

Exfoliated graphite decorated with ZrO₂ nanoparticles

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Abstract – Exfoliated graphite decorated with zirconium oxide particles has been synthesized through electrochemical oxidation of natural graphite in aqueous zirconyl nitrate/nitric acid solutions followed by a heat treatment. ZrO₂ is mainly formed as nanoparticles of 10-20 nm diameter though sometimes associated in 200 nm agglomerates. These nanoparticles are linked together to form ZrO₂ particles of ~ 5µm diameter distributed preferentially at the graphite grain boundaries of the exfoliated graphite support.

Carbon composites with improved mechanical and thermal properties are appealing materials for high temperature applications. Herein we report the synthesis and characterization of exfoliated graphite modified by ZrO₂ which is widely used as high-temperature material.

Expandable graphite was synthesized by anodic polarization of graphite at stabilized current with a total electrical charge transfer of Q=500-1500 C/g. Aqueous zirconyl nitrate/nitric acid solutions of various concentrations: C ZrO(NO₃)₂=1-20%, C NO₃⁻_{total}=17-60% - were used as electrolytes. According to X-ray diffraction data the phase composition of all the expandable graphite samples is represented by graphite and ZrO(NO₃)₂·6H₂O whereas the wide halo attributed to (001) reflection of graphite oxide is additionally detected in the samples obtained at Q>1000 C/g. SEM experiments revealed that 5-10 µm ZrO(NO₃)₂·6H₂O particles of various shape are randomly distributed on the surface but concentrated preferentially at the edges of the expandable graphite particles.

Thermal exfoliation of expandable graphite samples in air at 600 °C resulted in formation of exfoliated graphite containing *t*-ZrO₂ (*EG*_{ZrO₂}). The properties of *EG*_{ZrO₂}, i.e. its bulk density (1.4-6 g/l), the content of ZrO₂ (6-34 wt. %) and specific area (20-120 m²/g) strongly depend on the expandable graphite synthesis conditions. The increase of both total NO₃⁻ concentration in the electrolyte and the electrical charge transferred through it lead to reduction of the bulk density of later on obtained *EG*_{ZrO₂}. The increase of ZrO(NO₃)₂ concentration in the electrolyte with C NO₃⁻_{total}=60% resulted in the increase of ZrO₂ content in *EG*_{ZrO₂} which is accompanied by the reduction of its specific area attributed to the influence of ZrO₂ nanoparticles filling the pores of the exfoliated graphite support.

TEM experiments revealed that ZrO₂ is mainly formed as nanoparticles of 10-20 nm diameter though some of them are associated in 100-300 nm agglomerates (Fig.1). These nanoparticles are linked together to form ZrO₂ particles of ~ 5µm diameter distributed preferentially at the graphite grain boundaries of the exfoliated graphite support which is in a good agreement with SEM investigation of expandable graphite. We believe that the predominant covering of *EG*_{ZrO₂} grain boundaries by ZrO₂ particles may contribute to the formation of a ceramic network within the exfoliated graphite matrix after proper thermal treatment resulting in enhancement of its mechanical properties.

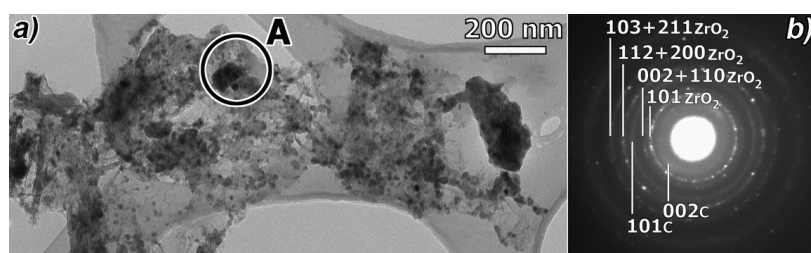


Figure 1: TEM image of ZrO₂ particles loaded on the surface of expanded graphite (a) and the diffraction pattern taken from the area A (b).