Experimental Protocols for Observation of Hydrogen Trapping in Atom Probe Tomography

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The presence of hydrogen within the microstructure of an alloy can lead to a serious reduction of ductility known as hydrogen embrittlement [1]. Some mitigation strategies against this phenomenon have been proposed such as minimizing hydrogen ingress with surface coatings or controlling internal hydrogen diffusion via the introduction of microstructural 'traps', e.g. second phase precipitates [2]. One example of interest in ferritic steels is a microstructure containing finely distributed nano-sized vanadium carbide precipitates, which shows good resistance to hydrogen environments [2]. However, there is a lack of experimental techniques to directly study the interaction between hydrogen and microstructural features, limiting ability to design effective microstructural hydrogen traps.

Atom Probe Tomography (APT) has been demonstrated to be able to image hydrogen within relevant microstructures [3]. In this experiment, a specifically modified atom probe instrument was used to provide *in-situ* deuterium (D) loading, as well as subsequent cryo-transfer capability. However, there exist only few such modified systems. Simplified experimental protocols requiring limited or no modification have enabled the direct observation of hydrogen via APT such as an electrolytic D charging protocol [4]. It has also been shown that D within the specimens can be retained using a proprietary cryo-transfer chain to suppress hydrogen diffusion [5].

Herein we demonstrate a protocol that is adaptable to commercial atom probe instruments, without modification (Fig. 1). This approach involves a cryogenic treatment in liquid nitrogen (LN_2) after electrochemical D loading and then an incorporation of glove box surrounding the LN_2 to reduce the effect of environmental contamination. The problematic ice contamination from the condensation of moisture is proved to be able to fully removed within atom probe chamber. This non-vacuum simplified protocol demonstrates comparable results with the vacuum cryo-transfer protocol in term of the content of retained D (Fig. 2). In this presentation, the difference between the approaches will be discussed in details, and the reproducibility of the approaches in other atom probe instruments will be shown.

Reference:
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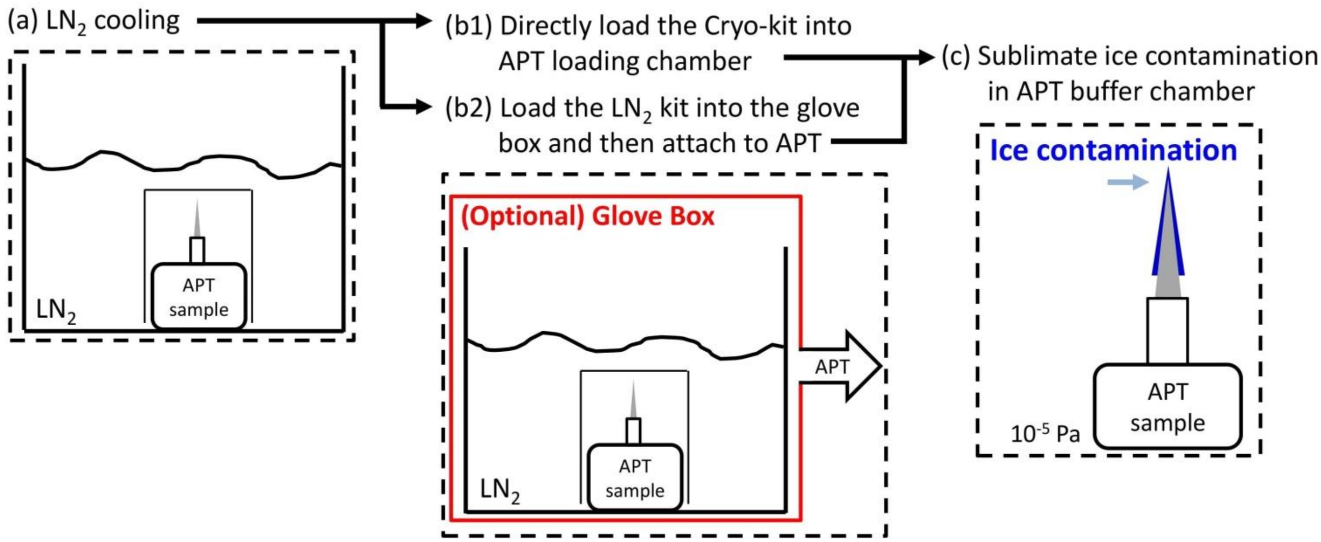

Figure 1. Experimental configuration of non-vacuum cryo-transfer technique

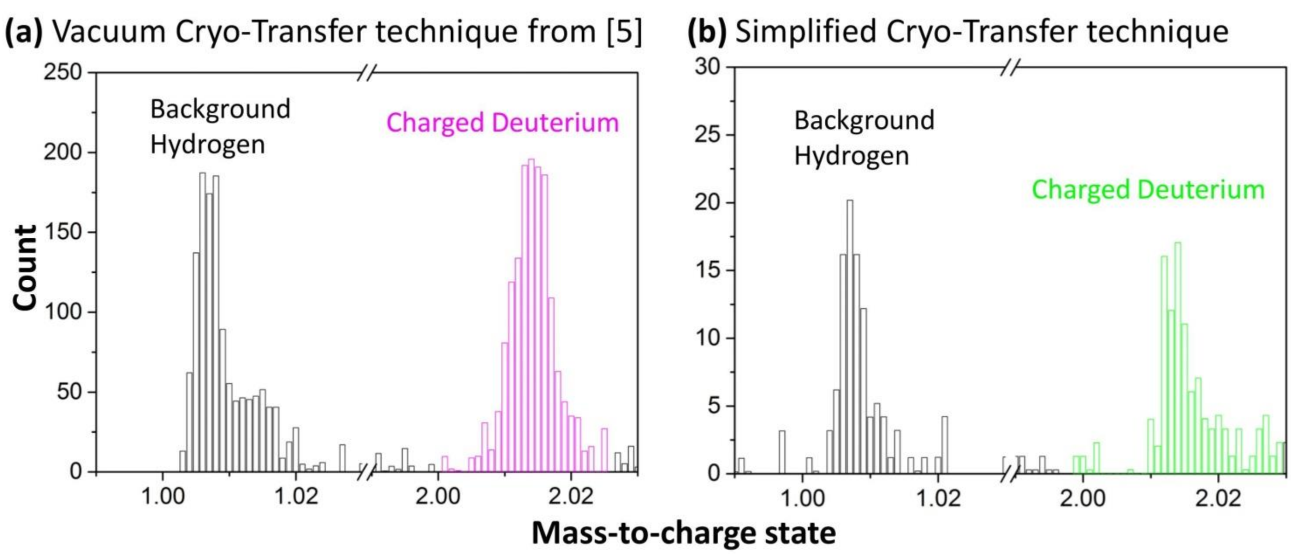

Figure 2. Comparison of the resulting global deuterium content in a ferritic sample containing 10-20nm fine V-Mo-Nb carbides as hydrogen traps from both cryo-transfer approaches.