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In situ synchrotron investigation of $MoSi_2$ formation mechanisms during current-activated SHS sintering

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Abstract

The development and control of the mechanically activated spark plasma sintering (MASPS) process is the main objective to be achieved in producing bulk nano-organized materials with perfectly controlled composition and microstructure. Consequently, it was essential to develop original experiments, such as time resolved X-ray diffraction with the X-ray synchrotron beam (D2AM-BM2, ESRF Grenoble) coupled to IR thermography to monitor in situ structural and thermal evolution during the current-activated SHS sintering process in the Mo–Si system. A new sample holder was designed in order to reproduce the synthesis conditions of the MASPS process, obviously without the consolidation step. The versatility of the set-up and the new possibilities offered by the design of new sample holders allow a new interpretation of $MoSi_2$ formation mechanisms during current-activated SHS sintering.

Keywords: Bulk molybdenum disilicide; Ball milling; Self-propagating high-temperature synthesis; Phase transitions

1. Introduction

Molybdenum disilicide ($MoSi_2$) is a promising material for high-temperature applications. It has a high melting point (2030 °C), high hardness and good oxidation resistance compared with other refractory silicides and intermetallic compounds [1]. Thus, this material is used mainly for heating elements and coating materials on molybdenum or other refractory metals. Many techniques have been developed to produce nanostructured massive molybdenum disilicide, such as solid-state reactions [2], spray forming [3] and mechanical alloying [4,5]. The large negative formation enthalpy of $MoSi_2$ ($\Delta H_f = -131.8 \pm 8.4$ kJ mol^{-1} [6]) allows the synthesis of this compound by self-propagating high-temperature synthesis (SHS) from a mixture of Mo and Si powders. In this technique, a sample is made by cold compaction of a stoichiometric elemental powder mixture. This green pellet is heated on one side; a reaction is first initiated in a small volume and then gradually spreads through the sample. When the reaction is initiated, a combustion front with a heating rate ranging from 800 to 3300 °C s^{-1} travels through the whole sample and leads to the formation of the expected compound. In addition, in the SHS process, the time required for the synthesis is much shorter than that of other powder metallurgy techniques [7–9].

Nevertheless, to obtain a dense material for high-temperature applications, a consolidation step [10,11] has to be added. Prior work on the consolidation of nanopowders obtained by mechanical alloying aimed to accomplish this challenge [12]. The mechanically activated spark plasma sintering (MASPS) process seems to be more attractive than the others for the preparation of intermetallic compounds, combining, at the same time, SHS reactions and

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