

Combustion Synthesis of Multiphase Metal Borides and Silicides

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ABSTRACT

Formation of niobium silicides (Nb-Si), niobium borides (Nb-B) and molybdenum silicides (Mo-Si) was investigated by self-propagating high-temperature synthesis (SHS) in this study. Effects of sample green density, preheating temperature, and starting stoichiometry on the combustion characteristics, as well as on the composition of final products were studied. In the first part of this study, production of niobium silicides (Nb₃Si, Nb₅Si₃, Nb₅Si₃, and NbSi₂) and Nb₅Si₃/Nb composites was conducted from elemental powder compacts. XRD analysis showed that the compact with a composition of Nb:Si = 5:3 yielded predominantly Nb₅Si₃, whereas the sample made up of Nb:Si = 3:2 produced largely Nb₅Si₃. The sample of Nb:Si = 1:2 produced NbSi₂ with some Nb and Si left unreacted. Experimental observations indicated that except for the sample of Nb:Si = 3:1, upon ignition self-sustained combustion was well established and proceeded throughout the entire sample. Moreover, the reactant compact of Nb:Si = 5:3 had the highest flame-front propagation velocity, followed sequentially by the powder compacts with Nb:Si = 3:2, 1:1, and 1:2. Variation of the combustion temperature with sample initial stoichiometry is in a manner consistent with that of the flame-front velocity. In the synthesis of Nb₅Si₃/Nb composites, the increase of elemental Nb content in the final composition lowered the combustion temperature and thereby reduced the flame-front velocity. Nb₅Si₃/Nb composites with the concentration of Nb ranging from 5 to 15 mol.% were produced by SHS in this study. XRD analysis of the Nb₅Si₃/Nb composite identifies the formation of Nb₅Si₃ dominated by the Nb₅Si₃ form, along with the existence of elemental Nb. Based upon the data measured combustion temperature and combustion wave velocity, the activation energies associated with combustion synthesis of Nb₅Si₃ and NbSi₂ were determined to be 259.2 and 160.8 kJ/mol, respectively. In the second part of this study, a comparative study on the preparation of specific niobium borides (including Nb₃B₂, NbB, Nb₅B₆, Nb₃B₄, and NbB₂) in the Nb-B system was conducted from elemental powder compacts of their corresponding stoichiometries. Reactant compacts of Nb:B = 1:1 and 1:2 were shown to yield single-phase NbB and NbB₂, respectively. In contrast, multiphase products consisting of Nb₃B₄, NbB, and NbB₂ were produced from the powder compacts with Nb:B = 3:2, 3:4 and 5:6. However, it was found that two boride phases Nb₃B₂ and Nb₅B₆ did not appear in the end products from any of the reactant compacts. Combustion of the samples with Nb:B = 3:2 was characterized by a localized reaction zone propagating along a spiral trajectory, due largely to the low combustion temperatures which further resulted in a poor degree of phase conversion with a significant amount of Nb left unreacted. Based upon the measured combustion temperature and combustion wave velocity, the activation energies associated with combustion synthesis of NbB and NbB₂ were determined to be 151.8 and 132.4 kJ/mol, respectively. In the third part of this study, production of molybdenum silicides (Mo₃Si, Mo₅Si₃, and MoSi₂) was conducted from elemental powder compacts. Reactant compacts of Mo:Si = 1:2 were shown to yield single-phase MoSi₂. However, reactions were extinguished on the compacts of Mo:Si = 3:1 and 5:3. Multiphase products consisting of Mo₅Si₃, MoSi₂ and unreacted Mo were produced from the powder compacts with Mo:Si = 2:3 and 1:1. Measured results showed that the reactant compact of Mo:Si = 1:2 and 2:3 had higher flame-front propagation velocities than that of Mo:Si = 1:1. The activation energy 34.9 kJ/mol was determined for MoSi₂.

Keywords : Nb-Si, Nb-B, Mo-Si, Self-propagating High-temperature Synthesis, Activation Energy, Preheating, XRD

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