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# Spontaneous deposition of Ir nanoparticles on 2D siloxene as high-performance HER electrocatalyst with ultra-low Ir loading

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Herein, 2D siloxene terminated by surface functional groups of Si–H and Si–OH, is demonstrated to be an effective support for the spontaneous deposition of iridium nanoparticles (Ir NPs) to promote HER electrocatalysis. With ultra-low Ir loading, the obtained Ir NPs/Siloxene electrocatalyst shows superior electrocatalytic activity toward HER.

It is everlasting and formidable challenge to explore advanced electrocatalysts for hydrogen evolution reaction (HER) and thus reduce the energy consumption for sustainable water splitting. As the benchmark electrocatalysts for HER, platinum (Pt) catalysts have been commercialized for decades. However, the scarcity and high cost are the major barriers to their widespread applications and force us to explore new alternatives. Although superior progress has been achieved for earth-abundant catalysts, the intrinsic electrocatalytic activities of most candidates are still inferior to noble metals.<sup>1</sup> Consequently, designing cost-effective noble-metal-based HER electrocatalysts with Pt-like activity by tailoring the architecture to enhance the intrinsic activity and decrease the noble metal usage gradually evolves as a central challenge and ultimate goal.

Iridium (Ir), as one neighbour of Pt in the volcano plot, is emerging as an impressive candidate for electrocatalyzing HER. Significant progress recently has been focused on seeking various supports for Ir nanoparticles. Among which, carbon materials, especially N-doped carbon matrix, have attracted considerable attention to boost the electrocatalytic performance of Ir based on metal-support interaction.<sup>2-6</sup> However, the doping type of N heteroatoms (pyrrolic, pyridinic and graphic N) has a different effect on the electrocatalytic performance of the supported Ir. And it is difficult to control the doping type and content of N heteroatoms in carbon framework.<sup>7, 8</sup> Therefore, it is urgent to further explore new effective and easy scale-up supports for Ir catalysts. Recently, Sheng et al. reported a binary Ir/Si nanowire composite (17.7 wt. % Ir) as efficient HER electrocatalyst, due to the strong interaction between Si and Ir demonstrated by experiments and theory calculations.9 Particularly, in the binary Ir/Si system, Si support can significantly suppress the aggregation tendency of Ir with large enthalpy of atomization, and thus improve its dispersity. As the second most earth-abundant element, silicon has a similar electronic structure with carbon, which is cheap, nontoxic and biocompatible. Hence, Si-based materials may be a kind of promising candidates to support Ir nanoparticles (NPs). Due to the rigorous and complicated technology along with high cost in the fabrication of Si nanowires, 10, 11 many challenges still remain to be solved to guarantee their dependable and large-scale implementation in electrochemical catalysis.<sup>12</sup> Besides, the intrinsic hydrophobic surface and HFderived reducibility (Si-H groups) are also the bottleneck for the green synthesis of noble metal nanoparticles/Si nanowires hybrids. In consequence, it would be highly desirable to develop new silicon-based materials for supporting active Ir in view of wide-range applications in industries.

Compared to 0D/1D and 3D bulk, 2D materials have distinct allure and superiority in catalysis, like large specific surface area and remarkable anisotropic properties. More interior atoms would be exposed and inevitably accompanied by forming defects on the surface of nanosheets, providing abundant nucleation sites for metal growth.13 As an emerging 2D Si-based material, siloxene is hydride- and hydroxylterminated layered silicon, composed of strong covalent bonding by Si chains interconnected with oxygen bridges within intralayer.14, 15 Based on the superiority of abundant functional groups, surface tunability, hydrophilicity, high surface area, and unique electronic properties, 2D siloxene has been preliminarily explored and shows promising in the field of supercapacitors,<sup>15, 16</sup> CO<sub>2</sub> methanation,<sup>17</sup> Mizoroki–Heck reaction<sup>18</sup> and so on.<sup>19, 20</sup> However, the exploration of 2D siloxene in electrocatalysis field is rarely found and deserves further concerns.

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Scheme 1 Schematic illustration for the synthesis of Ir NPs/Siloxene.

Motivated by the aforementioned considerations, herein, we explore the potential of 2D siloxene as an effective substrate to support Ir NPs for high-performance HER electrocatalysis. As depicted in Scheme 1, 2D siloxene sheets was firstly fabricated via topotactic transformation of layered calcium silicide (CaSi<sub>2</sub>)<sup>16</sup> to accomplish deintercalation of Ca<sup>2+</sup> interlayers, and simultaneous functionalization with unique surface functional groups of Si-H and Si-OH. Subsequently, the Ir NPs/Siloxene catalyst was prepared via in-situ hydrothermal reduction of IrCl<sub>6</sub><sup>2-</sup> by Si–H groups on siloxene. The abundant surface functional groups also serve as the anchoring sites to control the nucleation and growth of Ir NPs without any capping agents, consequently guaranteeing the highdispersion. Different from the traditional liquid synthesis, the spontaneous deposition without any additional reductants or capping agents endows the Ir NPs/Siloxene catalyst with clean surface active sites to express high electrocatalytic activity.

The pristine CaSi<sub>2</sub> presents densely packed and layered structure as observed from the scanning electron microscopy (SEM) image (Fig. S1, ESI<sup>+</sup>). Via topotactic transformation, 2D siloxene with folded and crumpled lamellas structure was obtained (Fig. 1a). After in-situ reduction reaction, ultrafine Ir nanoparticles with average diameter of ~2.4 nm are uniformly attached on the surface of siloxene sheets without any obvious aggregation (Fig. 1b). The dark-field scanning transmission electron microscopy (STEM) image and corresponding EDX mapping (Fig. S2, ESI<sup>+</sup>) also declare the homogeneous distribution of Ir, O and Si elements in Ir NPs/Siloxene. The high-resolution TEM image (Fig. 1c) depicts clear lattice fringes for the nanoparticles with characteristic interplanar spacing of 0.22 nm and 0.19 nm, indexed to (111) and (200) planes of Ir (PDF#46-1044). Also, the ring-like selected-area electron diffraction (SAED) of Ir NPs/Siloxene (Fig. 1d) further verifies the successful formation of Ir NPs.

The crystalline structures and chemical composition of Ir NPs/Siloxene were confirmed by X-ray diffraction (XRD) analysis. As clearly indicated in Fig. S3 (ESI<sup>+</sup>), the broad peak at 23.5° declares the preservation of siloxene after hydrothermal treatment.<sup>16</sup> Three characteristic peaks at 40.7°, 47.3° and 69.1° respectively correspond to (111), (200) and (220) crystal planes of Ir (PDF#46-1044), further providing solid evidence for

the successful reduction of  $IrCl_6^{2-}$  to Ir NPs on 2D siloxene The Ir loading mass in Ir NPs/Siloxene hybrids is measured to be 2.9 wt.% by inductively coupled plasma optical emission spectrometry. The noble-metal loading of Ir NPs/Siloxene is much lower than that of commercial Pt/C catalyst (20 wt.% Pt) and the reported Ir/Si nanowire composite (17.7 wt. % Ir).<sup>9</sup>

To better understand the spontaneous deposition of Ir nanoparticles on siloxene, the control experiments were conducted via synthesizing the Ir-based sample by similar hydrothermal procedure without the addition of siloxene. As illustrated by XRD pattern, SEM and TEM images in Fig. S4-6 (ESI<sup>+</sup>), the obtained product is the aggregated IrO<sub>2</sub> particles instead of Ir particles. The generation of IrO<sub>2</sub> is further demonstrated by Ir 4f XPS spectrum (Fig. S7, ESI<sup>+</sup>) with two characteristic peaks of Ir<sup>4+</sup> at higher binding energies.<sup>21</sup> Therefore, the Ir-based sample obtained under similar synthesis procedure without the addition of siloxene is IrO<sub>2</sub>. And it is rationally deduced that siloxene play an important role in the in-situ reduction of IrCl<sub>6</sub><sup>2-</sup> to Ir NPs.

The reductivity of siloxene is supposed to be attributed to the abundant surface functional groups. Fourier transform infrared (FT-IR) spectroscopy was used to characterize the functional groups on siloxene and Ir NPs/Siloxene (Fig. 2a). For siloxene, the bands at 437, 877, 1056, 1633, and 2126 cm<sup>-1</sup> respectively correspond to the vibrations of v(Si-Si), v(Si-H), v(Si-O-Si), v(Si-OH), and v(OSi<sub>2</sub>=Si-H), proving the presence of abundant Si-H and Si-OH groups on siloxene.<sup>15, 16</sup> The surface functional groups, serving as the anchoring sites to control the nucleation and growth of Ir, is responsible for the highdispersion of Ir NPs on siloxene. It will be beneficial to the increased atom utilization and the metal–support interaction



Fig. 1 (a) TEM image of siloxene; (b-c) TEM images and (d) SAED pattern of Ir NPs/Siloxene.

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Fig. 2 (a) FT-IR spectra of Ir NPs/Siloxene and siloxene; (b) XPS survey spectrum of Ir NPs/Siloxene and corresponding (c) Ir 4f spectrum; (d) The contrastive Si 2p XPS spectra of Ir NPs/Siloxene and siloxene support.

for alterable electronic structure,<sup>22</sup> finally enhancing the electrocatalytic performance. After the hydrothermal reaction, the band attributed to v(Si-H) disappears, potentially resulting from depletion in the reduction reaction of IrCl<sub>6</sub><sup>2-</sup>. It is further confirmed by the Raman analysis with the disappearance of the characteristic Si-H vibrations (Fig. S8, ESI<sup>+</sup>). Therefore, spontaneous deposition of Ir NPs on siloxene is ascribed to the in-situ redox reaction between the surface Si-H groups and IrCl<sub>6</sub><sup>2-</sup> based on the reported similar mechanism.<sup>22, 23, 24</sup> In addition, the oxygen-containing functional groups such as Si-OH are still held after hydrothermal treatment, which will be beneficial to the hydrophilicity of catalyst, potentially facilitating mass transfer. According to above analysis, spontaneous deposition of high-dispersed Ir NPs on 2D siloxene is achieved through in-situ reduction and anchoring by the intrinsic surface functional groups of siloxene under green and facile hydrothermal synthesis. Different from traditional liquid synthesis of high-dispersed metal nanoparticles,<sup>25</sup> the spontaneous formation of Ir NPs/Siloxene catalyst without any additional reductants or capping agents would avoid blocking of surface active sites and thus express high electrocatalytic activity.

The chemical states and electronic structure were investigated by X-ray photoelectron spectroscopy (XPS). Fig. 2b clearly shows the presence of Si, O and Ir elements in Ir NPs/Siloxene. In Ir 4f spectrum of Ir/siloxene (Fig. 2c), the intense doublet peaks at 61.3 eV and 64.2 eV are ascribed to Ir<sup>0,4, 22</sup> agreeing well with TEM, SAED and XRD results. The weak peaks located at 62.0 eV and 64.6 eV belongs to Ir4+, potentially due to slightly superficial oxidation. The O 1s spectrum in Fig. S9 (ESI<sup>+</sup>) presents a peak around 533.2 eV, corresponding to the oxygen- and hydroxyl-functionalized siloxene.<sup>15, 26</sup> In particular, the characteristic Si 2p peak of Ir NPs/Siloxene shifts toward higher binding energy compared to that of siloxene support (Fig. 2d). The variation in binding energy provides a significant evidence for the electronic interaction between Ir NPs and siloxene, induced by the electron transfer from siloxene to Ir NPs. It will result in a

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higher electron density around Ir NPs to promote, hydrogen adsorption.<sup>2, 27</sup> This metal–support interaction 05% modulating the electronic structure has been demonstrated to be helpful to the improvement of electrocatalytic activity.<sup>2, 28, 29</sup>

The electrocatalytic HER performance of Ir NPs/Siloxene were then evaluated in Ar-saturated 0.5 M H<sub>2</sub>SO<sub>4</sub> solution. Fig. 3a shows the LSV curves without iR compensation for different samples. In the investigated potential range, siloxene is inactive for HER. The Ir NPs/Siloxene catalyst with 2.1 wt.% Ir loading exhibits remarkably superior HER activity with 31 mV overpotential to achieve a current density of 10 mA cm<sup>-2</sup>, comparable to that of Pt/C (20 wt.% Pt). Insight into the kinetics of Ir NPs/Siloxene is achieved from Tafel plots. As shown in Fig. 3b, Ir NPs/Siloxene displays a Tafel slope of 29.4 mV·dec<sup>-1</sup>, which is approximate to that of Pt/C (28.1 mV·dec<sup>-1</sup>), declaring rapid reaction kinetics. Besides, the significantly improved charge-transfer kinetics for Ir NPs/Siloxene is also observed (Fig. S10, ESI<sup>+</sup>), despite a low electrical conductivity of siloxene (4.11×10<sup>-6</sup> S·m<sup>-1</sup>).

To further quantify the HER activity and evaluate the potential practicability, the mass activity of Ir NPs/Siloxene and Pt/C catalysts were calculated by normalizing to noble metal loading. As shown in Fig. S11 (ESI<sup>+</sup>) and Fig. 3c, Ir NPs/Siloxene presents admirable mass activity of 5.51 A·mg<sub>metal</sub><sup>-1</sup> at overpotential of 60 mV, almost 9.5 times higher than that of Pt/C (0.58 A·mg<sub>metal</sub><sup>-1</sup>). By normalizing the current with ECSA (0.28 cm<sup>2</sup>) estimated from the CV results in Fig. S12 (ESI<sup>+</sup>), Ir NPs/Siloxene reveals significantly better specific activity compared to Pt/C (Fig. S13, ESI<sup>+</sup>). Furthermore, the TOF of Ir NPs/Siloxene is calculated to be 11.05 s<sup>-1</sup> at an overpotential of 50 mV, which is much higher than that of Pt/C (2.07  $s^{-1}$ ), demonstrating greatly superior intrinsic catalytic activity of Ir supported on siloxene sheets. Overall, the high HER activity of Ir NPs/Siloxene catalyst is potentially associated to following factors: (i) the high-dispersion of Ir NPs, thus proliferating the catalytically active sites; (ii) the clean surface active sites, profiting from the spontaneous deposition of Ir NPs on 2D



Fig. 3 (a) LSV curves and (b) Tafel plots of different samples; (c) The metal mass activity of Ir NPs/Siloxene and Pt/C at different overpotential; (d) Stability test of Ir NPs/Siloxene.

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siloxene without any additional reductants or capping agents; (iii) the enriched electron state of Ir NPs due to the interaction between Ir NPs and siloxene support; (iv) the hydrophilicity derived from abundant oxygen-containing functional groups of siloxene to facilitate mass transfer; (v) the small energy barrier for H diffusion between Ir and Si, and accelerated hydrogen desorption from Si, as reported previously for Ir/Si system.<sup>9</sup>

The stability of Ir NPs/Siloxene catalyst in HER catalysis is assessed by an accelerated degradation test with continuous cyclic voltammetry (CV) scanning. After 1000 CV cycles, the Ir NPs/Siloxene catalyst exhibits a negligible loss of HER activity (Fig. 3d) with no significant aggregation of Ir NPs (Fig. S14, ESI<sup>+</sup>). Also, a slight decrease of current density is observed on the prolonged electrolysis at a constant overpotential of 31 mV (Fig. S15, ESI<sup>+</sup>). These demonstrate good catalytic stability of the Ir NPs/Siloxene catalyst in acidic environment. Therefore, the siloxene sheets is a promising choice as the support material of Ir NPs for promoting HER electrocatalysis.

In summary, we demonstrate 2D siloxene could serve as an effective support for high-dispersed Ir NPs to enhance the HER performance. Benefiting from the unique and abundant surface functional groups, 2D siloxene shows strong reducibility and anchoring ability to enable a facile in-situ spontaneous deposition of ultrafine Ir NPs on its surface. Without any additional reductants or capping agents during the synthetic process, it endows the Ir NPs/Siloxene catalyst with clean surface active sites to express high intrinsic catalytic activity. The as-prepared Ir NPs/Siloxene catalyst with low Ir loading exhibits excellent electrocatalytic activity with good stability toward HER in acidic media. Also, dramatically enhanced mass activity with 9.5 times higher than that of commercial Pt/C at 60 mV overpotential is eventually achieved by the Ir NPs/Siloxene catalyst. This work presents the potential of 2D layered siloxene as an ideal candidate to design and synthesize high-performance electrocatalysts for sustainable energy conversion applications.

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#### **Conflicts of interest**

There are no conflicts to declare.

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Based on reducibility of siloxene, Ir nanoparticles were spontaneously deposited on siloxene and showed superb performance for HER.