

Article

Substrate Holder Material-Driven Microstructure Evolution and Hydrogenation Behavior of Pd/Mg Thin Films Prepared by Magnetron Sputtering

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Abstract

Mg-based thin films are promising candidates for hydrogen-responsive optical devices. However, their performance is strongly influenced by microstructural evolution during deposition. In this work, Mg thin films were deposited onto glass substrates placed on different substrate-holder materials (Si and 304 stainless steel) to investigate the influence of substrate-holder configuration on microstructure formation. Fluorocarbon (FC)/Pd/Mg multilayer films were subsequently fabricated to evaluate hydrogenation and dehydrogenation behaviors. The results show that the substrate-holder material significantly affects film morphology and hydrogenation performance. Mg films prepared using the Si holder exhibit relatively uniform hexagonal-like surface morphologies, whereas those prepared using the stainless-steel holder show a transition from granular to hexagonal-like morphologies with increasing sputtering power. Hydrogenation measurements reveal that FC/Pd/Mg films prepared using the stainless-steel holder exhibit superior performance, including a reflectance modulation of approximately 70%, a transmittance modulation exceeding 40%, and a hydrogenation time of about 30 s. In contrast, films prepared using the Si holder show reduced optical modulation and slower hydrogenation kinetics. The observed differences in hydrogenation behavior are closely correlated with variations in film microstructure induced by different substrate-holder configurations. The results suggest that substrate-holder-dependent growth conditions may influence defect formation and hydrogen diffusion pathways in Mg-based thin films. This study highlights the importance of substrate-holder configuration as a processing parameter affecting microstructure evolution and hydrogen-responsive performance in FC/Pd/Mg multilayer films.



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Keywords: Mg thin films; Pd/Mg multilayers; hydrogenation kinetics; substrate microstructure evolution

1. Introduction

Magnesium-based thin films have attracted extensive attention as promising hydrogen-responsive functional materials due to their low density, high hydrogen storage capacity [1], and strong optical contrast upon hydrogen absorption [2–4]. In particular, Pd-catalyzed

Mg multilayer systems have been widely investigated for applications in smart windows, optical sensors, and hydrogen detection devices, where reversible switching between metallic Mg and insulating MgH_2 enables large changes in optical transmittance and reflectance [5–7]. However, despite considerable progress, the practical application of Mg thin films is still limited by sluggish hydrogen absorption/desorption kinetics at room temperature, which mainly originate from the high activation barrier for hydrogen dissociation and transport [8,9].

To address these limitations, significant efforts have been devoted to optimizing catalytic Pd layer thickness, improving interface engineering, and controlling deposition parameters [10–12]. It is well established that the microstructure of Mg films—such as grain size, defect density, and crystallinity—plays a crucial role in determining hydrogen absorption and diffusion behavior [13–20]. In magnetron-sputtered films, these structural characteristics are highly sensitive to the deposition environment, including sputtering power, substrate temperature, and thermal conditions during growth [21,22]. Among these factors, the thermal properties of the substrate system can significantly influence adatom mobility and nucleation processes [23,24], thereby governing the resulting film morphology.

During magnetron sputtering, the local growth environment near the substrate surface can influence adatom mobility, nucleation density, and grain coalescence behavior. Variations in substrate-holder configuration may affect interfacial growth conditions and microstructural evolution, leading to differences in defect formation, grain boundary density, and surface morphology of Mg films. Since hydrogen transport in Mg-based thin films is highly sensitive to microstructural defects and grain boundaries [14], such microstructural variations may subsequently influence hydrogen absorption kinetics and optical switching performance.

Although the influence of substrate temperature has been widely discussed [25,26], the role of substrate holder material—particularly its substrate-dependent growth conditions and interfacial effects—in regulating microstructure evolution has received far less attention. In practical sputtering systems, heat dissipation during film growth is not only determined by the substrate itself but also strongly affected by the substrate holder, which can alter the local thermal environment at the film growth interface. This variation may lead to significant differences in grain growth behavior, defect formation, and ultimately hydrogen transport pathways in Mg-based multilayers.

In this work, we systematically investigate the effect of substrate holder materials (Si and 304 stainless steel) on the microstructure evolution and hydrogenation behavior of Pd/Mg thin films prepared by magnetron sputtering. By combining structural characterization with hydrogenation–dehydrogenation optical measurements, we reveal the critical role of substrate-dependent microstructure regulation in determining film morphology and hydrogen transport efficiency. Our results demonstrate that substrate holder material is a key but often overlooked parameter that governs hydrogen-responsive performance in Mg-based multilayer systems, providing new insight for the rational design of high-performance hydrogen optical devices.

2. Experimental Procedure

Si (100) wafers and 304 stainless-steel plates were employed as substrate-holder materials during film deposition. Prior to sputtering, glass substrates were sequentially ultrasonically cleaned in acetone, ethanol, and deionized water, followed by drying under a nitrogen flow.

Pd/Mg multilayer films were deposited using a Denton Explore-14 magnetron sputtering system (Moorestown, NJ, USA). Before deposition, the chamber was evacuated to a base pressure of approximately 7.1×10^{-4} Pa, and all targets were pre-sputtered for 10 min to remove surface contaminants. High-purity Mg and Pd targets (99.99 at%) were used throughout the study. During deposition, Ar was introduced as the sputtering gas, and the working pressure was maintained at approximately XX Pa. The target-to-substrate distance was fixed at XX mm. All depositions were performed without intentional substrate heating, and the substrate temperature remained close to room temperature.

A 70 nm Mg layer was first deposited onto glass substrates by DC magnetron sputtering at powers of 40, 150, and 300 W. For the hydrogenation study, FC/Pd/Mg multilayer films deposited at 40 W were selected for further investigation. The Mg deposition rate at 40 W was 0.285 nm s^{-1} . Subsequently, a 5 nm Pd layer was deposited by RF magnetron sputtering at 100 W with a deposition rate of 0.297 nm s^{-1} . The Pd layer served both as a catalytic layer for hydrogen dissociation and as a protective layer against Mg oxidation.

To further improve environmental stability, a 30 nm fluorocarbon (FC) layer was deposited on top of the Pd/Mg structure. The FC coating acts as a hydrophobic barrier that suppresses moisture-induced degradation while preserving reversible hydrogen-induced optical switching behavior, as reported previously [27]. To ensure a fair comparison, identical FC deposition conditions were applied to all samples.

The resulting FC/Pd/Mg multilayer films were characterized using a KLA-Tencor P7 profilometer (Milpitas, CA, USA) for thickness measurements and a Regulus 8100 scanning electron microscope (SEM) (Hitachi High-Tech Corporation, Tokyo, Japan) for surface morphology observations. Optical transmittance and reflectance spectra were measured using a Hitachi UH-4150 UV-Vis-NIR spectrophotometer (Tokyo, Japan) in the wavelength range of 300–2500 nm. For hydrogenation/dehydrogenation measurements, the optical response was continuously monitored in situ at a characteristic wavelength of 980 nm, where a pronounced optical contrast was observed during hydrogen-induced switching. All transmittance and reflectance measurements were performed on the glass-supported films after deposition. It should be noted that the Si wafer and stainless-steel plate functioned solely as substrate holders during sputtering and were not part of the optical measurement configuration.

Hydrogenation experiments were carried out by exposing the samples to a flowing gas mixture containing 4% H_2 in Ar (200 sccm, 0.1 MPa), whereas dehydrogenation was performed in flowing air (300 sccm, 0.1 MPa) at room temperature ($\sim 25^\circ\text{C}$). Optical transmittance and reflectance were monitored in situ throughout the hydrogenation/dehydrogenation cycles. The switching time was defined as the time required for the optical signal to reach 90% of its total modulation range.

3. Results and Discussion

3.1. Effect of Substrate Holder Material and Sputtering Power on Mg Microstructure

Mg thin films were deposited by DC magnetron sputtering under different powers (40, 150, and 300 W) using different substrate holder materials (Figure 1), including Si and 304 stainless steel. The surface morphologies were characterized by SEM (Figure 2). For Mg films deposited on Si substrates, a relatively uniform hexagonal-like morphology is observed at all sputtering powers. With increasing sputtering power, the hexagonal features become more pronounced, indicating enhanced crystallization and grain growth.

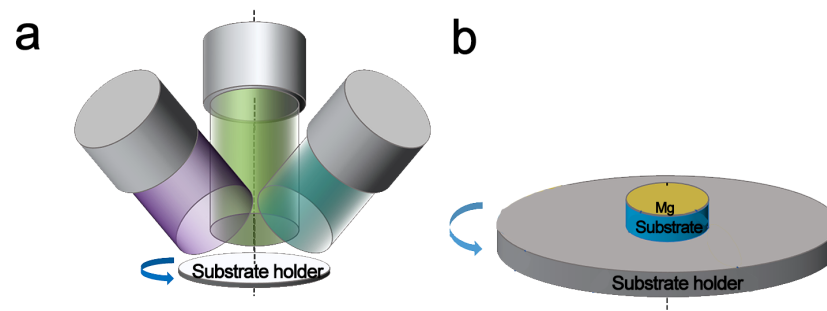


Figure 1. (a) Magnetron sputtering system; (b) location of the substrate holder.

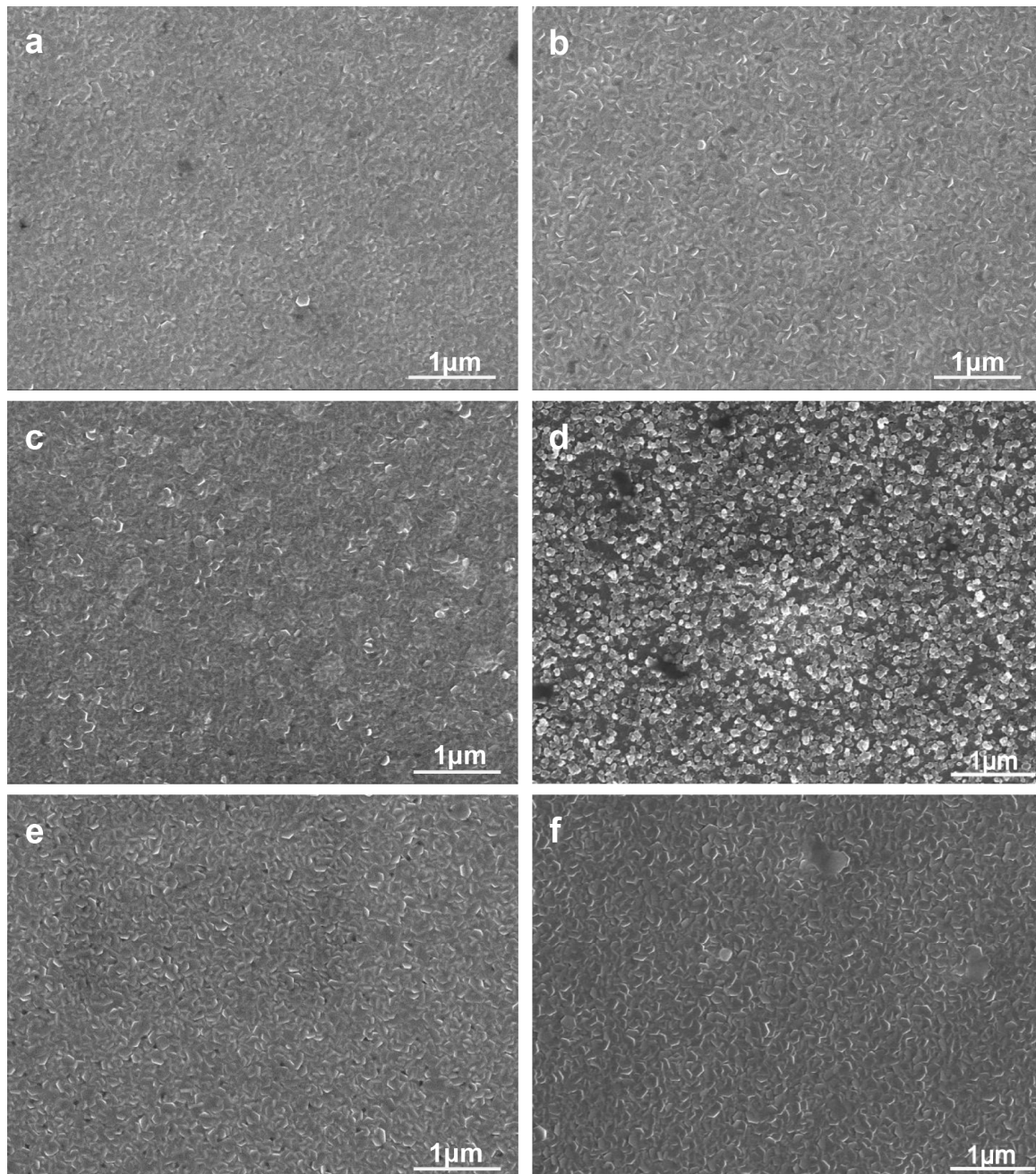


Figure 2. Surface morphologies of Mg thin films deposited under different sputtering powers and substrate holder material onto glass substrates. Mg films deposited on Si substrates holder at (a) 40 W, (b) 150 W, and (c) 300 W. Mg films deposited on stainless steel substrates holder at (d) 40 W, (e) 150 W, and (f) 300 W.

In contrast, Mg films deposited on stainless steel substrate holders exhibit a strong dependence on sputtering power. At 40 W, a granular morphology with poorly defined grain boundaries is observed. Such microstructures generally contain a high density of structural defects and disordered grain boundary regions, which can provide additional diffusion pathways for hydrogen [14]. At 150 W, a transition from granular to mixed granular/hexagonal-like surface morphology occurs. At 300 W, a well-developed hexagonal-like surface morphology dominates the film surface. Similar hexagonal grain features have been frequently reported in sputtered Mg thin films and are generally associated with the preferential growth behavior of hcp Mg crystals under suitable deposition conditions [14]. However, it should be noted that the present SEM observations primarily reveal surface morphology rather than crystallographic structure, and therefore the term “hexagonal-like morphology” is used in this work.

These results demonstrate that substrate holder material significantly influences Mg film growth behavior. The observed morphology evolution can be interpreted using the structure zone model (SZM) for sputtered films [21]. The observed morphology differences are likely associated with substrate-dependent growth conditions [14]. It should be noted that the present SEM observations provide primarily qualitative information on morphology evolution. More comprehensive quantitative analyses, including grain-size statistics and crystallographic characterization, will be required in future work to establish a more rigorous relationship between microstructure and hydrogen transport behavior.

3.2. Hydrogenation Behavior of Pd/Mg Films on Different Substrate Holder Materials

To evaluate the effect of substrate holder material on hydrogenation performance, FC/Pd/Mg multilayer films deposited at 40 W were investigated on Si and stainless steel substrates. For stainless steel substrates, the films exhibit excellent hydrogen-responsive optical properties (Figure 3). The reflectance modulation reaches approximately 70%, and the transmittance modulation exceeds 40%. The hydrogenation process completes within ~30 s, indicating fast hydrogen uptake kinetics. After six hydrogenation/dehydrogenation cycles, the optical modulation decreases by approximately 30%, indicating that degradation may occur during repeated hydrogen cycling. However, the limited number of cycles investigated in the present study does not allow a comprehensive assessment of long-term cycling stability. Although the present work mainly focuses on hydrogenation kinetics, the observed degradation after repeated cycling also suggests that microstructure-dependent stress accumulation may influence long-term durability. Further optimization of interfacial engineering and defect regulation is therefore necessary.

In contrast, films deposited on Si substrates show significantly degraded hydrogenation performance. The optical modulation is strongly suppressed, with reflectance changes of only ~10% at 980 nm (Figure 4), and the transmittance conversion range is strongly suppressed (Figure 5). In addition, as shown in Figure 6, hydrogenation kinetics are significantly slowed, and complete switching behavior is not achieved. The hydrogenation time increases to nearly 100 s, indicating a significant slow down in hydrogen uptake kinetics.

These results confirm that substrate holder material plays a decisive role in hydrogen transport efficiency and optical switching performance. Grain boundaries and structural defects in Mg films can act as preferential diffusion pathways for hydrogen atoms. Therefore, the defect-rich microstructure formed on stainless steel substrates is beneficial for accelerating hydrogen diffusion and improving hydrogenation kinetics. Although formal statistical analysis was not performed in the present study, the differences observed between samples prepared using different substrate-holder configurations were sufficiently large to allow clear comparison of their hydrogenation behavior and optical response.

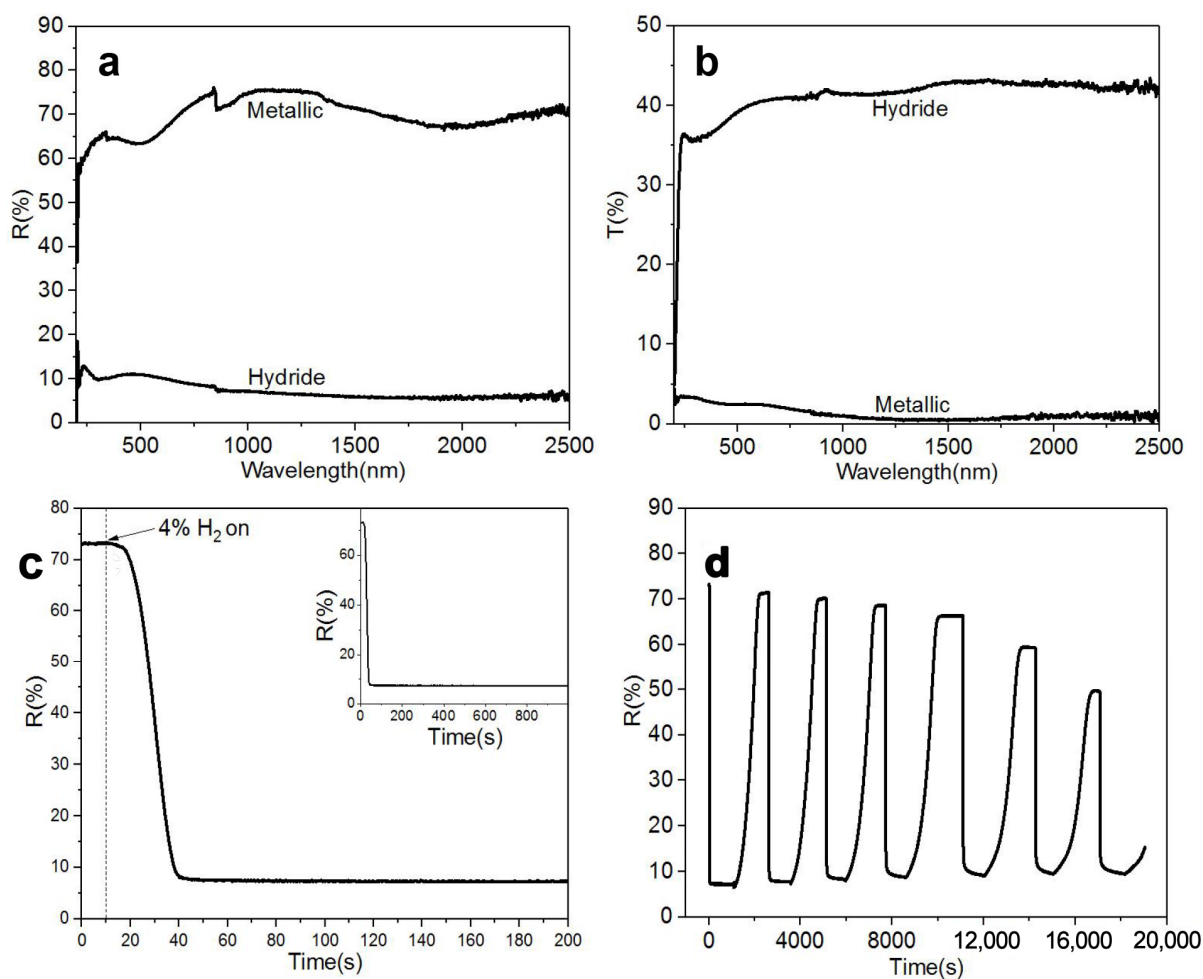


Figure 3. Optical switching performance of FC/Pd/Mg multilayer films deposited using a stainless-steel substrate holder. (a) Reflectance spectra in the hydrogenated and dehydrogenated states; (b) Transmittance spectra in the hydrogenated and dehydrogenated states; (c) Hydrogenation properties monitored at 980 nm; (d) Cycling durability evaluated by repeated hydrogenation/dehydrogenation measurements at 980 nm.

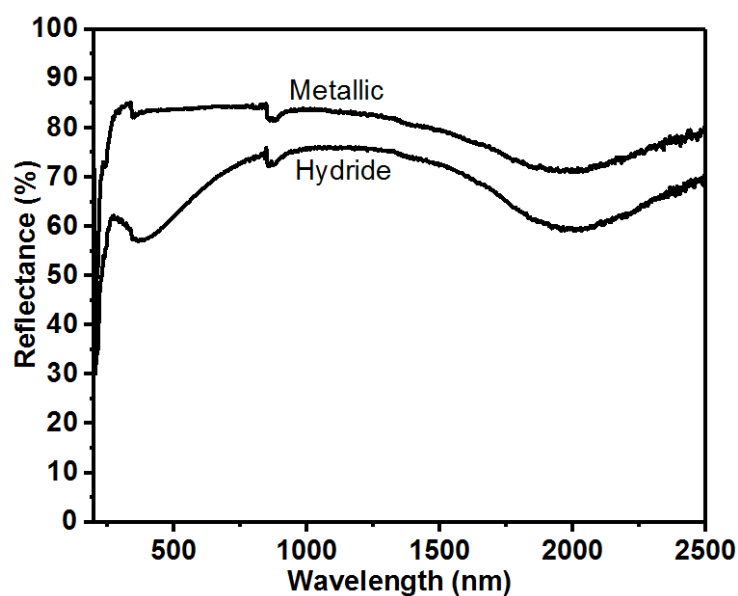


Figure 4. Time-dependent reflectance variation at 980 nm during hydrogenation and dehydrogenation of FC/Pd/Mg multilayer films deposited using a Si substrate holder.

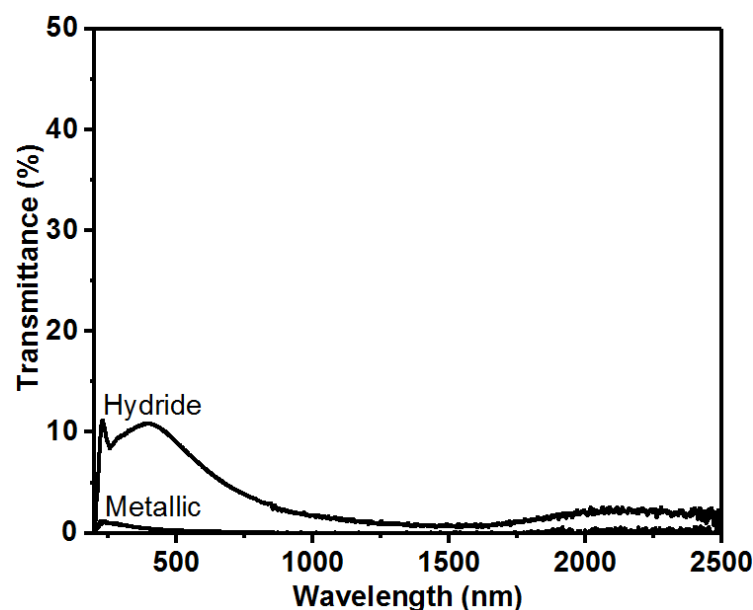


Figure 5. Time-dependent transmittance variation at 980 nm during hydrogenation and dehydrogenation of FC/Pd/Mg multilayer films deposited using a Si substrate holder.

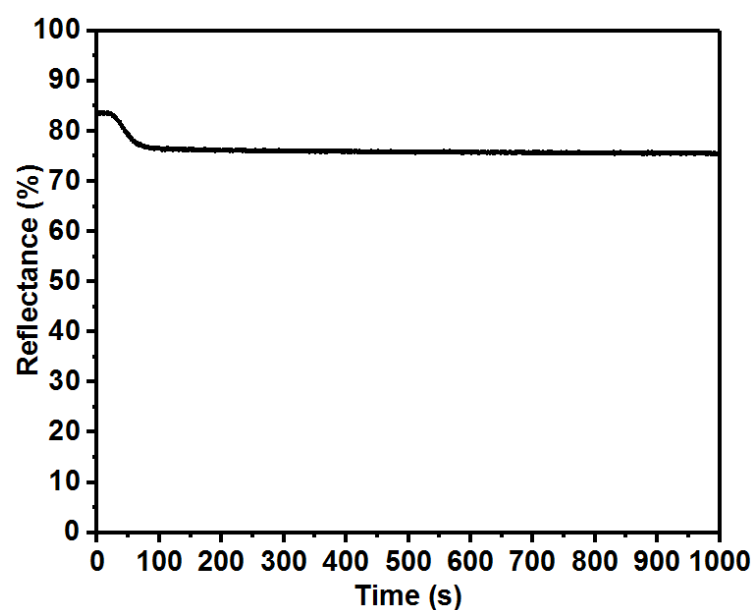


Figure 6. Hydrogenation kinetics of FC/Pd/Mg multilayer films deposited using a Si substrate holder, showing the temporal evolution of optical response at 980 nm during hydrogen absorption.

It should also be noted that Mg oxidation may influence hydrogen absorption behavior. However, the Pd catalytic layer and FC protective coating were introduced to minimize environmental degradation. Since all samples were prepared using identical Pd and FC layer configurations, oxidation effects are expected to be comparable among the investigated samples and are therefore unlikely to be the dominant factor responsible for the observed differences in hydrogenation performance.

3.3. Relationship Between Microstructure Evolution and Hydrogen Transport

The hydrogenation performance of FC/Pd/Mg multilayer films is primarily governed by the Mg microstructure, which is strongly influenced by the substrate-holder configuration and the resulting microstructural evolution during deposition (Figure 7). The observed differences in hydrogenation behavior are closely related to the microstructural charac-

teristics of the Mg layer. Films prepared using different substrate-holder configurations exhibit distinct surface morphologies and defect structures, which can influence hydrogen diffusion pathways. The defect-rich microstructure observed in the stainless-steel-holder configuration may provide additional diffusion channels for hydrogen transport, resulting in faster hydrogenation kinetics and enhanced optical modulation. In contrast, the relatively uniform hexagonal-like morphology observed in the Si-holder configuration may reduce the density of effective diffusion pathways, leading to slower hydrogen transport and weaker optical response. These structural features provide abundant hydrogen diffusion pathways, leading to fast hydrogenation kinetics and pronounced optical modulation. In contrast, Si substrates promote a different growth environment characterized by more stable thermal conditions during film formation, resulting in denser and more ordered microstructures with lower defect density. Consequently, hydrogen diffusion is hindered, leading to slower kinetics and weakened optical response.

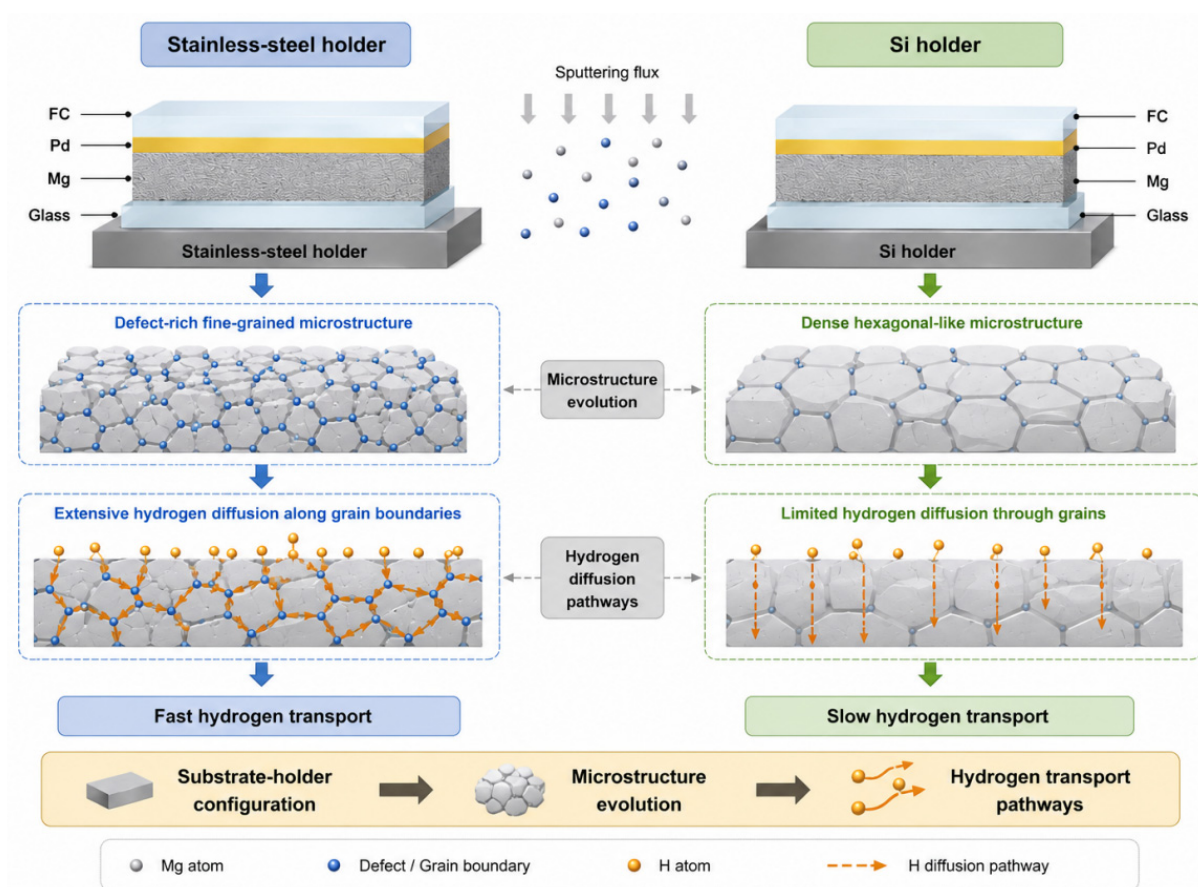


Figure 7. Schematic illustration of the correlation between microstructure evolution and hydrogen transport behavior in FC/Pd/Mg thin films prepared using different substrate-holder configurations.

4. Conclusions

FC/Pd/Mg multilayer films were fabricated by DC magnetron sputtering using different substrate holders (Si and 304 stainless steel) to investigate their influence on microstructure evolution and hydrogenation behavior. The results demonstrate that the substrate holder materials play a dominant role in determining film morphology and hydrogen transport properties. Mg films prepared using stainless-steel substrate holders exhibit relatively defect-rich surface morphologies, whereas films prepared using Si substrate holders show comparatively uniform hexagonal-like morphologies. Accordingly, FC/Pd/Mg films on stainless steel substrates show significantly improved hydrogenation performance, including faster switching (~ 30 s), higher reflectance modulation ($\sim 70\%$), and

stronger transmittance variation (>40%). In contrast, Si-based samples exhibit markedly suppressed hydrogenation kinetics and reduced optical modulation. The present hydrogenation measurements were conducted on films deposited at 40 W; therefore, the direct influence of sputtering power on hydrogenation behavior requires further investigation.

The present findings suggest a close correlation between substrate-holder-dependent microstructure evolution and hydrogen transport behavior in Mg-based thin films. These observations indicate that controlling the deposition environment through substrate-holder configuration may provide a useful approach for tailoring the hydrogen-responsive performance of Mg-based thin-film devices. This strategy may provide a practical route for designing high-performance hydrogen-responsive optical devices and other hydrogen storage-related thin-film systems.

5. Future Perspectives and Potential Applications

The present study shows that different substrate-holder configurations can significantly influence the microstructure and hydrogenation behavior of FC/Pd/Mg thin films. These findings suggest that substrate-holder design may provide an additional approach for tailoring the performance of hydrogen-responsive thin-film devices.

Future work should focus on quantitative characterization of microstructure evolution and its relationship with hydrogen transport behavior. In particular, crystallographic analysis, defect quantification, and in-situ diagnostic techniques will be valuable for clarifying the mechanisms responsible for the observed differences in hydrogenation performance. In addition, hydrogenation measurements on films prepared under different sputtering powers will help establish a more comprehensive understanding of the relationship between deposition conditions, microstructure, and optical switching behavior.

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