

Hydrogen Microscopy - Distribution of Hydrogen in Buckled Niobium Hydrogen thin Films

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Hydrogen storage is still the main key for hydrogen economy. Hence, many material systems are investigated regards their fundamental parameters that influence hydrogen mobility or adhesion. For example, hydrogen absorption in thin niobium metal films clamped to rigid substrates is a systems where hydrogen absorption results in mechanical stress that changes the hydrogen's chemical potential. The stress can be released globally by film peeling or locally by buckle formation (figure 1).

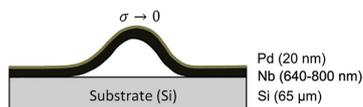


Figure 1: Cross section of an ideal buckle, resulting from local detachment of a film from the substrate. In niobium hydrogen thin films isolated buckles reach lateral dimensions of about 100 μm and heights of several μm .

At the microprobe SNAKE we have the unique possibility to study how the local hydrogen solubility of the film changes with its local stress state by mapping the buckled film fraction and using proton-proton (pp-) scattering with μm resolution for a sensitive hydrogen signal [1]. The high proton energies up to 25 MeV that are focusable by SNAKE and available at the MLL are essential in order to use this method at required substrate thickness (65 μm thick Si-waver). With this we show that local stress relaxation by the detachment of niobium hydrogen thin films from the substrate affects the chemical potential on the local scale revealing that hydrogen is not homogeneously distributed in the film as shown in figure 2.

In niobium hydrogen thin films loaded up to nominal concentrations in the two-phase coexistence region, the clamped film fraction remains in the solid solution phase, while the buckles represent the hydride phase as shown in figure 3. These results are compared to a simple model taking the stress impact on the chemical potential into account.

REFERENCES

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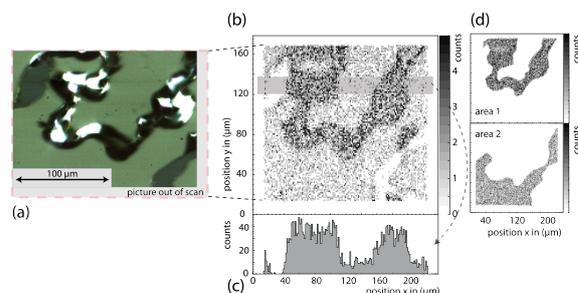


Figure 2: (a) CCD camera view onto a buckled area of an 800 nm NbH film with initial concentration $c_H = 0.30H/Nb$. (b) Hydrogen events detected in the film area in (a). (c) Profile of the density of scattering events in the film area marked in (b). (d) For enough statistics we investigate the map in (c) with two area cuts that are shown in (d); area 1 for the buckles and area 2 the clamped film.

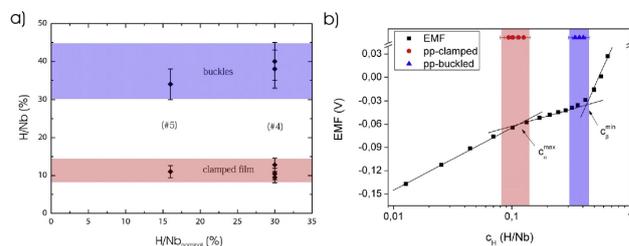


Figure 3: (a) Background corrected hydrogen concentrations determined by pp-scattering for clamped and buckled film fractions of two samples with initial nominal concentrations of 0.16H/Nb and 0.30H/Nb. A clear grouping into high (buckles) and low (clamped film) concentrated areas is visible. (b) Hydrogen concentrations determined by pp-scattering for clamped and buckled film fractions of NbH thin films, compared to the EMF-curve measured for an 800 nm NbH thin film. The inflection points of the EMF-curve, marked by the crossing points of the tangent lines, represent the phase boundaries of the film [3]. As it appears, the concentrations determined by pp-scattering fairly well agree with the phase boundaries of the films.

[2] S. Wagner, M. Moser, C. Greubel, K. Peeper, P. Reichart, A. Pundt, G. Dollinger. Hydrogen microscopy - Distribution of hydrogen in buckled niobium hydrogen thin films. Int. J. of Hydrogen Energy 38 (2013) 13822.

[3] U. Laudahn, A. Pundt, M. Bicker, U. v. Hülsen, U. Geyer, T. Wagner et al.. H induced stress in Nb single layers. J. Alloys Compd. 293 (1999) 490.