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The electrochemical reduction of carbon dioxide (CO<sub>2</sub>RR) offers potential for sustainable production and greenhouse gas mitigation, particularly with renewable energy integration. However, its widespread application is hindered by expensive catalysts, low selectivity, and limited current density. This study addresses these challenges by developing a low-mass-loading two-dimensional (2D) Bi<sub>2</sub>O<sub>2</sub>Se catalyst via chemical vapor deposition (CVD). The catalyst achieves a formate faradaic efficiency (FE) of 47.1% with a high current density of 4649 mA mg<sup>-1</sup> at -1.15 V (vs. RHE), significantly outperforming bulk Bi<sub>2</sub>O<sub>2</sub>Se. Pressurizing CO<sub>2</sub>, a condition commonly encountered in industrial processes, further enhances formate selectivity and current density, increasing from 2189 mA mg<sup>-1</sup> at ambient pressure (1.01 bar) to 7457 mA mg<sup>-1</sup> at 40 bar. In situ Raman spectroscopy and DFT calculations reveal the intermediates and pathways involved, underscoring the critical role of pressure in regulating CO<sub>2</sub>RR pathways. These findings highlight the potential of 2D catalysts for sustainable and industrially relevant CO<sub>2</sub> conversion under high pressure.

Production of value-added chemicals *via* renewable-driven electrochemical reduction of  $CO_2$  ( $CO_2RR$ ) holds promise to deal with the pressing global warming and energy crisis.<sup>1–3</sup> To date, the predominant focus of  $CO_2RR$  research lies in examining its performance under ambient pressure conditions, while industrial  $CO_2$  is typically pressurized during capture, transport, and storage. Coincidentally,  $CO_2RR$  benefits from high pressure in aqueous solutions because low  $CO_2$  solubility under ambient pressure usually leads to the formation of unfavorable active carbon species, and subsequently causes diminished current density and reduced selectivity.<sup>4–6</sup> We have reported that higher  $CO_2$  pressure can significantly improve the formate selectivity during aqueous-based  $CO_2RR$  over commonly used catalysts

# Pressure regulated CO<sub>2</sub> electrolysis on two-dimensional Bi<sub>2</sub>O<sub>2</sub>Se<sup>+</sup>

Ruofan Sun,‡ Jiwu Zhao,‡ Hang Liu, 🕩 Yanrong Xue and Xu Lu 吵 \*

such as Cu, Au, Ag and Sn.<sup>7</sup> Cu<sub>2</sub>O<sub>(a</sub>Cu catalysts with a hollow sphere morphology were also found to produce ethanol when operating under pressure.<sup>8</sup> These studies pointed out the important role of high pressure in regulating the  $CO_2RR$  pathways.

Bismuth-based catalysts have garnered considerable interest in the CO<sub>2</sub>RR field because of their high selectivity toward formate, cost effectiveness and low toxicity, rendering them a capable material for large-scale applications.<sup>3,9</sup> Two-dimensional (2D) bismuth materials have aroused increasing attention in light of their high specific surface area, large atomic exposure rate and tunable electronic states.<sup>10</sup> Han et al. showcased the effectiveness of ultrathin BiNS catalysts, achieving superior formate selectivity (>90% FE), current density (24 mA cm<sup>-2</sup>), and durability.<sup>11</sup> Peng et al. synthesized ultrathin bismuth nanosheets (1.02 nm thick) with enhanced intrinsic activity and abundant active sites.<sup>12,13</sup> Recently, 2D Bi<sub>2</sub>O<sub>2</sub>Se has emerged as a robust semiconducting material with ultra-high electron mobility and quantum oscillations,<sup>14</sup> and we believe that 2D Bi<sub>2</sub>O<sub>2</sub>Se may hold the potential to catalyze the CO2RR effectively. Moreover, 2D Bi2O2Se may be highly cost-effective: on the one hand, Bi is a kind of nonnoble metal and on the other hand, 2D materials exhibit low mass loading.

Here we report the pressure-regulated  $CO_2RR$  to formate as catalyzed by 2D Bi<sub>2</sub>O<sub>2</sub>Se. The 2D Bi<sub>2</sub>O<sub>2</sub>Se catalyst with a layered "2D Zipper" structure is synthesized by a chemical vapor deposition (CVD) method on glassy carbon. While the catalyst loading is as low as 5.2 µg cm<sup>-2</sup>, our 2D Bi<sub>2</sub>O<sub>2</sub>Se manifests a CO<sub>2</sub>RR current density as high as 4649 mA mg<sup>-1</sup> with a formate FE of 47%, surpassing reported Bi based 2D catalysts and the Bi<sub>2</sub>O<sub>2</sub>Se bulk counterparts. By pressurizing CO<sub>2</sub> from ambient pressure (1.01 bar) to 40 bar, we steer the CO<sub>2</sub>RR pathway toward formate, achieving a record-high current density of 7084 mA mg<sup>-1</sup> with a formate FE of 78%. *In situ* Raman spectroscopy and density functional theory (DFT) calculations reveal the mechanism of CO<sub>2</sub>RR to formate under high pressure, evidencing a stronger CO<sub>2</sub> adsorption, enhanced \*OCHO intermediate formation and more favorable pathway.

Division of Physical Science and Engineering (PSE), King Abdullah University of Science and Technology (KAUST), Thuwal, 23955-6900, Kingdom of Saudi Arabia † Electronic supplementary information (ESI) available. See DOI: https://doi.org/

<sup>10.1039/</sup>d4cc05357e

<sup>‡</sup> These authors contributed equally.

Fig. 1 Schematic of (a) 2D  ${\rm Bi}_2{\rm O}_2{\rm Se}$  synthesis by CVD and (b) the atomic structure.

The 2D Bi<sub>2</sub>O<sub>2</sub>Se was synthesized through a two-step process: (i) preparation of bismuth oxide on glassy carbon via E-beam evaporation and (ii) selenization by the CVD method (Fig. 1a). Using glassy carbon as the substrate allowed the in situ synthesis of 2D Bi<sub>2</sub>O<sub>2</sub>Se, enabling its direct use as a working electrode in H-cell setups without the need for 2D material transfer. Fig. 1b illustrates the atomic structure of Bi2O2Se, which comprises alternating layers of bismuth atoms in hexagonal lattices, oxygen atoms positioned between these layers, and selenium atoms filling the interlayer gaps.<sup>15</sup> As documented in the literature, the Raman peak at the  $A_{1g}$  mode (159 cm<sup>-1</sup>) indicates the symmetric stretching vibration mode of the Bi-O bonds in the Bi<sub>2</sub>O<sub>2</sub> layer (Fig. S1, ESI<sup>+</sup>).<sup>16</sup> The lattice structure of Bi<sub>2</sub>O<sub>2</sub>Se was confirmed using high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM), which aligns with the proposed "2D Zipper" model (Fig. S2, ESI<sup>+</sup>).<sup>17</sup> This model suggests that the chemical bonds near the surface of Bi<sub>2</sub>O<sub>2</sub>Se are strengthened, enhancing the stability of the few-layer structures. Additionally, electron energy loss spectroscopy (EELS) mapping of Bi<sub>2</sub>O<sub>2</sub>Se revealed a uniform distribution of Bi, O, and Se elements across the entire area (Fig. S3, ESI<sup>+</sup>). Atomic force microscopy (AFM) characterization on Bi<sub>2</sub>O<sub>2</sub>Se confirmed an average thickness of 10.49 nm (Fig. S4, ESI<sup>+</sup>), revealing 17 layers of 2D monolayer.14 This thickness suggests a low catalyst mass loading, which was further validated by inductively coupled plasma mass spectrometry (ICP-MS), revealing a catalyst loading of 5.2 µg cm<sup>-2</sup>. X-ray photoelectron spectroscopy (XPS) was used to verify the presence of Bi, O, and Se (Fig. S5 and S6, ESI<sup>†</sup>). The spectra revealed two oxidation states of the Bi atom, represented by Bi 4f7/2 and Bi 4f5/2 at binding energies of 159 and 164 eV, respectively, as well as O 1s states at 530 eV and Se 3d states at 53 eV, consistent with previous reports.<sup>18</sup> These observations authenticated the successful synthesis of 2D Bi<sub>2</sub>O<sub>2</sub>Se and motivated us to conduct the subsequent electrochemical experiments.

In contrast to wet chemical reduction,<sup>19</sup> galvanic replacement reaction,<sup>20</sup> hydrothermal reaction,<sup>21</sup> and electrochemical conversion<sup>22</sup> methods, the direct CVD growth of 2D Bi-based catalysts on glassy carbon substrates offers low mass loading by depositing only a few layers of catalyst (Fig. S4, ESI†). This may also enhance electron transport from the conductive substrate to the 2D catalyst. The CO<sub>2</sub>RR activity of 2D Bi<sub>2</sub>O<sub>2</sub>Se was firstly assessed in 0.5 M KHCO<sub>3</sub> electrolyte by linear sweep voltammetry (LSV) at ambient pressure (1.01 bar), as shown in Fig. S7 (ESI†). In a CO<sub>2</sub> saturated electrolyte, the 2D Bi<sub>2</sub>O<sub>2</sub>Se demonstrates a higher current density compared to an N<sub>2</sub> saturated electrolyte at -0.7 V vs. reversible hydrogen electrode (RHE),

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Fig. 2 Effective utilization of the 2D catalyst. (a) CO<sub>2</sub>RR to HCOO<sup>-</sup> performance of 2D Bi<sub>2</sub>O<sub>2</sub>Se and Bi<sub>2</sub>O<sub>2</sub>Se bulk at 1.01 bar. (b) The comparison of some typical Bi-based 2D catalysts for CO<sub>2</sub>RR to HCOO<sup>-</sup> with 2D Bi<sub>2</sub>O<sub>2</sub>Se.

indicating a greater favorability for  $CO_2RR$  over the hydrogen evolution reaction (HER).

To assess the activity in terms of current density per unit mass  $(mA mg^{-1})$  and faradaic efficiency (FE), we compared the synthesized 2D Bi<sub>2</sub>O<sub>2</sub>Se with Bi<sub>2</sub>O<sub>2</sub>Se bulk prepared by the spin coating method with catalyst loading of 0.5 mg cm<sup>-2</sup> (Fig. 2a). As shown in Fig. S8 (ESI<sup>+</sup>), 1H nuclear magnetic resonance (NMR) analysis identified formate as the only liquid product from the CO<sub>2</sub>RR, while gas chromatography (GC) detected H<sub>2</sub> and CH<sub>4</sub> as the gas products. It was observed that 2D Bi2O2Se exhibits significantly higher current density per unit mass, reaching 4649 mA mg $^{-1}$  at -1.15 V (vs. RHE), which is 2 orders of magnitude greater than that of the bulk counterpart. However, the FE toward formate for both 2D Bi<sub>2</sub>O<sub>2</sub>Se and Bi<sub>2</sub>O<sub>2</sub>Se bulk remained at the same level and decreased with increased potential. This was attributed to the stronger dependence of the Volmer step potential on the overall potential compared to CO<sub>2</sub> adsorption, leading to a higher preference for the HER at more negative potentials (Fig. S8, ESI<sup>†</sup>).<sup>23</sup> To understand the specific surface area properties of 2D Bi<sub>2</sub>O<sub>2</sub>Se and Bi<sub>2</sub>O<sub>2</sub>Se bulk, electrochemically active surface area (ECSA) calculation (Fig. S9 and S10, ESI†) and non-massnormalized LSV (Fig. S7, ESI<sup>+</sup>) were performed. Both 2D Bi<sub>2</sub>O<sub>2</sub>Se and Bi2O2Se bulk exhibit similar surface area and non-massnormalized current density, indicating minimal effect of ECSA when comparing the mass-normalized CO<sub>2</sub>RR current density. More than that, we found that both Bi<sub>2</sub>O<sub>2</sub>Se and Bi<sub>2</sub>O<sub>3</sub> were firstly reduced to the Bi metal state and it is Bi (012) that acts as the main facet during the CO<sub>2</sub>RR (Fig. S11, ESI<sup>†</sup>). These findings suggested that 2D Bi<sub>2</sub>O<sub>2</sub>Se growth on glassy carbon enabled more effective utilization of the catalyst in light of the low mass loading, while maintaining comparable CO2RR activity compared to bulk Bi<sub>2</sub>O<sub>2</sub>Se. A similar trend can be found when comparing 2D Bi<sub>2</sub>O<sub>3</sub> and Bi<sub>2</sub>O<sub>3</sub>Se bulk (Fig. S12, ESI<sup>†</sup>). Furthermore, the CVD grown 2D Bi<sub>2</sub>O<sub>2</sub>Se catalyst greatly outperforms the benchmarks in the literature, including mesoporous Bi nanosheets,<sup>24</sup> freestanding 2D bismuth nanosheets,25 layered Bi nanosheets,26 electron-rich Bi nanosheets,<sup>27</sup> 3D network of interconnected 2D bismuthene arrays,<sup>28</sup> atomically thin bismuthene with rich defects,<sup>29</sup> heterostructured bismuth-based catalysts,<sup>30</sup> Bi<sub>2</sub>O<sub>2</sub>CO<sub>3</sub> nanosheets<sup>31</sup> and Bi-MOF<sup>32,33</sup> (Fig. 2b).

Pressurization of  $CO_2$  has been reported to steer the selectivity<sup>34,35</sup> and boost the  $CO_2RR$  current density.<sup>4,36</sup> To further



Fig. 3 Partial current density of pressure regulated CO\_2RR to formate on 2D  $\text{Bi}_2\text{O}_2\text{Se}.$ 

enhance the CO<sub>2</sub>RR performance of our 2D Bi<sub>2</sub>O<sub>2</sub>Se catalyst, we subjected the system to elevated CO2 pressures ranging from 1.01 to 40 bar. In general, the FE towards formate increased as the CO<sub>2</sub> pressure increased, rising from 79.5% at 1.01 bar to 95% at 40 bar at -0.85 V (vs. RHE) (Fig. S13, ESI<sup>+</sup>). This demonstrates the capability of pressurized CO<sub>2</sub> to regulate the selectivity towards formate on 2D Bi<sub>2</sub>O<sub>2</sub>Se, consistent with previous study.<sup>7</sup> The corresponding total and formate partial current densities at various CO<sub>2</sub> pressures are plotted in Fig. S14 (ESI<sup>†</sup>) and Fig. 3. In particular, the formate partial current density was improved from 2146 to 7308 mA mg<sup>-1</sup> when the pressure increased from 1.01 to 40 bar at -1.15 V, indicating a greatly promoted production rate of formate. To compare the 2D Bi<sub>2</sub>O<sub>2</sub>Se with Bi<sub>2</sub>O<sub>2</sub>Se bulk under high CO<sub>2</sub> pressure, we plotted current densities with corresponding formate FEs (Fig. 2a and Fig. S15, ESI<sup>+</sup>). The results align with prior research, underscoring the efficacy of our 2D Bi2O2Se catalyst with minimal mass loading in sustaining high current densities. As depicted in Fig. S16 (ESI<sup>+</sup>), Nyquist plots for CO2RR over the 2D Bi2O2Se catalyst showed a decrease in charge transfer resistance with increased CO<sub>2</sub> pressures. This indicates that a significantly accelerated electron transfer with higher CO<sub>2</sub> pressure as enhanced CO<sub>2</sub> solubility in electrolytes promoted reactant transportation to the electrodes, that means, despite HER being more favorable under more negative potentials according to previous reported works,<sup>37,38</sup> the elevated CO<sub>2</sub> pressure can still suppress the HER (Fig. S8 and S17, ESI†).

These findings motivated us to investigate the mechanism of the  $CO_2RR$  to formate under high  $CO_2$ . In general, the  $CO_2RR$  to formate pathway follows 4 steps:

$$CO_2 + * \rightarrow *CO_2 \tag{1}$$

 $*CO_2 + H^+ + e^- \rightarrow *OCHO$ (2)

\*OCHO + H<sup>+</sup> + e<sup>-</sup>  $\rightarrow$  \*HCOOH (3)

\*HCOOH 
$$\rightarrow$$
 \* + HCOOH (4)

In order to track reaction intermediates during  $CO_2RR$  over 2D Bi<sub>2</sub>O<sub>2</sub>Se, *in situ* Raman spectroscopy measurements were performed in ambient CO<sub>2</sub> pressure (1.01 bar) and pressurized CO<sub>2</sub> (20 bar) (Fig. 4a and Fig. S18, ESI†). In the scan of applied potential from -0.75 to -1.2 V  $\nu s$ . RHE over the 2D Bi<sub>2</sub>O<sub>2</sub>Se catalyst, two intrinsic Raman peaks were detected. The Raman peaks at 1052 cm<sup>-1</sup> were attributed to HCO<sub>3</sub><sup>-</sup>, respectively,





indicating a CO<sub>2</sub> saturated environment during the CO<sub>2</sub>RR.<sup>39,40</sup> In addition, the peak at 2898 cm<sup>-1</sup> was ascribed to the C-H stretching of \*OCHO radicals.<sup>41</sup> This revealed the favored \*CO<sub>2</sub><sup>-</sup> formation to activate CO<sub>2</sub> molecules and enhanced adsorption strength of \*OCHO intermediates, leading to superior activity and selectivity toward formate. Due to the low catalyst loading and strong Raman signal from glassy carbon, the D band and G band of glassy carbon were observed at 1346  $\text{cm}^{-1}$  and 1572  $\text{cm}^{-1}$ . To gain a deep understanding on high pressure CO<sub>2</sub>RR, the DFT and Ab initio molecular dynamic (AIMD) calculations were performed in the Vienna Ab initio Simulation Package (VASP). In light of the pressure dependent CO2 solubility, we sought to explore the influence of CO<sub>2</sub> coverage from 1/8 monolayer (ML) to 3/8 ML on the Bi (012) facet, the dominant facet of the prepared catalyst, confirmed by X-ray diffraction (XRD) measurement, as shown in Fig. S11 (ESI<sup>†</sup>).

The optimized structures and free energy diagram of adsorbed CO<sub>2</sub>RR to formate intermediates are depicted in Fig. S19 (ESI<sup>+</sup>) and Fig. 4b. In the initial step (CO<sub>2</sub> +  $* \rightarrow$ \*CO<sub>2</sub>), a smaller free energy difference for 3/8 ML CO<sub>2</sub> coverage (0.1 eV) indicated a stronger CO<sub>2</sub> adsorption when compared with that of 1/8 ML CO<sub>2</sub> coverage (0.32 eV). The first protoncoupled electron transfer process (\*CO<sub>2</sub> + H<sup>+</sup> + e<sup>-</sup>  $\rightarrow$  \*OCHO) was defined as the rate-determine-step (RDS) of the CO<sub>2</sub>RR to formate with 1/8 ML CO2 coverage due to the large uphill energy. Moreover, in this step, the required free energy for 3/ 8 ML coverage is 0.1 eV, notably lower than that for 1/8 ML coverage (0.25 eV). That is,  $CO_2RR$  under high pressure exhibits higher activity for \*OCHO formation due to the lower reaction energy barrier. For the CO2RR to formate with 3/8 ML CO2 coverage, the second proton-coupled electron transfer process (\*OCHO + H<sup>+</sup> + e<sup>-</sup>  $\rightarrow$  \*HCOOH) was defined as the RDS. This proton-coupled electron transfer process (\*OCHO +  $H^+$  +  $e^- \rightarrow$ \*HCOOH) and the desorption process (\*HCOOH  $\rightarrow$  \* + HCOOH) contributed to the final formate production. The free energy difference of these two steps with 3/8 ML CO<sub>2</sub> coverage is lower, revealing a more favorable pathway. DFT calculations on the HER under pressurized CO2 were also conducted, as shown in Fig. S20 (ESI<sup>†</sup>). HER exhibited decreased energy barrier for RDS from 1 V to 0.92 V with enhanced CO<sub>2</sub> coverage from 1/8 ML to 3/8 ML, whereas the CO<sub>2</sub>RR has an RDS energy barrier of

only 0.13 V under high pressure. As a result,  $CO_2RR$  is preferred compared to HER as the pressure increases, aligning with the experimental data (Fig. S8 and S17, ESI<sup>†</sup>).

In conclusion, we devise a low-mass-loading 2D Bi<sub>2</sub>O<sub>2</sub>Se catalyst and leverage the pressure to enhance CO<sub>2</sub>RR for formate production. The 2D catalyst exhibited a significantly higher current density of 4649 mA mg<sup>-1</sup> with 47.1% FE to formate at -1.15 V (vs. RHE) compared to the bulk phase, indicating its costeffectiveness. High CO<sub>2</sub> pressure, commonly encountered in industrial processes involving CO2 capture, transport, and storage, significantly enhances formate selectivity and current density during CO<sub>2</sub>RR. Under high pressure, the partial current density increases from 2146 mA  $\mathrm{mg}^{-1}$  at 1.01 bar to 7308 mA mg<sup>-1</sup> at 40 bar at -1.15 V. Moreover, *in situ* Raman measurements and DFT calculations under pressurized CO<sub>2</sub>RR revealed the intermediates and pathways involved. These findings highlight the potential of 2D catalysts for sustainable CO<sub>2</sub> conversion at high pressure with industrial relevance and underscore the importance of pressure in regulating the CO<sub>2</sub>RR pathways.

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#### Data availability

The data supporting the findings of this study are available within the article and its ESI.† Additional raw data and experimental details can be provided by the corresponding author upon reasonable request.

### Conflicts of interest

There are no conflicts to declare.

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