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Todeschini, Matteo; Jansen, Henri; Wagner, Jakob Birkedal; Bastos da Silva Fanta, Alice

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Use of elevated temperature in-situ transmission Kikuchi diffraction for the study of ultra-thin metal films

Matteo Todeschini, Henri Jansen, Jakob Birkedal Wagner, Alice Bastos da Silva Fanta
DTU Danchip/Cen, Technical University of Denmark, 2800 Kongens Lyngby, Denmark

Noble metallic thin films are widely used in the fabrication of nanostructures and nanodevices. The fabrication process of these devices includes thermal treatments that might affect the stability of the films: for example, lithographic processes expose the thin film to baking temperatures well above 100°C for several minutes. Furthermore, the device downscaling has pushed the thickness of the thin films into the nanometer regime, amplifying the problem of solid-state dewetting[1], the agglomeration of a solid film to form islands or nanoparticles when heated to sufficiently high temperatures. Dewetting is an undesirable phenomenon, which can have a harmful effect on the performances an time-stability of the fabricated devices. Dewetting has been characterized and described ex-situ with SEM, TEM and EBSD mostly above 400°C[2,3], while below 200°C the film stability has not been studied in detail.

In this study, we used transmission Kikuchi diffraction (TKD) to perform in-situ annealing experiments on Au thin films deposited on a MEMS-based heating system, with a particular focus on the demonstration of the capabilities of TKD for such in-situ heating experiments. 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 (part of the process is visible in Figure 1). 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 of the chip, plasma cleaning of the sample and thickness variation of the film. The in-situ TKD mapping was also used to observe 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 had a different impact on the final Au film nanostructure.

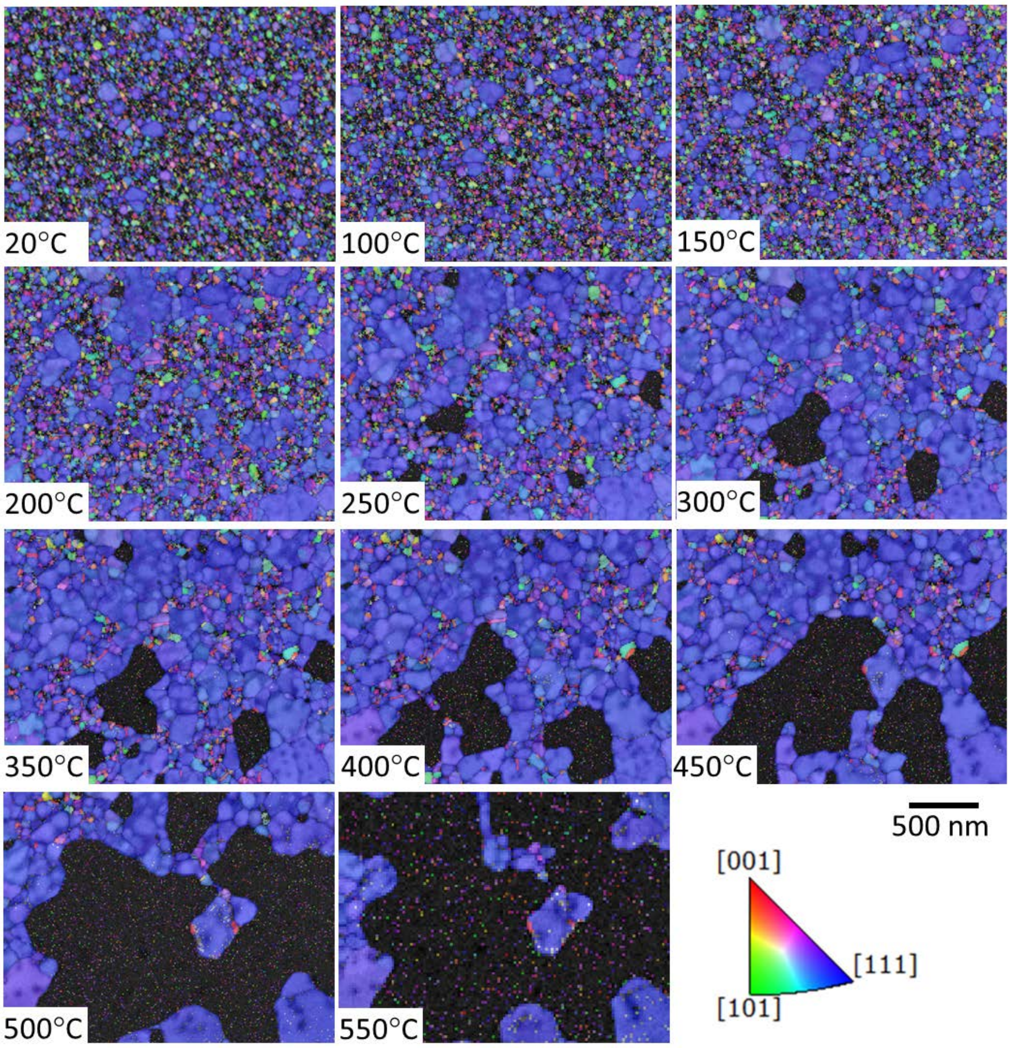

Figure 1: Out of plane inverse pole figure (IPF) in-situ TKD maps at selected temperatures showing the grain growth, the formation of holes and their subsequent growth in a 20 nm Au film.

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