Elevated temperature in-situ transmission Kikuchi diffraction for the characterization of ultra-thin metal films in nanofabrication applications

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Elevated Temperature In-situ Transmission Kikuchi Diffraction for the Characterization of Ultra-thin Metal Films in Nanofabrication Applications

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The fabrication process of increasingly complex multi-material and multi-layer structures and devices in nanofabrication includes thermal treatments that might affect the nanostructure and stability of the films heavily. In particular, pre-bake and post-bake processes involve the use of a heat treatment on the sample for periods varying from one to 30 minutes (and even hours for the case of very thick resists) and using temperatures ranging from 100˚C to 200˚C.

An important temperature-related problem for the application of such systems is solid-state dewetting[1], the agglomeration of the film to form islands or nanoparticles when heated to sufficiently high temperatures. The driving force for dewetting is the minimization of the total energy of the free surfaces of the film and substrate and of the film-substrate interface. Dewetting can have a harmful effect on the performances and time-stability of the fabricated devices, leading to the extreme case of interconnect failure and degradation of the electronic devices due to overheating. Dewetting has been characterized and described ex-situ with SEM and TEM mostly above 400˚C, while below 200˚C the film stability has not been studied in detail. The fundamental insight in the driving forces for changes in crystal structure, grain size and crystallographic texture of thin films due to thermal treatments requires the introduction of innovative techniques having nanoscale resolution and being able to operate in a wide range of temperatures. In this study, we demonstrate the in-situ capabilities of transmission Kikuchi diffraction (TKD) for the analysis of ultra-thin Au films at high temperature[2].

The metal thin films were deposited by e-beam evaporation on MEMS-based heating microchips having a Pt coil-based embedded heating system (Fig. 1a). The research area is formed by 20 nm-thick electron transparent Si_3N_4 membranes having dimensions of roughly 5 x 20 µm (Fig. 1b). The microchip is mounted inside the SEM using a purpose designed holder with a built-in 45˚ tilt. When inside the microscope, the holder is tilted to 45˚, in order to place the microchip perpendicular with respect to the electron beam (Fig. 1c). In this way an on-axis Bruker-Flash OPTIMUS detector is used to acquire TKD maps in an FEI Nova Nanolab 600 SEM. The TKD maps were acquired in low vacuum conditions (50 Pa).

The dewetting of an Au thin film into Au nanoparticles upon heating was followed with orientation mapping in a temperature range between 20˚C and 900˚C (Fig. 2). The evolution of grain size and film texture and the growth of holes in the film were tracked throughout the process with high resolution, accuracy and statistical significance. Several sources of influence on the quality and resolution of the acquired TKD maps were investigated: disturbance from infrared radiation, maximum measurable Au thickness, loss of crystalline order, thermal drift during heating, plasma cleaning of the sample and thickness variation of the film. A quasi in-situ TKD investigation was also performed to study the positive impact on the stability of the Au nanostructure at elevated temperatures due to the presence of Ti and Cr transition metals used as adhesion layers. The results showed that the continuity of the Au film was preserved up to 500˚C using either of the adhesion layers, but also how Cr and Ti have a different impact on the final Au film nanostructure, in particular with the Cr layer stopping the Au grain growth almost completely.

Further studies will include the high-resolution observation of the starting point of dewetting and the analysis of the stability of Au nanostructure when non-metallic adhesion layers, such APTMS[3], are used.

References:

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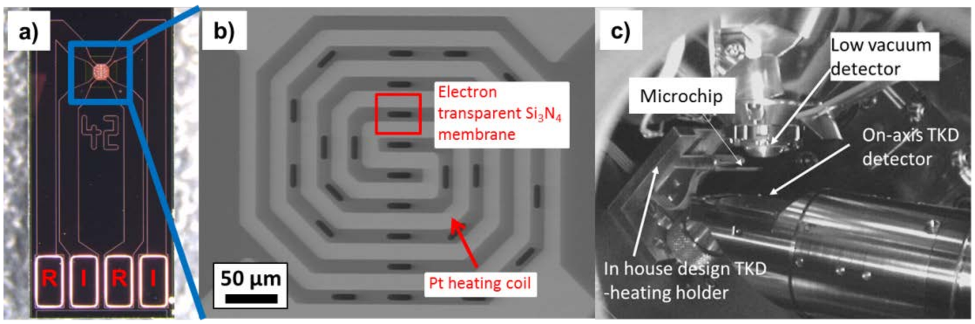

Figure 1. a) Structure of the MEMS microchip with resistance (R) and current (I) contacts highlighted. b) Zoom of the research area: the electron transparent Si_3N_4 membranes and the embedded Pt heating coils are visible. c) Image of the TKD system configuration. The 45˚ tilted holder supporting the microchip and the on-axis TKD detector are visible.

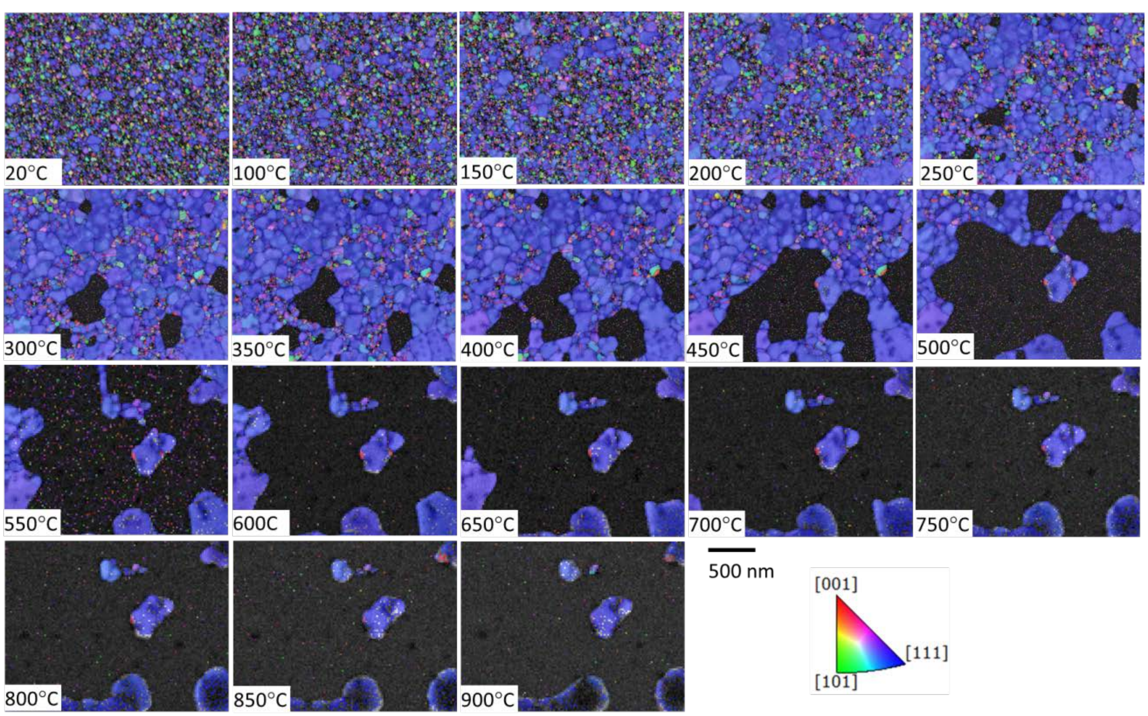

Figure 2. Out of plane inverse pole figure (IPF) in-situ TKD maps at selected temperatures showing the grain growth, the formation of holes and their subsequent growth in a 20 nm Au film.