Improvement of Some Physical and Sensing Properties of Nanostructured Cadmium Oxide Manganese Thin Films

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Abstract: The spray pyrolysis technique (SPT) was employed for depositing thin films of Mn-doped CdO. The structural analysis through XRD confirms films polycrystallinity with a cubic phase. AFM analysis reveals adecline in particle size and surface roughness. The optical transmittance spectra indicate that the films exhibit high transparency in the visible area. The absorption edge shifting toward longer wavelengths as Mn concentration increases. The band gap reduced from 2.75 eV for CdO to 2.65 eV for 4% Mn-doped CdO. The refractive index exhibits normal and abnormal dispersion regions, decreasing with higher Mn doping. The extinction coefficient follows the trend of the absorption coefficient, increasing with photon energy, confirming direct electronic transitions in the films. CdO:Mn films show p-type behavior; NO_2 exposure (250 ppm) reduces resistance. 4% Mn doping increases resistance by altering charge carrier concentration. As Mn concentration increases, the sensitivity of CdO films to NO_2 decreases. Sensitivity increases with NO_2 concentration due to stronger gas-film interactions and higher conductivity.

1 INTRODUCTION

CdO is an n-type semiconductor [1]-[3]. The behavior of CdO can be changed by doping with substances, such as F, Mn, Mo, and Sn, which enhance electrical conductivity and leading to a blue shift in band gap [4]-[9]. It is well known that doping efficiency depends on the difference in ionic radii between the dopant and the host element. Mn^{2+} ions (0.83 Å) have a smaller ionic radius compared to the larger Cd^{2+} ions (0.95 Å), leading to strong integration into the CdO lattice[10]-[12]. CdO films can be prepared via various techniques, including solution growth, MOCVD, sputtering, SPT, reactive evaporation, CBD, and PLD [13]-[26]. Among these techniques, SP is cost-effective for fabricating thin films with a large surface area. In this paper, SPT is considered an efficient and promising method widely used for depositing CdO films and analyzing their characteristics.

2 EXPERIMENTAL

CdO and CdO:Mn films were grown via (SPT). A 0.1M solution of $Cd[C_4H_6CdO_4]$ were prepared. $MnCl_3$, was dispersed in deionized water, and several drops of HCl were added to assure cleanliness. The optimal deposition parameters were as follows: The glass base was kept at 425°C, the nozzle and base were spaced 28 cm apart, and the spraying was done for 10 seconds, with a 65-second break to impede the cold-chain process. The spray rate was 4 ml/min, and (N_2) is the transporter gas. The film thickness, measured via weighing method, was determined to be 340 ± 25 nm. Structural properties were analyzed using (XRD), surface morphology was examined using (AFM), and transmittance was measured via a double-beam spectrophotometer . The CdO and CdO:Mn gas sensor was assessed by measuring the percentage change in resistance within a cylindrical chamber (radius: 8.2 cm, height: 15.2 cm).

3 RESULT AND DISCUSSION

The XRD patterns of of the intended films are presented in Figure 1, confimed a polycrystalline nature, with distinct peaks observed at 2θ = 33.12°, 38.26°, 55.237°, and 69.28°, corresponding to the (200), (220), and (222) planes of cubic CdO, as per ICDD Card No. 05-0640 [28]. The structural properties are presented in Table 1. Regardless of Mn doping level, the (111) plane showed the most development across all films. With increasing Mn content, the intensity of the (111) plane was found to marginally rise, indicating a shift towards lower angles due to Mn^{2+} substitution in the host lattice and a possible intense peak, indexed as the (111) plane, was utilized to determine the crystallite size (*D*) [30], [31]. Figure 2 presents the X-ray parameters of the extended films.

D was determined via Scherrer's formula [32]:

$$D_{hkl} = \frac{0.9\lambda}{\beta cos\theta}. \quad (1)$$

Where λ =1.541Å, β is FWHM, and θ is Bragg angle. The results indicate that as the Mn concentration increases, *D* increases from 15.43 nm to 17.71 nm. The films exhibit crystallite sizes in the nanometer range, confirming that CdO:Mn films qualify as nanomaterials. This change in crystallite size is attributed due to the variation in ionic radii between the dopant and host material [33, 34].

The formula for lattice microstrain is expressed as [35]:

$$\varepsilon = \frac{\beta cos\theta}{4}\ (lines^{-2}.m^{-1})\,. \quad (2)$$

The formula for dislocation densities is expressed as [36]:

$$\delta = \frac{1}{D^2}\left(\frac{lines}{m^2}\right). \quad (3)$$

As anticipated, a rise in *D* leads to a decline in microstrain (ε) and dislocation densities (δ). The microstrain (ε) was found to range from 2.2×10^{-3} to 2.0×10^{-3}, while the dislocation density (δ) decreased from 48.0×10^{14} to 31.8×10^{14}. T is result agrees well with Kaaviya et al.[37], [38].

Table 1: Structural styles of the extended films.

Specimen	2 θ°	Plane, (hkl)	FWHM, °	E_g, eV	D, nm	ε, $\times10^{14}$	Δ,$\times10^{-4}$
Undoped CdO	33.12	111	0.53	2.75	15.4	48.0	22.1
CdO: 2% Mn	33.09	111	0.50	2.71	16.5	36.3	20.9
CdO: 4% Mn	33.06	111	0.48	2.65	17.7	31.8	20.0

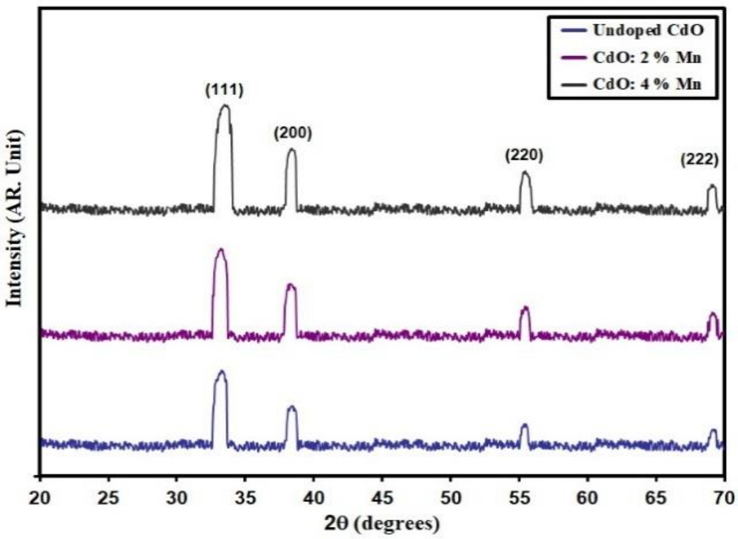

Figure 1: XRD styles of intended films.

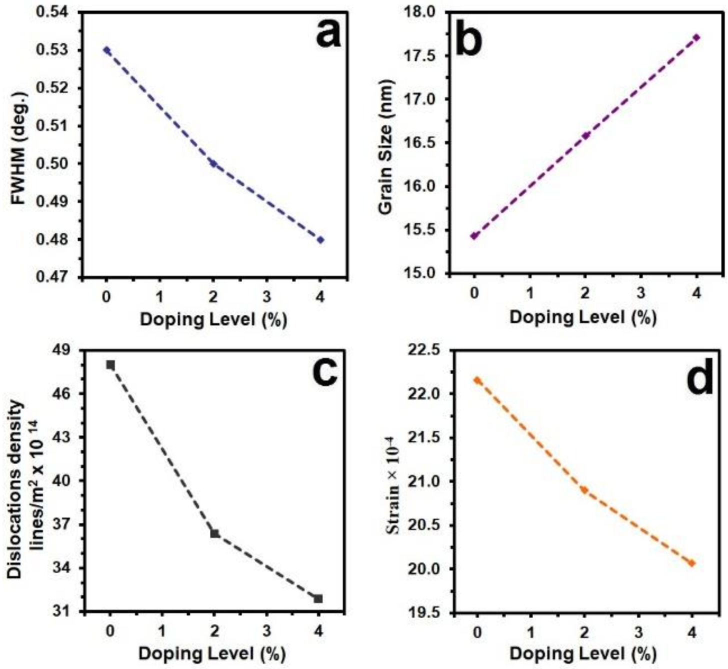

Figure 2: X-ray parameter of entended films.

The surface topography of the extended films was examined using AFM. All CdO thin film images were captured over an area of 0.39 µm × 0.39 µm. Figure 3 presents AFM images of the extended films within the same area. As illustrated in Figures 1a, 1b, and 1c, the CdO, 2% CdO:Mn, and 4% CdO:Mn thin films consist of small crystal grains with a nearly uniform distribution.

According to Table 2, the average particle size (P_{av}) is the highest among all samples at 87.6 nm, whereas the Pav of the 4% Mn-doped film is 32.3 nm. It is also evident that the roughness (R_a) values vary with increasing Mn doping. However, R_a values do not follow a consistent trend due to irregular grain distribution. Notably, the 4% Mn-doped CdO film exhibits the minimum R_a value, which is attributed to the uniform distribution of regularly shaped grains on the film surface. The Ra of the 4% Mn-doped film is 3.32 nm, the lowest among all samples, whereas the CdO thin film has an R_a of 8.62 nm. This finding aligns with previous literature [39], [40].

The AFM results confirm that Mn doping influences the surface quality of CdO films. The obtained AFM parameters (P_{av}) and root mean square (rms) values are summarized in Table 2.

Figure 4 presents transmittance (T) spectra of the entended films. T decreases as the Mn content rises. The films exhibit transparency in the visible region, with transmittance values ranging between 20% and 75% at wavelengths greater than 550 nm. Notably, the maximum T at 600 nm exceeds 65%. T is approximately 60% for undoped CdO and around 65% for films with 2% and 4% Mn doping. Below 550 nm, T sharply decreases due to the strong absorption in this area. The absorption spectra are displayed in Figure 5, as shown CdO films exhibit a distinct absorption edge. Additionally, the absorption edge shifts within the 300-500 nm range with increasing Mn doping, indicating a reduction in the band gap due to Mn incorporation[41]-[44].

The change of optical absorption coefficient (α) of different Mn doping concentrations is presented in Figure 6. The absorption coefficient (α) can be determined from T spectrum using the following (4) [45]:

$$\alpha = 2.303\frac{A}{T}. \tag{4}$$

Where A represents the absorbance, the calculated values α are on the order of 10^4 cm^{-1}. As shown in Figure 6, at higher photon energies α exhibits a high value, indicating a significant probability of allowed direct transitions. As the wavelength increases, α gradually decreases. Additionally, it is observed that A increases with the rise in Mn, with a dramatic rise close to the band gap (Eg) edge. As a result, the absorption edge shifts toward the longer wavelength area [46, 47].

Table 2: AFM parameter measurement of Mn dope.

Samples	P_{av} nm	R_a (nm)	rms (nm)
CdO	87.6	8.62	9.73
CdO: 2% Mn	48.2	8.09	8.23
CdO: 4% Mn	32.3	3.32	4.14

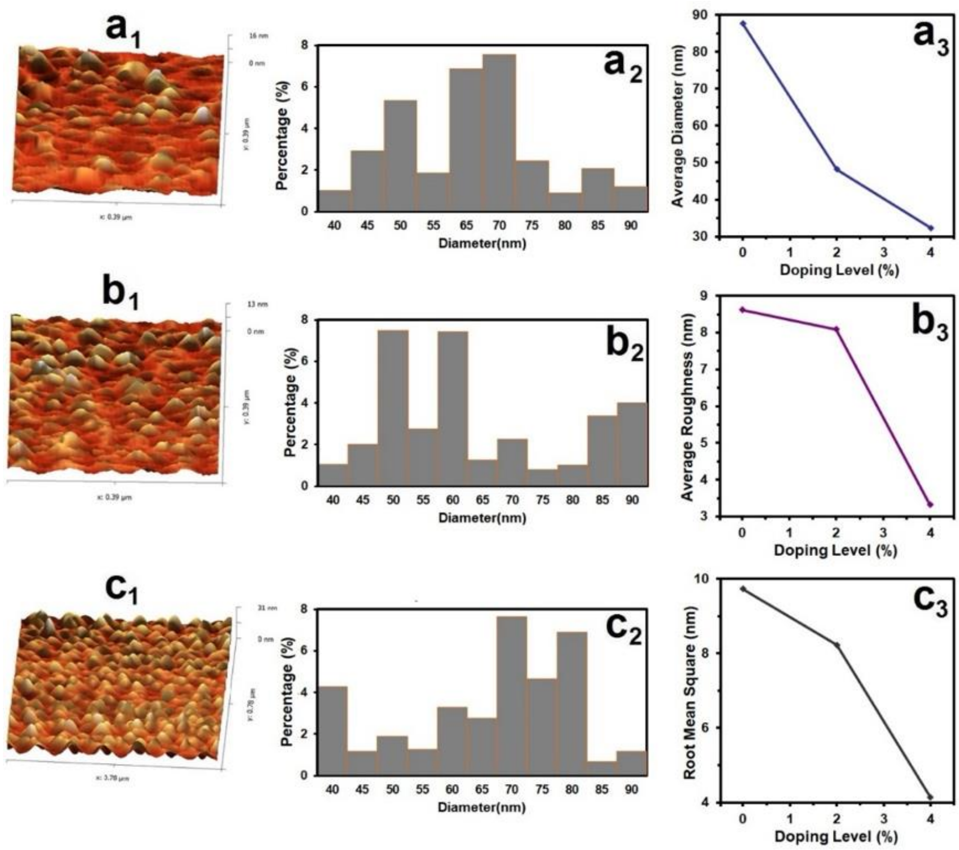

Figure 3: AFM of entended films.

The formula that follows is used to calculate the band gap (Eg). [48]:

$$\alpha h\nu = B(h\nu - E_g)^n. \quad (5)$$

Where B is constant, hν is the photon energy . The parameter n = 1/2 .Figure 7. illustrate E_g showing a value of 2.24 eV for CdO, which aligns well with several reported studies [36]. Mn doping in CdO reduces E_g to lower energy values due to the increased density of localized states within the Eg as the Mn content increases [49,50]. The E_g value were decided to be 2.75 eV, 2.71 eV, and 2.65 eV for undoped, 2 wt.% Mn and 4 wt.% Mn films, respectively, as illustrated in Figure 7.

The following relationship was previously employed to calculate the refractive index (n) via reflectance (R)[51]:

$$R = \frac{(n-1)^2}{(n+1)^2}\ . \quad (6)$$

The following formulas [52] are used to evaluate the extinction coefficient (k):

$$k = \frac{\alpha\lambda}{4\pi}\ . \quad (7)$$

Figure 8. presents n plot , n values of CdO thin film vary with Mn doping. n of CdO film is the highest, while n of the 4wt.% Mn film is the lowest among all the films. This indicates that n can either increase or decrease depending on the Mn doping content [53, 54]. Figure 9 illustrates the variation k. The behavior of (k) closely resembles primarily depends on (α), as described by (4). Consequently, an increase in photon energy leads to a rise in k due to the corresponding increase in α. This indicates that

direct electronic transitions occur in these films [55, 56]. Additionally, before reaching the Eg values, (k) exhibits significant changes with Mn doping, which can be attributed to structural modifications in the films [57],

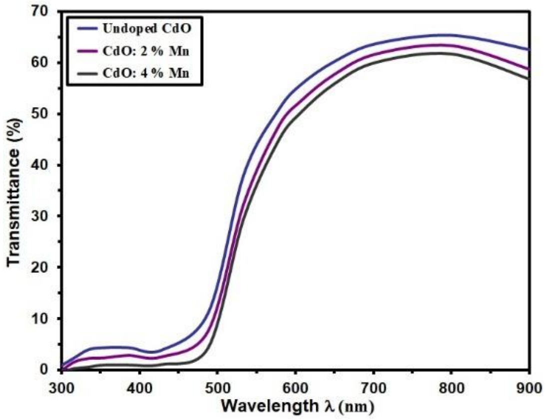

Figure 4: Transmittance (T) of the entended film.

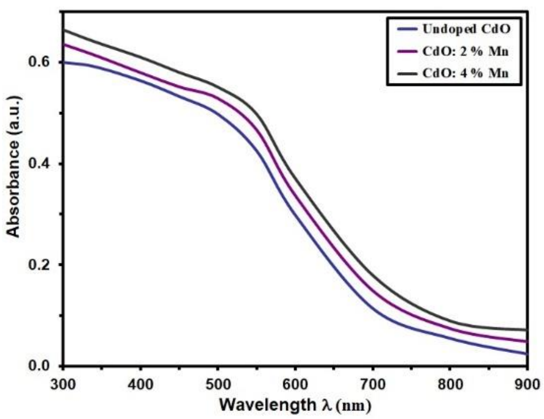

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Figure 5: Absorbance of the grown films.

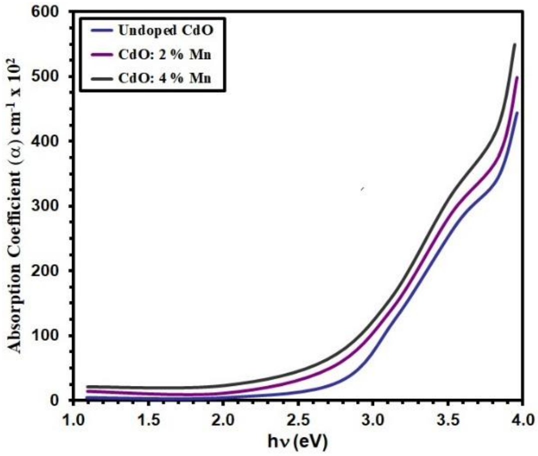

Figure 6: Absorption coefficient (α) of the grown films.

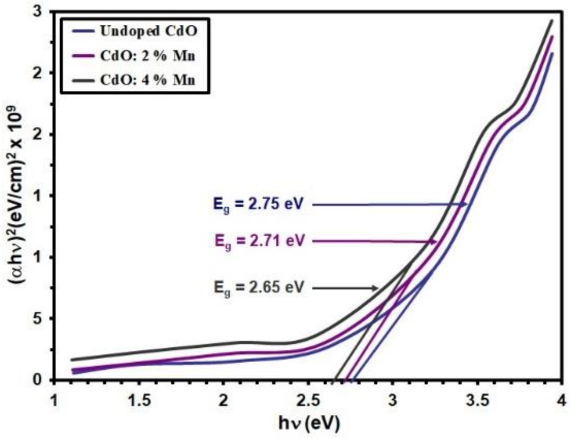

Figure 7: The gap energy (E_g) of the entended films.

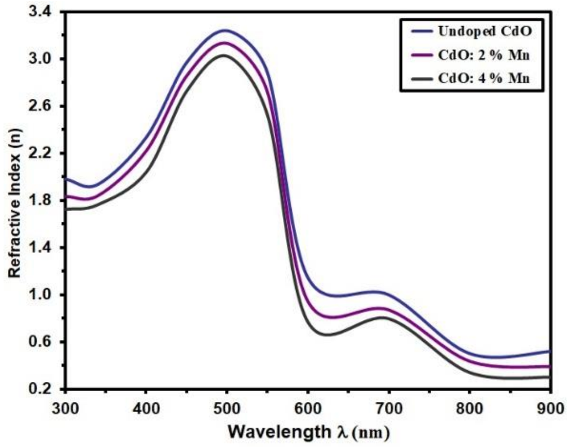

Figure 8: Refractive index (n) of the grown films.

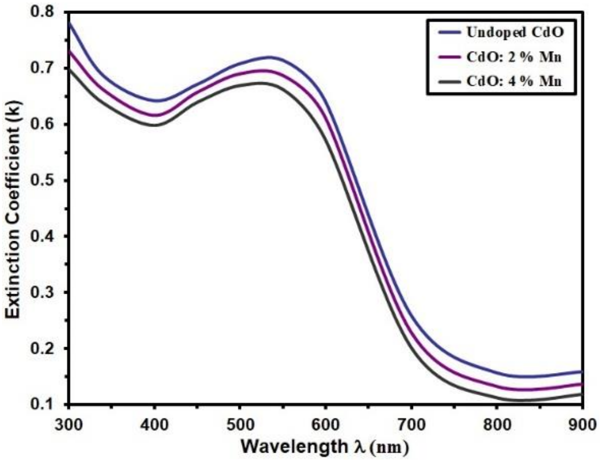

Figure 9: Extinction coefficient (k) of of the entended films.

The resistance variation over response time for deposit films is depicted in Figure 10. The p-type semiconductor behavior of CdO is indicated by the declined in resistance upon insertion of NO2 gas into the chamber.This indicates that oxidizing gases like NO_2 (at a content of 250 ppm) interact with film surface, capturing electrons from CB. As a result, hole concentration increases, leading to enhanced charge carrier density and reduced resistance. The semiconductor features were significantly impact by Mn content, as evidenced by the maximum resistance observed at a 4%wt Mn [59]. This increase in resistance can be ascribed to the alteration of charge carrier concentration and the introduction of defect states caused by Mn incorporation.

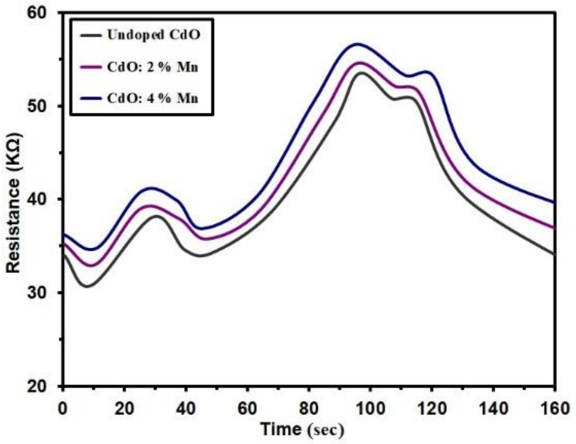

Figure 10: Resistance as a function of operating time for CdO thin films with different Mn concentrations.

The sensor's response can be determined using the following (8) [59]:

$$\text{Sensitivity} = \frac{\Delta R}{R_g} = \left|\frac{R_g - R_a}{R_g}\right| \times 100\ \% . \quad (8)$$

Figure 11 illustrates s of pure and Mn-doped CdO films to NO_2 exposure. As Fe concentration increases, sensitivity declines from 49.9% to 4.5% at 50 ppm, from 40.3% to 4.1% at 200 ppm, and from 48.7% to 11.4% at 250 ppm.This reduction can be ascribed to a larger nanocrystalline size and/or better crystallinity, which maintains the surface uniformity Additionally, the increase in carrier concentration leads to higher conductivity, further influencing sensitivity [60], [61]. Moreover, as shown in Figure 11, sensitivity increases as gas concentration rises from 150 to 250 ppm, suggesting a stronger interaction between the NO_2 molecules and the CdO film at higher contents.

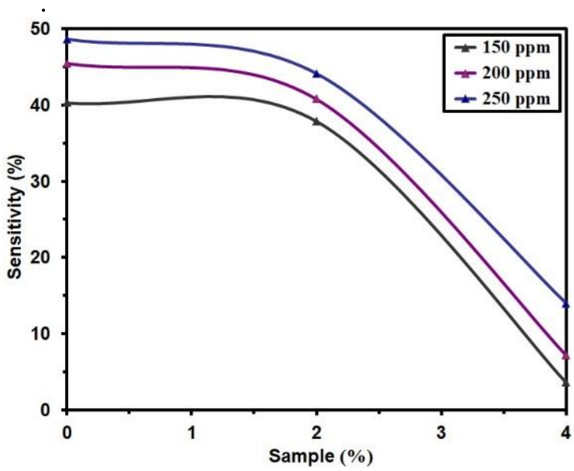

Figure 11: Sensitivity (S) as a function of CdO thin films with different Mn concentrations.

4 CONCLUSIONS

Mn-doped CdO thin films synthesized via the spray pyrolysis technique exhibited notable enhancements in structural, morphological, optical, and gas sensing properties. XRD analysis confirmed a polycrystalline cubic structure, where Mn doping led to increased crystallite size and a reduction in both microstrain and dislocation density, indicating improved crystal quality. AFM images showed that Mn incorporation reduced particle size and surface roughness, with the 4% Mn-doped film displaying the most uniform and smooth surface morphology. Optical characterization revealed high transparency in the visible region for all films, though transmittance slightly decreased with higher Mn content. A redshift in the absorption edge was observed, corresponding to a reduction in the optical band gap from 2.75 eV for undoped CdO to 2.65 eV for 4% Mn-doped CdO. The refractive index decreased with Mn doping.In terms of gas sensing behavior, the films exhibited typical p-type semiconductor characteristics. Exposure to NO_2 gas led to a decrease in resistance due to increased hole concentration. However, higher Mn content caused a rise in baseline resistance and a decline in sensitivity. Nevertheless, the sensitivity improved with increased NO_2 concentration, highlighting stronger gas-film interactions.

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