Intermetallic Phase Formation during Combustion Synthesis of Mechanically Activated Ni-Ti Alloy

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Abstract: Both mechanical activation (MA) and pre-heating conditions affect intermetallic phase formation during combustion synthesis (CS) of Ni-Ti equiatomic alloy. Input parameters are pre-heating temperature, activation duration and compaction pressure. Output variables are ignition time, temperature rise, reaction duration, product microstructure, constituent oxidation and intermetallic phase formation. Superelasticity and shape memory behaviors depend on austenite-rhombohedral and rhombohedral-martensite transformation temperatures. These temperatures were detected by differential scanning calorimetry (DSC) measurements. The phase changes were studied by scanning electron microscopy (SEM) and x-ray diffraction (XRD) analysis. Production of Ni_3Ti was detected when CS was performed on 1800 s milled sample pre-heated to 400°C. Compression tests indicated appearance of superelastic and superelastic/thermoelastic behaviors after CS of (a) 3600 s activated and (b) 7200 s activated samples at 400°C pre-heating, respectively. It was concluded that pre-combustion heating fostered mono-phase formation and titanium oxidation; while mechanical activation lowered ignition-start-time, duration of synthesis and Ni_3Ti formation.

Keywords: Combustion Synthesis, Intermetallics, Ni-Ti Alloy, Oxidation

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1 INTRODUCTION

In NiTi production, homogeneity and mono-phase formation need delicate control of the production procedure, environment and holder composition/ cleanliness due to reactivity of titanium with oxygen, nitrogen and carbon and great tendency of Ni and Ti for formation of intermetallic phases like Ni_3Ti and $NiTi_2$ [1-3]. The consequences of the structural heterogeneity of the matrix (caused by compositional effects, thermal and/or thermomechanical treatment steps and by the presence of impurities) in the transformation behavior of NiTi alloys and the effect of the presence of impurities, like oxygen and nitrogen, in the mechanical activation process evolution of Ni-Ti powder mixtures on properties of the product have been discussed by previous authors [4], [5].

Desirable properties like shape memory, superelasticity, bio-compatibility, bio-adaptation, resistance against corrosion and damping effect are bound to precise compositional control and mono-phase formation which depend on production procedure and subsequent thermo-mechanical treatments [6-10]. Combustion synthesis (CS) is a simply manageable fast process leading to contamination-free NiTi sample [11-15]. In bone implantation, large interconnected pores provide suitable medium for live tissue growth [12], [14]. Combustion synthesized (CS) scaffolds are especially beneficial because of their considerable intrinsic pores [16].

CS is achievable in two ways: self-propagation high-temperature synthesis (SHS) or thermal explosion (TE) [17]. The former belongs to the highly exothermic reaction of the constituents; while the latter is mainly due to the reactions having low heat-effects. The Ni-Ti combustion is categorized as a TE type process due to its insufficient heat of reaction. Pre-heating of NiTi is, hence, required for start of the CS reaction. Once the reaction starts, it would continue to the combustion end.

There are several methods for measurement of the transformation temperatures. The simplest method is DSC which measures the heats absorbed and released during cooling and warming of the samples. Because of absorption and release of the heat during martensite-austenite and reverse transformations, downward and upward peaks appear in the DSC curves of the NiTi system. Start and finish temperatures of the peaks show initiation and ending of the transformations. Tangent method can be used to evaluate the characteristic time and temperature in a CS process and, simultaneously, can be used in DSC to evaluate the characteristic peaks of the austenite and martensite transformation [18].

Both shape memory and superelastic behaviors depend on the matrix composition and intermetallics presence [19]. For a specified composition, the most appropriate tuning way for property change is aging heat treatment [19], [20]. Transformation temperatures can be altered by chemical composition, thermo-mechanical treatment, aging and mechanical activation [19-21]. Determination of the optimum conditions for production of the homogeneous NiTi mono-phase with both superelastic and/or thermoelastic effects are of considerable practical interest aimed to be approached in this research.

Changes of the transformation temperatures, duration of the combustion and the maximum achievable temperature are important parameters to be determined from the temperature profile of the combusting samples. Applying tangent method to the temperature-time diagram of the sample yields the specific CS times and temperatures [22]. The objective of the research is to determine the effects of pre-heating and mechanical activation on intermetallic phase formation and property change during combustion synthesis of the compacted equiatomic Ni-Ti mixtures. Results of morphological observations and compression tests are compared to determine the effect of pre-heating and the milling time on behavior of the CS specimens.

2 EXPERIMENTAL PROCEDURE

Equiatomic nickel powder (diameter: 15 µm, purity: 99.9%) and titanium particles (diameter: 80 µm, purity: 99.99%) were charged into cylindrical bottles and rotated three axially by an electrical machine for 10,800 s to obtain uniform mixtures. Mixed powder batches were used for ball milling, combustion synthesis and mechanical and morphological investigations. CS was performed on powders both un-milled and milled for 1800, 3600 and 7200 s. Mechanical activation was done by planetary ball milling at 250 rpm under argon protection. The powder to ball ratio was 1:40. Two ball diameters of 7.5 and 4.8 mm were used. The milling process was interrupted periodically (15 min interruption after each 15-min activation) to avoid excessive heating of the milling jars.

Powder samples were pressed at 150 MPa to form 17 mm diameter round pills. The pills were pre-heated to 350, 400, 500 and 600°C in a resistance furnace. The bead of a K-type thermocouple was located at the center of each pill to measure the change of the specimen temperature. A data acquisition device was used to help precise determination of temperature. All three milled batches of the samples were subjected to the same compaction conditions and heating cycles.

A small segment of each pill (25-50 mg) was cut-off and used for transformation detection by DSC measurements. Temperature rate during the tests was 10°C/min. X-ray diffraction (XRD) and scanning

electron microscopy (SEM) were used to determine the phases present in the samples. Compression tests were performed on cylindrical samples having 12 mm length and 6 mm diameter at initial strain rate of 3.33×10^{-3} s^{-1} using Zwick/roell (HCT 400/25) material testing equipment. Table 1 summarizes the experimental conditions used for different samples.

Table 1 Summary of the experimental conditions for different samples.

Sample No.	Milling Time (s)	Pre-heating Temperature (°C)
1	-	400
2	-	500
3	-	600
4	1800	400
5	3600	400
6	7200	400

3 RESULTS AND DISCUSSION

Variation of temperature of a CS sample preheated to 350°C is shown in Fig. 1. No heating due to the combustion reaction is observed at this stage.

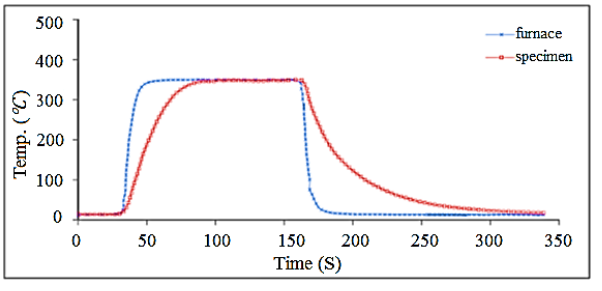


Fig. 1 Rise and fall of temperature of un-milled specimen placed inside a 350°C furnace which turned off at t=165s.

With pre-heating of 400°C (and higher), combustion reaction of un-milled sample started after around 70s, as is observable in Fig. 2. Exothermic nickel-titanium reactions result in sudden rise of the sample temperature, similar to the previous authors' observations [23]. Pre-heating temperatures above 400°C causes greater initial thermal gradient with larger heat transfer rate and combustion-temperature (~1150°C for 500°C and 1400°C for 600°C pre-heating). Fig. 2 indicates lowering of ignition time and acceleration of the combustion reaction with pre-heating temperature. Both phenomena are desirable for lessening the undesirable compounds and reduction of the unwanted pollutants.

Formation of Ni_3Ti assisted by titanium higher diffusivity is an undesirable incident that can be suppressed by elevation of the pre-heating [21], [24]. Fig. 3 compares XRD patterns of the specimens produced in pre-combustion temperatures of (1) 400°C, (2) 500°C and (3) 600°C. As shown in the figure, the Ni_3Ti peaks weaken with the pre-heating temperature so that no Ni_3Ti appears in the sample combusted at 600°C pre-heating. This is due to the lower chance of diffusion of titanium away from the nickel-titanium nucleates.

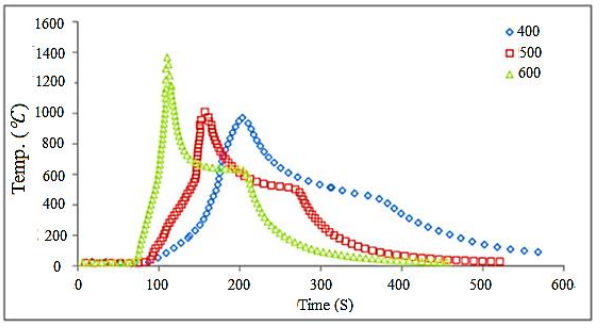


Fig. 2 Variation of temperature of the un-milled sample ignited at (1) 400°C, (2) 500°C and (3) 600°C pre-heating.

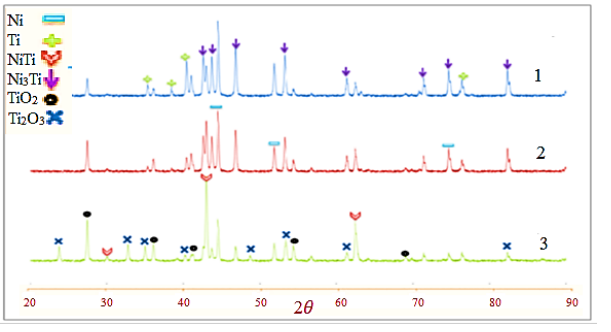


Fig. 3 XRD patterns of un-milled CS samples ignited at (1) 400°C, (2) 500°C and (3) 600°C pre-heating.

Ni_3Ti is brittle phase with no shape memory effect. Increasing pre-heating temperature improves shape memory effect of the samples by eliminating the chances of formation of this phase. This leads to a higher maximum temperature which causes greater titanium oxidation. Larger peaks of TiO_2 are observed in pattern 3 of Fig. 3. Ti_2O_3 peaks are also observable in this figure which indicates partial oxidation of the sample at higher temperatures. Competition between formation of Nitinol and titanium oxide results in lower elemental Ti and higher NiTi formation at higher temperatures.

Small peaks of the un-reacted nickel and titanium species are also observable in curves 1 and 2 of Fig. 3. This indicates that at lower temperatures, less reaction occurs due to the lower speed of diffusion of the elemental powders needed for completion of the combustion reaction. Combustion synthesis of the

elemental species is not fully achieved at small pre-heating, while considerable contents of the unwanted Ni_3Ti would be produced at grain boundary regions. Increasing the initial temperature enhances both NiTi formation and titanium oxygen reaction.

Fig. 4 specifies two distinct phases present in the structure of the specimen ignited at 600°C initial temperature. The region labelled A (dark gray color) indicates equiatomic NiTi phase. This confirms the appearance of the NiTi phase observed at the image shown in Fig. 3 for 600°C pre-heating temperature. The areas labelled B in Fig. 4 (light gray color) represent the Ti-rich phase Ti_2Ni, as assessed from EDS data. The XRD peaks of this phase appear at 2θ angles around 27.5 (222), 35 (331), 42 (333), 48 (600) and 63 (731) degrees which are barely observable in Fig. 3 because of their relatively small intensity and possible overlap with other peaks. This phase is also undesirable and sometimes appears in Ti rich boundaries of the sample. Mechanical activation of the powders may help eliminating this phase.

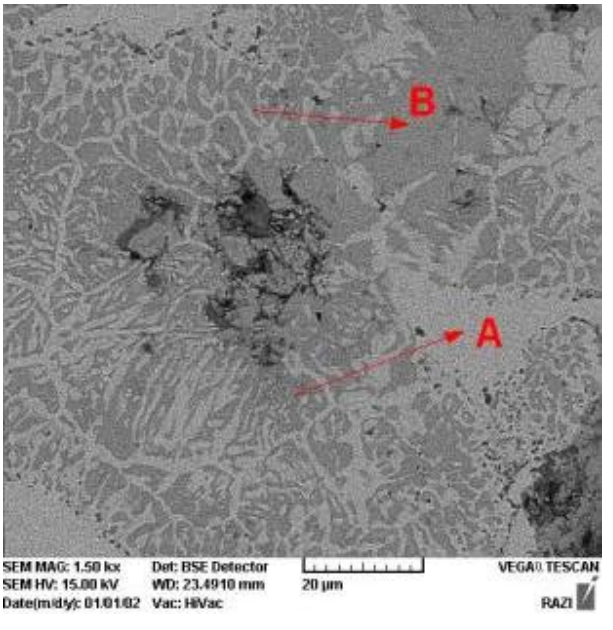


Fig. 4 SEM micrograph of the un-milled specimen 3 inserted into 600°C chamber: (A) NiTi and (B) Ti-rich phases

This paper introduces a novel method for diminution of the unwanted intermetallic phases by application of (a) mechanical activation, (b) powder compaction and (c) combustion by insertion into a pre-heated furnace chamber. Milling for (4) 1800, (5) 3600 and (6) 7200 seconds and then pre-heating to 400°C (the lowest initial temperature for ignition of an un-milled sample) resulted in temperature changes shown in Fig. 5, where XRD patterns are demonstrated in Fig. 6.

According to the previous investigations [25], short-time (less than 12h) mechanical activation does not lead to any intermetallic phase formation. The XRD patterns of the milled samples merely show the elemental peaks of the initial samples with small changes due to the preserved mechanical strain.

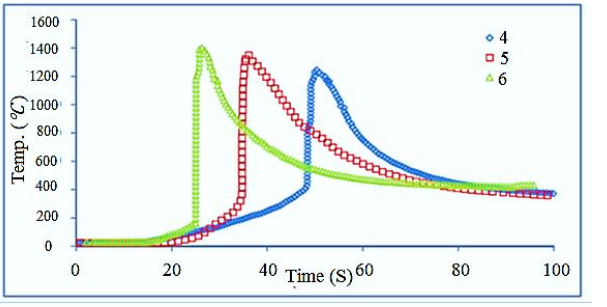


Fig. 5 Temperature changes of the specimens activated for (4) 1800, (5) 3600 and (6) 7200 s and then ignited at 400°C

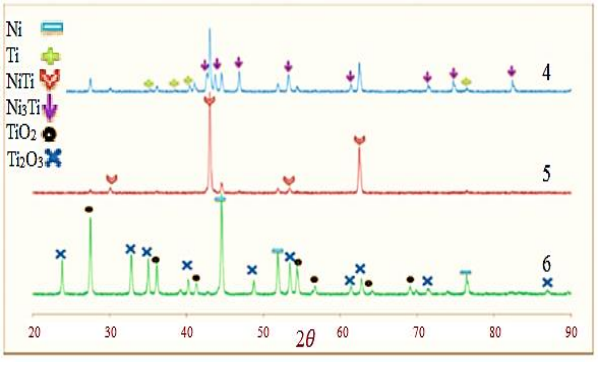


Fig. 6 XRD patterns for (4) 1800, (5) 3600 and (6) 7200 s milled samples ignited at 400°C pre-heating

The most significant effect of milling was hence lowering the duration of the combustion process illustrated in Fig. 4. With initial temperature of 400°C, the un-milled sample synthesized in around 100s (Fig. 2); while it took less than 50s for 7200s milled sample of Fig. 5. Since the synthesis reaction was well known to be very fast [26], lowering the combustion time was principally due to the facilitation of the mass transfer process. Ball milling increased the transfer rate of the interacting elements via the following mechanisms: (i) decreasing lattice order of the particles, (ii) upgrading the rapid transfer paths like dislocations and (iii) lowering the particle-sizes which decreases the diffusion distances.

A simple comparison of the consequences of (a) the initial temperature elevation with (b) the ball-milling duration on synthesis-time reduction indicates that the latter has much greater effect on ignition time than the former. The combustion peak-temperature increases with the ball milling, too. Less un-reacted elements are observable in the XRD diagrams of the specimens 4, 5 and 6 than the un-milled specimens (compare Fig. 2 with Fig. 5). This difference is attributed to the higher

maximum temperature and the lower synthesis time of the activated powders.

Despite lower ignition start time, specimen 4 has almost equal amount of Ni_3Ti to specimen 1. This can roughly balance with the un-reacted Ti and Ti rich phase in both specimens. There are more Ni_3Ti formed in less time in specimen 4 due to higher temperature and faster transfer rate as compared to the specimen 1. Due to mass transfer dependence of the ignition start and combustion reaction and increasing the diffusion rate with the ball-milling process, increasing the maximum temperature with pre-combustion milling-time seems understandable.

Mechanical energy saved in the sample facilitates initiation of this reaction. XRD results for specimen 6 reveal that titanium oxidation corresponds with intact nickel presence. This could be explained by short durations of initiation and progress of the synthesis reaction. Ball-milling provides more reactive areas with higher combustion temperature and more titanium oxidation which evolves heat besides the exothermic Nitinol formation.

Specimen 5 reaches a temperature of up to 1350°C; but contains much less titanium oxide than specimen 2 which shows maximum temperature of 1010°C. This is because of the short period of combustion in the former which does not allow recognizable titanium oxidation and intermetallic formation. Fig. 7 shows DSC cooling curves of ball-milled (for 1800, 3600 and 7200 s) samples after compaction and CS starting at 400°C pre-heating. Lattice imperfections caused by synthesis at 400°C prevents small temperature range for sharp austenite-martensite transformation.

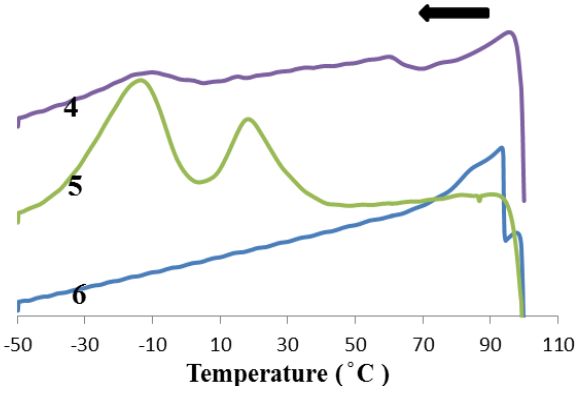


Fig. 7 DSC cooling curves of the specimens 4, 5 and 6 (milled for 1800, 3600 and 7200 s, pressed and then ignited at 400°C pre-heating)

Fig. 7 indicates that martensite transformation in specimen 4 occurs with a larger temperature change than the specimen 5. Both intermetallic phase formation and titanium oxidation cause compositional depreciation and disappearance of clear cut humps in the cooling curves. Fig. 7 indicates wrapping up of clear cut austenite-martensite transformation hump in specimen 6. This seems due to presence of unwanted oxides and un-reacted elements that hinder a straight forward phase transformation.

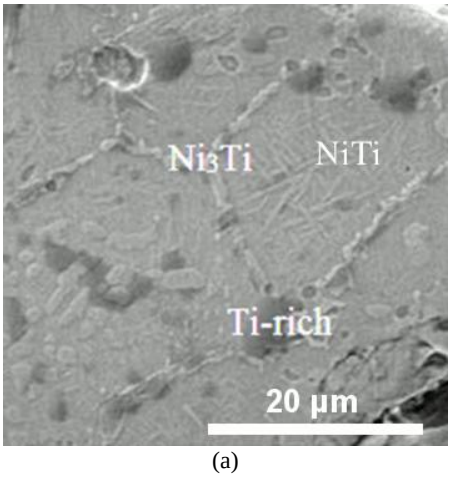
Transformation of austenite to martensite occurs when NiTi changes its crystallographic geometry from body-centered cubic to monoclinic structure. Presence of oxides and un-reacted elements in the sample widens the temperature range through which the transformation must occur. A two-step transformation is observed in Fig. 7 for all specimens, where it is more distinct for sample 5. Appearance of an intermediate rhombohedral structure prior to the martensite eventual phase corresponds with the first hump; while the rhombohedral-martensite conversion imputes the second hump.

Two-step transformations generally occur in the Ni-rich samples usually facilitated by the heat treatment [26]-[28]. High contents of oxides and elemental substances in specimens 4 and 6, prevent direct martensite transformation in absence of rhombohedral intermetallic phase. Fig. 8 shows SEM images of the specimens 4 and 5. Ti_2Ni and Ni_3Ti intermetallics exist alongside martensitic NiTi phase in specimen 4 produced via 1800 s ball-milling and CS after compaction (Fig 8a). Martensite start transformation occurs in specimen 4, at approximately 3°C (Fig. 8). Ball-milling for 3600 s prevents the formation of too many unfavorable intermetallics in the system. This sharpens the austenite-rhombohedral and rhombohedral-martensite transformation humps of the sample (Fig. 8b).

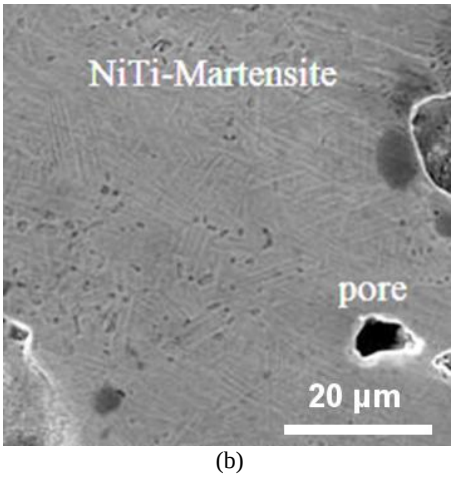
Fig. 9 shows compressive stress-strain curve of the specimens 4, 5 and 6. All specimens are tested at a temperature higher than their austenite finish temperatures ($T>A_f$). The compressive stress–strain curves of specimen 5 clearly exhibit superelastic behavior due to the formation of a nickel rich NiTi mono-phase. Specimens 4 and 6 exhibit lower flow stress levels and less superelastic behavior due to lower nickel dissolved in the matrix embedding intermetallic and elemental nickel phases. Because of higher porosity of the specimens 6, it has the lowest flow stress. The specimens 4 and 6 save around 3% thermoelastic strain in their structures after loading-unloading cycles due to the formation of nickel rich and elemental nickel phases which results in slight depletion of the matrix from the dissolved nickel constituent.

A desirable application around room temperature of the superelastic and shape memory Nitinol alloys is in medical systems. Porous NiTi samples can be used for manufacture of different scaffolds with close to bone mechanical properties, low and medium stiffness, bio-

compatibility and corrosion resistance in contact with natural body electrolytes and environments. Sample 5 is a well suited porous superelastic alloy designable for use under such delicate conditions.



(a)



(b)

Fig. 8 SEM micrographs of NiTi samples (a) 1800 and (b) 3600 s ball-milled, compacted and ignited at 400°C pre-heating

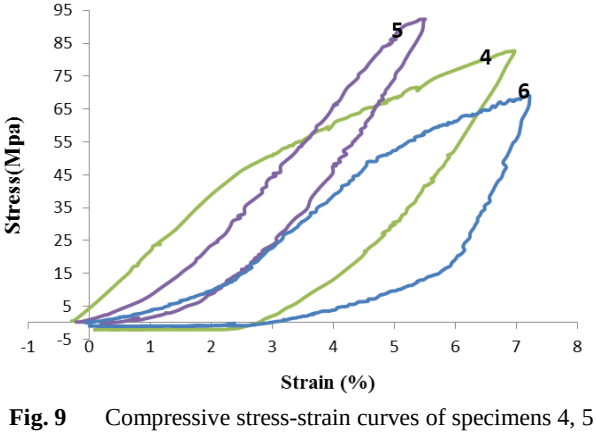


Fig. 9 Compressive stress-strain curves of specimens 4, 5 and 6 milled for 1800, 3600 and 7200 s, respectively and then ignited at 400°C pre-heating

4 CONCLUSION

a. For optimum combustion synthesis reaction, the initial furnace temperature must be set at 400°C.

b. No matter how much pre-heating is used for start of the combustion synthesis reaction, un-reacted elements and undesirable phases appear in the final product of the un-milled CS samples. The only difference is that the undesirable phase is Ni_3Ti with the initial chamber temperature of 400°C; while titanium oxide appears when the initial chamber temperature is 600°C.

c. Ball-milling results in total superelastic behavior together with 2 stage austenite-rhombohedral and rhombohedral-martensite phase transformations.

d. Ball-milling for 7200 s is much more effective on combustion synthesis than 200°C increase in the initial furnace temperature.

e. Ball-milling for 3600 s results in formation of almost pure NiTi mono-phase with greatest superelastic behavior. This is accompanied by minimization of the un-reacted elemental substances and diminution of the reaction duration which provides no time for titanium oxidation.

Both pre-combustion temperature and milling activation had significant influence on the formation of undesirable intermetallic phases, morphology and oxidation of the Ni-Ti samples. These phases affected the properties of the produced material.

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