

Effect of temperature and illumination conditions on the photochromic performance of yttrium oxyhydride thin films

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ABSTRACT

Transparent yttrium oxyhydride (YHO) thin films exhibit photochromic effects under ambient conditions. The optical transmission in such materials can be strongly modulated upon light absorption. This switchable optical property is interesting for technological applications, such as energy-saving smart windows and optical sensors. For the practical use of photochromic YHO materials, it is crucial to understand how the kinetics of coloration and the resulting photochromic contrast are influenced under different illumination conditions at both room and elevated temperatures. In the present study, we have prepared yellow, transparent, photochromic YHO thin films on glass substrates by using reactive magnetron sputtering. We investigated the photochromic performance of both as-deposited and annealed YHO thin films using illumination sources with varying photon energies and intensities. The photochromic contrast and the coloration were found to decrease considerably with increasing annealing temperature and with decreasing intensity and photon energy of the illumination sources. Moreover, time-resolved optical transmission measurements revealed a nearly logarithmic time dependence of the coloration of YHO thin films, indicating rapid coloration in the beginning of light exposure, followed by slow coloration with increasing illumination time.

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I. INTRODUCTION

Thin films of yellow transparent yttrium oxyhydride (YHO) have recently been demonstrated to exhibit photochromic effects under ambient conditions.¹ The optical transmission in such materials can be strongly modulated upon absorption of electromagnetic radiation. The transparency of YHO films can be reduced from typically ~80%–90% in the clear state to ~30%–40% in the photodarkened state in the visible and infrared regions of the light spectrum. The photodarkened YHO film thermally bleaches back to its initial yellow clear state upon dark annealing at room temperature and atmospheric pressure. This switchable optical property is interesting for a wide range of technological applications, such as energy-saving smart windows,² displays,³ optical sensors,⁴ spectacle lenses,⁵ and optical memories for data storage.⁶ Consequently, there has been a great effort in recent times to study the structural,⁷ compositional,^{8–11} surface,¹² electrical,¹³ and optical^{14–16} properties of YHO materials.

For the implementation of photochromic YHO materials in practical applications, it is essential to gain more insights on the

kinetics of the photodarkening and bleaching processes. Recently, it has been shown that thermal bleaching characteristics can be modified by doping YHO with Zr during thin film preparation,¹⁷ changing the oxygen concentration in a YHO compound,¹³ or increasing the heating temperature to 60–100 °C.¹⁸ In the present study, we focus on the kinetics of coloration and photochromic contrast in YHO. We have prepared yellow, transparent, photochromic YHO thin films on glass substrates by using reactive magnetron sputtering. We investigated how the photochromic performance of both as-deposited and annealed YHO thin films is affected when the photon energy and intensity of illumination sources are varied. Time-resolved optical transmission data unveiled a nearly logarithmic time dependence of the coloration of YHO thin films, indicating rapid coloration in the beginning of light exposure, followed by slow coloration with increasing illumination time.

II. EXPERIMENTAL DETAILS

Thin films of YHO were grown on glass substrates by employing reactive magnetron sputtering in an inline sputter-coating

system from Leybold Optics (model A550V7). The glass substrate size was $76 \times 26 \text{ mm}^2$ with a thickness of 1 mm. The total gas flow of argon and hydrogen was 180 sccm with an Ar:H₂ gas ratio of 8:1. The film deposition pressure was 0.6 Pa. We note that the oxygen gas was not injected during the film growth. The oxygen incorporation into the film was taking place when the yttrium hydride film reacted with air right after it was removed from the vacuum chamber.¹⁹ The substrate carrier was operated in the oscillation mode in front of the fixed Y target during deposition in order to obtain YHO films with a homogeneous composition across the whole sample volume.²⁰ By applying the above-mentioned deposition condition, we managed to achieve black YHO samples after the initial oxidation process. Upon prolonged oxidation in air (typically after a few hours), however, the black YHO samples gradually transformed to yellow transparent due to an increased oxygen concentration in the film. Such black-to-yellow transformed YHO thin films have previously been demonstrated to exhibit an enhanced photochromic response as compared to yellow YHO films that are formed immediately in contact with air, when their oxygen concentration exceeded a threshold level.¹⁶ The film thickness was determined using a stylus surface profilometer.

The optical properties (transmission and reflection) of YHO thin films before light exposure were characterized using an Ocean Optics QE65000 spectrometer equipped with an integrating sphere and deuterium and tungsten halogen light sources. In order to induce the photochromic effect in YHO, we used an advanced light engine in a WAVELABS solar simulator equipped with a total number of 21 LED light sources with different photon energies and tunable intensities. As can be seen in Fig. 1, six LED light sources were selected to illuminate the YHO samples: UV, blue, green, yellow, orange, and red LEDs. The detection LED with a center

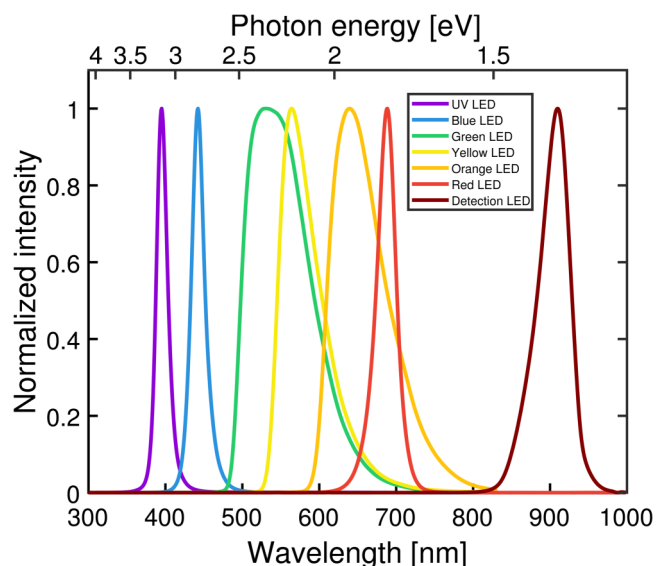


FIG. 1. Normalized intensity profiles plotted as a function of wavelength or photon energy for different LEDs used to illuminate the YHO thin film samples.

wavelength of 910 nm was chosen as the probing beam to measure the changes in optical transmission during illumination because the photon energy of this near-infrared LED is not strong enough to induce any photochromic reaction in the film. Note that three longpass colored glass filters, Thorlabs FGL495, FGL550, and FGL610, were applied to cut off a blue light component from the green, yellow, and orange LEDs, respectively, in order to avoid unwanted photodarkening contribution from the blue light. The maximum intensity, photon energy at the center of the normalized intensity profile, and spectral width (determined as the FWHM of the peak) for the LEDs are shown in Table I. Time-resolved optical transmission measurements of YHO thin films were carried out during continuous illumination up to 1 h at ambient conditions. The transmission data were acquired every second. Moreover, to study how the photochromic properties are influenced by sample heating, four small ($\sim 19 \times 26 \text{ mm}^2$ in size) nearly identical YHO samples (cut from one large YHO film) were annealed at 50, 100, 150, and 200 °C on a hotplate for 30 min in darkness under ambient conditions. The UV LED, operated at the maximum intensity, was used to illuminate the annealed samples.

III. RESULTS AND DISCUSSION

Figure 2(a) shows the optical transmission, T , and reflection, R , data for the as-deposited YHO sample at room temperature (RT) and YHO samples annealed at 50, 100, 150, and 200 °C. The film thickness of the as-deposited YHO was $\sim 870 \text{ nm}$. The absorption, A , data for the same YHO samples, determined as $A = 1 - R - T$, are shown in Fig. 2(b). The glass substrate is also included for comparison. As can be seen, the absorption edge is shifted to lower wavelengths with increasing annealing temperature, demonstrating a widening of the optical bandgap, in accordance with the results reported previously by Moldarev *et al.*⁹ The ion beam analysis performed by Moldarev *et al.* also revealed that upon annealing YHO thin films at elevated temperatures above $\sim 100^\circ\text{C}$, the oxygen concentration in YHO increased, whereas the hydrogen was partially released simultaneously. Note that their films were found to be uniformly oxidized. Thus, the reason for the annealing-induced bandgap widening can be ascribed to the increased oxygen concentration in the film, which is also consistent with oxygen-induced bandgap engineering observed in YHO films prepared with varying deposition pressures in our previous study.¹⁵

TABLE I. The maximum intensity, photon energy, and spectral width of the LED light sources.

LED	Max intensity (mW cm^{-2})	Photon energy (eV)	Spectral width (eV)
UV	5.1	3.14	0.13
Blue	9.7	2.80	0.12
Green	9.9	2.34	0.40
Yellow	4.9	2.20	0.22
Orange	16.3	1.94	0.24
Red	3.5	1.80	0.07
Detection	3.1	1.36	0.07

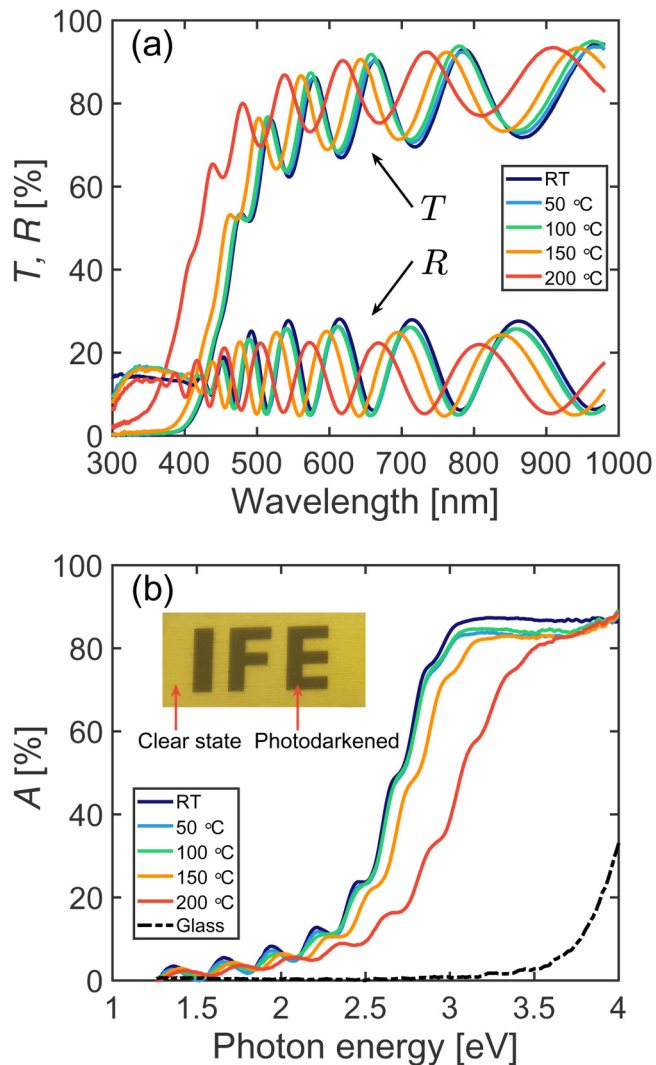


FIG. 2. (a) Transmission, T , and reflection, R , data obtained from as-deposited (RT) and annealed YHO thin films. The YHO samples were annealed at four different temperatures of 50, 100, 150, and 200 °C for 30 min on a hotplate under ambient conditions. (b) Absorption, A , data obtained from the same set of YHO samples, including the glass substrate. The inset photograph displays a YHO thin film in the yellow transparent state without light absorption and the photodarkened state visualized by our institute logo, IFE, which has been obtained after illuminating the film through a shadow mask.

In this work, the indirect optical bandgap energy, as determined by a Tauc plot²¹ for indirect allowed transitions, was found to increase from 2.39 eV in the as-deposited YHO to 2.57 eV in the YHO sample annealed at 200 °C. Fitting our data for direct allowed transitions,²¹ the direct bandgap was found to be approximately 0.5–0.6 eV larger than the indirect bandgap. The absorption spectra in Fig. 2(b) clearly show that the light absorption reduced considerably at the photon energy above ~ 2.5 eV when the annealing

temperature exceeded 100 °C. Note that almost no light absorption occurred in the glass substrate for the photon energy below ~ 3.2 eV. The inset photograph in Fig. 2(b) displays a representative YHO thin film in the yellow transparent state before illumination and the photodarkened state visualized by our institute logo, IFE, which has been obtained after illuminating the film through a shadow mask.

For smart-window applications under outdoor, realistic conditions, photochromic YHO materials are irradiated by sunlight, which consists of photons with a wide range of wavelengths and energy. The photochromic windows are also subjected to varying light intensities and ambient temperatures depending on the weather condition. Thus, it is important to study the photochromic performance of YHO under different illumination conditions and temperatures in a controllable manner. Figure 3(a) presents time-resolved optical transmission, $T(t)$, as a function of illumination time, t , for YHO thin films using various illumination sources: UV, blue, green, yellow, orange, and red LEDs. Note that the LEDs were chosen to operate at their maximum intensity in order to achieve largest possible photochromic contrast. The transmission data show that the contribution of the red and orange lights to the photochromic performance of YHO is negligible, whereas the effect of UV, blue, green, and yellow lights is substantial. Figure 3(c) displays $T(t)$ obtained from YHO thin films illuminated by UV LED operated at four intensities ranging from 0.5 to 5.1 mW cm⁻². Moreover, Fig. 3(e) shows $T(t)$ for YHO thin films annealed at 50, 100, 150, and 200 °C for 30 min on a hotplate in darkness under ambient conditions. Figures 3(b), 3(d), and 3(f) show time-resolved photochromic contrast, $\Delta T(t)$, as a function of illumination time obtained under the same experimental conditions as described in Figs. 3(a), 3(c), and 3(e), respectively. $\Delta T(t)$ is defined as the difference between the average transmission values in the spectral range of 900–920 nm measured before (T_0) and after illumination at time t (i.e., $T(t)$), and normalized with T_0 : $\Delta T(t) = [T_0 - T(t)]/T_0$. As shown by the dotted lines in Figs. 3(b), 3(d) and 3(f), the changes in $\Delta T(t)$ with increasing illumination time can be fitted by a logarithmic function with the following expression:

$$\Delta T(t) = C \ln\left(\frac{t}{\tau_0}\right), \quad (1)$$

where the coloration parameter, C (i.e., the slope), can be viewed as a measure of the strength of photodarkening and τ_0 is the characteristic time. C and τ_0 were determined by using the non-linear least squares method with the Levenberg–Marquardt algorithm in MATLAB. The goodness-of-fit was evaluated in terms of the coefficient of determination, R^2 . Table II displays the fitting parameters extracted from the logarithmic function for different photodarkening conditions. R^2 values show that the fitting accuracy is best when high intensity UV or blue illumination sources were employed. It should be pointed out that the accuracy of the fitting is relatively poor for the experimental data acquired at the beginning of the photodarkening process (typically $t < 30$ s, which depends on the illumination condition). However, after illuminating YHO thin films for more than ~ 30 s, it can be seen from Figs. 3(b), 3(d), and 3(f) that the photochromic contrast increased

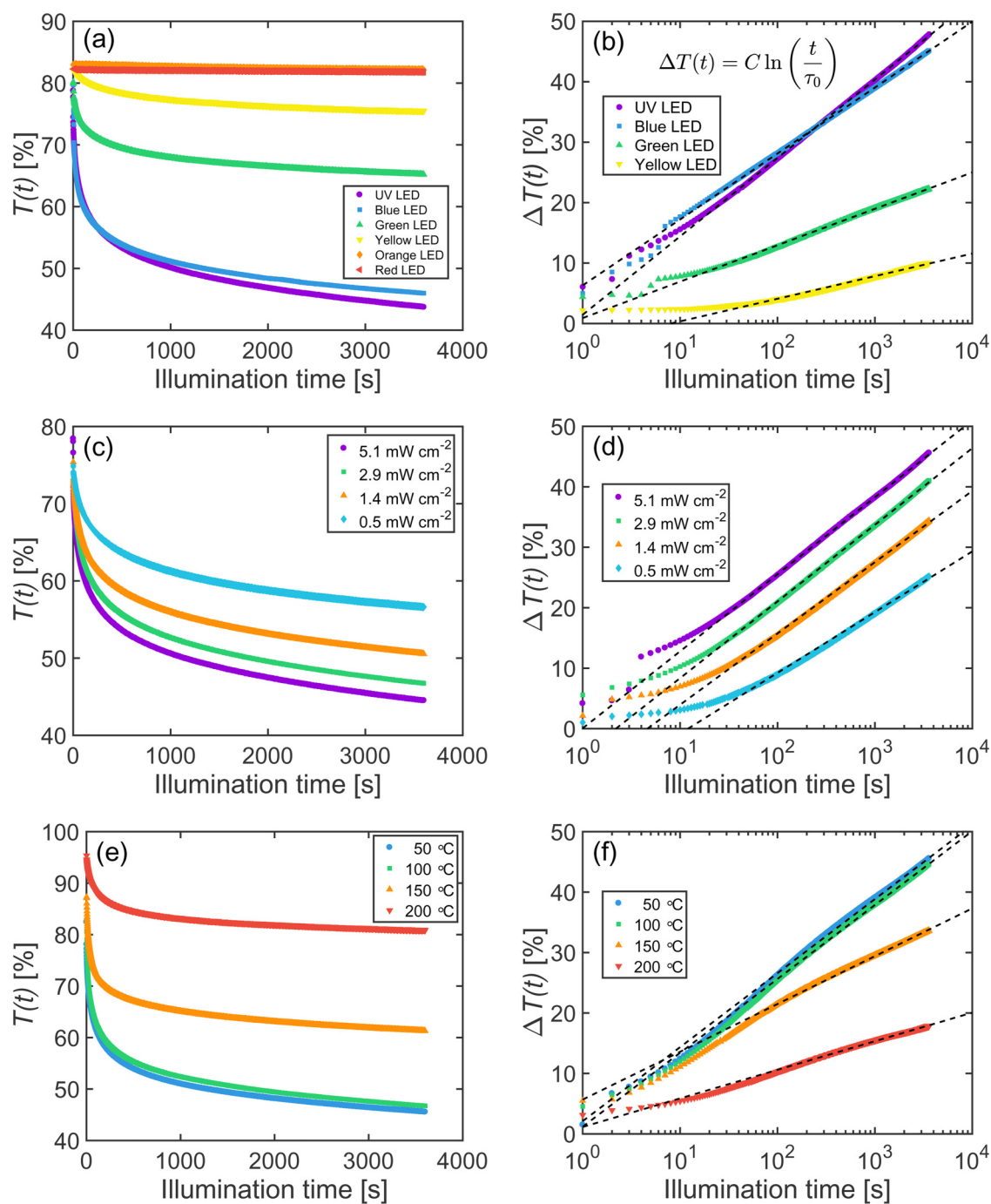


FIG. 3. (a) Time-resolved optical transmission, $T(t)$, plotted as a function of illumination time, t , for YHO thin films using various illumination sources: UV, blue, green, yellow, orange, and red LEDs. (c) $T(t)$ plotted as a function of illumination time for YHO thin films using UV LED operated at four different intensities ranging from 0.5 to 5.1 mW cm^{-2} . (e) $T(t)$ plotted as a function of illumination time for YHO thin films annealed at 50, 100, 150, and 200 $^{\circ}\text{C}$ for 30 min on a hotplate under ambient conditions. (b), (d), and (f) Time-resolved photochromic contrast, $\Delta T(t)$, plotted as a function of illumination time obtained under the same experimental conditions as described in (a), (c), and (e), respectively. $\Delta T(t)$ is defined as the difference between the average transmission values in the spectral range of 900–920 nm measured before (T_0) and after illumination at time t [i.e., $T(t)$] and normalized with T_0 : $\Delta T(t) = [T_0 - T(t)]/T_0$. The changes in $\Delta T(t)$ after an illumination time of ~ 10 –30 s can be fitted by a logarithmic function with the following expression: $\Delta T(t) = C \ln(t/\tau_0)$. The fits to the experimental data are shown by the dotted lines.

TABLE II. Fitting parameters extracted from the logarithmic function used to fit the photodarkening data obtained at different annealing temperatures and illumination conditions.

Parameter	C (%)	τ_0 (s)	R^2
<i>Photon energy (eV)</i>			
3.14	5.6324	0.7730	0.9990
2.80	4.7370	0.2639	0.9996
2.34	2.6161	0.7074	0.9977
2.20	1.6238	8.0242	0.9772
<i>Intensity (mW cm⁻²)</i>			
5.1	5.5306	0.9889	0.9987
2.9	5.5056	2.1910	0.9969
1.4	5.1189	4.6144	0.9942
0.5	4.3619	12.053	0.9877
<i>Temperature (°C)</i>			
50	5.3188	0.6734	0.9974
100	5.3102	0.8001	0.9983
150	3.4343	0.1916	0.9940
200	2.0541	0.5735	0.9947

in a nearly logarithmic fashion with increasing illumination time. In general, the logarithmic function is able to predict the main features of the evolution of photodarkening in YHO thin films, in which the coloration is rapid in the beginning of light exposure, followed by slow coloration with increasing illumination time.

Figure 4(a) shows photochromic contrasts, $\Delta T(60)$ and $\Delta T(3600)$, obtained after illumination times of 60 s and 3600 s, respectively, plotted as a function of the photon energy of various LED illumination sources. The coloration parameter, C , is also shown. Three interesting features can be seen: (i) The photochromic contrast is enhanced with increasing photon energy despite the fact that the LEDs were operated at different maximum intensities, as displayed in Table I. For instance, the intensity of orange LED was more than three times stronger as compared to the UV and yellow LEDs, but the resulting photochromic contrast acquired after 1 h illumination was found to be very small, i.e., $\Delta T(3600) \approx 1\%$, which is close to the measurement uncertainty originating from the thermal instability of the detector. Hence, this finding suggests that for the photon energy below ~ 2 eV, the photochromic effect was either not triggered in YHO thin films due to weak light absorption as indicated in Fig. 2(b) or the photodarkening was not strong enough to compete with thermal bleaching. (ii) $\Delta T(60)$ was nearly half of $\Delta T(3600)$. This optical characteristic might be relevant for technological applications where a high photochromic contrast accompanied by a relatively fast switching speed is desirable. (iii) The coloration parameter was found to increase with increasing photon energy as well.

The intensity of illumination also plays a vital role for the photodarkening process in YHO thin films. As shown in Fig. 4(b), the general trend is that both photochromic contrasts, $\Delta T(60)$ and $\Delta T(3600)$, and the coloration parameter decreased when the intensity of UV LED dropped. Obviously, the photochromic contrast was strongest at the maximum intensity of 5.1 mW cm^{-2} , in which $\Delta T(3600) = 46\%$. Interestingly, it appears that YHO thin films are

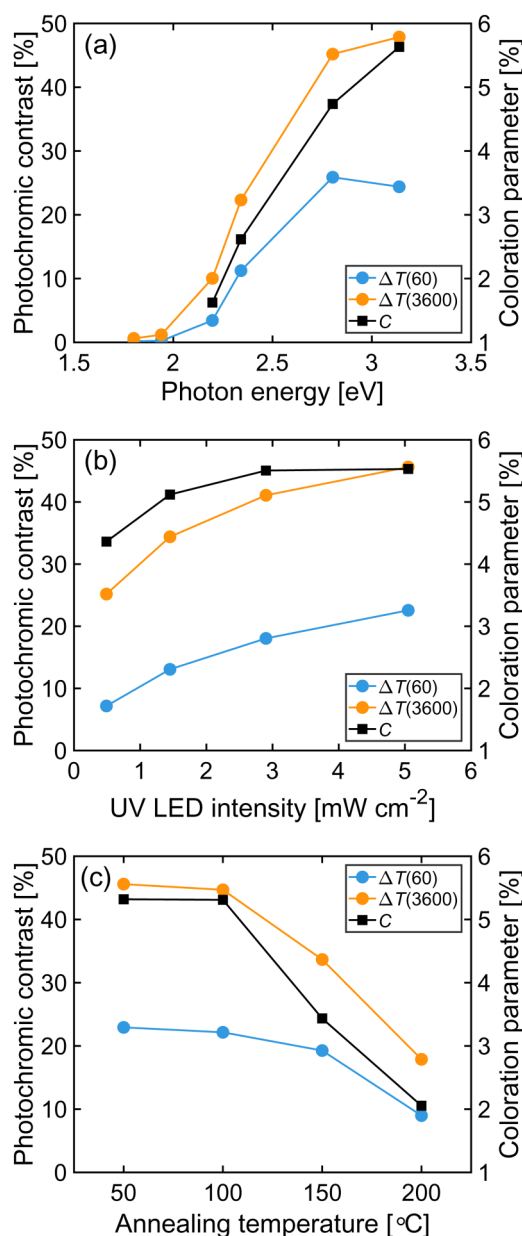


FIG. 4. Photochromic contrasts, $\Delta T(60)$ and $\Delta T(3600)$, obtained after illumination times of 60 s and 3600 s, respectively, plotted as a function of photon energy of the LED illumination sources (a), intensity of the UV LED (b), and annealing temperature for YHO samples (c). The coloration parameter, C , as determined from Fig. 3, is also shown. The lines serve as a guide to the eye.

highly reactive even at a relatively low illumination intensity. For instance, at an intensity of 0.5 mW cm^{-2} , which is about ten times weaker than the maximum intensity, $\Delta T(3600)$ was found to be 25%. This amounts to more than half of the largest photochromic contrast obtained after 1 h illumination.

In order to exploit YHO materials in device applications, it is crucial to investigate the possible degradation in photochromic performance at elevated temperatures. Figure 4(c) presents photochromic contrasts, $\Delta T(60)$ and $\Delta T(3600)$, and the coloration parameter for YHO samples annealed in the temperature range of 50–200 °C. As can be seen, the photochromic contrasts and the coloration parameter were found to decrease considerably when the annealing temperature raised above 100 °C, while no noticeable changes have been observed below 100 °C. However, it should be pointed out that our YHO samples were annealed prior to illumination. It has previously been shown that when YHO is subjected to simultaneous illumination and heating, the photo-darkening is found to be increasingly suppressed as the heating temperature increases from 22 to 84 °C.²² Such temperature-dependency of the photochromic effect is well known for photochromic lenses where these devices are not able to achieve true darkness as experienced in sunglasses in a very hot weather condition. The degradation in photochromic contrast observed at annealing temperatures above 100 °C can be mainly attributed to a considerable reduction in optical absorption in annealed samples due to an increased oxygen concentration, as evidently shown by the absorption spectra in Fig. 2(b). As can be seen, at a UV LED photon energy of 3.14 eV, the optical absorption reduced from 85% in the as-deposited YHO sample at RT to 56% in a sample annealed at 200 °C. Consequently, in order to overcome such a detrimental heat-induced effect, an appropriate cooling scheme might be necessary for high-temperature applications of photochromic YHO materials.

IV. CONCLUSIONS

We have prepared yellow, transparent, photochromic YHO thin films on glass substrates by using reactive magnetron sputtering. We investigated the kinetics of photodarkening in YHO thin films. Time-resolved optical transmission data show a nearly logarithmic time dependence of the coloration of YHO thin films, indicating rapid coloration in the beginning of light exposure, followed by slow coloration with increasing illumination time. The photochromic performance of both as-deposited and annealed YHO thin films was found to be strongly dependent on the photon energy and the intensity of illumination. Both the photochromic contrast and the coloration decreased considerably with increasing annealing temperature and with decreasing intensity and photon energy of various LEDs used to illuminate the YHO samples. The present findings are useful for identifying both advantages and limitations associated with the implementation of photochromic YHO materials in potential technical devices, such as energy-saving smart windows and optical sensors.

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DATA AVAILABILITY

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

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