Nanocrystalline and Amorphous Tungsten Oxide Powder Prepared by Gas Condensation Technique

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Inert gas condensation technique has been used widely to prepare nanocrystalline powders including metals, alloys, and ceramics. For instance, nanocrystalline tungsten oxide powders can be prepared by gas condensation and may exhibit unique gas-sensing properties. The grain size of the nanocrystalline tungsten oxide powders can be manipulated by processing parameters or post heat treatment.

In the present study, nanosized amorphous tungsten oxide powders were synthesized by a two-step oxidation and gas condensation process. Pure tungsten raw materials were first oxidized at 1000 °C in a furnace and then put into a gas condensation high vacuum chamber to produce nanosized tungsten oxide powders. Gas sensing properties of tungsten oxide films prepared by corresponding powders were investigated. Experimental results showed that tungsten oxide powders prepared by two-step gas condensation method consisted of nanosized amorphous aggregates. While those prepared by direct gas condensation was nanocrystalline. Conventional XRD patterns were shown in Fig. 1. Figure 2 shows the in situ XRD patterns of the amorphous powders that transformed into WO₃ phase after heat treating them at 400°C.

Synchrotron X-ray absorption spectroscopy studies was used to evaluate these tungsten oxide nanomaterials. Figure 3 shows XANES spectra at W $L_{\rm III}$ edge of as-prepared amorphous, nanocrystalline tungsten oxide and tungsten trioxide. The larger the edge position reveals the higher valence of the tungsten oxide.

Though not shown here, when tested under 5 ppm NO₂ gas, the sensitivity was ~69.9 at 125 °C and decreased to 1.1 at 240 °C. When tested at 100 °C, it reached a high sensitivity of 53.6 and 129.4 for 1 ppm and 3 ppm NO₂, respectively. The sensitivity decreased to ~37.7 after 10 repetitive gas adsorption-desorption cycles. The gas sensing ability can be recovered by heating at 300 °C. Nano-sized amorphous tungsten oxide films exhibited not only good gas sensing ability but stability.

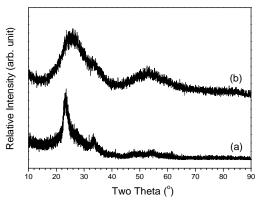


Fig. 1: XRD patterns of crystalline and amorphous tungsten oxide powders.

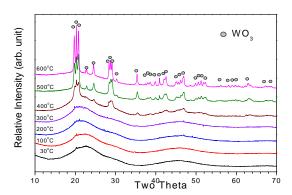


Fig. 2: In situ XRD patterns of amorphous tungsten oxide powders.

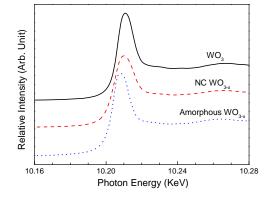


Fig. 3: XANES spectra at W $L_{\rm III}$ edge of as-prepared amorphous, nanocrystalline tungsten oxide and tungsten trioxide.