Properties of $ZnMoO_4 \cdot 0.8H_2O$ synthesized by ultrasonic method

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Background

The $ZnMoO_4 \cdot 0.8H_2O$ has used as anode material for lithium ion batteries. The $ZnMoO_4 \cdot 0.8H_2O$ is a precursor of the $ZnMoO_4$ that is perspective material for bolometers, scintillation detectors, humidity sensors, microwave dielectric devices, battery electrodes and high effective catalyst oxidation ethanol to acetaldehyde. The traditional method of synthesis of the $ZnMoO_4 \cdot 0.8H_2O$ is based on the interaction of soluble salts: $Na_2MoO_4 \cdot 2H_2O$ and $Zn(NO_3)_2 \cdot 6H_2O$ in aqueous solution. Traditional synthesis demand a lot of water, characterized more long synthesis time and more expensive raw materials, but don't guarantee pure product.

Experimental conditions

The dynamics formation phase $ZnMoO_4 \cdot 0.8H_2O$ from oxides ZnO and MoO_3 by ultrasonic treatment shows in this poster. The mechanical mixture of oxides ZnO and MoO_3 with molar ratio 1:1 was used as raw material. Ultrasonic treatment (UST) was carried out USDN-A (УЗДН-А) frequency: 22 kHz, reaction medium – water at room temperature. Time of treatment was 5, 10 and 20 min. After US treatment samples were dried at 100°C and characterized by XRD, adsorption of N_2, SEM, TEM, thermal programmed reducing in a H_2-Ar mixture (TPR-H_2) in the temperature range of 30-800°C at a heating rate of 10 °C/min and thermal programmed heat in Ar (measurement error is 0.5%).

Properties of initial oxide mixture ZnO and MoO_3

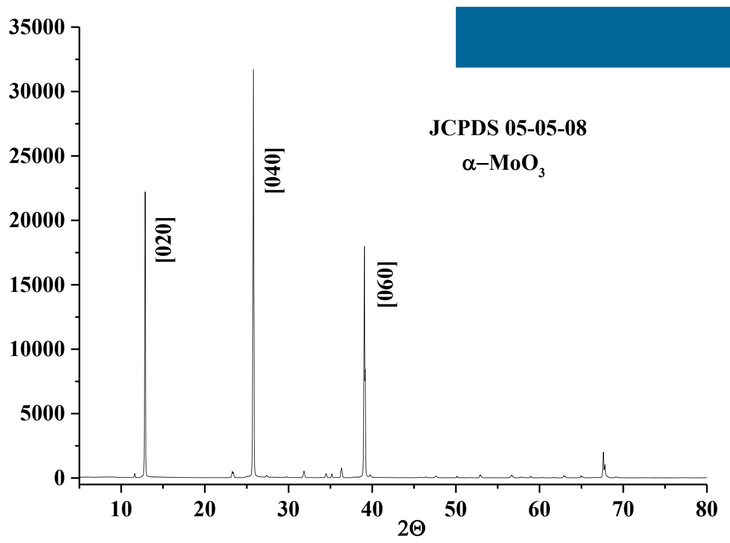

Fig. 1. XRD data of initial oxide mixture ZnO/MoO_3

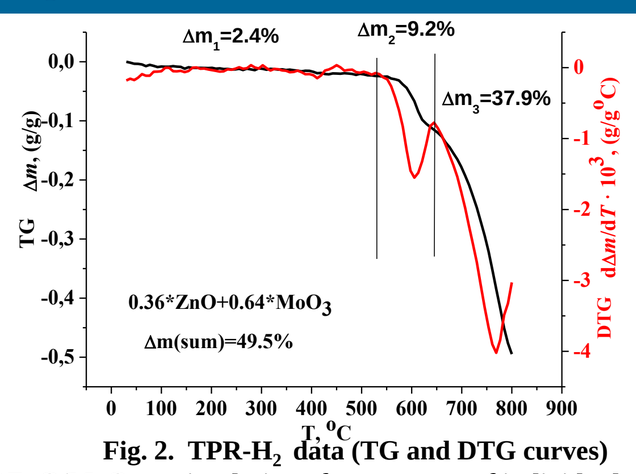

Fig. 2. TPR-H_2 data (TG and DTG curves) of ZnO/MoO_3 as simulation of sum curves of individual oxides for equimolar mixture

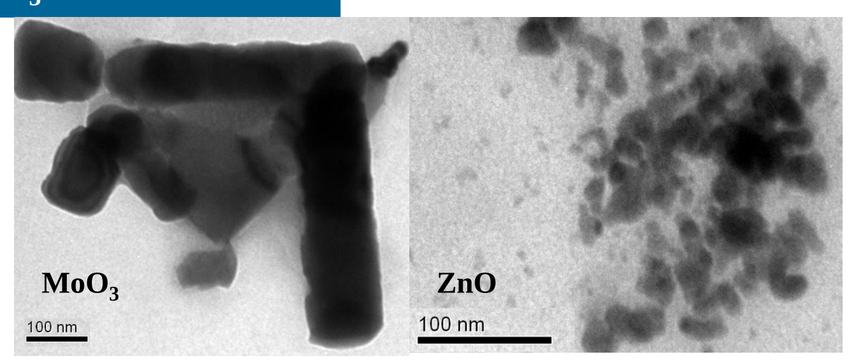

Fig. 3. TEM of individual oxides MoO_3 and ZnO

Properties of ZnO/MoO_3 after ultrasonic treatment during 5 minutes

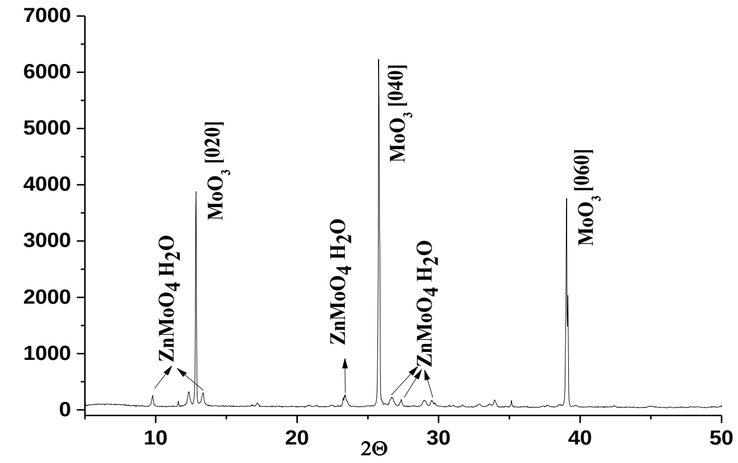

Fig. 4. XRD data of ZnO/MoO_3 UST _5

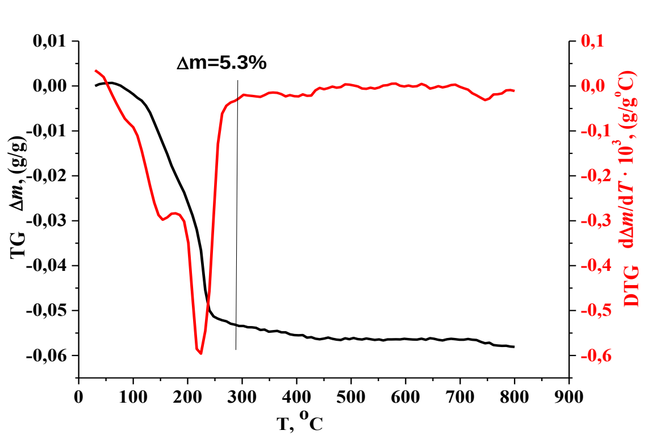

Fig. 5. TG and DTG curves of ZnO/MoO_3 UST_5 in Ar

After US treatment of oxides during 5 minutes XRD patterns (Fig. 4) represent weak diffraction peaks of the $ZnMoO_4 \cdot 0.8H_2O$ phase and strong diffraction peaks of initial MoO_3. However, TP heating in an Ar medium (Fig. 5) shows the weight loss up to T-300 °C is 5.3%, that corresponds to the loss of hydration water. Theoretical calculation of the weight loss effect of hydration water for the pure $ZnMoO_4 \cdot 0.8H_2O$ is 6%. Consequently, the content of initial oxides is 22%, and $ZnMoO_4 \cdot 0.8H_2O$ is 88% for sample of ZnO/MoO_3 UST_5.

Properties of ZnO/MoO_3 after ultrasonic treatment during 10 minutes

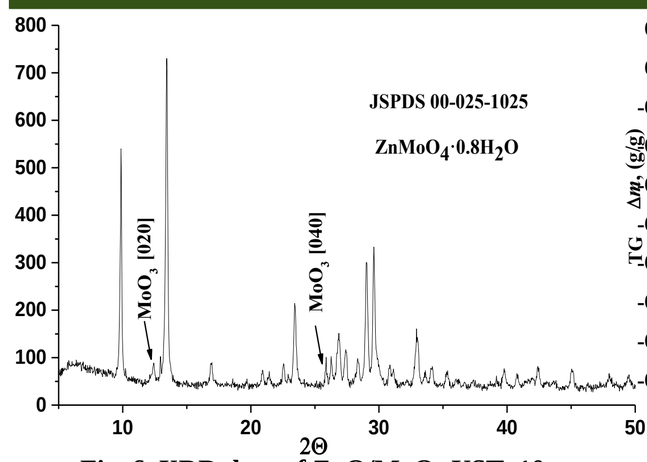

Fig. 6. XRD data of ZnO/MoO_3 UST_10

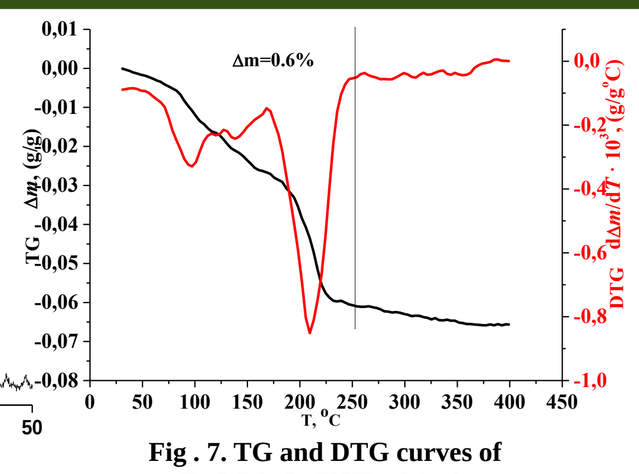

Fig . 7. TG and DTG curves of ZnO/MoO_3 UST_10 in Ar

Fig. 8. TEM ZnO/MoO_3 UST_10

After US treatment of oxides during 10 minutes was formed well crystallized phase $ZnMoO_4 \cdot 0.8H_2O$ with barely noticeable diffraction peaks of MoO_3. TEM data confirm that particles with the structure shown in Fig. 3, disappeared and nanostructures 100 nm wide and more than 2 µm long were formed (Fig. 8). The thermal programmed heating this sample in an Ar medium accords pure phase $ZnMoO_4 \cdot 0.8H_2O$ (Fig. 7).

An increase the UST time to 20 minutes leads to the fact that the diffraction peaks of MoO_3 completely disappear (Fig. 9). CEM data demonstrate morphology like fabric that composed from long interweaving nano-filamentary structures (Fig. 12) .

Properties of ZnO/MoO_3 after ultrasonic treatment during 20 minutes

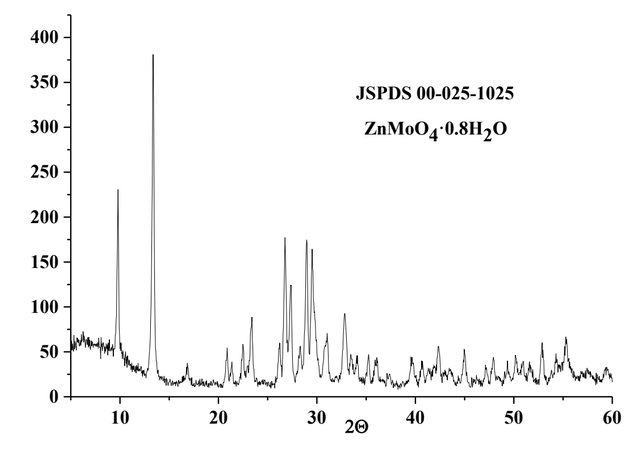

Fig. 9. XRD data of ZnO/MoO_3 UST_20

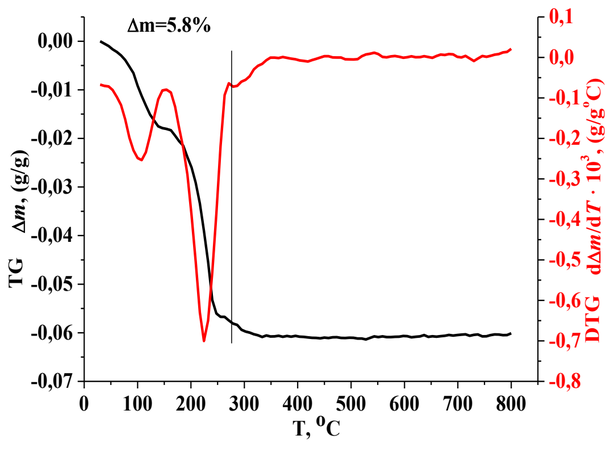

Fig. 10.TG and DTG curves of ZnO/MoO_3 UST_20 in Ar

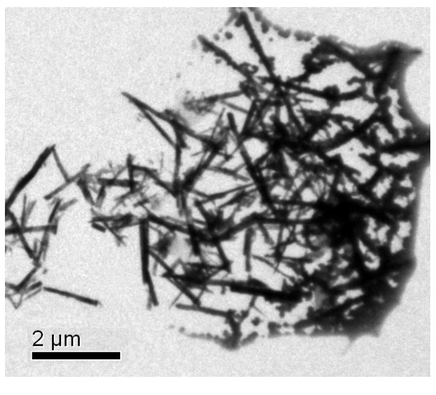

Fig. 11. TEM ZnO/MoO_3 UST_20

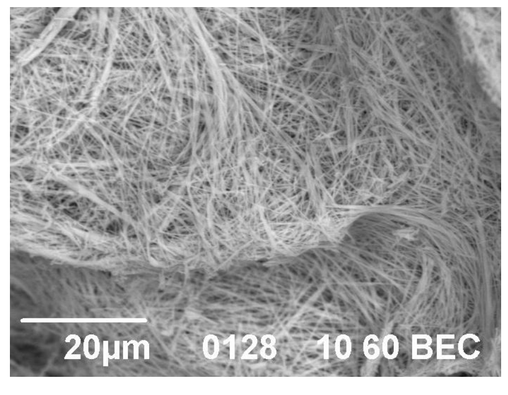

Fig.12. SEM ZnO/MoO_3 UST_20

Conclusions

Sonochemical synthesis allows to create a pure and nanostructured $ZnMoO_4 \cdot 0.8H_2O$ phase using cheap oxides ZnO and MoO_3 as raw material just in 20 minutes. The proposed US synthesis is eco-friendly, because by-products are not formed in the synthesis process in contradistinction to traditional synthesis. Moreover, water was used in synthesis can be recycling without cleaning. In addition, $ZnMoO_4$ can be obtained from $ZnMoO_4 \cdot 0.8H_2O$ avoiding high temperatures and long-lasting synthesis.