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Thin Solid Films

journal homepage: [www.elsevier.com/locate/tsf](http://www.elsevier.com/locate/tsf)

## In-situ investigation of optical transmittance in metal thin films

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### ARTICLE INFO

Available online xxx

#### Keywords:

Metal thin film deposition  
Sputtering  
Localized surface plasmons  
Transmittance of thin metal films

### ABSTRACT

The results of in-situ spectroscopic investigation of metal thin films transmittance during the growth process are presented. Thin films of Au, Ag, Cu, and Ni were deposited on sapphire substrates by dc sputtering method in the triode sputtering setup. The sputtering process was provided at an argon pressure of 1 mTorr using the constant sputtering voltage of 1.5 kV. The transmittance spectra were recorded in the 200–1100 nm wavelength range through the growing thin metal films.

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### 1. Introduction

Thin metal films play an important role in the nowadays micro- and nanoelectronics, optoelectronics and photonics. These films are widely used in various applications: transparent conductive coatings for ultra-violet (UV) spectrum [1], transparent electrodes for “smart windows” and solar cells [2], catalytic coatings for growing nanostructures [3], active component of surface plasmon resonance (SPR) based biosensors [4,5] etc. Ultrathin metal films may represent discontinuous, island-like structures that exhibit excitation of localized plasmon–polaritons under light illumination [6–14]. The type of deposited metal, the shape, the size and the surface density of the metallic islands are the key factors affecting the electrical and optical properties of thin metal film, including the efficiency of plasmon–polaritons excitation. The structure and the properties of metal film on a given substrate strongly depend on the method of film deposition. As illustration for such dependence, Fig. 1 represents transmittance of thin films of four metals: gold, silver, copper and nickel [14]. Three first metals are noble metals having fully filled penultimate electron shell and only one electron on the last electron shell. Nickel is the transition metal, such as its penultimate electron shell is not fully filled. As can be seen from the Fig. 1, ultra-thin films of noble metals demonstrate features that are not found in the films of transition metals.

A specific interest is in the early stage of film growth, which determines the structure of discontinuous island-like metal films [15]. Various techniques were proposed for in-situ, real-time monitoring of optical properties of thin metal films during deposition process. The spectroscopic ellipsometry method was used for real-time monitoring in magnetron sputtering of noble metals on SiO<sub>2</sub> [16,17] and Si [18] substrates and to monitor growth of Ag nanoparticles in a polymer

matrix [19]. The surface differential reflectivity spectroscopy was demonstrated [20,21] as an effective tool for the real-time, plasmonic-based characterization of thin metal films growth on Al<sub>2</sub>O<sub>3</sub> substrate. The visible spectroscopy and Fourier transform infrared spectroscopy were used to record in real-time reflectance and transmittance spectra of Au nanostructures during wet-chemical preparation [22]. In-situ monitoring of optical reflectivity [23] was used in magnetron sputtering of ultrathin Ag layers on fused silica substrates.

In the present work, the real-time optical transmittance spectroscopy was implemented to probe optical properties of thin metal films deposited by low-pressure direct current (DC) plasma sputtering. A low pressure, about 1 mTorr, is provided by triode sputtering method, which supports a ballistic type of mass transfer between the sputtering target and the substrate. This method allows fine control over the film thickness and its density. Thin films of various pure metals, Ag, Au, Cu, and Ni, were deposited on sapphire substrates and their transmittance spectra in the 200–1100 nm wavelength range were recorded in-situ while sputtering. Analysis of time evolution of in-situ recorded transmittance spectra allows comparison of growth processes and estimation of growth rate and sputtering yield for various metals.

### 2. Experimental details

Experiments with low-pressure plane plasma discharge and sputtering of metal targets were done using a laboratory setup equipped with a standard two-stage vacuum system providing a residual vacuum of  $3\text{--}5 \times 10^{-5}$  Torr. The sputtering system consists of a thermo-emissive tungsten cathode, a tantalum anode, placed opposite to the cathode, a water-cooled sputtering target-holder, arranged in parallel with the cathode–anode axis, and a substrate-holder, placed opposite to the target. Two external coaxial electromagnetic coils create a homogeneous magnetic field in the vacuum chamber. This field confines the plasma discharge between cathode and anode, thus allowing

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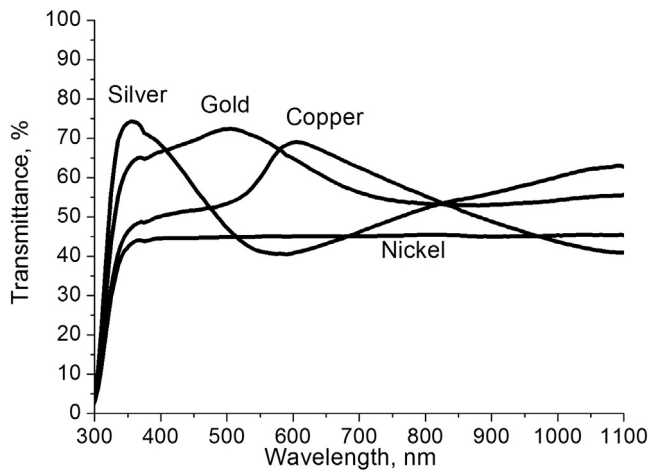


Fig. 1. Transmittance characteristics of very thin metal films.

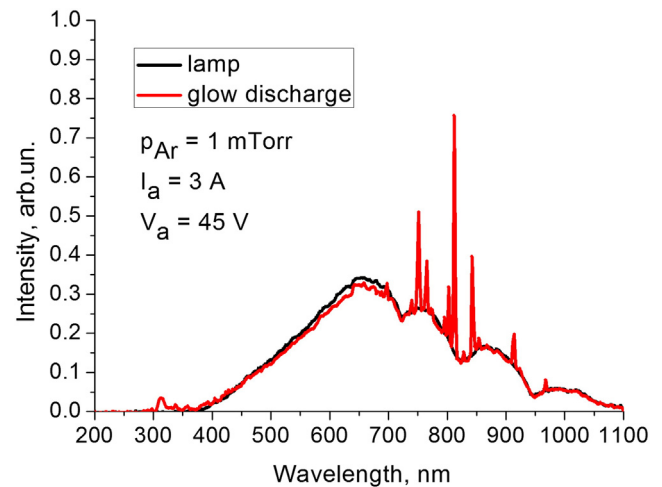


Fig. 3. Experimental emissivity spectrum of the forced glow discharge in argon.

for a lower working pressure while sputtering. A detailed description of this sputtering system is given in our previous work [24]. The experimental setup, based on the triode sputtering system, is presented in Fig. 2.

In order to illuminate the growing thin films, an incandescent lamp was installed inside the vacuum chamber. The light, transmitted through deposited metal film, was coupled to fiber-optic cable at the opposite side of the sapphire substrate and out-coupled from the vacuum chamber via “Accu-Glass” fiber-optic vacuum feedthrough providing a homogeneous transmission in the 200–1000 nm wavelength range. The position of illuminating lamp inside the vacuum chamber was carefully aligned relative to the substrate in order to maximize the intensity of light, collected by fiber-optic cable, and minimize the influence of direct and scattered background radiation from the cathode and plasma discharge.

The transmittance spectral characteristics of thin metal films growing on the sapphire substrates were in-situ recorded using Newport OSM-400 spectrometer. The surface structure of the deposited films was studied using the scanning-probe microscopy (SPM) DI300 in atomic-force microscopy (AFM) contact mode and the computerized metallurgical microscope “Nicon-Optiphot 100” with optical magnification of up to  $\times 1600$ . Sputtering deposition for all metal films was carried-out under the same conditions of argon pressure (1 mTorr), plasma current (3–3.5 A), confining magnetic field ( $\sim 150$  Oe)

and sputtering voltage (1500 V). The sputtering experiments were done using pure metal targets (99.999) of 50 mm in diameter and of 1–6 mm in thickness.

### 3. Results and discussion

The spectra, recorded in our experiments, represent superimposition of spectra of different sources: incandescent lamp, installed inside the vacuum chamber, heated tungsten cathode, plasma discharge, recombination and relaxation processes through sputtering of metal targets. The spectrum of the incandescent lamp was used as reference one, allowing to record transmittance spectra of different metals practically free of noise. Fig. 3 represents recorded experimental spectrum of a raw plane plasma discharge spectrum in argon atmosphere of 1 mTorr superimposed on the spectrum of the incandescent lamp defined as the reference light. The peaks observed in the emission spectrum correspond to recombination processes in the plasma of a glow discharge in argon [25]. Subsequently, the intensity of the reference light source (incandescent lamp) was increased in order to reduce the relative contribution to recorded spectra from the glow discharge radiation.

Fig. 4 represents the spectra of light, transmitted through growing gold film, recorded in-situ with 20-s time intervals while sputtering. Each curve in Fig. 4 represents the emission spectrum of the incandescent lamp superimposed with the spectral lines of the plasma discharge.

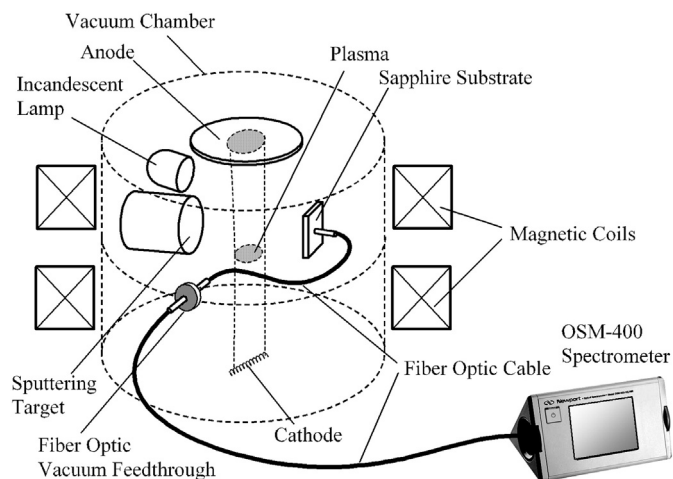


Fig. 2. Experimental setup.

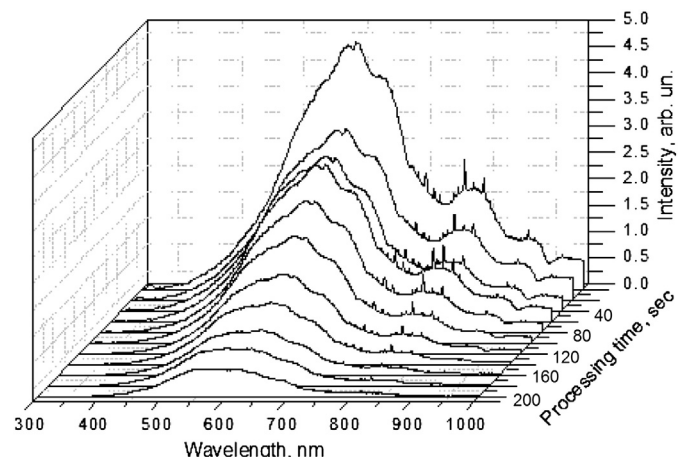


Fig. 4. Intensity of light transmitted through growing thin gold films.

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