

A new setup for optical measurements under controlled environment

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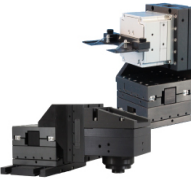
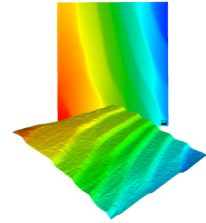
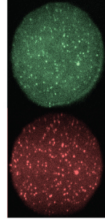
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ABSTRACT

We present a new analytical instrument for studying the optical properties of materials in different gaseous environments at room and controlled elevated temperatures. The system consists of a vacuum chamber, which is equipped with temperature and pressure controllers, a heating band, and a residual gas analyzer and is connected to a gas feeding line via a leak valve. Two transparent view ports located around a sample holder allow for optical transmission and pump-probe spectroscopy using an external optical setup. The capabilities of the setup are demonstrated by conducting two experiments. In the first experiment, we study the photodarkening and bleaching kinetics of photochromic oxygen-containing yttrium hydride thin films illuminated in ultra high-vacuum and correlate it with changes in partial pressures inside the vacuum chamber. In the second study, we investigate changes in the optical properties of a 50 nm V film upon hydrogen absorption.

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I. INTRODUCTION

The study of optical characteristics of materials provides unique information on intrinsic properties, such as microstructure, impurities and defect levels, lattice vibration, and electronic structure.^{1,2} Furthermore, macro-scale parameters, such as roughness and thickness, have an impact on optical properties and can be determined.³ Many substances are modified upon changes in their environment as they undergo, for example, temperature- or pressure-induced phase transitions⁴ or photo-induced reactions.⁵ The observation of the optical response under different conditions (like gaseous environment, illumination, and heating) enables the investigation of physical processes and their kinetics.

Since, in some cases, changes in optical properties indicate a phase transition, optical spectrometry can be used to reconstruct a phase diagram, as reported in Ref. 6 for the binary mixture N₂-CO. By means of pressure-dependent reflectivity measurements, increasing metallicity with increasing pressure was observed for Cd₃As₂.⁷ The measurement of optical absorption is commonly used to study photocatalytic degradation of redox indicators (e.g., Methylene blue) and thereby assess the efficiency of photocatalysts.⁸

The photochromic effect in oxygen-containing rare-earth metal hydrides (REMHO), i.e., photo-induced reversible modulation of

their optical absorption, has been first observed in YHO⁹ and subsequently in other REMHO, like GdHO, DyHO, ErHO, and NdHO.^{10,11} Despite numerous works on photochromic REMHO over the last decade, the origin of the photochromic effect remains under discussion. Baba *et al.*¹² proposed the reversible release and uptake of oxygen as the underlying mechanism of photochromic reactions. Authors observed a tremendously reduced bleaching rate of photochromic YHO films in the N₂ atmosphere and connected this to the lack of oxygen in the environment necessary for the recovery process. In the same work, photo-induced lattice contraction, first reported in Ref. 13, was discussed. While these results were explained by O release in the surrounding, direct evidence of any outgassing were not provided. Using the effective medium theory in Ref. 14, changes in the optical absorption under illumination have been explained by the formation of metallic hydride domains in the insulating matrix. Employing atomic probe tomography (APT) and transmission electron microscopy (TEM), Hans *et al.* demonstrated that photochromic GdHO films consist of two phases: an H-enriched phase and an O-enriched one.¹⁵ It was also suggested that the migration of H atoms between the two phases is responsible for the photochromic effect. Evidence of H₂ release from YHO films under illumination was presented in Ref. 16; however, the correlation between the photodarkening process and potential outgassing,

as well as the effect of the environment on photodarkening kinetics, were not established due to technical limitations. In Ref. 17, authors studied photochromism in YHO at low temperatures (as low as 5 K), while the effect of elevated temperatures also remains unexplored.

The use of optical transmission spectrometry for the investigation of thin film metal hydrides has been intensified over the last decades to complement gravimetric,¹⁸ temperature-programmed desorption,¹⁹ and volumetric²⁰ techniques as *in situ* concentration measurements.^{21,22} The effect of hydrogen uptake on the electronic structure and consequently the optical response exhibits large variations for different metals and structures while the underlying fundamental mechanisms are not yet fully revealed.²³ For some metals, the effect of hydrogen is so profound; switchable mirrors can even be made from, e.g., hydrides of Y or Mg–Ni alloys^{24,25} due to metal–insulator transitions. For the case of V–H, an interstitial metal hydride, hydrogen is found to induce *s*–*d* hybridization located around 6 eV below the Fermi level.²⁶ However, optical photons in the visible range only excite electrons from the upper d-band close to the Fermi energy. Thus, observed changes in the optical transmission²⁷ might not necessarily reflect changes in the band structure due to hybridization. Droulias *et al.*²⁸ suggest that volume effects cause shifts in the orbital overlap, which drive the changes in optical transmission upon deuterium absorption rather than rearrangements of electronic states around the Fermi level. While relying on calibration, optical transmission spectrometry for indirect concentration measurement and simultaneous measurement of the hydrogen pressure and temperature at equilibrium conditions can further reveal information on thermodynamic properties.²⁹

Materials and phenomena like the above mentioned require optical measurements combined with well controllable environments. To allow such measurements, we present a new setup for optical measurements under a controlled environment, denoted as OSFOLD (Optical Setup FOR gas LoadIng experiments). The equipment is designed to conduct measurements of the optical transmission while the sample is under different gaseous environments and temperatures. Additionally, the setup features a residual gas analyzer (RGA) and good vacuum conditions; therefore, thermal and photo desorption spectroscopy can be realized. To demonstrate the capabilities of the system, the photochromic effect in YHO thin films and H absorption in V thin films have been investigated.

II. INSTRUMENTATION

OSFOLD consists of two stainless steel chambers separated by a gate valve (Fig. 1). The first chamber is coupled to a turbomolecular pump (Pfeiffer HiPace 400) backed by a dry vacuum pump (Kashiyama NeoDry 15E), which enables ultra high-vacuum (UHV) conditions. The chamber is further equipped with a Pirani/cold cathode gauge (Pressure gauge 1), which is working in the range from 1000 to 1×10^{-9} mbar. The installed RGA (Extorr XT100) scans in the mass range from 1 to 100 amu with a nominal sensitivity of 10^{-11} mbar for mass 28 and is located above the turbomolecular pump.

The second chamber is designed to host the sample and allows optical characterization via two kodial glass viewports that are bakeable at temperatures up to 350 °C. The pressure is monitored by a capacitance gauge (Pressure gauge 2) situated on top of the chamber.

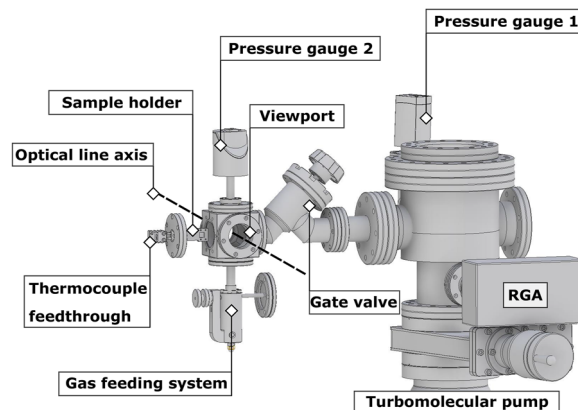


FIG. 1. Schematics of OSFOLD, consisting of the following main components: pressure gauge 1 (a Pirani/cold cathode gauge); pressure gauge 2 (a capacitance gauge); a gate valve; two kodial glass viewports; RGA (Extorr XT100); a turbomolecular pump; a K-type thermocouple feedthrough; a leak valve; and a sample holder.

Despite having a limited working pressure range (1100–0.1 mbar), the reading of capacitance gauges is not subjected to gas type. A leak valve located at the bottom of the chamber is connected to a gas feeding line and controls the flow of different gases (e.g., H₂, Ar, or O₂) injected into the system. Heating bands surrounding the chamber allow measurements at elevated temperatures, which are measured by two K-type thermocouples. In this approach, when the entire chamber is heated, the measurement can be conducted under thermal equilibrium between the sample and the environment. The first thermocouple with a diameter of 0.8 mm is installed at the sample holder via the thermocouple feedthrough, while the second one of 1.5 mm diameter measures the temperature at the heating band. A proportional integral derivative (PID) temperature controller (Eurotherm 3508) adjusts the heating power based on the reading from both thermocouples. An additional override algorithm is implemented to ensure gradual heating of the sample and prevent the exceedance of temperature thresholds.

Optical characterization is performed using the external optical line shown in Fig. 2. A stabilized tungsten-halogen source emitting light in the range of 360–2600 nm is used as a probe. The light is collimated by a biconvex lens with a 75 mm focal distance, while the beam diameter is adjusted by an iris diaphragm. The parallel beam is then transmitted through the sample and focused by another biconvex lens onto a compact CCD spectrometer (Thorlabs CCS200), which is sensitive to light from 200 to 1000 nm with an average spectral resolution of 4.4 points per nm. For pump–probe experiments, an additional high-intensity light source of wavelength <500 nm is installed perpendicular to the optical line. A longpass dichroic mirror with a 490 nm cut-on wavelength is oriented at 45° with respect to the optical line and reflects the light towards the sample creating two coaxial beams: the reflected pump light (<490 nm) and the transmitted probing light (>490 nm). A longpass filter with a cut-on wavelength of 500 nm is positioned in front of the spectrometer to prevent saturation of the CCD sensors by the pumping light.

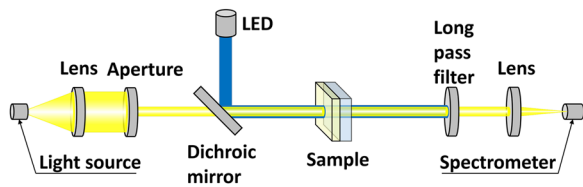


FIG. 2. Schematics of the optical line of OSFOLD. The main constituents are a white light source irradiating in the range of 360–2600 nm; a CCD spectrometer working in the range of 200–1000 nm; two biconvex lenses with 75 mm focal distance; an iris diaphragm; a longpass dichroic mirror reflecting light <490 nm; a longpass filter with a cut-on wavelength of 500 nm; and high-intensity LED.

III. EXPERIMENTS

A. Hydrogen absorption in V thin films

A V film of 50 nm was grown epitaxially on a single-crystalline MgO(001) substrate by DC magnetron sputtering following previous studies.²⁸ The film is capped with 5 nm of Pd, which prevents surface oxidation and catalyzes the dissociation of molecular hydrogen.³⁰ The sample was placed in the OSFOLD, and optical transmission spectra were continuously recorded with an integration time of 900 ms after UHV conditions had been reached. The temperature was gradually increased to 150 °C. When the sample temperature was stabilized, a reference transmission spectrum I_0 , averaged over several minutes, was extracted from the dynamically recorded optical transmission. After decoupling the vacuum pumps with the gate valve, molecular hydrogen was introduced into the chamber (19 Pa and 6.7 kPa as used in Ref. 27 for a similar sample). The hydrogen pressure was registered time-resolved with both pressure gauges. Averaged transmission spectra $I(p_{H_2})$ were recorded after equilibrium had been reached, i.e., stabilized transmission,

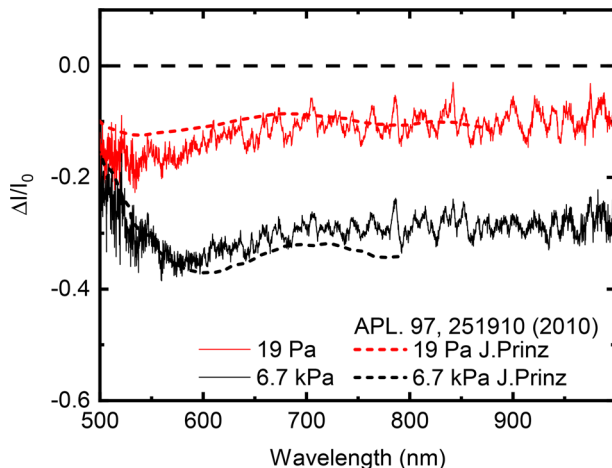


FIG. 3. Relative change in optical transmission spectra through a 50 nm V film on MgO due to H absorption measured at 150 °C. I_0 is the transmission at vacuum conditions averaged over several minutes, $I(p_{H_2})$ is averaged transmission spectra at a certain hydrogen pressure, and $\Delta I = I(p_{H_2}) - I_0$. The dashed line represents data reproduced with permission from Prinz *et al.*, "Combined light and electron scattering for exploring hydrogen in thin metallic films," Appl. Phys. Lett. 97, 251910 (2010). Copyright 2010 AIP Publishing for comparison.

pressure, and temperature. Figure 3 presents the change in optical transmission spectra $\Delta I = I(p_{H_2}) - I_0$ due to hydrogen absorption in the 50 nm V film at 150 °C normalized by the reference spectrum I_0 taken in vacuum. Hydrogen absorption in the Pd layer can be neglected due to a high heat of solution under these conditions.³¹ Furthermore, it is assumed that (multiple) reflections and scattering can be neglected. The optical transmission decreases with increasing hydrogen pressure while showing a wavelength dependence for both applied pressures. The maximum change in the transmission is about 35% and observed around 575 nm. Upon comparison to the experiments conducted by Prinz *et al.*²⁷ (data points are replicated in Fig. 3), we find good agreement between the results. Obtaining consistent results using the same methodology for the hydrogen loading experiments of thin vanadium films demonstrates excellent reproducibility and quality of the experimental method, setups, and samples.

B. Photochromic effect of YHO in UHV

A photochromic YHO sample was prepared in two steps. First, a 300 nm thin film of YH₂ was deposited on a soda-lime substrate using reactive magnetron sputtering in Ar:H₂ atmosphere. The total pressure during the deposition was 6.2×10^{-3} mbar, while the Ar and H₂ flow was kept at 20 and 4 SCCM, respectively. The DC power of 100 W with a pulsed frequency of 70 kHz was applied to the 2 in. Y target to create a plasma. After deposition, the film was oxidized in air forming YHO. More details on sample preparation can be found in Ref. 10. Chemical composition analysis employing ToF-E ERDA³² revealed that the sample composition is Y_{1.14}O_{0.86} and, thus, is in the range where photochromic properties are observed (i.e., oxygen to yttrium ratio $0.45 < \delta < 1.5$).³³

After the sample was prepared, it was transferred into OSFOLD, and the chamber was evacuated to $<10^{-9}$ mbar. The RGA was set to scan in the range of 0–44 amu with a sweeping rate of ≈ 0.166 scans per second. The transmittance of the sample was measured with an integration time of 60 ms and averaged in the wavelength range of 500–900 nm (which corresponds to 1718 data points). To start the photochromic transition, a 405 nm LED with a nominal irradiance of $14.53 \mu\text{W}/\text{mm}^2$ was utilized. The sample was illuminated for 10 min, and the changes in normalized transmittance T_{norm} are presented in Fig. 4(a). T_{norm} reduced by 24% under illumination and recovered to 95% of its initial value after 20 min of bleaching. Note that the sample was illuminated for the first time ever, thus the kinetics of the photodarkening is not affected by any memory effects.⁹ The photochromic reaction is accompanied by changes in the total pressure in the chamber P_{tot} [Fig. 4(b)]. A rapid increase and decrease of P_{tot} (within 1–2 s) with a subsequent gradual decrease is observed when the light turned on and off, respectively.

The RGA signal shown in Fig. S1 indicates that the major constituents of the residual gas are CO₂, H₂O, and H₂ (masses 44, 16, and 2 amu, respectively). Other peaks can be assigned to products of the fragmentation of the aforementioned molecules. As seen in Fig. 4(d), the illumination significantly affects the partial pressure of CO₂ ($P_{m=44}$). The increase of $P_{m=44}$ by 2.5×10^{-9} mbar within the first seconds of illumination corresponds to the initial rise of P_{tot} and is attributed to the photodesorption of CO₂ from the walls of the chamber. A similar effect was reported for different types of stainless steel exposed to the light of energy exceeding 2.92 eV.³⁴

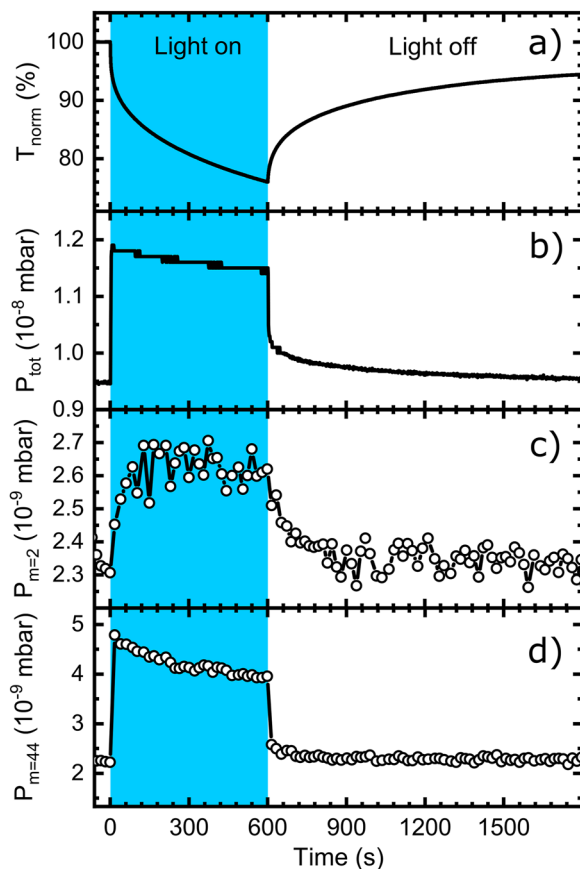


FIG. 4. (a) Time evolution of the normalized optical transmittance T_{norm} of the YHO sample exposed to 10 min illumination with a 405 nm LED, followed by 20 min bleaching. Corresponding changes in total pressure in the chamber P_{tot} (b), partial pressure of H_2 $P_{m=2}$ (c), and partial pressure of CO_2 $P_{m=44}$ (d).

Apart from the desorption of CO_2 , illumination with LED gives rise to the increase in hydrogen partial pressure [Fig. 4(c)]. This process happens at a lower rate in comparison to photodesorption and might be related to the photochromic reaction occurring in the YHO sample. According to the model proposed in Refs. 15 and 35, photodarkening arises from the transfer of hydrogen between two sub-phases of YHO and residual stress in the hydride phase, while some fraction of H especially at the surface can leave the film. An analogous effect, an increase in $P_{m=2}$, was reported in Ref. 16 where photochromic YHO was exposed to 455 nm light in UHV. Note that the apparatus used in that study did not allow simultaneous measurement of transmittance and partial pressure under illumination, i.e., during photodarkening.

IV. CONCLUSIONS

In this work, we present the optical setup for gas loading experiments (OSFOLD). Its capacity is exemplified by conducting two experiments, both requiring simultaneous measurements of the optical transmission and control of the gaseous environment.

Photochromic properties of a YHO film were studied in a UHV environment simultaneously monitoring the optical transmittance of the sample as well as changes in the partial pressures in the chamber. While the photochromic effect in an oxygen-free environment has previously been reported, OSFOLD permitted the establishment of the correlation between an H_2 release and illumination. As a second example, we have investigated a 50 nm V film to show the reliability and reproducibility of measurements with our setup. *In-situ* hydrogen gas loading experiments were performed, and the recorded optical transmission spectra are in good agreement with previously published data.

SUPPLEMENTARY MATERIAL

See the [supplementary material](#) for Fig. S1.

ACKNOWLEDGMENTS

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Dmitrii Moldarev: Conceptualization (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Writing – original draft (lead); Writing – review & editing (lead). **Kristina Komander:** Conceptualization (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Writing – original draft (equal); Writing – review & editing (equal). **Radek Holeňák:** Conceptualization (equal); Formal analysis (supporting); Investigation (supporting); Resources (equal); Visualization (equal); Writing – original draft (supporting); Writing – review & editing (supporting). **Max Wolff:** Conceptualization (equal); Project administration (equal); Resources (equal); Supervision (equal); Writing – original draft (supporting); Writing – review & editing (supporting). **Daniel Primetzhofer:** Conceptualization (equal); Project administration (lead); Resources (lead); Supervision (equal); Writing – original draft (supporting); Writing – review & editing (supporting).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

REFERENCES

- 1 G. Ding, C. Clavero, D. Schweigert, and M. Le, "Thickness and microstructure effects in the optical and electrical properties of silver thin films," *AIP Adv.* **5**, 117234 (2015).

- ²T. Tyner and J. Francis, "Infrared spectroscopy," in *ACS Reagent Chemicals* (American Chemical Society, 2008), Chap. 2.
- ³R. Swanepoel, "Determination of the thickness and optical constants of amorphous silicon," *J. Phys. E: Sci. Instrum.* **16**, 1214 (1983).
- ⁴H.-W. Chen, C.-Y. Huang, G.-J. Shu, and H.-L. Liu, "Temperature-dependent optical properties of CuFeO₂ through the structural phase transition," *RSC Adv.* **11**, 40173–40181 (2021).
- ⁵M.-F. Wang, Y. Mi, F.-L. Hu, H. Hirao, Z. Niu, P. Braunstein, and J.-P. Lang, "Controllable multiple-step configuration transformations in a thermal/photoinduced reaction," *Nat. Commun.* **13**, 2847 (2022).
- ⁶M. Vetter, H.-J. Jodl, and A. Brodyanski, "From optical spectra to phase diagrams—The binary mixture N₂–CO," *Low Temp. Phys.* **33**, 1052–1060 (2007).
- ⁷E. Uykur, R. Sankar, D. Schmitz, and C. A. Kuntscher, "Optical spectroscopy study on pressure-induced phase transitions in the three-dimensional Dirac semimetal Cd₃As₂," *Phys. Rev. B* **97**, 195134 (2018).
- ⁸P. Rong, Y.-F. Jiang, Q. Wang, M. Gu, X.-L. Jiang, and Q. Yu, "Photocatalytic degradation of methylene blue (MB) with Cu₁–ZnO single atom catalysts on graphene-coated flexible substrates," *J. Mater. Chem. A* **10**, 6231–6241 (2022).
- ⁹T. Mongstad, C. Platzer-Björkman, J. P. Maehlen, L. P. A. Mooij, Y. Pivak, B. Dam, E. S. Marstein, B. C. Hauback, and S. Z. Karazhanov, "A new thin film photochromic material: Oxygen-containing yttrium hydride," *Sol. Energy Mater. Sol. Cells* **95**, 3596–3599 (2011).
- ¹⁰S. Aalsteinsson, M. Moro, D. Moldarev, S. Droulias, M. Wolff, and D. Primetzhof, "Correlating chemical composition and optical properties of photochromic rare-earth oxyhydrides using ion beam analysis," *Nucl. Instrum. Methods Phys. Res., Sect. B* **485**, 36–40 (2020).
- ¹¹F. Nafezarefi, H. Schreuders, B. Dam, and S. Cornelius, "Photochromism of rare-earth metal-oxy-hydrides," *Appl. Phys. Lett.* **111**, 103903 (2017).
- ¹²E. M. Baba, J. Montero, E. Strugovshchikov, E. O. Zayim, and S. Karazhanov, "Light-induced breathing in photochromic yttrium oxyhydrides," *Phys. Rev. Mater.* **4**, 025201 (2020).
- ¹³J. P. Maehlen, T. T. Mongstad, C. C. You, and S. Karazhanov, "Lattice contraction in photochromic yttrium hydride," *J. Alloys Compd.* **580**, S119–S121 (2013).
- ¹⁴J. Montero, F. A. Martinsen, M. García-Tecedor, S. Z. Karazhanov, D. Maestre, B. Hauback, and E. S. Marstein, "Photochromic mechanism in oxygen-containing yttrium hydride thin films: An optical perspective," *Phys. Rev. B* **95**, 201301 (2017).
- ¹⁵M. Hans, T. T. Tran, S. M. Aðalsteinsson, D. Moldarev, M. V. Moro, M. Wolff, and D. Primetzhof, "Photochromic mechanism and dual-phase formation in oxygen-containing rare-earth hydride thin films," *Adv. Opt. Mater.* **8**, 2000822 (2020).
- ¹⁶D. Moldarev, L. Stolz, M. V. Moro, S. M. Aðalsteinsson, I.-A. Chioar, S. Z. Karazhanov, D. Primetzhof, and M. Wolff, "Environmental dependence of the photochromic effect of oxygen-containing rare-earth metal hydrides," *J. Appl. Phys.* **129**, 153101 (2021).
- ¹⁷E. M. Baba, P. M. Weiser, E. Ö. Zayim, and S. Karazhanov, "Temperature-dependent photochromic performance of yttrium oxyhydride thin films," *Phys. Status Solidi RRL* **15**, 2000459 (2021).
- ¹⁸R. Feenstra, D. G. de Groot, J. H. Rector, E. Salomons, and R. Griessen, "Gravimetric determination of pressure-composition isotherms of thin PdHc films," *J. Phys. F: Met. Phys.* **16**, 1953–1963 (1986).
- ¹⁹D. Broom, "The accuracy of hydrogen sorption measurements on potential storage materials," *Int. J. Hydrogen Energy* **32**, 4871–4888 (2007).
- ²⁰Y.-W. Lee, B. M. Clemens, and K. J. Gross, "Novel Sieverts' type volumetric measurements of hydrogen storage properties for very small sample quantities," *J. Alloys Compd.* **452**, 410–413 (2008).
- ²¹R. Gremaud, C. P. Broedersz, D. M. Borsa, A. Borgschulte, P. Mauron, H. Schreuders, J. H. Rector, B. Dam, and R. Griessen, "Hydrogenography: An optical combinatorial method to find new light-weight hydrogen-storage materials," *Adv. Mater.* **19**, 2813–2817 (2007).
- ²²J. I. Avila, R. J. Matelon, R. Trabol, M. Favre, D. Lederman, U. G. Volkman, and A. L. Cabrera, "Optical properties of Pd thin films exposed to hydrogen studied by transmittance and reflectance spectroscopy," *J. Appl. Phys.* **107**, 023504 (2010).
- ²³K. J. Palm, J. B. Murray, T. C. Narayan, and J. N. Munday, "Dynamic optical properties of metal hydrides," *ACS Photonics* **5**, 4677–4686 (2018).
- ²⁴J. N. Huiberts, R. Griessen, J. H. Rector, R. J. Wijngaarden, J. P. Dekker, D. G. de Groot, and N. J. Koeman, "Yttrium and lanthanum hydride films with switchable optical properties," *Nature* **380**, 231–234 (1996).
- ²⁵A. Borgschulte, R. Gremaud, S. de Man, R. J. Westerwaal, J. H. Rector, B. Dam, and R. Griessen, "High-throughput concept for tailoring switchable mirrors," *Appl. Surf. Sci.* **253**, 1417–1423 (2006).
- ²⁶Y. Fukai, S. Kazama, K. Tanaka, and M. Matsumoto, "Hydrogen-induced states in VH_x and VD_x observed by soft X-ray emission spectroscopy," *Solid State Commun.* **19**, 507–509 (1976).
- ²⁷J. Prinz, G. K. Pálsson, P. T. Korelis, and B. Hjörvarsson, "Combined light and electron scattering for exploring hydrogen in thin metallic films," *Appl. Phys. Lett.* **97**, 251910 (2010).
- ²⁸S. A. Droulias, O. Grånäs, O. Hartmann, K. Komander, B. Hjörvarsson, M. Wolff, and G. K. Pálsson, "Influence of deuterium-induced volume changes on optical transmission in Fe/V (001) and Cr/V (001) superlattices," *Phys. Rev. B* **105**, 195438 (2022).
- ²⁹X. Xin, G. K. Pálsson, M. Wolff, and B. Hjörvarsson, "Finite-size effects: Hydrogen in Fe/V(001) superlattices," *Phys. Rev. Lett.* **113**, 046103 (2014).
- ³⁰Y. Wang, G. K. Pálsson, H. Raanaei, and B. Hjörvarsson, "The influence of amorphous Al₂O₃ coating on hydrogen uptake of materials," *J. Alloys Compd.* **464**, L13–L16 (2008).
- ³¹F. D. Manchester, A. San-Martin, and J. M. Pitre, "The H-Pd system," *J. Phase Equilib.* **15**, 62–83 (1994).
- ³²P. Ström and D. Primetzhof, "Ion beam tools for nondestructive in-situ and in-operando composition analysis and modification of materials at the Tandem Laboratory in Uppsala," *J. Instrum.* **17**, P04011 (2022).
- ³³D. Moldarev, M. V. Moro, C. C. You, E. M. Baba, S. Z. Karazhanov, M. Wolff, and D. Primetzhof, "Yttrium oxyhydrides for photochromic applications: Correlating composition and optical response," *Phys. Rev. Mater.* **2**, 115203 (2018).
- ³⁴A. Mesarwi and A. Ignatiev, "Photodesorption from stainless steels," *J. Vac. Sci. Technol. A* **6**, 3140–3143 (1988).
- ³⁵A. Ohmura, A. Machida, T. Watanuki, K. Aoki, S. Nakano, and K. Takemura, "Photochromism in yttrium hydride," *Appl. Phys. Lett.* **91**, 151904 (2007).