Synthesis of ZrC-TiC nanostructures

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Materials science and engineering has experienced a tremendous growth in the field of development of nanostructured materials with enhanced chemical, mechanical, and physical properties.

The binary solid carbide mixtures of (ZrC–TiC) are of special interest for ultrahigh temperature applications due to their refractoriness, high hardness, low thermal expansion, good thermal shock resistance and chemical stability at high temperatures. Formation of the heterogeneous finely dispersed structures in ZrC–TiC mixtures also are of a great interest because their possible contribution into development of superplastic ceramic-based composites [1-5]. Conventionally, the binary solid carbide compounds are synthesized by carbothermal reduction of metal oxides (ZrO_2 and TiO_2) and amorphous carbon mixture in controlled atmospheres at a temperature between (1700 – 2100 ^oC). The conventional method is energy and time consuming (10 – 24h) and a final product suffers from impurities and is inhomogeneous because the powders are mixed on a relatively coarse scale (e.g., micrometer-scale) [5-7].

In our work we use combination of sol-gel method and conventional carbothermal reduction to synthesize nanopowders for preparation of binary carbides material. Combination of these offers some advantages compared to conventional powder processing, such as the lower reaction temperatures/ shorter reaction times due to the intimate contact of the reactants.

The present study is novel way for synthesis of the mixed ZrC–TiC powders precursor from corresponding metal alkoxides by using of sol-gel method. The main advantage of the used sol-gel process is the reduction of the kinetic barriers between the formed metal oxide and the carbon particles created in pyrolysis of metal alkoxide polymer due to the homogeneous dispersion of reactants in the precursor material. The increased contact area of the nanograins results in carbothermal reduction between the metal oxide and carbon particles at lower temperature and shorter time as compared to conventional carbide synthesis methods. Moreover, the use of molecular precursors and the control of the synthesis conditions make possible to prepare homogeneous and pure multicomponent systems.

Also, we clarify the mechanism of carbothermal reduction of the binary solid carbides precursors in argon and vacuum environments. Titanium and zirconium carbides blend were synthesized by carbothermal reduction from polymer precursor of ZrC–TiC at 1500 ^oC in an argon atmosphere and vacuum. The resulting products had small crystallite size (~40 nm Fig.1.). However, these products contained little ZrO_2.

The structural transformation of the polymeric materials into the carbides was characterized by sacning electron microscope (SEM), X – ray diffraction analysis and Raman spectroscopy. Characterizations of heat treated samples at 800 ^oC to 1500 ^oC in argon and vacuum has showed that the carbothermal reduction of the binary solid carbide mixture (ZrC–TiC) polymeric precursor began in vacuum at lower temperature (1100 ^oC Fig. 2.) than in argon environment.

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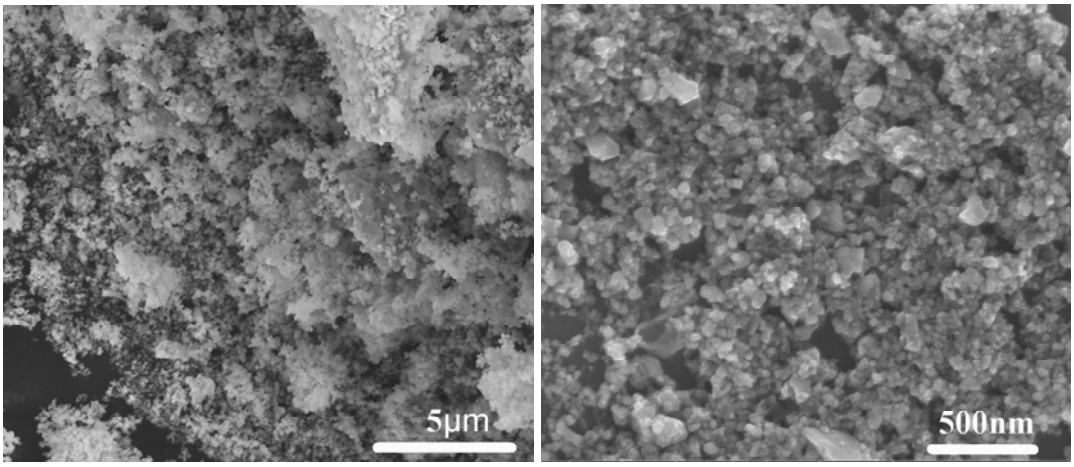

Figure.1. . SEM images of synthesized typical ZrC – TiC powders at 1500 ^{o}C at various

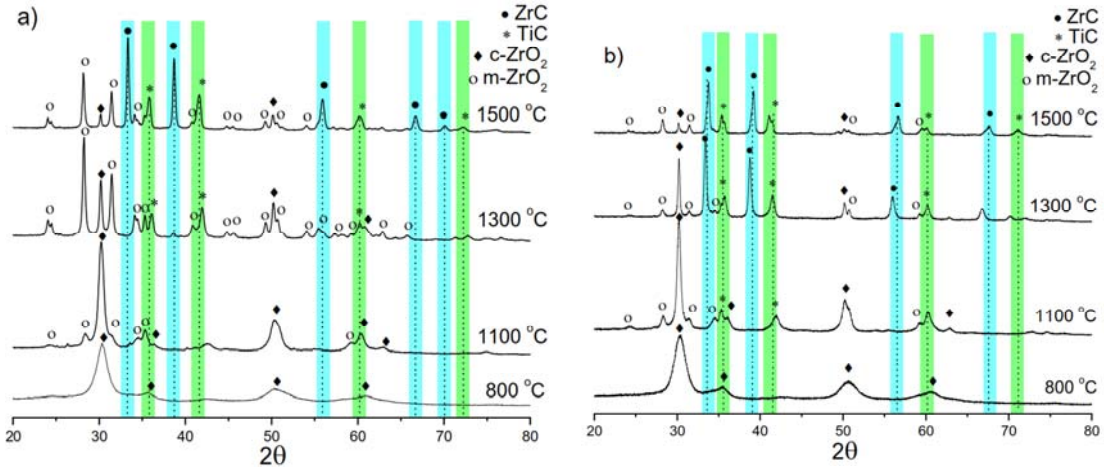

Figure. 2. X-ray diffraction patterns of a) ZrC-TiC polymeric precursor annealed in argon; b) ZrC-TiC polymeric precursor annealed in vacuum.