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# Copper thin films deposited under different power delivery modes and magnetron configurations: A comparative study

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## ABSTRACT

This study investigated the topological, structural, mechanical (hardness and Young's modulus) and tribological (critical loads and coefficient of friction) properties of copper (Cu) thin films with nanocrystalline structure, smooth and uniform surfaces and thickness of 800 nm, deposited on silicon and graphite substrates by conventional dc magnetron sputtering (dcMS) and HiPIMS operated with single ultra-short pulses (3 μs), in the absence and presence, respectively, of an additional magnetic field. Operating the HiPIMS discharge with such short pulses optimizes the deposition rate and provides a higher fraction of ionized metal flux, while the presence of the additional magnetic field facilitates the transport of charged particles towards the substrate, leading to a higher deposition rate. The density of the deposited thin films was obtained by measuring the areal atomic density using Rutherford backscattering spectrometry and the film thickness by cross-sectional scanning electron microscopy. The nanoindentation results were initially analyzed using the Oliver-Pharr method, and then, in order to correct de pile-up errors, other methods have been employed. Compared to the other samples, mainly due to the high energetic bombardment during the growing process and intense surface diffusion, the Cu thin films deposited using HiPIMS assisted by an additional magnetic field exhibit significantly smoother surfaces, higher crystallinity, denser microstructure, higher hardness and Young's modulus, and lower coefficient of friction.

## 1. Introduction

The new demands of emerging technology, including miniaturization, interconnection and integrations of certain devices with micron-, sub-micron-, or nano-scale functionalities require materials in the form of thin films whose microstructure and mechanical properties are key parameters on the reliability. Understanding the deformation mechanisms, as well as the mechanical and tribological behaviour of thin films is highly important, especially for microelectronic and micromechanical device development [1,2]. One of the problems implied by their small dimensions is the impossibility to use traditional methods to test their mechanical properties because these methods do not scale properly into the micron- and nano-scales.

With an endless variety of applications, especially in electrical industry, copper (Cu) is a preferred material over aluminum, not only because it has a better thermal and electrical conductivity, a higher melting point and a better mechanical strength, but also because Cu interconnects provide higher current densities and better electromigration performance. However, some of the disadvantages of using Cu thin films are their low hardness and adhesion to the substrates which are of prime importance for satisfying the durability requirements for practical applications [3–5].

The mechanical properties of materials are size-dependent and, therefore, the mechanical behaviour of thin films often differs from that of their bulk counterparts [6]. Nanoindentation and nanoscratch testing have gained increasing interest in recent years in the characterization of materials at small scales due to their many advantages over the conventional methods, including the possibility to determine the individual parameters of the thin film and substrate, respectively. The most commonly employed nowadays method for nanoindentation data processing is the Oliver-Pharr (O&P) model [7–11]. The nanoscratch testing is a flexible method which can overcome some of the atomic force microscopy limitations and it can be performed by moving an indenter laterally under a constant or ramped applied load in a single or repetitive scratch mode. Adhesive properties, critical breakthroughs, delamination events, as well as the coefficient of friction of thin films can be investigated by this method [12].

The mechanical and tribological properties of the coatings depend on many structural factors including packing density, microstructure, nature of the chemical bonds and surface morphology, which are mainly affected by the energy transfer from the plasma to the substrate during

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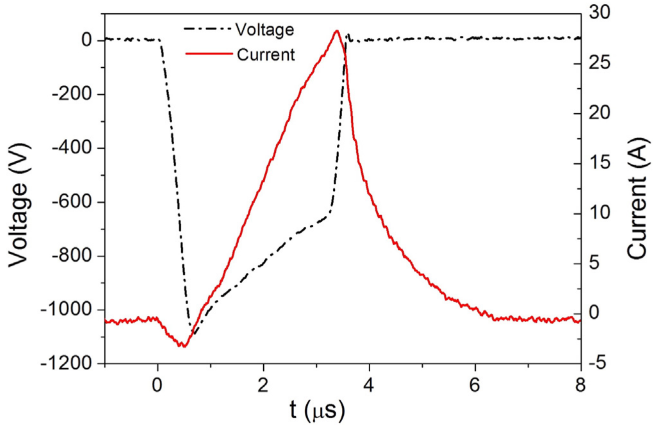

**Fig. 1.** Typical discharge current intensity and voltage waveforms for the HiPIMS discharge operated using an external magnetic field.

film growth process. Therefore, manufacturing of thin films with advanced properties needed to cope with the demands of nowadays nanotechnology brings with it the need to develop new deposition techniques. High power impulse magnetron sputtering (HiPIMS) is a sputtering technique where the high density power (>kW/cm$^2$) applied to the magnetron target in unipolar pulses at low duty cycle and low repetition frequency facilitates generation of high-density plasma with high ionization degree (>80%) and broad energy distribution function of the sputtered species [13]. The high ion-to-neutral ratio and intense energetic particle bombardment have been shown to enable the deposition of ultra-dense and smooth metallic films [14,15], making possible to tailor the phase and elemental composition, microstructure and morphology, and subsequently the properties and functionality of the deposited coatings [16]. In HiPIMS, the deposition rate, ionization rate of sputtered species and the energetic ion flux towards substrate can be controlled through the pulsing scheme, as well as through the magnetic field's strength and configuration [17–19].

The main purpose of this paper is to emphasize the advantages of the HiPIMS discharge operated with ultra-short pulses (3 μs), in the presence of an additional magnetic field, in terms of manufacturing thin films with advanced properties. Our approach to achieve this goal is to present a comparative study containing results obtained from the property investigation of Cu films deposited by conventional dc magnetron sputtering (dcMS) and HiPIMS operated with ultra-short pulses in the absence and presence (m.f.-HiPIMS), respectively, of an additional magnetic field. The topological, structural, mechanical (Young's modulus and hardness), and tribological (critical loads and coefficient of friction) properties were investigated for all the deposited films. The present work summarizes only the results capable to highlight clear and reliable differences between the obtained thin films.

## 2. Experimental details

Nanocrystalline Cu thin films, with thickness of approximately 800 nm, were deposited using the dcMS, HiPIMS/m.f.-HiPIMS techniques on silicon (Si) and graphite substrates not intentionally heated during the sputtering process. All the deposition runs were carried out by sputtering a circular Cu target in high-purity argon atmosphere, keeping constant the gas mass flow ratio at 20 sccm, the working gas pressure at 1 Pa, the average power at 100 W and the target-to-substrate distance at 100 mm.

For the HiPIMS discharge, the magnetron was operated in standard HiPIMS mode with ultra-short pulses of 3 μs, using a high power pulse generator with pre-ionization [20]. The magnetic field's strength and configuration in the vicinity of the magnetron cathode were changed by adding a permanent magnet with thoroidal shape (external diameter 10 cm, internal diameter 6 cm and height 3 cm). The magnet, placed axially above the magnetron cathode, at a distance of 4 cm, changes the magnetic balance degree of the magnetron cathode from balanced to un-balanced of type II by weakening the magnetic field strength of the central pole and raising the B strength of the outer pole with 600 G (the magnetic field strength in the racetrack region remains unchanged). Unipolar ultra-short voltage pulses (3 μs duration), with amplitude of −1 kV, were applied to the magnetron cathode, using a repetition frequency of 3650 Hz. The value of 3 μs for the pulse duration was chosen in order to optimize the HiPIMS deposition rate [21]. More than that, the fraction of ionized metal flux increases as the pulse duration decreases. Time evolution of magnetron voltage and discharge current intensity during one discharge pulse is presented in Fig. 1. During the pulse, the discharge power develops to a peak of 17.5 kW, while the average power during thin films deposition was kept constant at 100 W.

Time averaged ion energy distributions were recorded by energy-resolved mass spectrometry (ERMS) using an EQP 1000 Hiden Analytical spectrometer placed in a position directly facing the target's surface at a distance of 10 cm. The deposition rates were estimated by using an Inficon Q-pod quartz crystal microbalance, placed in the virtual place of the substrate, but, in order to get the exactly thickness of thin films and to study their microstructure, cross-sectional scanning electron microscopy (SEM, VEGA-TESCAN) was used. In order to determine the fraction of ionized metal species, a well-established technique consisting of a quartz crystal microbalance (QCM) in combination with a two-gridded energy analyzer has been used [22].

Fig. 2 presents the schematic view of the experimental setup including the HiPIMS system (left), quartz crystal microbalance (QCM), and mass spectrometer (right).

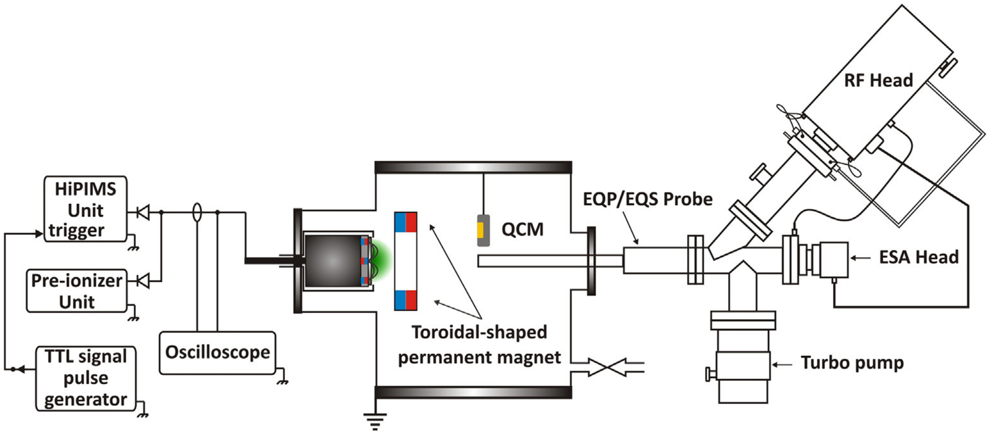

**Fig. 2.** Simplified schematic diagram of the experimental setup.

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