). The imaging was carried out under an accelerating voltage of 5 kV.

Transmittance, reflectance and loss spectra of continuous copper layers were calculated from optical constants taken from Ref. [17], using the "OptiLayer" software from OptiLayer Ltd [18].

Transmittance, *T*, and reflectance, *R*, spectra of films were measured with the angle of incidence (in reflection) 8° in the 200 < λ < 1400 nm wavelength range using a Photon RT (Essent Optics) spectrophotometer. The loss, *L*, values were calculated as *L* = 100-*T*-*R*.

3. Results and discussion

3.1. Evolution of growing copper films optical properties

Optical properties dependence on copper film thickness (Figs. 1 and 2) is non-monotonic and illustrates different stages of the film growth: separate islands, coalescence and continuous. It is well

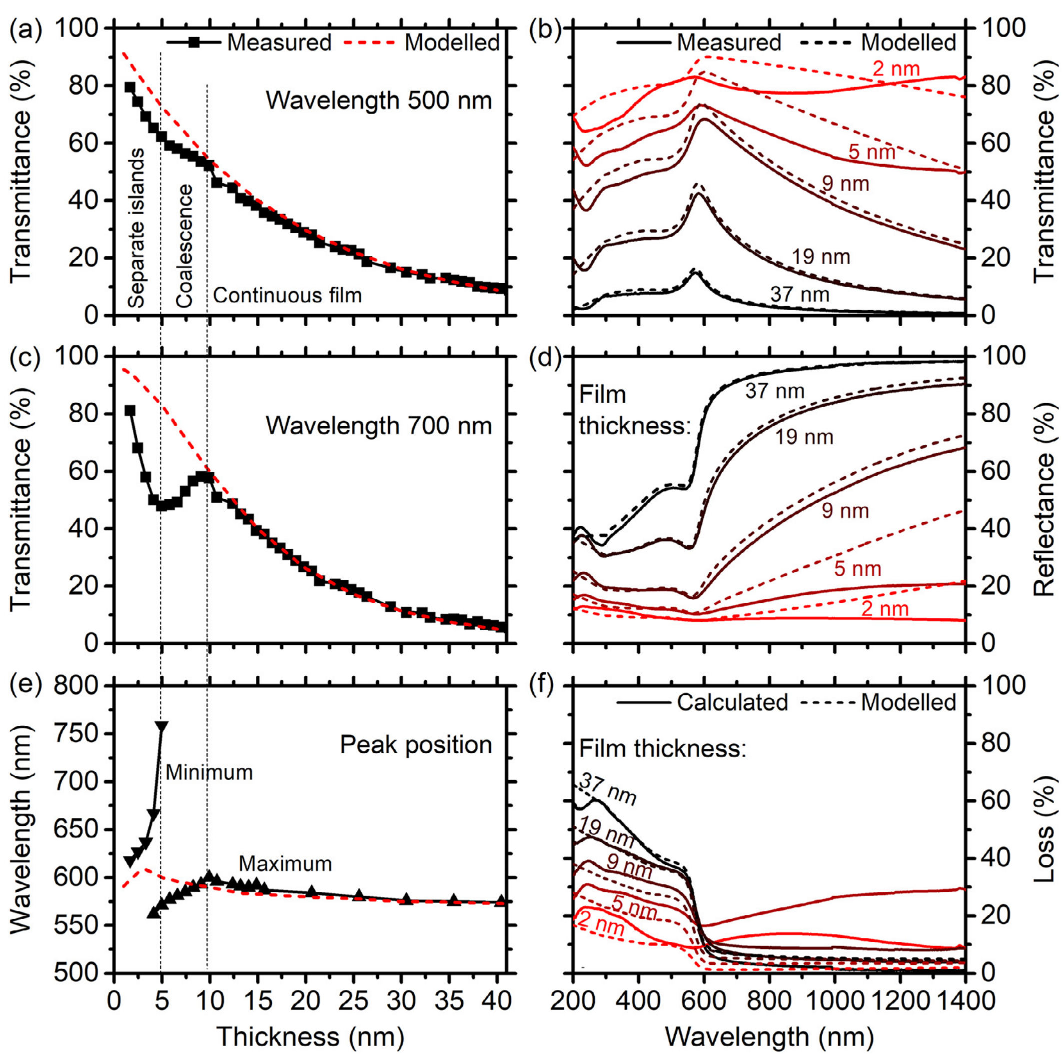

Fig. 2. Real-time *in-situ* transmittance values measured at the wavelengths 500 nm (a), 700 nm (c) and transmittance peaks position (e) during copper film growth. Measured transmittance (b), reflectance (d) and calculated loss (f) spectra for deposited films with various thickness.

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