

Combining in situ heating and serial block face SEM approaches to investigate the 3D thermal evolution of nanoporous gold

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Nanoporous gold (NPG) - in the form of network of nanosized ligaments and pores (figure 1) - is well-known to display remarkable properties, such as high surface area-to-volume ratio, catalytic activity, electrical conductivity and mechanical strength that make them very attractive for several applications. [1-3] However, any application involving thermal treatment requires an in-depth understanding of the NPG ligaments width's evolution over both temperature and time. In this work we analyzed the internal structural features change, upon thermal treatment, of NPG 20 μm -thick films fabricated by electrochemically dealloying. [4] At first, by *in situ* environmental SEM (ESEM) experiments, we investigated the NPG ligaments coarsening during isothermal annealing performed at 630°C, 580°C and 550°C, respectively (figure 2). Afterwards, with an approach based on Serial Block Face (SBF)-SEM applied to as-dealloyed NPG samples, both before and after the isothermal annealing treatment, we investigated the 3D structure (figure 3).

Such a combined approach allowed us to highlight the following points: 1) isothermal kinetics temperature affects both the time needed to accomplish the coarsening and the final NPG structure; 2) the coarsening kinetics exhibited approximately two stages: in the first one, which lasted about 15 minutes, the ligaments growth was fast, then gradually decelerated. Besides, NPG 3D structure after thermal treatment shows that the main coarsening effect on it consists in a not uniform porosity reduction across the entire sample thickness. The aforementioned results can help to point out the mechanisms governing thermal coarsening in NPG, paving the way for a deeper understanding of the influence of characteristic lengths on physical and chemical properties of this material.

References

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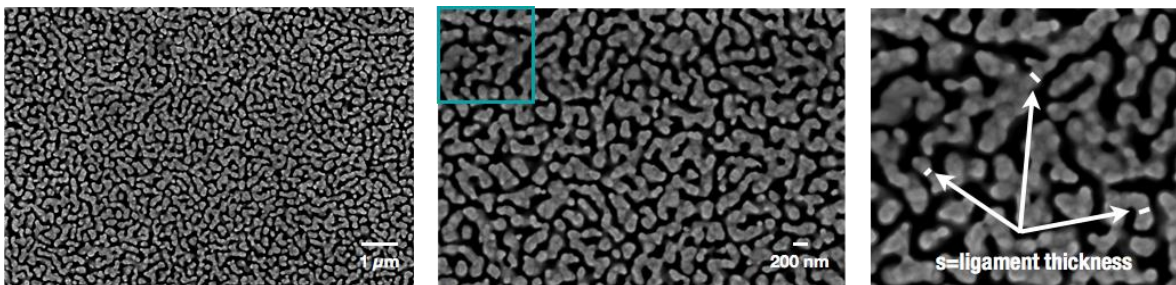


Figure 1: SEM images showing the surface of the NP Au film before the annealing

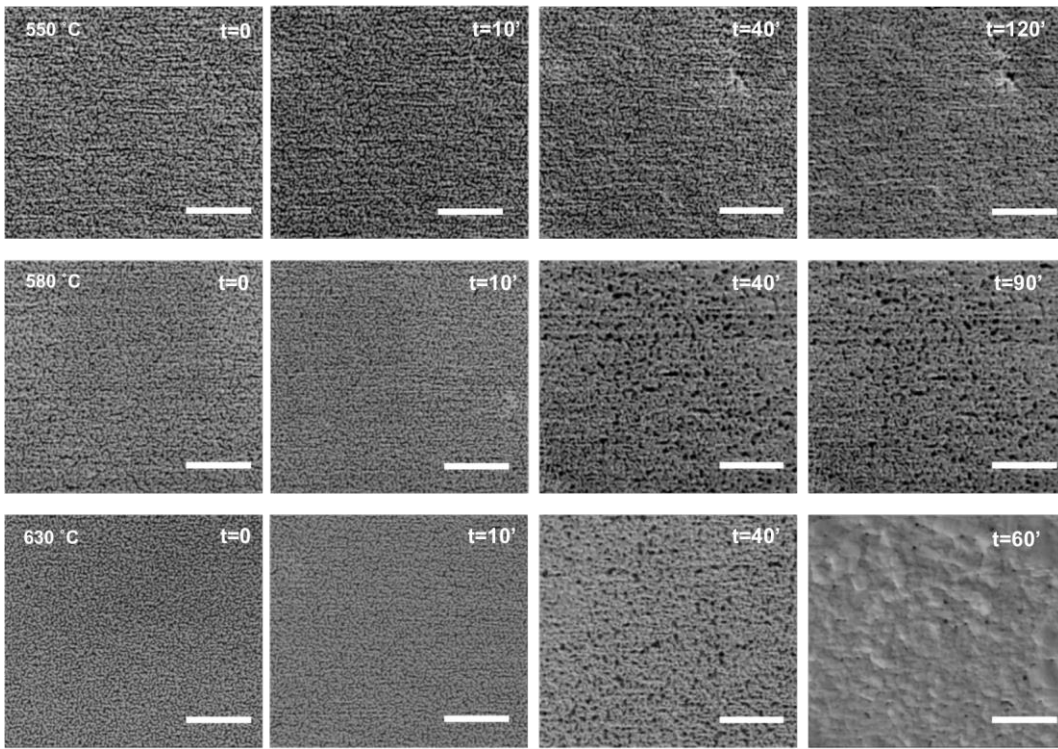


Figure 2: *In situ* ESEM: isothermal coarsening at 3 different temperatures, scale bar 5 μm

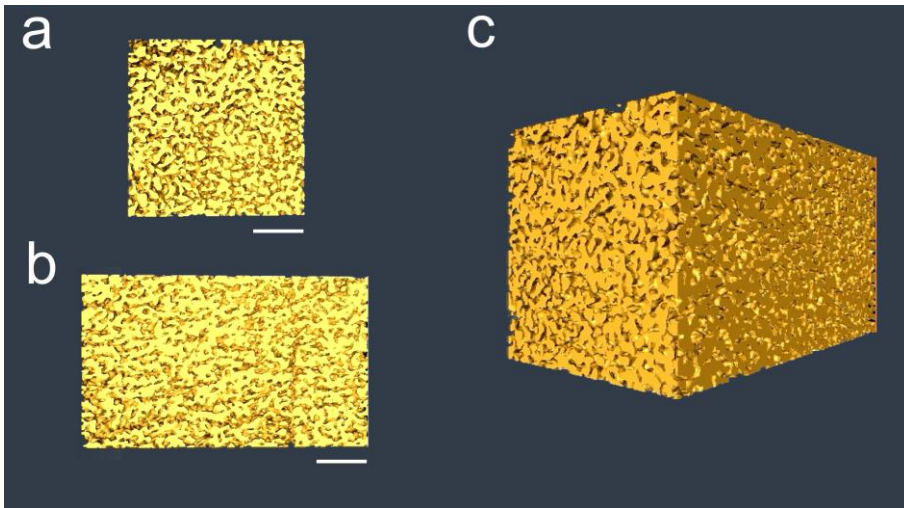


Figure 3: As-dealloyed NPG 3D reconstruction, scale bar 2 μm