

# Correlating Photoconductivity and Optical Properties in Oxygen-Containing Yttrium Hydride Thin Films


Suraya Kazi, Dmitrii Moldarev, Marcos V. Moro, Daniel Primetzhofer,\* and Max Wolff\*

Oxygen-containing yttrium hydride (YHO), primarily known for its photochromic properties, simultaneously shows a photoconductive response. Polycrystalline YHO thin films are deposited by reactive magnetron sputtering onto glass substrates and their optical and electrical properties are correlated under illumination with light. While photoconductivity is found in a broader range of compositions than photochromism, maximum photoconductive response is found for samples exhibiting photochromism. This photoconductive effect can be observed above a certain cutoff energy of light and a stronger response is triggered by shorter wavelengths. After the illumination is stopped, the slow recovery (few minutes) of the optical transmission and electrical transport suggests that the underlying process is not purely electronic. A memory effect is found depending on the previous exposure of samples to light which supports the hypothesis of local hydrogen diffusion during the photochromic reaction. An unprecedentedly fast bleaching speed is reported for samples stored in vacuum immediately after film deposition.

## 1. Introduction

Rare-earth metals are being exploited in a wide range of high-tech electric devices since long time ago mainly because of their peculiar magnetic properties.<sup>[1]</sup> Several oxygen-containing rare-earth metal hydrides (REMHO) like YHO, gadolinium oxyhydride (GdHO), dysprosium oxyhydride, erbium oxyhydride etc., exhibit a light-induced reversible decrease in optical transmission and a simultaneous increase in conductivity.<sup>[2,3]</sup> Combining and controlling the photochromism and the photoconductivity in REMHO thin films can facilitate the development of optical displays, transistors, smart windows, optical sensors, memory devices, etc.<sup>[3–6]</sup> However, the underlying physics is still to be established.

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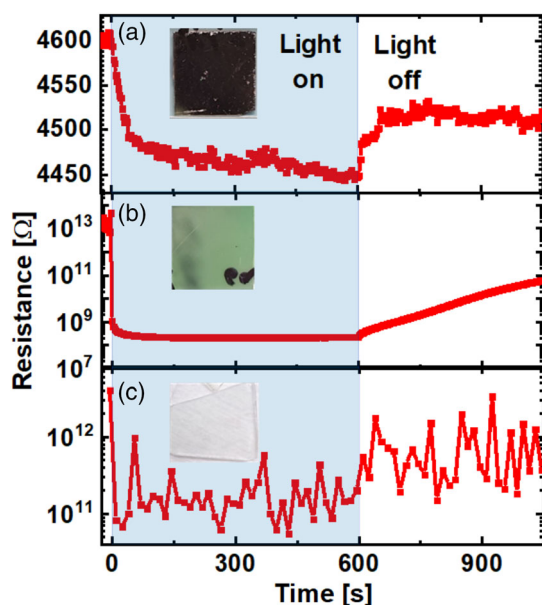
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The color neutral photochromism of REMHO materials was first observed at ambient conditions in yellowish transparent YHO thin film in 2011.<sup>[3]</sup> These YHO thin films are produced by at first creating metallic black YH<sub>2</sub> thin films by reactive magnetron sputtering of Y in Ar:H<sub>2</sub> atmosphere and later exposed to air to incorporate oxygen.<sup>[7]</sup> YHO thin films exhibit face-centered cubic (fcc) lattice structure similar to metallic black YH<sub>2</sub>, unlike YH<sub>3</sub> which is transparent insulator but has a hexagonal close-packed lattice structure.<sup>[3,8]</sup> The bulk nature of photochromism of YHO was hypothesized in a recent study observing its thickness dependence.<sup>[9]</sup> However, the photochromic response saturates for films above 600 nm indicating a limitation of this effect for bulk YHO material.<sup>[9]</sup>

Employing ion beam-based techniques, it has been demonstrated that the chemical composition of thin REMHO films can be altered by changing deposition parameters and can be described by the formula REMH<sub>2–δ</sub>O<sub>δ</sub> (where  $\delta$  is oxygen to rare-earth metal concentration ratio), as first demonstrated for YHO<sup>[10]</sup> and later confirmed for other REMHO.<sup>[11]</sup> A slightly different chemical formula (REM<sub>3–2δ</sub>O<sub>δ</sub>) was proposed by Cornelius et al., where a single-phase oxyhydride nature was assumed and, consequently, charge neutrality and stoichiometry were considered.<sup>[12]</sup> A photochromic behavior of REMHO was observed in samples with an oxygen concentration of  $0.45 < \delta < 1.5$ .<sup>[10]</sup> In this range of compositions, films are tinted transparent (see inset in Figure 1b) and semiconducting with a bandgap of approximately 2.6 eV.<sup>[10,13]</sup> Despite the implications for light adsorption resulting from these values, it has been demonstrated that also light of energies lower than the bandgap can trigger the photochromic effect.<sup>[14]</sup> For  $\delta < 0.45$ , the sample appears black and metallic similar to YH<sub>2</sub> (inset in Figure 1a) whereas for  $\delta \geq 1.5$ , the sample is a fully transparent (inset in Figure 1c) and insulating oxide with a bandgap of up to 3.8 eV.<sup>[10]</sup>

Atom probe tomography (APT) and transmission electron microscopy (TEM) performed by Hans et al. revealed a columnar microstructure of photochromic GdHO thin films and a dual phase nature, that is, coexistence of an O-rich insulating phase and an H-rich metallic phase.<sup>[15]</sup> They hypothesized that H-diffusion between the two phases may be the explaining mechanism behind the photochromic effect.<sup>[15]</sup> A similar hypothesis suggesting hydrogen transfer between two phases (coexisted fcc YH<sub>2</sub> and hexagonal YH<sub>3</sub>) was made by Ohmura et al. to describe photochromism in an yttrium hydride foil under high pressure ( $> 5.1$  GPa).<sup>[16]</sup>



**Figure 1.** Measured resistance plotted versus time of illumination with light of 455 nm wavelength and under relaxation for three oxygen-containing yttrium hydride (YHO) samples: a) opaque, b) photochromic, and c) fully transparent. The insets show photographs of the corresponding samples. The blue-shaded parts represent the time under which samples were illuminated.

Since YHO thin films are semiconducting in the range of compositions for which they show photochromism, they also show a strong photoconductive response. When the photochromic effect of YHO thin films was demonstrated for the very first time in 2011, an associated photoconductive response of these materials was also reported.<sup>[3]</sup> However, even though the photochromism of YHO has been studied by several research groups since then,<sup>[11]</sup> this photoconductive behavior has not been examined vividly regarding its dependence on wavelength and intensity of triggering light and the composition of the films. Recent work by Shimizu et al.<sup>[17]</sup> is the first demonstration of the reversible metallization of epitaxial YHO thin films in the photoconductive state which delineates that there is much more to understand about this phenomenon.

In this study, we simultaneously examined the wavelength and the intensity dependences of the photochromism and photoconductivity of polycrystalline YHO thin films. As a result, both effects clearly showed similar dependence on illumination conditions indicating a correlation between these two phenomena. We observed relaxation on a typical timescale of several minutes

for the photoconductive effect when illumination was stopped. Furthermore, repeated measurements after 15 min on the same sample indicated a memory effect of the film depending on the intensity of the previous light exposure to the sample. These findings rule out the possibility of the photoconductivity of REMHO thin films being a solely electronic process and are in line with the hypotheses of elemental diffusion (most likely, hydrogen) and/or structural rearrangement of O and H.<sup>[15,18]</sup>

## 2. Results

By studying YHO samples of different thicknesses, we found a thickness dependence of photoconductivity which saturates for films thicker than 500 nm (see Figure S1, Supporting Information) qualitatively similar to what has been reported for photochromism in a recent study.<sup>[9]</sup> Since photochromism is a bulk effect,<sup>[9]</sup> it is conceivable that photoconductivity may be so as well considering its thickness dependence. However, we note that any high conductivity layer would dominate our measurements.

### 2.1. Composition Dependence

Figure 1 depicts the reversible decrease in resistances under illumination of three YHO samples with different optical properties. The films are found opaque, photochromic, and fully transparent, which arises from the difference in their chemical compositions.<sup>[10,11]</sup> The oxygen concentration of the photochromic sample extracted from time-of-flight energy elastic recoil detection analysis (ToF-E ERDA) is  $\delta = 0.74 \pm 0.01$ , while for the chemical composition and growth parameters of the transparent sample we refer to sample HII in ref. [10]. In addition, in refs. [10,11], it was established that opaque samples are typically formed with  $\delta < 0.45$ . The growth parameters of the opaque and the photochromic samples can be found in Table 1, referred as Batch 1 and Batch 2, respectively. The opaque sample exhibits the lowest initial resistance of  $4.60 \pm 0.01$  k $\Omega$ , while the resistances of the photochromic sample and the transparent sample are in the order of  $10^{12}$ – $10^{13}$   $\Omega$ . Similar changes in resistance by several orders of magnitude with a change in O to H ratio were reported for GdHO.<sup>[19]</sup> The samples are illuminated with 455 nm light-emitting diode (LED) ( $I \approx 19.5$  W m $^{-2}$ ) for 600 s and allowed to bleach for up to 450 s, while measuring their resistance after terminating the illumination. All films, irrespective of their photochromic properties, show photoconductivity with the largest response for the photochromic ones. For example, the photochromic film from Batch 2 shows a decrease in resistance by

**Table 1.** Growth parameters of the samples.

Batch #	Date of deposition	Target distance [cm]	Base pressure [10 $^{-5}$ mbar]	Hydrogen flow [arb. Unit]	Growth pressure [10 $^{-3}$ mbar]	Growth time [sec]	Current [mA]	Color of the films
1	2020-11-20	5	4	20	10	1800	120	black
2	2020-12-16	6	4.5	20	8	900	120	greenish
3	2021-05-07	6.5	4	20	10	2100	120	yellow
4	2021-06-01	6.5	6	16	8	1800	120	yellow

4 orders of magnitude already after a few seconds of illumination (see Figure 1b).

The opaque sample initially relaxes quicker than the photochromic sample after terminating the illumination, but the resistance saturates without reaching the original values. This kinetics of the process indicates both a fast electronic component and another slow/irreversible process. In contrast, for the photochromic sample, the relaxation of the resistance after illumination is taking place in the order of a few minutes and not almost instantaneous as expected for an electronic process. To compare, the expected lifetime of charge carriers in semiconductors such as Si or Ge is found in the milliseconds or seconds range for indirect bandgap and in nanoseconds for direct bandgap transitions.<sup>[20]</sup> However, comparably slow relaxation or long lifetime of charge carriers can be seen in materials with a defect-rich structure where vacancies act as charge-carrier traps. The slow relaxation of the photoconductive response in YHO thin films may be an indication of the hydrogen diffusion and structural rearrangements inside the film during and after illumination.<sup>[15]</sup> The relaxation time scale also supports the recent work by Wu et al. where involvement of a multistep process during photodarkening is proposed, which includes the creation of H vacancies and localization of  $e_g$  electrons resulting in metallic domain formation.<sup>[8]</sup> According to Wu et al., under light exposure, hydrogen is liberated which creates positively charged vacancies at the octahedral sites with charge-compensating localized  $e_g$  electrons in the vicinity of Y ions.<sup>[8]</sup> Band formation might occur due to overlap between the orbitals when the concentration of the localized  $e_g$  electrons is high enough at certain domains leading to metallicity of the domains of the YHO film.<sup>[8]</sup>

Additionally, we observed that some samples even with weak photochromism (6.6%) can feature strong photoconductivity (decrease in resistance by 4 orders of magnitude). After 1 month, when the measurement was repeated, the photochromism dropped to  $3.0\% \pm 0.5\%$  but still maintained almost the same photoconductive response. Here, photochromism is calculated as  $PC = \frac{T_b - T_a}{T_b}$  where  $T_b$  and  $T_a$  are the transmittance of the film before and after the illumination, respectively.

## 2.2. Temperature Dependence

Recently published works<sup>[17,21]</sup> have shown that epitaxial thin films of YHO (originally insulating) exhibit metal-like temperature dependence of resistivity after UV laser exposure. In our study, polycrystalline YHO thin films photodarkened by 455 nm LED have showed similar behavior, that is, the resistance of the photodarkened film increased with temperature as expected for a metal (see Figure S2, Supporting Information). In contrast, for an undarkened sample, the resistance decreases with temperature like a semiconductor (see Figure S3, Supporting Information). This observation implies that during photodarkening of polycrystalline YHO thin films, the semiconducting phase transforms into a metallic phase (or more precisely, an admixture of metallic phase since YHO film is not homogeneous single-phase material, rather dual phase<sup>[15]</sup>). As proposed by Wu et al. and Hans et al.,<sup>[8,15]</sup> photo-triggered transport of H might increase the H-rich metallic regions (or the metallic domains) in the film which not only increase the

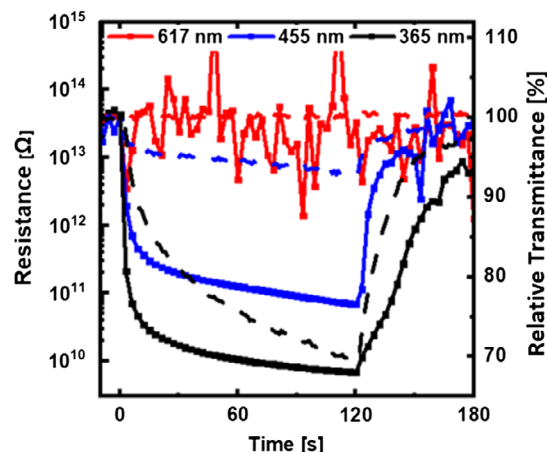
conductivity of the sample but also probably provide a metallic-like electronic structure to the H-rich domains. Thus, a dual-phased semiconductive YHO thin film can be turned into a material having an admixture of metallic phase by simply illuminating it. A similar semiconductor-to-metallic phase transition is not observed for other photoconductive materials which also manifest reversible photoconductivity but maintain a semiconductor-like temperature dependence of resistivity when illuminated.<sup>[22]</sup>

## 2.3. Wavelength Dependence

To study the effect of the energy of photons on the photoconductivity, samples from Batch 3 (see Table 1) with oxygen concentration  $\delta = 1.06 \pm 0.02$  (i.e., exhibiting photochromic behavior) were used. Figure 2 illustrates the relative optical transmittance and resistance of the three identical samples illuminated for 2 min separately by three LEDs with wavelengths of 617 nm red, 455 nm blue, and 365 nm violet, that is, 2.01, 2.72, and 3.40 eV photon energies, respectively.

Both the photoconductive and photochromic responses are more pronounced for shorter wavelengths. No photochromic and photoconductive effects are found for the longest wavelength (617 nm) which suggests that there is a cutoff wavelength between 455 and 617 nm to trigger both effects. This wavelength dependence can be explained by the bandgap of the sample, which is expected to be larger than the energy of blue light (2.72 eV) according to ref. [14].

Note that the samples of Batch 3 recovered exceptionally fast from the photodarkening, returning to 96–98% of their initial relative transmittance within 1 min after switching off the light. This fast recovery of the relative transmittance is, to the best of our knowledge, the fastest relaxation ever achieved. For comparison, check ref. [14] where the relaxation time constant for YHO samples in air is hours. These specific samples were kept in an evacuated desiccator right after synthesizing and the measurements were done within a few minutes, which could be an explanation

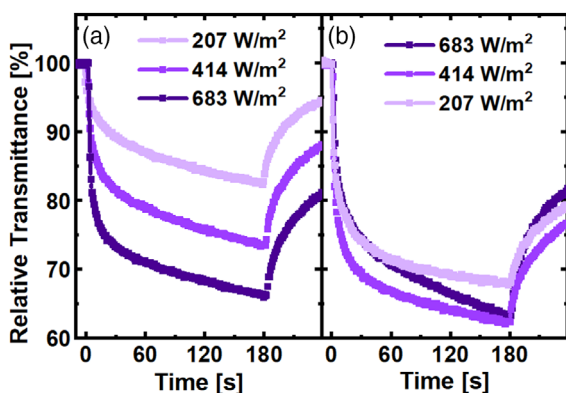


**Figure 2.** Resistance (dashed lines) and relative transmittance (solid lines) of YHO films (Batch 3) plotted versus time. The samples have been illuminated with light of different wavelengths but equal intensity ( $124 \text{ W m}^{-2}$ ) for 2 min followed by 1 min of relaxation.

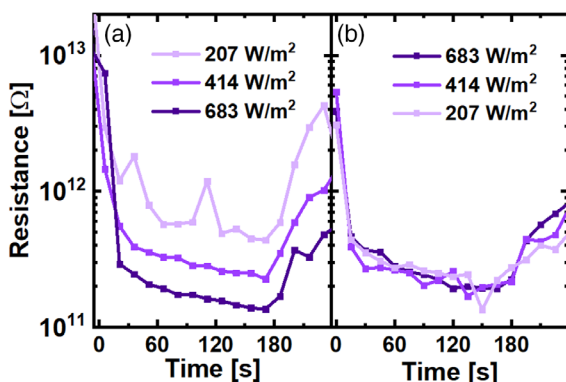
for this fast recovery and suggests that there is room to improve the performance of these materials; however, more systematic studies are needed to understand the bleaching dynamics.

## 2.4. Intensity Dependence

A sample from Batch 4 in Table 1 was illuminated for 3 min with 405 nm violet light three times with different intensities (207, 414, 683 W m<sup>-2</sup>). After each measurement, the sample was allowed to bleach to at least 97% of its initial transmittance. When the sample was illuminated with increasing intensity in subsequent illuminations, the strength of the photodarkening increased almost linearly (see Figure 3a). However, in case an identical sample was first illuminated with high intensity, followed by lower intensity in the subsequent illumination, it was found to darken to almost the same level (see Figure 3b). For example, the photochromic response triggered by the lowest intensity (207 W m<sup>-2</sup>) had almost doubled from 17.4% ± 0.2% to 32.0% ± 0.3% as a consequence of a prior illumination with higher intensity (see Figure 3). Qualitatively, we found a similar



**Figure 3.** Relative transmittance of a YHO film plotted versus time for 3 min of illumination with 405 nm violet LED and subsequent relaxation: a) starting from the lower intensity and proceeding toward the higher intensity as well as b) in reverse order.



**Figure 4.** Resistance of YHO plotted versus time during and after 3 min of illumination with 405 nm violet LED and subsequent relaxation: a) starting from the higher light intensity and proceeding toward lower intensity as well as b) in reverse order.

trend in the corresponding resistance measurement shown in Figure 4 where photoconductive responses are plotted versus time. Both these measurements indicate a memory effect, that is, the magnitudes of the photochromic and photoconductive responses are affected by previous illumination cycles. One explanation for this may be found in the hypothesis of hydrogen transfer from one phase to another as the proposed mechanism of photochromic reaction.<sup>[15]</sup> In this case, the number of H migrated under illumination is dependent on the light intensity. If a large amount of hydrogen changed their location (e.g., another phase) during illumination with high intensity, probably not all hydrogen atoms return to their previous position while relaxing. Some may still be trapped, for example, near a phase boundary and in the next cycle, when the lower intensity is used, easily diffuse back making the photochromic response faster.

## 3. Discussions

In this work, we demonstrate that all YHO samples irrespective of their chemical compositions and appearances (opaque, photochromic, or fully transparent) exhibit photoconductivity where the photochromic ones show the largest relative photoconductive response. Both photochromic and photoconductivity effects showed higher response for shorter wavelength, and a cutoff region (between 455 and 617 nm) to trigger the effects. These common characteristics could be related to the bandgap of the YHO samples. Both effects can be visible for the energy of light slightly lower than their bandgap which indicates the involvement of other processes such as defect formations, during illumination.<sup>[8]</sup>

Furthermore, we have seen that both effects recover close to the initial value almost at similar time constants, but with different scaling, indicating that they might follow different kinetics or might scale differently with the induced change in concentrations, charge carriers, etc. Moreover, the recovery time scales for both effects being in the range of minutes shows that they are not purely electronic processes, but rather multistep mechanisms as proposed by Wu et al.<sup>[8]</sup> as previously mentioned.

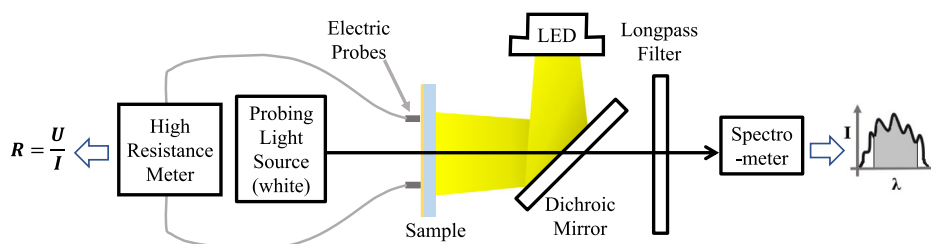
Both responses being affected by a previous exposure of the sample to illumination, that is, the existence of a clear memory effect is another prominent finding of this study which validates that none of these effects is expected to solely occur due to electronic transfer but rather involves more complex multistep processes such as short-range hydrogen transfer in between phases and possible structural modifications in line with the hydrogen diffusion hypothesis claimed by Hans et al.<sup>[15]</sup> or the H liberation resulting in metallic domain formation proposed by Wu et al.<sup>[8]</sup>

A proposed way to corroborate the present interpretation could be to perform experiments targeting the dynamics of elements at the atomic level during illumination to validate the hypothesis of hydrogen transfer between phases. Also, the unprecedented fast switching observed in this work should be optimized further to create high-quality samples for applications.

## 4. Summary and Conclusion

We produced YHO thin films by reactive magnetron sputtering in Ar:H<sub>2</sub> environment and characterized their chemical, optical,





**Figure 5.** Schematic diagram of the setup for simultaneous optical and electrical characterization.

and electrical properties by means of ion-beam analysis (IBA), UV–visible spectrometry, and two-point probe-resistance measurement. We found indications that photoconductivity can be a bulk effect since it increases with increasing YHO film thickness and saturates above 500 nm of thickness. In this work, we grew samples with unprecedented fast bleaching kinetics which is promising for application. We observed photoconductivity in all types of YHO thin films (opaque, photochromic, or fully transparent) with the largest response at the level of 4 orders of magnitude for photochromic samples. Both effects, photoconductivity and photochromism showed similar wavelength dependence with respect to the triggering light. Furthermore, we observed a memory effect (dependence on previous exposure) in the samples which is not expected for a single excitation electronic process. Moreover, the time scale of the relaxation curve of the photoconductive response, being in minutes, is another indication of the photochromic and photoconductive effect to be a multi-step process. Some species, most likely hydrogen moving over short distances or formation of metallic domain can be involved according to earlier hypotheses.<sup>[8,15]</sup>

## 5. Experimental Section

**Synthesis:** Yttrium hydride thin films were synthesized on glass substrates (1 mm thick microscopy slides) through reactive magnetron Ar-sputtering inside a compact Balzer Union DC sputtering device. An yttrium disc (99.99% pure) was installed as a target in the upper part of the sputtering chamber connected to the cathode and the glass substrates were mounted on the substrate holder. High vacuum ( $\approx 10^{-5}$  mbar) was achieved in the chamber prior to the deposition with the help of a turbomolecular pump. A mix of Ar and H<sub>2</sub> gas was introduced (pressure  $\approx 10^{-3}$  mbar), and a negative voltage was applied to the target to ignite a plasma. The nominal purity of the hydrogen gas was 99.998%. Detailed theoretical descriptions and working principles of reactive magnetron sputtering method can be found elsewhere.<sup>[7,23–25]</sup> After being exposed to air at ambient conditions, the yttrium hydride thin films (black in color) turned into tinted transparent films of YHO with various colors (mostly yellowish) due to oxidation. The growth conditions of different samples used for this work are summarized in Table 1.

**Characterization:** Samples were characterized with respect to their chemical, optical, and electrical properties. For determining the chemical composition, ToF-E ERDA using a 36 MeV I<sup>16+</sup> beam from the 5 MV NEC SSDH-2 pelletron accelerator at the Tandem Laboratory, Uppsala University, was employed.<sup>[26]</sup> Thicknesses of the films were calculated from the areal densities determined by Rutherford backscattering spectrometry (RBS) using 2 MeV He<sup>+</sup> and assuming bulk densities calculated in ref. [9]. Details about IBA and determining the chemical composition of similar REMHO films can be found elsewhere.<sup>[10,11,26]</sup> Depth-resolved

chemical composition profiles of some samples obtained from IBA can be found in Figure S4, Supporting Information.

**Optical Characterization:** To examine the photochromic response of the samples, UV–visible spectroscopy was employed. White light from a compact stabilized tungsten–halogen light source was used as probing light and was detected and recorded by a UV–vis spectrometer (Kvant spectra-1). The photochromic effect in the samples was induced by nearly monochromatic LEDs of different wavelengths with tunable intensity. Lights from these LEDs were reflected toward the sample by a dichroic mirror which was placed at 45° angle with respect to both the LED and the sample surface (see Figure 5). This dichroic mirror transmitted the probing light and the intensity spectra of the light transmitted through the film during a full cycle of photodarkening and bleaching were recorded. A longpass filter was placed in front of the detector of the spectrometer to avoid any light from the LED being recorded. The relative transmittance of the film was calculated by normalizing the averaged intensity within a specific range of wavelengths (700–900 nm to exclude the effect from the triggering lights) with respect to the value of the averaged intensity before illumination.

**Electrical Characterization:** The resistance of the samples was measured by a two-point probe since it was easier to develop than the more common four-point probe and also because of the limitation of our instrumentation. Two gold-plated contact pins distanced by 1 cm from each other were pressed against the surface of the film. To minimize the possible mechanical damage of the film, probes were attached to compressing springs. The probes were connected to an HP 4339B high-resistance meter which allows precision high-resistance measurements with a sampling rate of 10 ms. The resistance was calculated by Ohm's law from the measured current and the applied voltage. As the resistance of the sample is extremely high, contribution from the contacts as well as possible changes in the contact resistance due to changes in a work function of the film under illumination<sup>[27]</sup> should be comparably small. The setup illustrated in Figure 5 was used to perform optical and electrical characterization simultaneously on the same sample. More details on the setup can be found elsewhere.<sup>[28]</sup> The spectrometer and the resistance meter were connected to computers equipped with a LabVIEW<sup>[29]</sup> Virtual Instrument (VI) program to automate the measurements and save the results.

## Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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## Conflict of Interest

The authors declare no conflict of interest.

## Data Availability Statement

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

## Keywords

memory effects, oxygen-containing yttrium hydrides, photochromism, photoconductivity, thin-film characterization

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