碳负载的 Pd-Cu-Fe（Pd/Cu-Fe/C）催化剂是由 Pd^{2+} 部分刻蚀可调二元 Cu-Fe 合金模板（Cu_{9}Fe_{1}, Cu_{8}Fe_{2}, Cu_{7}Fe_{3}）的新策略制成的。与 Pd/ C 催化剂相比，由 Cu_{9}Fe_{1}, Cu_{8}Fe_{2} and Cu_{7}Fe_{3} 模板部分刻蚀所得的催化剂的阳极峰电位分别负移了 51 mV, 88 mV 和 110 mV。其中，由 Cu_{7}Fe_{3} 模板所制得的催化剂表现出很高的催化活性和稳定性。表明它是甲酸燃料电池的一个很好的催化剂候选物。本次实验研究使用了 X 射线衍射分析，透射电子显微镜和电化学测量的方法对 Cu-Fe/C 三元催化剂进行了相关性质的分析。
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Abstract in English

Carbon loaded Pd-Cu-Fe catalysts (Pd-Cu-Fe/C) were fabricated with a novel strategy of partly etching carbon loaded tunable binary Cu-Fe alloy templates (Cu$_9$Fe$_1$, Cu$_8$Fe$_2$, Cu$_7$Fe$_3$) with Pd$^{2+}$. Compared with Pd/C catalysts, the anodic peak potential at the catalysts started from Cu$_9$Fe$_1$, Cu$_8$Fe$_2$ and Cu$_7$Fe$_3$ shifted toward negative value by 51 mV, 88 mV and 110 mV, respectively. Among them, catalyst started from exhibited the highest catalytic activity and a high stability which suggest the Cu$_7$Fe$_3$ template is a good candidate for the formic acid fuel cell. The characteristics of the Pd-Cu-Fe/C ternary catalysts were correlated using X-ray diffraction analysis, transmission electron microscopy, and electrochemical measurements.

Key words: Carbon based palladium catalyst; Formic acid oxidation; Fuel cell; Etching ; Ternary catalysts.

Abstract in Chinese

碳负载的Pd·Cu·Fe (Pd·Cu·Fe / C) 催化剂是由Pd$^{2+}$部分刻蚀可调二元Cu·Fe合金模板（Cu$_9$Fe$_1$, Cu$_8$Fe$_2$, Cu$_7$Fe$_3$）的新策略制成的。与Pd / C催化剂相比，由 Cu$_9$Fe$_1$, Cu$_8$Fe$_2$ and Cu$_7$Fe$_3$ 模板部分刻蚀所得的催化剂的阳极峰电位分别负移了51 mV, 88 mV 和 110 mV。其中，由Cu$_7$Fe$_3$模板所制得的催化剂表现出很高的催化活性和稳定性。表明它是甲酸燃料电池的一个很好的催化剂候选物。本次实验研究使用了X射线衍射分析，透射电子显微镜和电化学测量的方法对Cu·Fe / C 三元催化剂进行了相关性质的分析。
Chapter 1. Introduction

1.1 Characteristics of Fuel cells

Fuel cells [1-5] is efficient, convenient and environmentally friendly green energy devices. In today's society, improving energy efficiency, reducing environmental pollution, are urgent need to solve the major problems. Fuel cell is a kind of device which is used for transferring the chemical energy of fuel and oxidizing agent directly into electrical energy through electrode reaction[6], with the characteristics of fuel diversification, exhaust clean, low noise and less pollution to the environment. Due to the reaction process does not involve burning. Accordingly, its energy conversion rate is not limited by carnot cycle, as high as 60%~80%. Fuel cells have several advantages compared with the traditional energy [7, 8]. (1) high efficiency, decentralized power generation device. (2) clean and safe power generation device.(3) “the power and waste heat utilization system” can be used at the same time (4) power generation environment friendly.(5) high flexibility, wide application, convenient and safe operation, etc. Fuel cells can be used in more places because of its simple structure. Generated Power depends on the number of single battery power and battery;

1.2 The advantages of direct formic acid fuel cells

Direct formic acid fuel cell (DFAFC) has many advantages [9-11]. Formic acid can be used as an additive to food and medicine, it is a non-polluting environmentally friendly material, formic acid is not flammable, safe for storage and transportation. Formic acid is a strong electrolyte which can promote the transmission of electrons and protons. It is conducive to increasing the solution proton conductivity of anode roobm and formic acid can be ionized into formate, formate and Nafion membrane in the sulfonate mutual Rejection,
which partially hinders the transmission of formic acid, the permeability formic acid on the Nafion membrane is only 1/5 of methanol, so the low transmittance of formic acid can improve the efficiency of the battery. It’s conducive to manage and allow the use of high concentrations of formic acid. The use of formic acid as fuel is very suitable mobile power for use at room temperature or low temperature. Because of the formic acid with high energy conversion efficiency, low permeability of formic acid, non-toxic, non-flammable, etc.

### 1.3 The working principle and structure of direct formic acid fuel cell (DFAFC)

Direct formic acid fuel consist of anode, cathode, proton exchange membrane and bipolar plate. The working principle is as follows:

**Anode:**

\[
\text{HCOOH} \rightarrow \text{CO}_2 + 2\text{H}^+ + 2e^- \quad (1)
\]

**Cathode:**

\[
\frac{1}{2}\text{O}_2 + 2\text{H}^+ + 2e^- \rightarrow \text{H}_2\text{O} \quad (2)
\]

The total battery reaction is:

\[
\text{HCOOH} + \frac{1}{2} \text{O}_2 \rightarrow \text{CO}_2 + \text{H}_2\text{O} \quad (3)
\]

Direct formic acid fuel cell works as shown in Figure 1-1: formic acid is oxidized to produce \(\text{CO}_2\) on the anode, electrons and protons was released at the same time, electrons from the external loop through the load conduction to the cathode, while hydrogen protons through the electrolyte to the cathode. On the cathodes, electrons and protons react with oxygen, which is electrochemically reduced to water and consumes electrons and protons.

The structure is as follows:
Chapter 2 Experimental

2.1 Introduction of Experimental part.

With hydrogen and light hydrocarbons as the fuel of the proton exchange membrane fuel cell has become an important technology of the next generation of power systems [12]. However, direct formic acid fuel cells with formic acid as a fuel for its good safety and environmental protection has become a powerful alternative for portable electronic devices [13, 14]. Formic acid has the advantages characteristics of safe, non-toxic, nonflammable and transportation convenient. The Nafion membrane permeability of formic acid is far lower than that of methanol [15] Because of the repulsive force between HCOO and SO3 ions. The research of electric catalytic oxidation catalyst of formic acid has been for a half century [16-17]. The are platinum catalyst [18] and palladium catalysts among them[19]. In the field of electrochemical catalysis, Pd is a very important metal [20-22]. Especially for formic acid oxidation, Pd and pt showed high catalytic activity and low toxicity of CO [23-25].People have explored some methods to improve the catalyst performance and and reduce consumption of Pd. Among them,one of the effective method is to change the composition of catalyst. Such as form binary and ternary Pd catalysts by joining the proper metal, such as Pd, Ag [22], Pd - Ni [23], Pd, Co, Pd - Co - Ir [24], etc.

Compared with the binary catalysts, ternary catalysts have much more elements and microstructure. With the increase of elements composition of catalysts, we can get quite a lot different kinds of catalysts. Thus, we need to prepare many samples for screening test. In this case, the experiment's work is very difficult. At present, Pd-Cu and Pd-Fe have been reported for the oxidation of formic acid. Cu and Fe and their alloys are essential for people's daily life and industrial production, and are well-stocked and inexpensive. We attempted to prepare Pd-Fe-M or Pd-Cu-M (M is a doped third metal). One of the easiest ideas is the simplest preparation of Pd-Cu-Fe ternary catalyst.
There are many methods for preparing multivariate catalysts, such as etching, alloying, electrodeposition, etc. [43-45]. Etching method has been applied more than ten years [46-48]. The etching process is usually carried out by a displacement reaction. In this way, a series of multifunctional, well-controlled hollow, porous structures and multicomponent heterogeneous structures with adjustable composition can be obtained [49]. In addition to the use of Pd\(^{2+}\) partial or total corrosion of Cu to prepare a monovalent palladium-based electrocatalyst [18] or a binary palladium-based electrocatalyst [50], we have developed a method for the preparation of ternary Pd- Catalyst method. According to the preparation principle, at least one metal in the alloy needs to be reduced by Pd\(^{2+}\). In this paper, the Pd-Cu-Fe / C catalyst was prepared by partially etching the Cu-Fe alloy via Pd\(^{2+}\). The etch process has at least the following process:

\[
\begin{align*}
Pd^{2+} + Cu & \rightarrow Pd + Cu^{2+} \quad (2.1) \\
Pd^{2+} + Fe & \rightarrow Pd + Fe^{2+} \quad (2.2) \\
Cu^{2+} + Fe & \rightarrow Cu + Fe^{2+} \quad (2.3)
\end{align*}
\]

After etching for several hours, more than half of the atoms in the alloy are replaced by Pd atoms, so the resulting samples exhibit the catalytic performance of a typical Pd-based catalyst. Since the etching is not complete, the performance of the resulting catalyst is partially affected by the template. Through the simple preparation method, we obtain the complex multi-catalyst, these catalysts have good catalytic activity for formic acid oxidation and a certain stability.

Compared with the Pd/C catalyst, the anode potential of the Pd-Cu-Fe ternary catalyst is obviously negatively shifted, which shows a high catalytic activity. Among them, the Pd-Cu-Fe ternary catalyst prepared by Cu\(_7\)Fe\(_3\)/C template is negatively shifted by 110 mV than the Pd/ Cpeak potential. The catalytic activity of these ternary catalysts is higher than that of binary catalysts, such as Pd\(_2\)Co [31], Pd\(_{0.97}\)Co\(_{0.03}\) [51], Pd\(_{0.8}\)Cr\(_{0.2}\) [52], compared to the peak potential of binary catalysts in some literature and the previously mentioned Pd-Cu and Pd-Fe catalysts. The negative shift of formic acid of the Pd-Cu-Fe/C catalyst obtained by etching the Cu\(_7\)Fe\(_3\)/C template from Pd\(^{2+}\) is 110 mV which is similar to that of 140 mV of the ternary Pd\(_4\)Co\(_2\)Ir/ C. Pd\(_4\)Co\(_2\)Ir catalyst is known to have the greatest displacement of formic acid electrocatalytic oxidation peaks. Cu, Fe is the most widely used and most readily
available metal. Cu and Cu alloys have good mechanical properties, such as excellent corrosion resistance and thermal conductivity [53]. Fe is one of the most commonly used metals in modern industry and is the most abundant metal on earth. The relative stability of Fe-based alloys is due to magnetic interactions [54]. Cu-Fe alloys are widely used in electronic components due to their strong electrical conductivity and low cost [55]. The preparation of this catalyst can also be extended to more electrochemical reactions, such as ethanol oxidation, oxygen reduction and so on.

2.2 Experimental part

2.2.1 Experimental device

<table>
<thead>
<tr>
<th>Instrument name and model</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrochemical analyzer (CHI832B)</td>
<td>Shanghai Chen Hua Instrument Company</td>
</tr>
<tr>
<td>Electronic balance (FA1004N type)</td>
<td>Shanghai Precision Science Instrument Co., Ltd</td>
</tr>
<tr>
<td>Ultrasonic cleaning machine (KQ-50B type)</td>
<td>Kunshan City ultrasound Instrument Co., Ltd.</td>
</tr>
<tr>
<td>Vacuum drying oven (DZF-6020)</td>
<td>Gong Yi City Yuhua Instrument Co., Ltd.</td>
</tr>
<tr>
<td>Rotary vane vacuum pump (2XZ-1 type)</td>
<td>Zhejiang Huangyan Ningxi Medical Equipment Co., Ltd</td>
</tr>
<tr>
<td>X-ray diffractometer (D8advance type)</td>
<td>Germany bruker-axs company</td>
</tr>
<tr>
<td>Transmission electron microscopy (JEM-2010F)</td>
<td>Japan Electronics Co., Ltd. (JEOL) company</td>
</tr>
<tr>
<td>X-ray photoelectron spectroscopy (XSAM-800)</td>
<td>Kratos, Japan</td>
</tr>
<tr>
<td>Inductively Coupled Plasma Emission</td>
<td>American PE company</td>
</tr>
</tbody>
</table>
Table 2-2 Reagents and drugs used in the experiment

<table>
<thead>
<tr>
<th>name</th>
<th>Chemical formula</th>
<th>Manufacturer</th>
<th>purity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Activated carbon</td>
<td>Vulcan XC-72R</td>
<td>United States Cabot Corporation</td>
<td></td>
</tr>
<tr>
<td>Palladium chloride</td>
<td>PdCl₂</td>
<td>Chinese medicine Chemical reagents</td>
<td>analytical grade</td>
</tr>
<tr>
<td>Anhydrous copper sulfate</td>
<td>CuSO₄</td>
<td>Shanghai Qiang Shun Chemical Reagent Co., Ltd.</td>
<td>analytical grade</td>
</tr>
<tr>
<td>Ferrous sulfate</td>
<td>FeSO₄·7H₂O</td>
<td>Shanghai Shanhai Mission Experimental Plant</td>
<td>analytical grade</td>
</tr>
<tr>
<td>Sodium borohydride</td>
<td>NaBH₄</td>
<td>Shanghai layer Chemical Co., Ltd.</td>
<td>analytical grade</td>
</tr>
<tr>
<td>Anhydrous ethanol</td>
<td>C₂H₅OH</td>
<td>Chinese medicine Chemical reagents</td>
<td>analytical grade</td>
</tr>
<tr>
<td>Sodium hydroxide</td>
<td>NaOH</td>
<td>Chinese medicine</td>
<td>analytical grade</td>
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</table>
### Chemical reagents

<table>
<thead>
<tr>
<th>Reagent</th>
<th>Description</th>
<th>Source</th>
<th>Grade</th>
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<tr>
<td>Nafion solution</td>
<td>5% Nafion</td>
<td>United States</td>
<td>DuPont company analytical grade</td>
</tr>
<tr>
<td>sulfuric acid</td>
<td>H₂SO₄</td>
<td>Laiyang fine chemical plant</td>
<td>analytical grade</td>
</tr>
<tr>
<td>hydrochloric acid</td>
<td>36% HCl</td>
<td>Tianjin Guangcheng Chemical Reagent analytical grade</td>
<td></td>
</tr>
<tr>
<td>Formic acid</td>
<td>HCOOH</td>
<td>Chinese medicine</td>
<td>Chemical reagents analytical grade</td>
</tr>
</tbody>
</table>

#### 2.2.2 Preparation of catalyst

The preparation of carbon-supported ternary catalysts is shown in Figure 2.1.
The carbon supported Cu-Fe alloy template was prepared by the sodium borohydride reduction method we had used before [52, 56]. CuSO$_4$ and FeSO$_4$ were used as precursors of Cu and Fe, respectively. The atomic ratios of Cu and Fe in Cu and Fe alloys are Cu: Fe = 10: 0, 9: 1, 8: 2 and 7: 3, respectively.

The alloy template was etched in a 20 mmol L$^{-1}$ PdCl$_2$ solution at room temperature for 3 hours. The PdCl$_2$ solution was formulated with 0.2 mol L$^{-1}$ HCl. Adjust the pH to about 7 before use. The catalyst was filtered and washed thoroughly with three times distilled water. Finally, the catalyst was vacuum dried at 130 °C for 6 hours. For ease of discussion, the catalysts obtained by etching Cu$_{10}$Fe$_0$, Cu$_9$Fe$_1$, Cu$_8$Fe$_2$ and Cu$_7$Fe$_3$ are named NPCu$_{10}$Fe$_0$, NPCu$_9$Fe$_1$, NPCu$_8$Fe$_2$ and NPCu$_7$Fe$_3$, respectively. The Pd / C catalyst was prepared using sodium borohydride reduction. PdCl$_2$ is a precursor of Pd. The method of washing and drying is the same as the above-mentioned ternary catalyst.

2.2.3 Electrochemical test method

All electrochemical tests were performed in a three-electrode system by CHI832B electrochemical workstation. High purity carbon rod serve as the Counter electrode, Hg/HgSO$_4$ electrode as the reference electrode. 10 mg of the catalyst powder was added to 0.5 mL of ethanol and 0.5 mL of water, and the 20 minutes’ ultrasound for forming a homogeneous suspension. 20 μL (5 μL × 4 times) suspension was added dropwise to a 3 mm diameter glassy carbon electrode surface. The glassy carbon electrode was polished with 0.05 μm Al$_2$O$_3$ powder before use and rinsed with three times distilled water. After the catalyst was dried naturally, 2 μL of Nafion solution (5wt%) was added dropwise to the electrode. Cyclic voltammetry was performed in 0.5 mol L$^{-1}$ H$_2$SO$_4$ solution. The linear sweep voltammetry curve and the time current curve were carried out in 0.5 mol L$^{-1}$ H$_2$SO$_4$ and 0.5 mol L$^{-1}$ HCOOH solution. The potentials in this paper are converted to standard hydrogen electrodes. The electrolyte solution was purged with N$_2$ for 20 minutes before testing, and the electrolyte was kept in the N$_2$ atmosphere during the test.
2.3 Results and discussion.

2.3.1 Characterization of the catalyst.

Figure 2.2 shows the XRD patterns of Pd-Cu-Fe/C and Pd/C catalysts. In the figure, the peak of about 24.8° on the spectrum of all the catalysts is the peak of C(002). (111), (200), (220) and (311) planes of Pd are about 40°, 46°, 68° and 82°, respectively. There is a relatively slow diffraction peak of Cu₂O near 35° [51]. No peaks on the Fe element were found in the figure, and this phenomenon also appears in some Pd-Fe electrocatalysts report[41, 57-58]. They point out that this is due to the heavy atom effect of Pd [41, 59], which forms Pd-coated Fe nanoparticles. The Pd-Cu-Fe catalyst is more complex than the binary catalyst, and the partially dissolved Cu²⁺ can be reduced by Fe on the surface. The catalyst was dried at 130℃, Pd, Cu, Fe three elements will spread each other, forming Pd-Cu-coated Cu-Fe nanoparticles of the boundaries are not obvious in the layers Pd-Cu and Cu-Fe. The linear position in the graph is the position of the diffraction peak of Pd in the Pd/C catalyst. The movement of the Pd peak position also indicates the formation of the alloy. The particle diameters of the particles can be calculated from the Hiller formula[60]. The calculated diameters of Pd/C, NPCu₁₀Fe₀, NPCu₉Fe₁, NPCu₈Fe₂ and NPCu₇Fe₃ catalysts are 5.0 nm, 4.9 nm, 4.4 nm, 4.8 nm and 4.5 nm, respectively.
Figure 2.2 The XRD pattern of (a) Pd / C, (b) NPCu_{10}Fe_{10}, (c) NPCu_{9}Fe_{1}, (d) NPCu_{8}Fe_{2} and (e) NPCu_{7}Fe_{3}. The straight line is the diffraction peak position of Pd in Pd/C catalyst.

2.3.2 Electrochemical test and morphology analysis of the catalyst.

Figure 2.3 shows the cyclic voltammograms of Pd-Cu-Fe/C and Pd/C catalysts in 0.5 mol L\(^{-1}\) sulfuric acid. The peak in the range of 0 to 0.4 V is the desorption peak of hydrogen, and the peak after 0.5 V is the oxidation of the metal and the reduction peak of the oxide. The current density in the electrochemical curve is based on the electrochemically active area. The desorption peak of hydrogen is usually used to calculate the electrochemically active area [62-63]. However, since hydrogen easily enters the lattice of Pd, it becomes difficult to calculate the electrochemically active area using the desorption peak of hydrogen [64]. In this paper, the electrochemical active area is calculated by the reduction peak of Pd oxide [65]. The charge density of single layer PdO is 424 \(\mu\)C cm\(^{-2}\). The electrochemical active area of the Pd-based catalyst can be obtained using the following formula [66]

\[
\text{ECSA} = \frac{Q_R}{Q_{MR}}
\]  

(2.4)

\(Q_R\) is the reduced power of PdO, obtained by the reduction peak of PdO in the cyclic voltammogram in the sulfuric acid. \(Q_{MR}\) is a single layer PdO with a charge density of 424 \(\mu\)C cm\(^{-2}\).
Figure 2.3 The cyclic voltammetry curves of (a) Pd/C, (b) NPCu$_{10}$Fe$_0$, (c) NPCu$_9$Fe$_1$, (d) NPCu$_8$Fe$_2$ and (e) NPCu$_7$Fe$_3$ catalysts in the solution of 0.5 mol L$^{-1}$ H$_2$SO$_4$. Scan speed 10 mV s$^{-1}$.

The electrochemical properties were observed by linear scanning voltammetry curves of catalyst in 0.5 mol L$^{-1}$ HCOOH and 0.5 mol L$^{-1}$ H$_2$SO$_4$ solution. Figure 2.4 shows the linear canned voltammograms of each catalyst in 0.5 mol L$^{-1}$ H$_2$SO$_4$ + 0.5 mol L$^{-1}$ HCOOH solution. It can be seen that there is a large peak in the range of 0.2-0.4 V. There is an indistinct slow peak in the range of 0.7-0.9 V. The peak in the range of 0.2-0.4 V is a direct oxidation peak of formic acid, which does not have CO toxicity. The peaks at 0.7-0.9 V are attributed to the oxidation of CO. The CO accumulated in the process of formic acid oxidation may be derived from the indirect oxidation of formic acid or the reduction of surface-generated CO$_2$. Based on the formation of CO in the electrooxidation of formic acid on Pd-based catalysts, some scientists have carried out spectroscopic studies. These spectroscopic studies [96-98] show that the reduction of CO$_2$ from formic acid dehydrogenation is the main source of CO. However, some literature suggests that the oxidation of formic acid through both the direct route and the CO pathway [23-34, 37, 70]. Based on the theory in the literature, we believe that part of the CO is obtained by the reduction of CO$_2$ from the oxidation product of the formic acid, since Ojani [39] pointed out that the oxidation of formic acid on the Pd-Cu porous catalyst produces CO intermediates by dehydration. The peak potential of NPCu$_7$Fe$_3$, NPCu$_9$Fe$_1$, NPCu$_8$Fe$_2$ and NPCu$_{10}$Fe$_0$ catalysts were negative shift by 110 mV, 88 mV, 51 mV and 46 mV, respectively, and the current densities were 48.3%, 27.9%, 15.6% and 17.2% higher than that of Pd/C. The peak of the NPCu$_7$Fe$_3$ catalyst is sharp, indicating that there are more accessible active sites on the catalyst [71]. The NPCu$_7$Fe$_3$ catalyst exhibits the highest catalytic activity in some catalysts.
Figure 2.4 shows the Linear scan voltammetry curve of (a) Pd/C, (b) NPCu_{10}Fe_0, (c) NPCu_9Fe_1, (d) NPCu_8Fe_2 and (e) NPCu_7Fe_3 catalyst in 0.5 mol L\(^{-1}\) H\(_2\)SO\(_4\) + 0.5 mol L\(^{-1}\) HCOOH solution; scan speed: 10 mV s\(^{-1}\).

Figure 2.5 shows the time current curves for the Pd/C and NPCu_7Fe_3 catalysts in 0.5 mol L\(^{-1}\) H\(_2\)SO\(_4\) + 0.5 mol L\(^{-1}\) HCOOH solution at an operating voltage of 0.3 V. The stable current density of NPCu_7Fe_3 catalyst is higher than that of Pd/C, which indicates that it has high stability.
2.4 Conclusion

In general, Pd-Cu-Fe/C ternary catalysts can be prepared by partial etching of alloy. The resulting ternary catalyst has a higher catalytic activity of formic acid. The Pd-Cu-Fe/C catalyst obtained from Cu_7Fe_3/C template exhibits the highest catalytic activity. [72]

Bibliography


