Praparation of CuO with Wing-Like Morphology of Cicada and Its Absorption Properties to Organic Volatiles

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Abstract: In order to obtain some advanced functional materials with unique morphology for nanoelectronic/microelectronic device applications or catalysis fields, a study on preparation of CuO with controlled morphologies under hydrothermal reaction condition was carried out. The experimental results showed that CuO nanosheet with wing-like morphology of cicada was obtained. Series of examinations were performed with TEM (transmission electron microscopy), XRD (X-ray diffraction), and so on. A chemical prototype sensor was constructed based on CuO nanosheet with wing-like morphology of cicada and highly sensitive quartz resonators. The gas-sensing behavior of the sensor to some common organic volatiles (such as toluene, formaldehyde, chloroform, alcohol vapor and other some common organic volatiles) operating at room temperature was investigated. The results showed that the CuO with unique structure morphology would have many important potential applications in chemical sensor to organic volatiles or catalysis fields.

Keywords: CuO; wing-like morphology of cicada; hydrothermal preparation; chemical sensors; absorption properties; organic volatiles

蝉翼状 CuO 纳米片的制备及其对有机挥发份气体的吸附特性研究

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摘　要：为获得特有形貌纳/微米结构的功能材料用于有机物的催化氧化、有机物的催化降解、电子器件应用等领域，采用水热方法制备了可控形貌的 CuO，并用所制得的 CuO 材料构筑了 QCM 结构化学传感器原型器件。采用透射电镜（TEM），X-射线衍射（XRD）等进行了相应表征，对器件的吸附响应性能进行了初步考察。透射电镜结果显示， 所制得的 CuO 为蝉翼状纳米片形貌。该形貌的 CuO 对常见的有机挥发份气体（如：甲苯，甲醛，氯仿，乙醇等挥发份气体）显示出明显的吸附响应特性。这些考察结果表明，该形貌的 CuO 材料由于具有高的比表面积和不同的活性场所等将在化学传感和催化等领域有重要的潜在应用。

关键词：蝉翼状 CuO；水热制备；化学传感；吸附特性；有机挥发份

1 引言

由于纳、微米结构的材料具有一些独特的功能性能，研究纳、微米结构功能材料的制备，性能考察及其应用是当前的一大研究热点[1-16]，其中太阳能电池，催化领域，化学传感，生物传感等[6-22]方面备受广泛关注。

在研究新材料的制备、改性、加工工艺及其性能考察中，考虑到很多材料的性能强烈依赖于其形貌，因此，为获得优异性能的这些新材料，重要的是制备高比表面

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积的多种功能材料及其先进的制备技术等。迄今，对这些热点材料的研究大都集中在：金属氧化物，碳纳米管，有机功能材料及有机-无机复合功能材料等[15,16,18-24]，其中，无机功能材料是一大类重要的多功能材料，对其研究涉及面较广，多数集中在 TiO_2, ZnO, CuO, CdS 等体系的方方面面。

在如此众多的无机功能材料中，CuO是一类重要的有代表性的多功能材料，在有机催化反应、有机物的催化降解等领域有重要的应用。为改善CuO材料的性能，对CuO材料的形貌开展了大量的研究与调控工作。如：Zhou and co-workers [1]通过管状糖脂类化合物的自组装制备了可控CuO纳米线覆盖物的纳米电缆阵列。Tan and co-workers [2]报道了一简便的溶液方法制备Cu_2O单晶纳米线，该纳米线具有可控的直径、不同的形貌和高的长径比。SU and co-workers [25]用多孔氧化铝模板和溶胶-凝胶方法合成了高度有序的CuO纳米线阵列。Wu and co-workers [26]采用阳极氧化法制备了氢氧化铜纳米针、纳米管阵列。Jana and co-workers [3]在碱性溶液中氧化铜泊室温下获得了纳米结构的CuO。Kajari and co-workers [27]采用溶剂热途径制备了Cu_2O纳米线，并考察了其荧光和光电导性能。UV－vis结果显示出明显的蓝移；光电导比达16。Huang and co-workers [28]研究了单根锥形CuO纳米线的荧光性能。Chen and co-workers [29]通过碱辅助氧化技术获得了$Cu(OH)_2$纳米管阵列，采用表面处理实现了由超疏水到超亲水的转变。Jang and co-workers [4]采用软模板自组装的方式大量制备了CuO和ZnO纳米片，提供了一简便的制备方法。Liu and co-workers [30]采用聚乙二醇还原途径制得了Cu_2O纳米线阵列。Zhu and co-workers [5]采用Cu-Zn合金直接氧化共合成了ZnO－CuO纳米结构复合材料。Liu and co-workers [31]合成了CuO/Fe_2O_3杂化微孔球。Derekaya and co-workers [32]制备了CeO_2负载的CuO，该复合材料可选择性地氧化CO。Barreca and co-workers [33]研究了负载的Cu_2O， CuO纳米系统用于光催化产生H_2。Wang and co-workers [34]制备了Ag/CuO纳米复合材料，并开展了系列表征和催化性能的研究，结果显示，该纳米复合材料对分解H_2O_2具有高的催化特性。Patake and co-workers [35]采用电化学方法沉积了多孔和非晶CuO薄膜，并用于超级电容器的制作。Zhang and co-workers [36]采用低温水热途径高产率地制备出CuO纳米带，该纳米带显示出高的放电电容量。Chen and co-workers [6]采用垂直CuO纳米线阵列构筑化学传感器，并考察了其对H_2, CO, H_2S, NH_3气体的敏感响应，显示出对H_2S气体的高选择性。Kim and co-workers [7]采用CuO纳米线气体传感器进行了车内空气质量的控制。Jia and co-workers [8]采用直接加热途径得到了CuO纳米线垂直阵列，该阵列可用作很好的电化学传感器平台，CuO纳米线电极显示出对H_2O_2极好的电催化响应、高的灵敏度、快速响应，且大大降低了其氧化-还原的过电位。Hansen and co-workers [9]报道了大面积、低成本途径制备超高致密的CuO纳米线阵列。该纳米线传感器显示出对NO_2和NH_3气体的高敏感性，同时对白光有强的响应。Hoa and co-workers [10]合成了多孔CuO纳米线，并研究了其在测定氢中的应用。Zhang and co-workers [11]采用种子调控生长途径外延生长了CuO纳米线阵列，并构筑了生物传感器，可用于测定H_2O_2 和葡萄糖。同时研究了不同形貌的CuO生物传感器的敏感性能[12]。

为获得室温敏感、快速响应的化学传感器，我们曾采用软模板途径对导电聚合物进行了系列形貌调控和改性研究，通过掺杂、有机-无机杂化复合等技术途径，取得了一些有意义的结果[20,21]。为拓展材料研究领域，作者采用水热法制备了 TiO_2 纳米线，并考察了其模拟化学战剂的敏感响应性能[22]。考虑到超支化结构材料具有高的比表面积和相互贯穿的电荷传输通道，我们通过水热法制备了超支化结构的 CdS，构筑了 QCM 结构的化学传感器原型器件，并对代表性的有机挥发性气体的室温敏感行为进行了考察，取得了一些有意义的结果。为积累研究数据、比较不同材料、不同形貌对材料界面吸附响应特性的影响，便于有机物的催化反应、有机物的催化降解、化学传感等领域应用借鉴，同时探讨材料的制备、工艺及性能相关性，便于指导新材料的设计与性能调控，在此报道蝉翼状 CuO 纳米片的制备及其对有机挥发份气体的吸附特性研究结果。

2 实验部分

2.1 原材料

硫酸铜($CuSO_4\cdot5H_2O$, AR)；六次甲基四胺($N_4(CH_3)_6$, AR)。

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2.2 CuO的水热制备

将硫酸铜与六次甲基四胺以适当的摩尔比混合于内衬为聚四氟乙烯容器中，加去离子水至填充度为80的高压釜中，在120-130℃下反应10-20 h， 然后冷却至室温。对其用去离子水洗涤4-6 次,并在室温下自然干燥，待测定。

2.3 透射电镜观察

透射电镜观察采用 JEM-1230，操作电压为 160 kV。样品分散在去离子水中，采用挂铜网的办法观察。

2.4 XRD 表征

采用日本岛津 XRD-6100X 射线衍射仪(工作电压为 40KV， 工作电流时 30mA；用单色 CuKa 射线照射，扫描范围 10°-80°），对产物进行结构粉末衍射表征。

2.5 QCM结构化学传感器原型器件的构筑及其吸附响应特性的表征

考虑到 QCM（石英晶体谐振器）具有超敏感特性，文献曾报道可以用于称量单个原子的重量。为此，我们选用 QCM 结构构筑化学传感器原型器件用以表征所设计的材料对有机挥发份气体的吸附特性，这将对有机物的催化反应、有机物的催化降解和传感器等领域有参考价值。QCM（石英晶体谐振器）结构化学传感器原型器件的构筑及其敏感性的表征参见文献［22］。敏感性的测量采用吸附后引起谐振差频（测量沉积 CuO 的石英晶体谐振器和空白石英晶体谐振器的差频对应吸附气体量的变化）的变化来表征。

3 结果与讨论

本研究采用水热方法制得的 CuO 形貌见图 1。

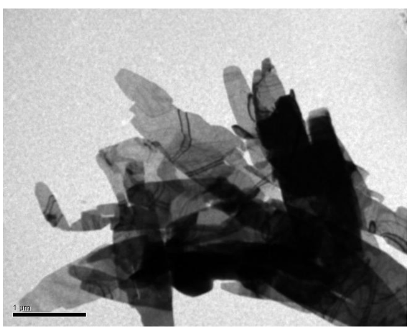

图 1 采用水热方法制得的 CuO 透射电镜形貌。

Figure1. The TEM image of CuO obtained.

这种蝉翼形状 CuO 的 XRD 结果见图 2。

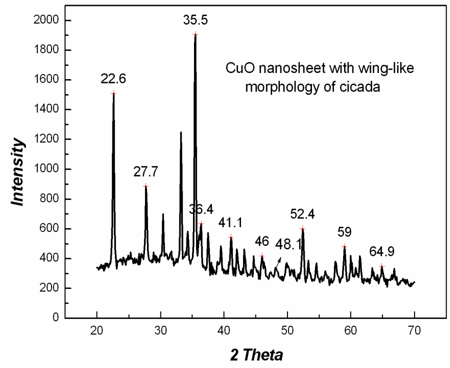

图 2 蝉翼形状 CuO 的 XRD 结果。

Figure 2. The XRD results of CuO nanosheet with wing-like morphology of cicada.

由图 2 可见，这种蝉翼形状 CuO 的 XRD 其 2θ 在 35.5， 46， 48.1， 53.3， 61.3， 66.6 等处有较强的衍射峰，属于单斜晶相的 CuO。

此外，由于本研究所制得的 CuO 为蝉翼形状结构，这种特有的形貌暗示着将具有高的比表面积和良好的吸附性能，在多种有机物的催化降解，催化氧化，超敏感化学传感器、太阳能电池等领域有潜在的重要应用。考虑到石英晶体谐振器的超敏感吸附响应性，为此，我们构筑 QCM（石英晶体谐振器）结构化学传感器原型器件，并选择有代表性的有机挥发份进行了性能考察，结果见图 3-7。

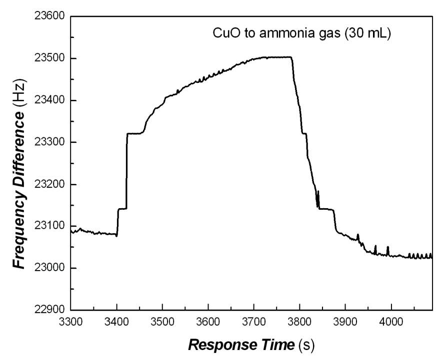

图 3 在浓度 30 ml / 1000 mL 饱和氨气气氛时蝉翼形状 CuO 的气敏响应曲线。

Figure 3. Response behavior of CuO nanosheet with wing-like morphology of cicada to 30 mL saturated ammonia vapor diluted in a 1000 mL chamber with N_2.

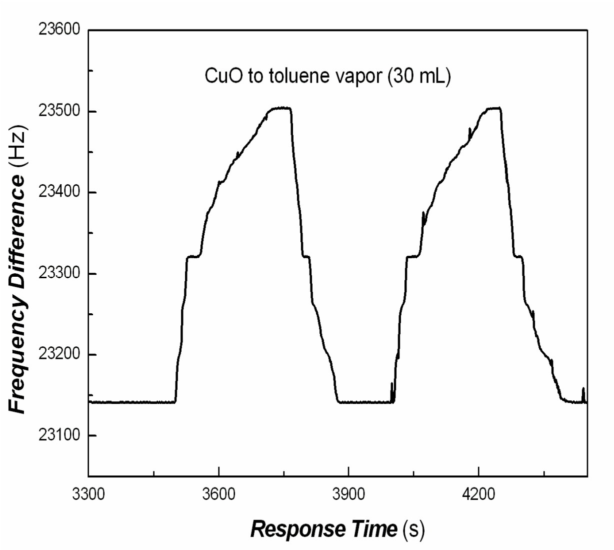

图 4 在浓度 30 ml / 1000 mL 饱和甲苯气氛时蝉翼形状 CuO 的气敏响应曲线。

Figure 4. Response behavior of CuO nanosheet with wing-like morphology of cicada to 30 mL saturated toluene vapor diluted in a 1000 mL chamber with N_2.

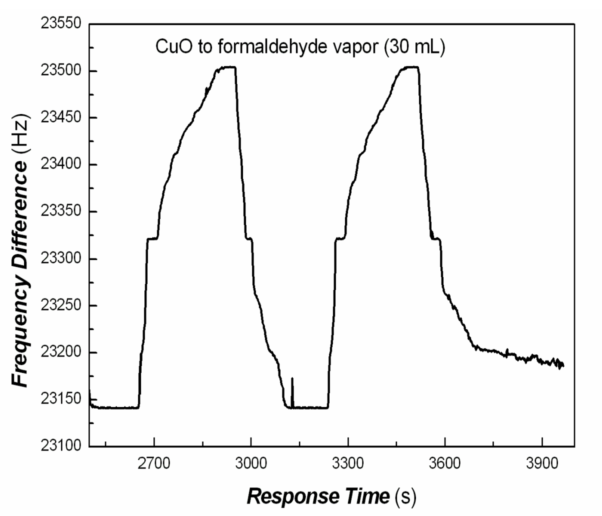

图 5 在浓度 30 ml / 1000 mL 饱和甲醛气氛时蝉翼形状 CuO 的气敏响应曲线。

Figure 5. Response behavior of CuO nanosheet with wing-like morphology of cicada to 30 mL saturated formaldehyde vapor diluted in a 1000 mL chamber with N_2.

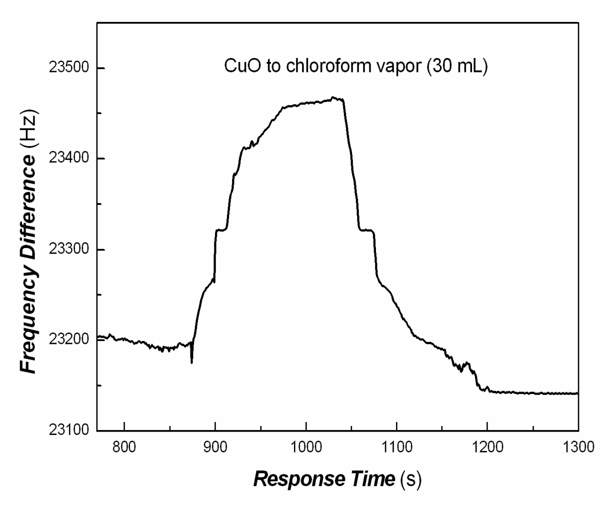

图 6 在浓度 30 ml / 1000 mL 饱和氯仿气氛时蝉翼形状 CuO 的气敏响应曲线。

Figure 6. Response behavior of CuO nanosheet with wing-like morphology of cicada to 30 mL saturated chloroform vapor diluted in a 1000 mL chamber with N_2.

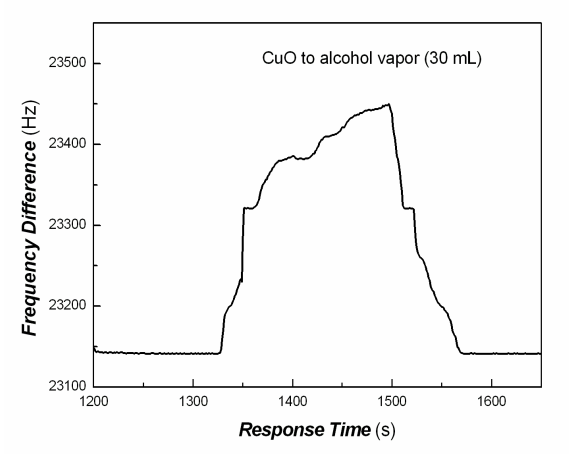

图 7 在浓度 30 ml / 1000 mL 饱和乙醇气氛时蝉翼形状 CuO 的气敏响应曲线。

Figure 7. Response behavior of CuO nanosheet with wing-like morphology of cicada to 30 mL saturated alcohol vapor diluted in a 1000 mL chamber with N_2.

对不同有机挥发份和不同浓度的有机挥发份气氛下蝉翼形状 CuO 化学传感器原型器件吸附响应特性也进行了考察，其结果见图 8，9。

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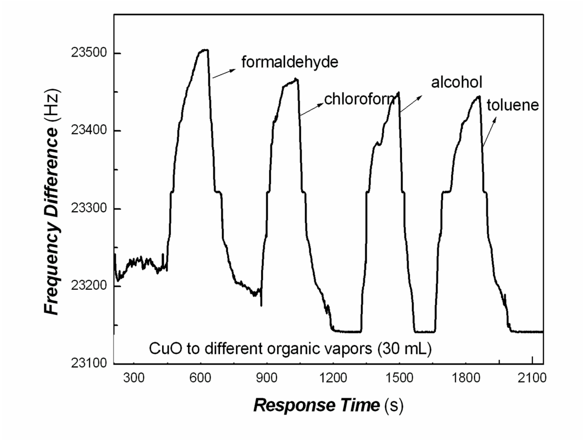

图 8 在浓度 30 ml / 1000 mL 不同有机挥发性气体气氛时蝉翼形状 CuO 的气敏响应曲线。

Figure 8. Response behaviors of CuO nanosheet with wing-like morphology of cicada to different 30 mL saturated organic vapors diluted in a 1000 mL chamber with N_2.

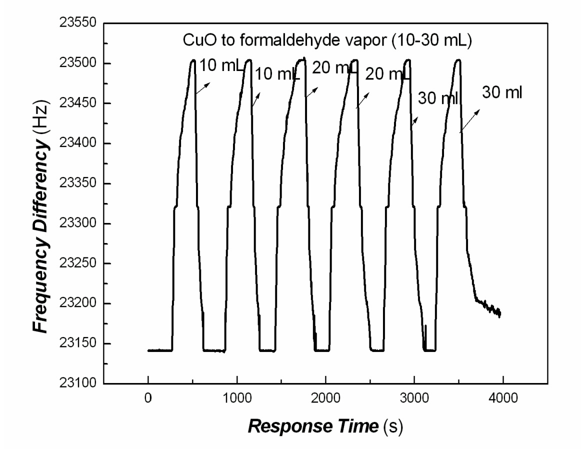

图 9 不同浓度甲醛气体对蝉翼形状 CuO 的气敏响应性能的影响。

Figure 9. Effects of concentrations of formaldehyde vapor on the response behaviors of CuO nanosheet with wing-like morphology of cicada.

由图 8 可见，本研究所制得的蝉翼形状 CuO 对不同有机挥发份气体的吸附有很大的类似性，说明选择性较差，活性较高，有待进一步改性。

由图 9 可见，本研究所制得的蝉翼形状 CuO 化学传感器原型器件在实验浓度范围内对有机挥发份的响应存在的浓度依赖性不显著，有待进一步降低浓度进行考察，这也预示着该材料将具有低的测定限。另外，本研究所制得的蝉翼形状 CuO 对不同有机挥发份气体的动态吸附响应较好。

4 结 论

总之，采用水热方法制备了蝉翼形状结构的CuO，并用所制得的CuO材料构筑了QCM结构化学传感器原型器件。采用透射电镜（TEM）， X-射线衍射（XRD）等进行了相应表征，对器件的室温吸附响应性能进行了考察。研究结果表明，该蝉翼形状结构的CuO对常见的有机挥发份气体（如：甲苯，甲醛，氯仿，乙醇等）显示出明显的特有的吸附响应特性。这些考察结果显示，该形貌的CuO材料由于具有高的比表面积和不同的活性场所将在有机物的催化反应、有机物的催化降解、化学传感和其他电子器件等领域有潜在广泛的应用。

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