

MM 41: Poster SYM Hydrogen in Materials: New Developments

Time: Thursday 18:00–20:00

Location: Poster B

MM 41.1 Thu 18:00 Poster B

Beyond the featureless particle approximation: An extension of quantum-liquid density-functional theory to account for rotational effects. — ●SERGEI YURCHENKO¹, SERGUEI PATCHKOVSKI², THOMAS HEINE¹, and GOTTHARD SEIFERT¹ — ¹Institut für Physikalische Chemie und Elektrochemie, TU Dresden, D-01069 Dresden, Germany — ²Steacie Institute for Molecular Sciences, NRC Canada, 100 Sussex Drive, Ottawa, Ontario, K1A 0R6 Canada

In a different contribution we present QLDFT (Quantum Liquid Density Functional Theory), a Kohn-Sham based extension to DFT for liquids, to describe the adsorption properties of molecules in host structures under consideration of quantum effects. In QLDFT the individual molecules are treated as featureless particles, and rotational contributions to the free energy are neglected. However in the vicinity of strong charges the featureless particle approximation breaks down and rotational contributions become important even at ambient conditions.

We present an extension of QLDFT to account for rotational effects. Then, we apply this new method to examine the effect of electric field-induced interactions on hydrogen adsorption. These interactions initiated by the atomic charges are stronger than dispersive interactions and can facilitate the H₂/host binding, which can potentially lead to the increase of the hydrogen adsorption. We present results of simulations of the hydrogen adsorption for a number of model and real structures. We analyze the rotational and spatial probability distributions of the adsorbed hydrogen both with slow and fast varying atomic charge fluctuations.

MM 41.2 Thu 18:00 Poster B

Hydrogen Adsorption in Metal-Organic Frameworks — ●IRENA SENKOVSKA and STEFAN KASKEL — Department of Inorganic Chemistry, Technical University Dresden, Germany

Metal-Organic Frameworks (MOFs) have recently received considerable attention because of their high specific micropore volume and the ability to store gas molecules exceeding the storage capacity of traditional adsorbents. A variety of differences in the MOFs structures makes it difficult to analyze the influence of different factors on hydrogen uptake capabilities in MOFs. We have investigated the influence of the minor structural changes of the MOFs on their hydrogen storage capacity. The influence of the incorporated metal was shown for following isostructural compounds: Cu₃(BTC)₂ (BTC=1,3,5-benzenetricarboxylate) and Mo₃(BTC)₂; Zn₂(BDC)₂DABCO and Co₂(BDC)₂DABCO (BDC=1,4-benzenedicarboxylate, DABCO=1,4-diazabicyclo[2.2.2]octane). Our research interest is directed also towards the discovery of new MOFs, as well as adjusting the pore dimensions of MOFs, using different building blocks, solvent and solvent mixtures, in order to improve gas uptake and adsorption properties. Magnesium-based MOFs were found with the same network topology, very small pore size and selective adsorption behaviour. They show a guest-induced reversible structure transformation due to the flexibility of the Mg₃-cluster and the organic linkers. This effect could be used for fitting the pore sizes and for the increase of gas sorption capability in Mg contained MOFs after all. The hydrogen adsorption was also studied in several Al-based IRMOFs.

MM 41.3 Thu 18:00 Poster B

H₂ Adsorption in Metal-Organic Frameworks — ●AGNIESZKA KUC¹, THOMAS HEINE¹, GOTTHARD SEIFERT¹, and HELIO DUARTE² — ¹Physikalische Chemie TU, Dresden, Germany — ²UFMG, Belo Horizonte, Brazil

We report the results of calculations on molecular hydrogen binding in so called IRMOF-1 (Isorecticular Metal-Organic Framework). MOFs are a new class of porous and stable materials that can act as hydrogen storage media. Density Functional Theory (DFT) was employed for the calculations of geometry of a connector and a linker (building blocks). Complete periodic systems and electronic structure of MOFs were calculated within Density Functional based Tight-Binding (DFTB) method. Second order Møller-Plesset (MP2) perturbation theory was used to calculate the interaction energies between H₂ and model building blocks.

Two different model structures were chosen to mimic the environment of extended IRMOF-1 crystal. Systematic study of the influence of the basis set size on the results was performed. The results were

corrected by employing the basis set superposition error (BSSE) to describe more precisely weak interactions. The mixed basis sets were used, depending on the interaction regions. The dipole moments and the electrostatic potential were estimated, as well.

MM 41.4 Thu 18:00 Poster B

Hydrogen on planar and curved graphitic surfaces — ●HELMUT HERMANN, MELANIE HENTSCHKE, ANDREI TOUZIK, and OLGA KHVOSTIKOVA — Leibniz-Institute for Solid State and Materials Research IFW Dresden, PF 270116, D-01171 Dresden, Germany

For planar graphene, recent theoretical work has predicted a maximum amount of about 4.5wt% hydrogen storage capacity [1]. Thermodynamic estimates have shown that the storage capacities do not exceed 1wt% at 100K and rapidly decrease at higher temperatures [2]. Experimental investigations of the hydrogen storage capacity of nanostructured graphitic material in the temperature range of 30K to 300K point to the assumption that the storage capacity is considerably enhanced at low temperatures by the presence of small open micropores as compared to planar graphene [3]. Therefore, we are looking for graphitic materials with particularly high fraction of micropores. Expandable graphite seems to be a promising starting material for this purpose.

[1] T. Heine, L. Zhechov, G. Seifert, Phys. Chem. Chem. Phys. 6 (2004) 980.

[2] A. Touzik, H. Hermann, Chem. Phys. Lett. 416 (2005) 137.

[3] M. Hentsche, H. Hermann, D. Lindackers, G. Seifert, Int. J. Hydrogen En. 32 (2007) 1530.

MM 41.5 Thu 18:00 Poster B

Hydrogen loading of nano crystalline niobium thin-films. — ●KAI NÖRTHEMANN and ASTRID PUNDT — Institut für Materialphysik, Universität Göttingen

This contribution presents the phase transition in hydrogen loaded nano crystalline thin films.

As model the system of niobium with hydrogen was measured during gas phase loading. The phase transition is monitored in situ with the surface sensitive scanning tunneling microscopy (STM). The α and hydride phase can be distinguish due to the expansion of the hydride phase. We will show the arrangement of the hydride phase in the surrounding α phase. Due to the continuous taken STM images we also show the time dependency of the phase transition.

This work is financially supported by the DFG via SFB 602.

MM 41.6 Thu 18:00 Poster B

Optical and magnetic characterisation of rare earth hydride films — ●HELGE SCHRÖTER, DIRK MENZEL, and JOACHIM SCHOENES — Institut für Physik der Kondensierten Materie, TU Braunschweig, Mendelssohnstraße 3, 38106 Braunschweig

It is well known that rare earth metals like yttrium or europium show a metal insulator transition, if they are exposed to a hydrogen atmosphere. Yttrium changes from a reflective metal to a very weakly transparent metal (YH₂) and finally to a transparent insulator (YH₃). Europium also undergoes a metal to insulator transition however, in addition a transition from antiferromagnetism to ferromagnetism has been reported to occur in powder samples [1]. Due to the reactivity of the pure rare earth metals it is difficult to grow stable films. We have grown in-situ hydrogenated films of yttrium and europium by pulsed laser deposition in a hydrogen atmosphere. By variation of the hydrogen pressure thereby, we succeeded to grow layers with different hydrogen concentrations. The EuH_x films have been covered in-situ with a gold layer to protect against oxidation and deterioration. After removal from the vacuum chamber the optical and magnetic properties of the films have been determined by spectroscopic ellipsometry and SQUID magnetometry, respectively. The former measurements demonstrate the formation of the optical gap, while the latter indicate an increasing moment with increasing hydrogen content. For the highest hydrogen concentrations a semiconductor with a gap of 1.9 eV and a ferromagnetic transition at 17 K is obtained.

[1] P. Wachter et al. J. Magn. Magn. Mat. 31-34 (1983) 255.

MM 41.7 Thu 18:00 Poster B

Mechanical stress impact on thin films thermodynamic prop-

erties - investigated by H-loading — ●STEFAN WAGNER and ASTRID PUNDT — Institut für Materialphysik der Universität Göttingen, Friedrich-Hund-Platz 1, 37077 Göttingen

Thermodynamic properties of thin films deviate strongly from those of bulk. The deviations are reported to originate from microstructure and from mechanical stress, while the contribution of both is unknown in particular. Focussing on the mechanical stress contribution and by using Pd-H and PdFe-H as model systems, it is shown that mechanical stress strongly changes phase transition pressures. For the Pd-H films the measured loading pressures shift up to 140 mbar in contrast to 18 mbar for bulk. These shifts relate to the film bonding to the substrate and can be affected by film detachment.

MM 41.8 Thu 18:00 Poster B

Optimization of the magnetic properties of Co/Pd multilayers by applying a large persistent in-plane biaxial stress — ●SENTHILNATHAN MOHANAN and ULRICH HERR — Institut für Mikro- und Nanomaterialien, Universität Ulm, 89081 Ulm

Co/Pd multilayers exhibiting perpendicular magnetic anisotropy (PMA) are promising candidates for perpendicular recording media. In these multilayers, stress induced magnetic anisotropy contributes significantly to the PMA due to the high negative magnetostriction constant. We present a study about the optimization of the magnetic properties of Co/Pd multilayers by changing the magnetoelastic contribution to the anisotropy energy [1]. An in-plane biaxial tensile stress was applied to the Co/Pd multilayers by loading the Ta substrate with hydrogen. In-situ x-ray analysis during hydrogen loading and unloading showed that the hydrogen can absorb and desorb from the Co/Pd multilayer under the conditions used, but that it remains in the Ta substrate after exposing the sample to air again. This leads to a persistent in-plane biaxial stress of up to 2.8 GPa in the Co/Pd multilayers. We observed an increase of H_c and the remanent magnetization with increasing biaxial stress. Analysis of the effective perpendicular anisotropy K_{eff} in terms of a simple model yields the corresponding magnetostriction constants. The authors gratefully acknowledge financial support by the Landesstiftung Baden-Württemberg, Germany.

1. S. Mohanan, and U. Herr, *J. Appl. Phys.* 102, 93903 (2007)

MM 41.9 Thu 18:00 Poster B

Remote control of the Fe magnetic moment in magnetic heterostructures — ●ARNDT REMHOF^{1,2}, GREGOR NOWAK¹, HARTMUT ZABEL¹, MATTS BJÖRCK³, MARTIN PÄRNASTE³, BJÖRGVIN HJÖRVARSSON³, and VALERY UZDIN⁴ — ¹Department of Physics, Ruhr-Universität, Bochum, Germany — ²Empa, Swiss Federal Laboratories for Materials Research, Dübendorf, Switzerland — ³Department of Physics, Uppsala University, Sweden — ⁴Saint-Petersburg State University, Russia

In magnetic superlattices with interlayer exchange coupling, not only the coupling strength but also the magnetic moment of the ferromagnetic layer can be altered non-locally by modifying the electronic structure of the non-magnetic spacer layer. Specifically, changes of the electronic structure of the V spacer layers in Fe/V (001) superlattices are seen to affect the adjacent Fe layers. By reversibly loading the V layer with hydrogen, the magnetic moment of Fe increases, whereas the induced magnetic moment in V remains unchanged. The nature of this remote and non-local control of the magnetic moment is connected with a d-electron charge transfer and effective shift of the Fermi level relative to the d-bands of Fe and V, as elucidated on the basis of self-consistent electronic structure calculations [1].

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[1] A. Remhof et al, *Europhys. Lett.* 79 37003 (2007).

MM 41.10 Thu 18:00 Poster B

High pressure techniques for synthesis and characterization of hydrogen storage materials — ●CARINE RONGEAT, ISABEL LLAMAS JANSA, and OLIVER GUTFLEISCH — IFW Dresden, Institute for Metallic Materials, P.O. Box 270116, D-01171 Dresden, Germany

High pressure reactive milling is a very effective synthesis technique for hydrogen storage materials. It was used successfully to synthesize light metal hydride by milling metal powder under high hydrogen pressure [1]. This technique can also be applied to the synthesis of novel promising complex hydrides. For example, one-step synthesis of doped-sodium alanate was performed from NaH, Al and 4

[1] Doppiu et al., *J. Alloys Compd.* 427 (2007) 204-208.

[2] Rongeat et al., *J. Phys. Chem. B* (2007) accepted.

MM 41.11 Thu 18:00 Poster B

Formation and Hydrogen Reactivity of Complex Mg-Ni-La-(Co)-H phases — ●ANGELIKA TERESIAK, ANNETT GEBERT, VALENTIN BUNEA, JÜRGEN THOMAS, JÜRGEN ECKERT, and LUDWIG SCHULTZ — Leibniz Institute for Solid State and Material Research Dresden,

Currently, $Mg * Ni * RE(La)$ alloys are of great interest as new electrode materials for Ni-MH batteries. The recently discovered Mg_2NiLaH_7 hydride in the Mg - Ni - La - H quaternary system shows with 2,8 wt% (109 g/l H) a much higher hydrogen content than the well-known commercial $LaNi_5H_x$ - and TiV_2H_x alloys.

In the present study we investigate the formation of the complex hydride $Mg_2NiLa(Co)H_{7*x}$ by using various process routes, which aim at achieving material with nanocrystalline microstructure. The Mg_2NiLaH_7 forms completely by intensive ball milling of an inductively molten master alloy of Mg_2NiLa under hydrogen for 7.5h. Also, the gradual electrolytic charging of the master alloy using a current density of $-1mA/cm^2$ succeeded in forming the hydride. Starting from melt-spun amorphous ribbons, the crystallisation to nanocrystalline Mg_2NiLa and a subsequent hydrogenation under 0.5MPa hydrogen for 26h were performed and monitored by means of in situ high temperature XRD. Furthermore, the influence of Co additives to Mg_2NiLa on the phase formation and hydrogen absorption processes was studied.

MM 41.12 Thu 18:00 Poster B

The role of hydrogen in the development of free-machining titanium alloys — CARSTEN SIEMERS, MARTIN BÄKER, ●HANS-RAINER SINNING, and JOACHIM RÖSLER — Institut für Werkstoffe, TU Braunschweig, Langer Kamp 8, 38106 Braunschweig

Recently considerable effort has been made to improve the poor machinability of titanium and titanium alloys like Ti6Al4V. One of different possibilities is the use of hydrogen as a temporary alloying element: specimens are etched to remove surface layers, and heat treated in hydrogen atmosphere at 600-850°C to charge the Ti matrix with hydrogen (ca. 10-20 at%). Cutting operations are performed in the hydrogen-loaded state, which reduces the cutting force progressively with increasing cutting speed, up to 50% under high-speed cutting (up to 100 m/s) conditions. Finally, the hydrogen is removed from the samples by a heat treatment in vacuum, to restore the mechanical properties of the original alloy.

Such H-induced reduction of the cutting force can be attributed to two effects: the stabilisation of the high-temperature bcc β phase of titanium, which is softer and more ductile than the hcp α phase, and the so-called HELP (hydrogen-enhanced local plasticity) mechanism. The latter results from the shielding of the elastic interaction between dislocations and obstacles due to hydrogen moving along with the dislocations, which eases the shear deformation in the cutting zone where the temperatures exceed 200°C. The effect becomes more pronounced at higher cutting speed, with higher local temperatures and therefore faster hydrogen diffusion in the cutting zone.

MM 41.13 Thu 18:00 Poster B

Microstructure and electrical conductivity of Cu-Ti alloys aged in hydrogen atmosphere — ●SATOSHI SEMBOSHI^{1,2}, RYOTA GEMMA², TALAAT AL-KASSAB², ASTRID PUNDT², and REINER KIRCHHEIM² — ¹Materials Science and Engineering, Osaka Prefecture University — ²Institut für Materialphysik, Uni. Göttingen

The influence of the aging in a hydrogen atmosphere on the electrical properties of Cu-1 and 3 at. % Ti alloys was investigated by means of electrical conductivity measurements and structural measurements using the X-ray diffraction, transmission electron microscopy and atom probe tomography. In the Cu-3 at.%Ti alloy, aging for 48 hours at 773 K in hydrogen atmosphere leads to an improvement of the electrical conductivity up to 65 % IACS (International Annealed Copper Standard), in comparison the value obtained for the alloy aged in a vacuum atmosphere. In addition, aging in hydrogen atmosphere leads to an efficient decrease of the lattice parameter of the Cu solid solution (Cuss) phase, suggesting a significant decrease of the Ti concentration in the Cuss phase. An important structural finding is the existence of not only a needle-shaped $Cu_{3.4}Ti$ phase but also some cuboidal $TiH_{2.2}$ phase precipitates in the alloy aged in the hydrogen atmosphere. We, therefore, conclude that the aging in a hydrogen atmosphere strongly promotes the depletion of Ti in the Cuss phase because of the $Cu_{3.4}Ti$ and $TiH_{2.2}$ precipitates formation, resulting in the significant improvement of the electrical conductivity. This work is

partly supported by Inamori Foundation in Japan.

MM 41.14 Thu 18:00 Poster B

First principles analysis of Hydrogen in Manganese-rich austenitic steels — ●LARS ISMER, TILMANN HICKEL, and JÖRG NEUGEBAUER — Max-Planck-Institut für Eisenforschung GmbH, Max-Planck-Straße 1, 40237 Düsseldorf, Germany

Austenitic Mn-rich steels denote a promising new class of steels, since they combine high strength with ductility. Like other high strength steels, however, they are highly sensitive to hydrogen embrittlement (HE). Various microscopic mechanisms have been discussed as source of HE but the dominant mechanism has not been resolved so far. For a quantitative understanding a description of thermodynamic as well as kinetic aspects of the hydrogen-steel interaction is essential. We have therefore employed density functional theory to investigate the solubility and mobility of interstitial H in the austenitic phase of Fe, Mn, and Fe(x)Mn(1-x). For Fe(x)Mn(1-x) in the paramagnetic state our results show a significant increase both in the solubility and mobility of the H impurity with increasing Mn concentration. A detailed analysis shows that this effect is exclusively related to larger lattice volume induced by Mn. In addition we investigated the interaction of H with interstitial carbon. Our results show that at the high C-concentrations characteristic for the Mn steels the H solubility and mobility increase. This effect is caused by an (anisotropic) volume expansion of the lattice.

MM 41.15 Thu 18:00 Poster B

In-situ deformation study of porous nanocrystalline Pd during electrochemical charging — ●VISWANATH RAGHAVAN NADAR¹, DOMINIK KRAMER¹, and JÖRG WEISSMÜLLER^{1,2} — ¹Forschungszentrum Karlsruhe, Institut für Nanotechnologie, Karlsruhe, Germany — ²Technische Physik, Universität des Saarlandes, Saarbrücken, Germany

The length of porous nanocrystalline palladium in a nearly neutral aqueous NaF (0.7 M) electrolyte was measured using a commercial dilatometer as a function of the applied electrode potential and of the charge. As expected, the sample expands during formation of the palladium hydride phases. However, the length is not just a simple function of the faradaic charge, but shows a remarkable fine structure which we discuss using a comparison with other nanoporous samples, e.g. gold and platinum. Even without hydrogen ad- or absorption, the length of nanometals varies as a function of charge due to the variation of surface stress with charge density, which causes large variations of the bulk pressure of nanomaterials due to their large ratio of the number of surface atoms relative to the number of bulk atoms. The surface stress behaviour of palladium observed at potentials positive of the hydrogen adsorption is qualitatively similar to that of porous platinum, although there is a quantitative difference if one relates the expansion to the charge density, e.g. by calculating the surface stress-charge coefficient. The highly reproducible cyclic strain caused by hydrogen absorption and -desorption in Pd is larger than the surface stress effects by more than an order of magnitude.

MM 41.16 Thu 18:00 Poster B

Absolutbestimmung der Wasserstoffkonzentration innerhalb der plastischen Zone der Aluminiumlegierung AA6013 — ●CHRISTIAN LENK, THORSTEN LOEWENHOFF, MATZ HAAKS und KARL MAIER — Helmholtz Institut für Strahlen- und Kernphysik, Universität Bonn, Nussallee 14-16, D-53115 Bonn, Germany

In ersten orts aufgelösten Messungen an einer wechsellagernden CT-Probe aus der Legierung AA6013 konnte bereits eine erhöhte Wasserstoffkonzentration in der plastischen Zone vor der Risspitze nachgewiesen werden. Findet die Wechsellagerung in einer korrosiven Umgebung statt, so dass Wasserstoff in die Probe diffundieren kann, führt dies zu einer erhöhten Rissausbreitungsgeschwindigkeit und einem verzögerten Ausheilverhalten von Fehlstellen in der plastischen Zone. Der Nachweis erfolgt dadurch, dass im UHV Späne mit einer Dicke von wenigen μm aus einer Probe herausgesägt und in situ ausgeheizt werden. Der folgende Anstieg des Partialdrucks wird mit einem

Massenspektrometer aufgenommen. Es wird eine Methode vorgestellt, mit der es möglich ist, die absolute Wasserstoffkonzentration im Span aus der plastischen Zone zu bestimmen.

MM 41.17 Thu 18:00 Poster B

Hydrogen in ZnO - a challenge to experiments and theory — ●GERHARD BRAUER¹ and JAN KURIPLACH² — ¹Forschungszentrum Dresden-Rossendorf e.V., Dresden, Germany — ²Charles University, Prague, Czech Republic

Positron lifetime spectroscopy, nuclear reaction analysis and X-ray diffraction have been combined to investigate various, nominally undoped, ZnO single crystals. Hydrogen is detected in all crystals in a bound state (0.3 - 0.8 at.-%), and in some cases also in an unbound state (0.7 * 1.9 at.-%), which can be removed by annealing. A single positron lifetime of 180-182 ps and 165-167 ps is measured for all hydrothermally and melt grown crystals, respectively. These lifetimes are attributed to zinc vacancy-hydrogen complexes, as deduced from ab initio studies of various vacancy-hydrogen defect configurations in ZnO and related positron calculations. In addition, various defect studies of hydrothermally grown (0001) oriented ZnO crystals electrochemically doped with hydrogen are presented. It is demonstrated that a very high amount of hydrogen (up to ~30 at.-%) can be introduced into the crystals by electrochemical doping. It is found that more than half of this amount is chemically bound, i.e. incorporated into the ZnO crystal lattice.

MM 41.18 Thu 18:00 Poster B

Hydrogen in V-8at%Fe single-layered films and Fe/V multi-layered films — ●RYOTA GEMMA, TALAAT AL-KASSAB, REINER KIRCHHEIM, and ASTRID PUNDT — Institut fuer Materialphysik, Friedrich-Hund-Platz 1, D-37077, Goettingen, Germany

The influence of out-of-plane lattice parameter a_0 of V layer on the plateau slope of electromotive force (EMF) curves of V-8at%Fe films and Fe/V multi-layered films was investigated by applying in-situ XRD and in-situ stress measurements equipped with electrochemical hydrogen loading set up. For the films with small a_0 , the plateau slope and hydrogen-induced stress were found to be larger than those with larger a_0 . This trend can be qualitatively understood if we consider that hydrogen atoms predominantly occupy O_z sites in vanadium hydride phase, unlikely for the materials with isotropic site occupation as Pd. The deuterium distribution in the same kind of (110) films was also investigated by atom probe tomographic analysis (APT). The depth profile of the Fe/V films showed D atoms concentrated in V layers, as expected from the fact that V has bigger enthalpy for hydrogenation than that of Fe. However, the D concentration was not homogeneous in a V layer and far below the expected value from EMF curve. A similar phenomenon was found also for V-8at%Fe single-layered film. The results will be discussed in terms of difference in the microstructure and initial stress of the films.

MM 41.19 Thu 18:00 Poster B

Wechsellagerungsmaschine für spezielle Anforderungen — ●THORSTEN LOEWENHOFF, CHRISTIAN LENK, MATZ HAAKS und KARL MAIER — Helmholtz Institut für Strahlen- und Kernphysik, Universität Bonn, Nussallee 14-16, D-53115 Bonn, Germany

Zur Untersuchung dünnwandiger Al-Legierungen für den Flugzeugbau wurde eine Wechsellagerungsmaschine konstruiert, um den speziellen Anforderung an die Probenabmessungen zu genügen. Der Aufbau erlaubt mittels einer Piezokeramik Wechsellagerungen im Bereich von einigen 10 μm Amplitude mit Frequenzen von 0,1 Hz bis 50 Hz. Die entwickelte Steuerungssoftware stellt verschiedene Signalformen (Sinus, Dreieck, Rechteck) zur Verfügung und nimmt Daten (Kraft auf die Probe, Verformungsamplitude) zur späteren Analyse auf. Im Gegensatz zu hydraulischen Verformungsmaschinen lässt die Piezokeramik Rechtecksignale mit guter Flankensteilheit zu. Dies ermöglicht Untersuchungen der Eindiffusion von Wasserstoff an Risspitzen in Al-Legierungen.