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Effect of Reaction Atmosphere and Heating Rate during Reactive Sintering of Ni–Ti Intermetallics

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Abstract

The effect of reaction atmosphere and heating rate during reactive sintering of nickel and titanium powder blend (Ni: 52 at.%, particle size <150 μm, >99.99% purity, Ti: 48 at.%, particle size <44 μm, >99.5% purity) were studied. The powder mixtures were uniaxially compressed under pressure of 637 MPa and sintered at temperatures 500, 650, 900 and 1100°C in air, vacuum, argon and nitrogen atmospheres. Two heating rates were applied: 200 and >300 $K.min^{-1}$. The results show that a significant effect of reaction atmosphere in terms of the quantity of the Ti_2Ni phase applies especially at very high heating rate. In terms of phase morphology, oxygen causes refinement of the structure, regardless of heating rate. Mechanism and kinetics of the reactions leading to Ni–Ti phases were determined by DTA and thermogravimetry. It was found that the Ti_2Ni phase forms already at 500 °C. At 650 °C this phase undergoes a reaction with nickel to NiTi and Ni_3Ti.

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Keywords: Intermetallic compounds; Powder metallurgy; Electron microscopy; Microstructure; Thermal analysis; Thermogravimetry

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1. Introduction

Solid-state reactive sintering is a cost effective method of synthesis of ceramics, composites and many intermetallic compounds [1-3]. One of its advantages is high purity of the synthesized material when pure powders and protective atmosphere are used. Often, reactive sintering results in porous products [3] owing to initial pores of the powder compact prior to the combustion reaction, the Kirkendall effect due to the difference in diffusion rates between the elemental powder components [4] and changes in the crystal lattice. Porous materials can be used in the production of orthopedic implants [5].

The purpose of this paper is to describe the effect of reaction atmosphere and heating rate during thermal explosion reactive sintering of Ni-Ti intermetallics.

2. Experimental details

Cylindrical shape green bodies (∅ 10 mm × 5 mm) were prepared by uniaxial cold pressing of the blends of 52 at.% of Ni powder (> 99.99% purity, particle size < 150 μm) and 48 at.% of Ti powder (> 99.5% purity, particle size < 44 μm) under pressure of 637 MPa during 5 minutes. Approximately 80 mg of green bodies was mechanically separated for differential thermal analysis (DTA) and thermogravimetry, carried out using the SETSYS Evolution-1750 device by heating from the laboratory temperature to 1200 °C with the heating rate of 30°C.min^{-1} and alumina crucible in various atmospheres.

The bulk green body samples were sintered at temperatures 500, 650, 900 and 1100°C in a resistance heating furnace in air or in evacuated silica ampoules with high heating rate of > 300°C.min^{-1}. The lower heating rate 200°C.min^{-1} was achieved in an induction furnace where sintering was performed in air, argon or nitrogen atmospheres. Microstructure of sintered samples was examined by TESCAN VEGA 3 scanning electron microscope equipped with OXFORD Instruments X-max EDS SDD 20 mm^2 detector (SEM-EDS). The microscope was mostly operated in backscattered electron signal (BSE) in order to distinguish phases in the composition contrast. Prior the microstructure observation, samples were mechanically ground, polished and etched using modified Kroll's reagent (10ml HF, 40 ml HNO_3 and 50 ml H_2O). Image analysis to evaluate porosity and area fraction of residual titanium were carried out by means of ImageJ software.

The phase analysis was carried out by X-ray diffraction (XRD) method using PANalytical X'Pert Pro diffractometer (CuKα radiation). The XRD patterns were processed and evaluated by PANalytical X'Pert HighScore Plus software with PDF-2 database.

3. Results and discussion

3.1. Differential thermal analysis

Differential thermal analysis (DTA) curves, as well as corresponding thermogravimetric curves of the green body compacts heated from the ambient temperature up to 1200°C in argon, air and argon, air and nitrogen atmospheres are in Fig. 1. If oxygen is present in the atmosphere (air and argon, air) a small exothermic peak is present on blue and red curves in Fig. 1a at the temperature of 530°C. This peak is due to the formation of the Ti_2Ni phase [6] which often contains oxygen [7,8]. At this temperature, the formation of Ti_2Ni phase is diffusion controlled. Owing to the presence of oxygen, the NiTi phase forms already at the temperature of about 750°C (the highest peaks in Fig. 1a). On the other hand in argon and nitrogen atmospheres the low temperature formation of the Ti_2Ni phase is suppressed and this phase forms at 950°C altogether with the NiTi phase as it follows from corresponding peaks on green and brown curves in Fig. 1a and electron microscopy micrographs in Fig. 3. At this temperature the phases form by a different mechanism, i.e. self-propagating high temperature synthesis (SHS). The thermogravimetric curves (Fig. 1b) indicate that during heating of the samples in the atmosphere containing oxygen the surface oxidation starts at about 600°C. In the presence of nitrogen, a TiN layer starts to form at about 800°C. In argon atmosphere almost no mass change was measured through the whole temperature range of heating (Fig. 1b).