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An Impregnated Active Layer Mode of Solution Combustion Synthesis as a Tool for the Solution Combustion Mechanism Investigation

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Abstract : Solution combustion synthesis (SCS) is the unique method which multiple times has proved itself as an effective and efficient approach for the versatile synthesis of a variety of materials. It has significant advantages such as relatively simple handling process, high rates of product synthesis, mixing of the precursors on a molecular level, and fabrication of the nanoproducts as a result. Nowadays, an overwhelming majority of solution combustion investigations performed through the volume combustion synthesis (VCS) where the entire liquid precursor is heated until the combustion self-initiates throughout the volume. Less amount of the experiments devoted to the steady-state self-propagating mode of SCS. Under the beforementioned regime, the precursor solution is dried until the gel-like media, and later on, the gel substance is locally ignited. In such a case, a combustion wave propagates in a self-sustaining mode as in conventional solid combustion synthesis. Even less attention is given to the impregnated active layer (IAL) mode of solution combustion. An IAL approach to the synthesis is implying that the solution combustion of the precursors should be initiated on the surface of the third chemical or inside the third substance. This work is aiming to emphasize an underestimated role of the impregnated active layer mode of the solution combustion synthesis for the fundamental studies of the combustion mechanisms. It also serves the purpose of popularizing the technical terms and clarifying the difference between them. In order to do so, the solution combustion synthesis of γ-FeNi (PDF#47-1417) alloy has been accomplished within short (seconds) one-step reaction of metal precursors with hexamethylenetetramine (HTMA) fuel. An idea of the special role of the Ni in a process of alloy formation was suggested and confirmed with the particularly organized set of experiments. The first set of experiments were conducted in a conventional steady-state self-propagating mode of SCS. An alloy was synthesized as a single monophasic product. In two other experiments, the synthesis was divided into two independent processes which are possible under the IAL mode of solution combustion. The sequence of the process was changed according to the equations which are describing an Experiment A and B below: Experiment A: Step 1. $Fe(NO_3)_3*9H_2O$ + HMTA = FeO + gas products; Step 2. FeO + $Ni(NO_3)_2*6H_2O$ + HMTA = Ni + FeO + gas products; Experiment B: Step 1. $Ni(NO_3)_2*6H_2O$ + HMTA = Ni + gas products; Step 2. Ni + $Fe(NO_3)_3*9H_2O$ + HMTA = Fe_3Ni_2+ traces (Ni + FeO). Based on the IAL experiment results, one can see that combustion of the $Fe(NO_3)_39H_2O$ on the surface of the Ni is leading to the alloy formation while presence of the already formed FeO does not affect the $Ni(NO_3)_2*6H_2O$ + HMTA reaction in any way and Ni is the main product of the synthesis.

Keywords : alloy, hexamethylenetetramine, impregnated active layer mode, mechanism, solution combustion synthesis
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