MM 15: Hydrogen in metals III: Experiments

Time: Monday 15:45–18:00 Location: TC 006

Topical Talk MM 15.1 Mon 15:45 TC 006

On the combination of different experimental techniques to increase understanding on the hydrogen/material interaction in iron based alloys — Tom Depover, Elien Wallaert, Aurélie Laureys, Emilie Van den Eeckhout, and •Kim Verbeken — Ghent University, Department of Materials Science and Engineering, Technologiepark 903, B-9052 Ghent (Zwijnaarde)

Hydrogen induced cracking might arise when metals are in contact with an H-containing environment. Although known for a long time, the explanation of the responsible mechanism still remains under discussion. New, high strength metals appear more prone to H-related failure. The microstructure complexity of such metals even complicates the study of H induced phenomena. Moreover, H interacts in a very specific way with each microstructural feature. Furthermore, H-related research is a very challenging task due to the low H solubility, high H mobility and difficult H visualization.

H-material interactions can be studied via evaluating the effect of hydrogen on the mechanical properties, done by tensile tests after or during charging, characterizing H trapping via thermal desorption spectroscopy, studying H diffusion by electrochemical permeation and advanced characterization of H-induced cracks by electron backscatter diffraction. In this work, an overview is given on some recent results on the H-material interactions in iron-based alloys by combining these techniques. It will be demonstrated that the combination of these data is an asset contributing to the elucidation of the complicated H-material interactions.

MM 15.2 Mon 16:15 TC 006

Hydrogen diffusivity as a measure for relative dislocation densities in palladium — •Martin Deutges[1], Hans Peter Barth[2], Yuzeng Chen[3], Christine Borchers[1], and Reiner Kirchheim[1,4] — [1]Institut für Materialphysik, Georg-August Universität Göttingen — [2]Now at: DLR Göttingen — [3]State Key Lab of Solidification Processing, Northwestern Polytechnical University, Xi'an, P.R. China — [4]International Institute for Carbon-Neutral Energy Research (WPI-I2CNER), Kyushu University, Japan

It is well known that hydrogen affects the defect formation energy [1] which is thermodynamically described by the defactant concept [2]. The palladium-hydrogen system was chosen to analyze the effect of hydrogen on the formation of dislocations [3,4]. For this purpose palladium was loaded with different amounts of hydrogen and subsequently cold rolled. The dislocation density was quantified by measuring hydrogen diffusivity, which depends on the amount of trap sites for hydrogen. The results were compared to conventional XRD-methods [4]. After cold rolling, residual hydrogen is removed. During reloading with hydrogen the diffusion through the material is measured. The change in diffusivity allows to calculate the relative dislocation density. The presence of hydrogen during cold rolling of palladium leads to an increase of dislocation density [3].

[1] M. Deutges et al., Scripta Mater.68 (2013) 71.
[2] R. Kirchheim, Acta Mater. 55 (2007) 5129.
[3] Y.Z. Chen et al., Scripta Mater. 68 (2013) 743.
[4] M. Deutges et al., Acta Mater. 82 (2015) 266.

MM 15.3 Mon 16:30 TC 006

The impact of the carbon content on hydrogen diffusion and its influence on hydrogen embrittlement for lab-cast bainitic Fe-C steels — •Emilie Van den Eeckhout, Tom Depover, and Kim Verbeken — Department of Materials Science and Engineering, Ghent University (UGent), Technologiepark 903, B-9052 Ghent, Belgium

The present work investigates hydrogen diffusion in lab-cast alloys in which a bainitic microstructure was introduced. The high diffusivity of hydrogen is a critical factor affecting hydrogen induced cracking since hydrogen is enabled to diffuse to highly stressed regions, leading to an accelerated failure. When hydrogen diffusion is impeded by introducing hydrogen traps, fracture is delayed and the susceptibility to hydrogen embrittlement is reduced.

The hydrogen diffusion coefficient is calculated using a permeation cell based on the Devanathan - Stachurski permeability cell. Decreasing the carbon content from 0.4 to 0.2 wt. % elevates the hydrogen diffusion, which can be attributed to less hydrogen traps of the latter bainitic steel. The impact of this higher diffusivity on the degree of embrittlement was evaluated by performing tensile tests on in-situ hydrogen charged samples at various cross-head displacement speeds. A correlation with melt extraction tests is made to demonstrate the combined effect of hydrogen content and hydrogen diffusion on hydrogen embrittlement. Additionally, the role of hydrogen diffusion is evaluated by calculating and visualizing, by in-depth fractography, the distance hydrogen can diffuse during a tensile test.

15 min. break

MM 15.4 Mon 17:00 TC 006

HYDROGEN EMBRITTLEMENT IN AEROSPACE MATERIALS — •Sathiskumar Jothi, Nick Croft, and Stephen GR Brown — Swansea University, Swansea, UK

Microstructures play a prominent role in aerospace components which are typically made of high toughness, corrosion resistant and high strength structural polycrystalline metallic materials such as nickel and nickel based super alloys. Nickel and nickel based super alloys are made up of complex microstructures which are susceptible to delayed failure caused by absorption of hydrogen produced either during fabrication in manufacturing process (i.e electrodeposition, welding etc*) or during operational use under environmental conditions. Several catastrophic failures have occurred in nickel and nickel based super alloys due to intergranular as well transgranular hydrogen embrittlement (HE) and hydrogen stress cracking (HSC) not only in aerospace industries but also in many other engineering sectors. HE depends on many factors including hydrogen diffusion and segregation, microstructural morphology and defects, stresses and texture morphological behaviour. Under EU FP7 *MultiHy* project, we employed multiscale technique to investigate the influence of these factors in hydrogen embrittlement both computationally and experimentally. The studies provide insights on the influence of these factors and control it strategically to reduce the susceptibility of materials to hydrogen embrittlement

MM 15.5 Mon 17:15 TC 006

Investigation of Crack and Blister Formation Due to Hydrogen Loading in Iron — •Marie Tiegel[1], Annegret Lehmberg[1], May L. Martin[1], Martin Deutges[1], Christine Borchers[1], and Reiner Kirchheim[1,2] — [1]Institut für Materialphysik, University Göttingen, Germany — [2]International Institute for Carbon-Neutral Energy Research (WPI-I2CNER), Kyushu University, Japan

Hydrogen-induced damage is a serious problem in various applications of metals and it is becoming more relevant as hydrogen is developed as a solution for energy storage. The mechanism of damage due to hydrogen is still ambiguous and requires further investigation. In this work, hydrogen- induced cracking (HIC) in high-purity iron was investigated. The samples were electrochemically loaded with hydrogen at different current densities. Hydrogen loading leads to cracks inside the bulk of iron, and the cracks near the surface create bubble-like defects on the surface above the cracks, called blisters. The microstructure of the cracks and blisters was investigated using SEM and TEM, including electron backscatter diffraction analysis. The surface curvature of the blisters consists of steps in the material, which is attributed to multiple individual crack advance events. TEM observations of the microstructure underneath the blister surfaces suggest that the formation of the blisters is due to the production of shear bands. Density measurements allow estimation of the pressure in the cracks, which is compareble to the yield stress of iron. In conclusion, the growth mechanism of blisters and cracks is due to a stress release following an increase of pressure in newly formed voids during hydrogen loading.

MM 15.6 Mon 17:30 TC 006

Hydride phase precipitation and growth in thin Nb-H films — •Vladimir Burlaka, Stefan Wagner, and Astrid Pundt — Institut für Materialphysik, Universität Göttingen, Göttingen, Germany

In thin Me-H films below the critical thickness, hydrogen absorption and hydride precipitation are expected to be controlled by mechanical stress arising because of the interface matching between the film and the substrate as well as between the hydride precipitates and the surrounding host-matrix [1,2].

In the present study we experimentally address the effect of the film

thickness on hydride precipitation and growth in Nb-H thin films of 15 - 40 nm. Hydrogen gas loading is performed for in-situ Scanning Tunneling Microscopy [1] and in-situ XRD measurements to study the hydride precipitates volume content, their lateral distribution and their mean lateral sizes. We found a strong change in the lateral distribution and the mean size at the critical film thickness, for similar pressures applied. It will be demonstrated that XRD pattern generally change when the film thickness is decreased below the critical thickness, making hydrides invisible [2]. But, STM images clearly verify the presence of hydrides even in the thin thickness range.

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[1] K. Nörthemann and A. Pundt, Phys. Rev. B 78 (2008) 014105. [2] V. Burlaka, S.Wagner and A. Pundt. accepted for publication in JALCOM.

MM 15.7 Mon 17:45 TC 006

Hydrogen loading kinetics of Mg Thin Films: effect of the driving force revisited — •Helmut Takahiro Uchida[1], Magnus Hamm[1], Stefan Wagner[1], Carsten Baehtz[2], Björgvin Hjörvarsson[3], and Astrid Pundt[1] — [1]Univ. Göttingen, IMP, Friedrich-Fund-Platz 1, 37077 Göttingen, Germany. — [2]Institute of Ion Beam Physics and Materials Research, HZDR, Germany — [3]Uppsala University, Dep. of Physics and Astronomy, Sweden.

Mg forms a dihydride with a high gravimetric hydrogen content of 7.6 wt% upon hydrogen absorption. This hydride is very stable and blocks hydrogen diffusion („Blocking effect“) [1,2]. This hinders the practical use of pure Mg for mobile applications, at low tempertures.

In this work, the impact of the driving force on the hydride formation kinetics in Mg films (200-2800 nm) is studied at room temperature, applying time-resolved *in-situ* XRD at different constant hydrogen gas pressures [3]. The evolution of inner stress is also monitored.

The hydrogen loading kinetics is found to strongly depend on the chemical potential. A heterogeneous hydride nucleation and growth model is suggested and evaluated by finite-element-calculatuion. The model allows qualitatively explaining the complex stress development, the different diffusion regimes and the blocking-layer thickness.

Financial support by the DFG via PU131/9 and PU131/10, as well as by the DESY/Hamburg and the ESRF/Grenoble are gratefully acknowledged. [1]J.Rydén *et al.*, *J. Less-Common Metals*, **152** (1989) 295. [2]H.T.Uchida *et al.*, *Script. Mater.*, **64** (2011) 935. [3]H.T.Uchida *et al.*, *Acta Mater.* (2014) (*Manuscript accepted*).