

Nano-chemical mapping of cobalt nanoparticles on the graphene-related membranes



莊程豪教授
淡江大學物理系

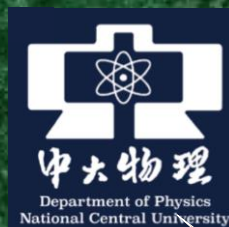
Prof. Cheng-Hao Chuang
Department of Physics
TamKang Univeristy

With the increasing demand in the sustainable energy, any possibility to substitute the fossil fuel becomes the key melody in the next generation technologies. Hydrogen or oxygen evolution reaction (HER or OER) over metal oxides are thought to own the promising power capability. It becomes crucial in the advanced electrode materials for the good catalytic performance, which is actually unclear in the reaction mechanism and catalytic optimization. Non-noble materials, such as transition metal oxide, have been heightened because of some reasons: (a) abundant hydroxyl groups on the surface, (b) size-diminished metal crystal, (c) anti-corrosion resistance in the alkaline solution. On the other hand, graphene sheet is considered as one of important 2D materials due to the unique electric transfer property and thinnest thickness. The ability to grow the functional nanomaterials on the top of graphene substrate has been proposed due to the ideal single-atom thickness, excellent electrical conductivity, transparent electrode, flexible nanosheet, and stable structure of graphene-based materials. Cobalt oxides as one of active electrocatalysts has been proven to be one class of HER/OER in alkaline environment. The facial functional groups (hydroxyl, carboxyl, and epoxide) of nanocomposite synthesis are characteristic for the interesting interaction with metal site.[1] By the strong covalent bonding between O and Co, the oxidation state of Co^{3+} in Co_3O_4 can be partially reduced to Co^{2+} state among individual Co_3O_4 nanocrystal/graphene.[2] Since the nano-scaled microscopic probing is still absent for the detail and direct observation, we will demonstrate the element-specific chemical images to promote the graphene-related electrode for the future liquid-cell applications. TEM image in Figure 1(a) shows many Co_3O_4 crystals on the graphene electrode by the electrochemical deposition. Precise area of Co_3O_4 crystals (green squares in Figure 1(b)(c)) reflects the extra-formation of Co_3O_4 crystals not only by the less transmission intensity (dark area) but also by the new crystal-phase coverage (amorphous boundary). Scanning Transmission X-ray Microscopy (STXM) equipped with the optical lens (Zone Plate and Order Sorting Aperture) can visualize the same interest of areas by the transmission mode of X-ray absorption spectroscopy. Figure 1(d) displays Co L-edge XAS at the regions of three interests. The area selection from “1”, corresponding to the highest optical density, has multi-peaks from 2p to 3d state absorption. The 3d-t_{2g} (3d-eg) bond with Oh symmetry are existed at PE 775.0 eV (777.3 eV), which is believed for the Co^{2+} state. The high-energy shoulder at PE 778.2 eV is the key peak to identify its Co^{3+} state with Oh symmetry. The neighbouring area “2” is diminished its the $\text{Co}^{3+}/\text{Co}^{2+}$ ratio of absorption; meanwhile, the outside area “3” is considered for the non- Co_3O_4 area by the different chemical structure mappings. The interaction between grown Co_3O_4 and graphene is characteristic of Co anchoring on the graphene, while carbon atom of graphene replaces oxygen ligands of Co_3O_4 as $\text{Co}(\text{CO})_x$ formation at the solid-liquid interface. The chemical reduction from Co^{3+} and Co^{2+} to Co^{0+} metal species has been investigated under the nano-scaled microscopy, consistent with new electrocatalytic activity and stability in Co_3O_4 /Graphene nanocomposition.³ Our work will also provide the reduced graphene oxide membrane as the electrochemical window materials used the liquid cell, in order to demonstrate the modification of interface property of Co and its HER performance.

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Colloquium