Melting metallurgy processes of NiTi shape memory alloys can lead to undesirable contamination of the melt and forming of oxide inclusions. One possible solution of this problem is reactive sintering using pure elemental powders. Reactive sintering method enables to produce high-purity materials. In this process, compressed mixture of metallic powders is transformed to bulk intermetallic phases via thermally activated exothermic reactions. The evolved heat sustains and helps to propagate the reaction front through the body of the reactants. Therefore this process is called “Self-sustainable High-temperature Synthesis” (SHS). This work aims to optimize the parameters of the SHS process for the preparation of NiTi shape memory alloy to obtain a high-purity, low-porosity material.

Results revealed that heating rate strongly affects the structure of this alloy. Using slow heating (20 °C.min⁻¹) leads to extremely heterogeneous structure composed of various Ni-Ti phases and high porosity (Fig. 1). The microstructure consisting of NiTi and Ti₉Ni phases can be obtained in this material by rapid heating (approx. over 300 °C.min⁻¹) (Fig. 2). Sufficient reactive sintering temperature to obtain NiTi phase is 900 °C. At 800 °C, the structure composed of Ni and Ti elemental powder particles was observed (Fig. 3). No Ni-Ti intermetallic phase was observed after sintering at 800°C. Further increase of the SHS initiation temperature to 1100 °C reduces porosity and the amount of the Ti₉Ni phase. The lowest porosity and lowest amount of the undesirable Ti₉Ni phase were achieved by the utilization of coarse titanium powder (200-600 µm) with fresh surface, produced by mechanical machining, or by the increase of nickel content in the alloy (Fig. 4). However, the change of the nickel content will affect the transformation temperatures and induce the formation of Ni₄Ti₃ phase. Reactive sintering process is completed during less than 20 min in the investigated material. It was proved that the formation of Ti₉Ni phase always accompanies this process, even though its content can be minimized by the proper choice of the SHS parameters. To eliminate this phase, further thermal or thermo-mechanical treatment will be required.

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Fig. 1: Microstructure of Ni-50at%Ti alloy prepared by SHS process at 1100°C for 20 min, heating rate of 20 °C.min⁻¹, Ti particle size < 10 µm, light microscopy.

Fig. 2: Microstructure of Ni-50at%Ti alloy prepared by SHS process at 1100°C for 20 min, heating rate > 300 °C.min⁻¹, Ti particle size < 10 µm, light microscopy.

Fig. 3: Microstructure of Ni-50at%Ti alloy prepared by SHS at 800°C for 20 min, heating rate > 300 °C.min⁻¹, Ti particle size < 10 µm, light microscopy.

Fig. 4: Microstructure of Ni-50at%Ti alloy at 1100°C for 20 min, heating rate > 300 °C.min⁻¹, Ti particle size 200 - 600 µm, light microscopy.