Report and Retrospect on Experiments on Enhanced PEC Performance in BFO Heterostructure

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Gel Configuration

To study enhanced photoelectrochemical performance in BFO heterostructure, the BFO thin films are prepared by the sol–gel method; consequently, BFO gel (25mL in volume and 0.2mol/L in concentration) is needed.

In the gel, Bi comes from $Bi(NO_3)_3{\cdot}5H_2O$ and Fe comes from $Fe(NO_3)_3{\cdot}9H_2O$. It is shown that 0.005mol of each substance is required, which means 2.42535g of $Bi(NO_3)_3{\cdot}5H_2O$ and 2.02005g of $Fe(NO_3)_3{\cdot}9H_2O$ respectively. In addition, 25mL of 2-methoxyethanol is needed as solvent.

Step 1:

2-methoxyethanol is measured with a syringe and pour into a beaker. In this case, a 5mL syringe is used 5 times to measure 25mL 2-methoxyethanol.

Step 2:

Firstly, a piece of paper is folded by diagonal and put onto a electrical balance. Then $Bi(NO_3)_3{\cdot}5H_2O$ crystals are measured accurate to ten thousandth gram. Such measurement should be as quick as possible since the crystal deliquesces very easily when exposed in the air.

Step 3:

Firstly, those crystals are poured into the beaker carefully to prevent the solvent from splashing. Then the beaker is covered with preservative film to reduce the loss of evaporation. Later the beaker is put onto the magnetic stirrer with a proper mixing speed until all of the crystals dissolve.

Step 4:

Same as step 2, $Fe(NO_3)_3{\cdot}9H_2O$ crystals are measured and poured into the beaker. Then 0.01mol (2.1014g) citric acid is added.

Step 5:

Same as step 3, the beaker is put onto the magnetic stirrer for 1h.

Gel Spin-Coating

Prepared gel was spin-coated onto Pt substrate for later PEC measurement.

Step 1:

Syringe is used to draw 5 mL BFO gel for later operations. Then Pt substrate is taken out from Acetic acid solution with tweezers.

Step 2:

Pt substrate is put onto the centrifuge; button ‘Vacuum’ is pressed to fix the substrate. Dust is firstly blown from its surface; q-tips are later used to absorb acetic acid to further clean its surface, during which blow drier is used to increase evaporation rate.

Step 3:

When the Pt substrate is completely cleared, the gel is slowly dropped, until the substrate, including 4 corners, is covered by an even and relatively thick layer of BFO gel. Any air bubble in this gel layer should be punctured by the needle, which should, at the same time, be kept from touching the surface of Pt substrate.

Step 4:

Subsequently, the centrifuge, which is initialized to spin at 1000rpm for 5s followed by spinning at 5000rpm for 30s, is activated. After such centrifugation, whether the gel left on the the Pt substrate is even in color and surface and is without air bubbles should be examined carefully.

Step 5:

The gel should be completely removed from the Pt substrate, ***restarting from step 2*** exactly as is described above, if there is any deficiencies. If there is not, one selected corner of the Pt substrate should be cleared completely using a clean q-tip in order to leave a small arc region at one corner for later photocurrent measurement.

Step 6:

Button ‘Vacuum’ is pressed to de-fix the substrate. Then, the wet Pt film is transferred onto the evaporating dish, which is later covered with a small piece of paper to separate dusts. They are dried on a electrothermal furnace at 150 degrees centigrade for 2.5min. When finished, the paper is uncovered to check whether the film is even in both surface and color. If not, the gel should be completely removed from the film, ***restarting from step 2***. Otherwise, the evaporating dish, along with the film in it, is put into another electrothermal furnace at 600 degrees centigrade for 5min.

Repeat step 2 to 6 for 9 times.

Film Annealing

Step 1:

The film is put into a electrothermal furnace at 620 degrees centigrade for 2h and later cooled to room temperature naturally.

Film Sealing

Step 1:

Firstly, a piece of wire is cut off from a copper coil. Its both ends (approximately 5cm) are later burned in the alcohol burner until black copper oxide is formed, after which they are polished with abrasive paper.

Step 2:

One end of the copper wire is curved to a circle (in order to increase contact area) and clung onto the corner of the film that is not covered with BFO. Meanwhile, copper foil is used to keep them close.

Step 3:

Glue is used to seal the film. It is mixed first and smeared on the edges of the film, around the face covered with BFO, and completely on the face without BFO. It should also be smeared to the place where the wire is polished and where the wire and the film are connected to each other in order to prevent photocurrent leakage.

PEC Performance Measuring

Photocurrent density is measured by electrochemical analyzer. The light source was a 300 W Xe lamp with tunable light intensity. The Nyquist plot was obtained using an AC voltage of 10 mV with a frequency range of 0.01 Hz to 10 MHz.

Step 1:

The film is put into 0.1 M Na_2SO_4 aqueous solution and connected to PEC workstation, which consists of equipment as mentioned above.

Analysis, Conclusion, and Retrospect

The film I made during the experimentation has a relatively inferior PEC performance during PEC measurements in that there is a tiny difference between photocurrent and dark current.

Factors that have an impact on the results of the experimentation are omnifarious. First, the ratio between the solutes and the solvent should be very accurate when configuring the gel. Additionally, the air temperature and humidity are significantly influential to the film when the gel is spin-coated. Also during 9 times of spin-coating, any unevenness at any time of the procedure, any difference in thickness of each BFO layer, and any dust on the Pt substrate will all affect the final outcome. Meanwhile, it is important to control the time of pre-heating at 150 degrees centigrade and the time of annealing. Insufficient contact between the copper wire and the film and deficiencies in the sealing process will cause photocurrent leakage and reduce PEC performance.

This experience in the laboratory in Soochow University gives me the opportunity to explore something unknown and the difficulty of experiments and research. Failure is quite common especially when any seemingly insignificant factor all has an effect on the final result.