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Crystal growth of TiC grains during SHS reactions

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Abstract

The microstructure formation of TiC during self-propagating high-temperature synthesis (SHS) reactions is investigated thanks to the quenching method. The morphology and chemical composition of titanium carbide grains are characterized, during the growth phase, from X-ray diffraction (XRD), scanning electron microscopy (SEM) and wavelength dispersion spectroscopy (WDS) analyses. It is demonstrated that TiC_x grains nucleate with a great deficit in carbon; the carbon composition evolves towards the stoichiometry during the growth phase. The TiC grain sizes are then coupled to thermal data acquired by infra red (IR) thermography. It is shown that the convolution of the temperature and of the time elapsed in the temperature range $T_{max}-1000\,K$ constitute a pertinent parameter to give account of the variation of TiC grain sizes.

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1. Introduction

Gasless combustion is one of the most interesting variations of self-propagating high-temperature synthesis (SHS). In this case, the initial reactants, intermediate, and final products remain in the condensed state (liquid or solid) during the reaction [1,2]. The reaction is ignited by heating a small region of the sample to the ignition temperature. As a result, the heat input required to ignite the reaction is relatively low. The SHS reaction, once ignited, propagates itself from one side to the opposite one without any other energy supply. While this synthesis method is very attractive from the energy point of view, a great number of industries is still reluctant to invest in this process as it is rather difficult to control the microstructure formation.

Since the end of the 1980s, most research articles, related to this topic, are focused on the morphology and microstructure control of SHS products (grain size, particle shape, etc.) [3–6]. Among these studies, the synthesis of nanomaterials becomes a particularly promising center of interest [7–13]. Nevertheless, for SHS to be used safely in the industry and to synthesize nanomaterials with controlled properties, microstructure and nanostructure formations have to be precisely understood. In recent articles proposed by Khina [14] and Locci [15], the validity of theoretical models of TiC nucleation and TiC growth mechanisms have been checked. In particular, the limit of applicability of the so-called ''solid–solid–liquid'' mechanisms is discussed in view of experimental data related to diffusion processes and thermal profiles. As a result, it is shown that a direct contact between a liquid phase containing dissolved reactants (Ti with dissolved carbon) and solid reactant (carbon) is needed for the reaction to be self-propagated. It is shown that TiC nuclei mainly results from heterogeneous nucleation. In Locci's paper, a model is built to simulate TiC formation kinetics during SHS reactions. An estimation of the solid–liquid interfacial energy is reached; its numerical value is close to the energy

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