Implantation in-situ et caractérisation de l'hydrogène en sonde atomique dans les matériaux métaliques

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Abstract

The investigation of hydrogen in atom probe tomography (APT) presents a challenge due to its low mass, high diffusion coefficient, and presence as a residual gas in ultra-high vacuum chambers, leading to multiple complications. Various solutions have been proposed in the literature, such as ex-situ loading coupled with cryotransfer or high-temperature hydrogen loading in an adjacent chamber. However, these solutions often face difficulties due to the complex control of the specimen temperature during hydrogen loading and the transfer of the specimen for analysis. This manuscript proposes an alternative approach for in-situ hydrogen loading in an APT chamber, derived from a method developed in field ion microscopy. By applying negative nanosecond pulses to the specimen in an APT chamber under low H_2 pressure, we demonstrate that a high dose of hydrogen can be implanted at depths ranging from 2 to 20 nm beneath the specimen surface. An APT chamber was modified to enable the direct application of negative pulses with controlled gas pressure, pulse repetition rate, and pulse amplitude. Through electrodynamic simulations, we show that the implantation energy falls within the range of 100 to 1,000 eV, and a theoretical implantation depth and density are predicted and compared to experiments. Subsequently, the work focused on investigating the consequences of these new loadings. We primarily show two effects: the first is swelling of about 10 to 15 % of the specimens observed through TEM studies, and the second is an "embrittlement" effect, with a reduction in the cohesive energy of 0.6 % to 20 % between the atoms of the target material depending on the theoretical model used