OPTIMIZATION OF THE MECHANICAL ACTIVATION PROCESS OF NiTi ALLOYS

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Overview

In this work the brittle nature of $NiTi_2$ was used to promote a better efficiency of the mechanical activation process of NiTi alloys. The mechanical activation of ($NiTi_2$ -Ni) powder mixtures resulted in the formation of a predominant amorphous structure after 2 h at 300 rpm . A sintered specimen consisting mainly of the NiTi phase was obtained after vacuum sintering at 1050 ºC for 0.5 h. The produced NiTi phase exhibited the martensitic transformation behavior. Using elemental Ti powders instead of pre-alloyed $NiTi_2$ powders, the structural homogenization of the synthesized NiTi alloys was delayed. Performing the mechanical activation at 300 rpm for the (Ti-Ni) powder mixtures gave rise to the formation of composite particles consisting in dense areas of alternate fine layers of Ni and Ti. No significant structural modification was observed even after 16 h of mechanical activation and only after vacuum sintering at 1050 ºC for 6 h the NiTi phase was observed to be the predominant phase. The higher reactivity of the mechanically activated ($NiTi_2$ -Ni) powder particles can explain the different sintering behavior of those powders compared with the mechanically activated (Ti-Ni) powders. The obtained results demonstrate an effective way to optimize the mechanical activation process of NiTi alloys.

Methods

Raw materials

Commercially pure Ni (99.9%, -200 mesh) and Ti (99.5%, -200 mesh) powders.

The pre-alloyed $NiTi_2$ powders were fabricated by vacuum induction melting of Ni ingots (99.9%) and Ti ingots (99.9%), followed by mechanical crushing and screening methods.

Target composition

Ni-50 at.%Ti

Two groups of powders blends from the starting $NiTi_2$, Ni and Ti powders: ($NiTi_2$-Ni) and (Ti-Ni).

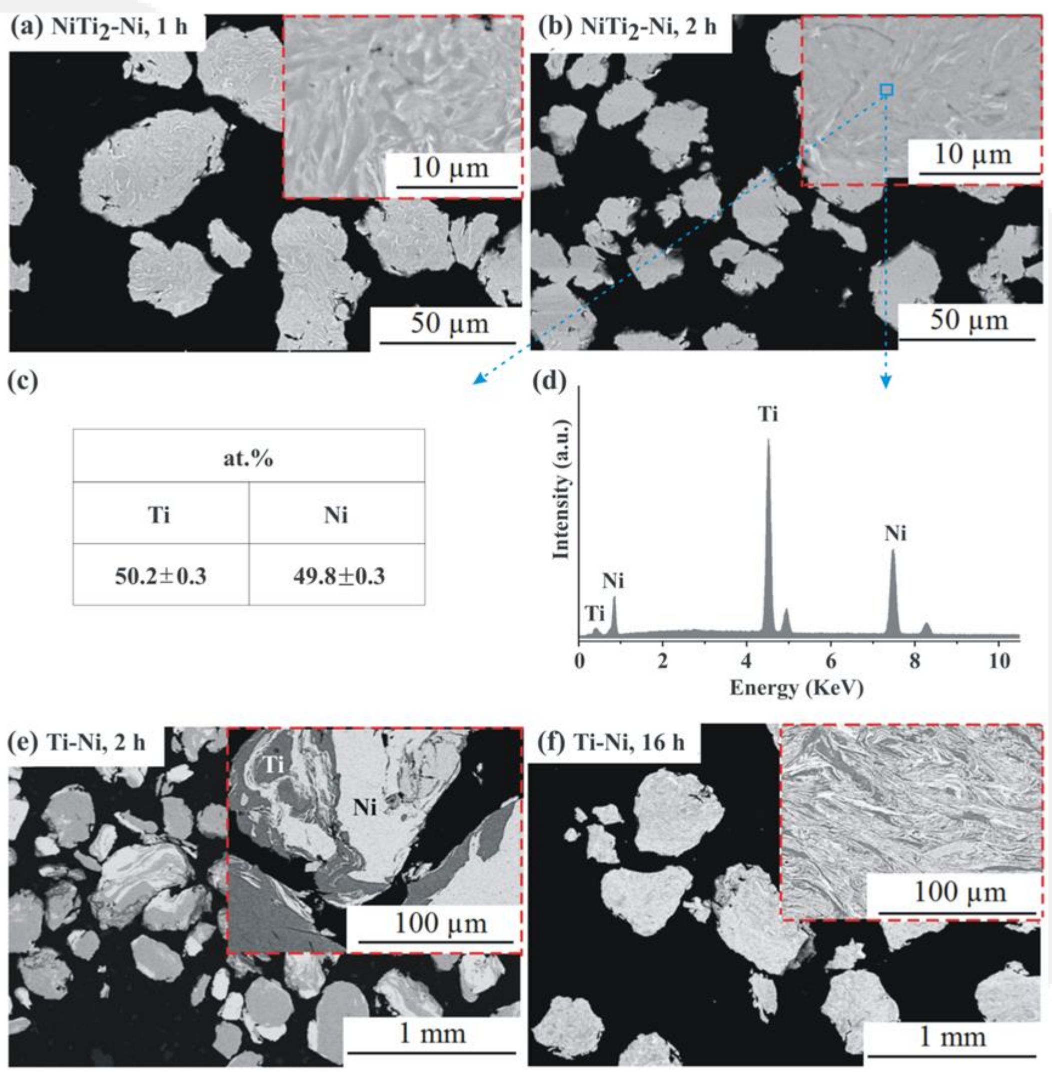
Mechanical activation conditions

High-energy planetary ball mill PM 400 from Retsch // speed: 300 rpm // time: up to 16 h // ball-to-powder ratio 20:1 // stainless steel containers (250 mL) and balls (15 mm diameter) // no additional fluid medium // containers evacuated and back-filled with argon // milling periods of 10 min and pauses of 5 min to cool the containers.

Sintering conditions

The mechanically activated powders were cold pressed, with a pressure of 300 MPa, into columnar specimens with 10 mm × 10 mm × 25 mm in size. The cold pressed columnar specimens were then sintered in a vacuum furnace (2.3×10^{-3} Pa) at 850 ºC and 1050 °C, with holding periods of 0.5 h and 6 h

Results

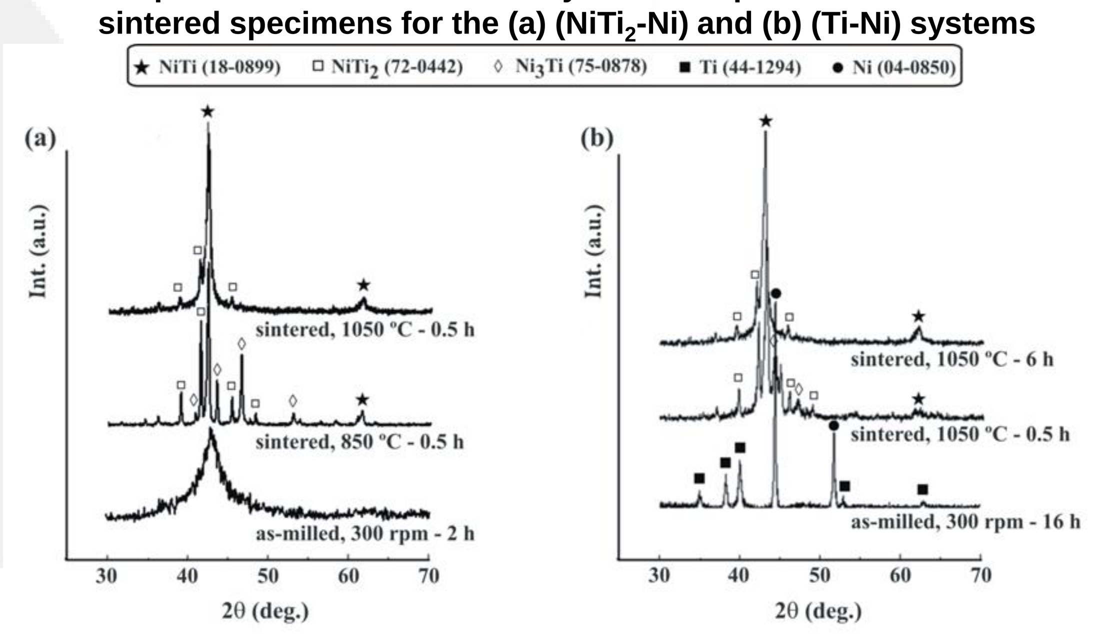


(a, b, c, f) Typical SEM/BSE images of the mechanically activated powder mixtures, (c) elemental composition (in at %) determined in areas equivalent to the blue square marked in (b) (average of 3 analysis), (d) typical EDS spectrum obtained in areas equivalent to the blue square marked in (b)

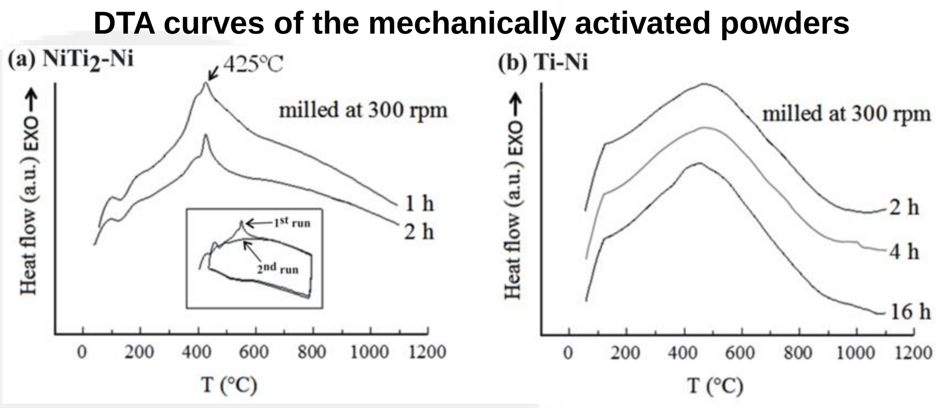
Oxygen contents of the pristine and mechanically activated powders

Powders		Oxygen (wt.%)
Starting powder	Ti	0.281 ± 0.009
	Ni	0.060 ± 0.002
	$NiTi_2$	0.101 ± 0.011
($NiTi_2$-Ni)	300 rpm – 1 h	0.176 ± 0.017
	300 rpm – 2 h	0.172 ± 0.002
(Ti-Ni)	300 rpm – 1 h	0.214 ± 0.026

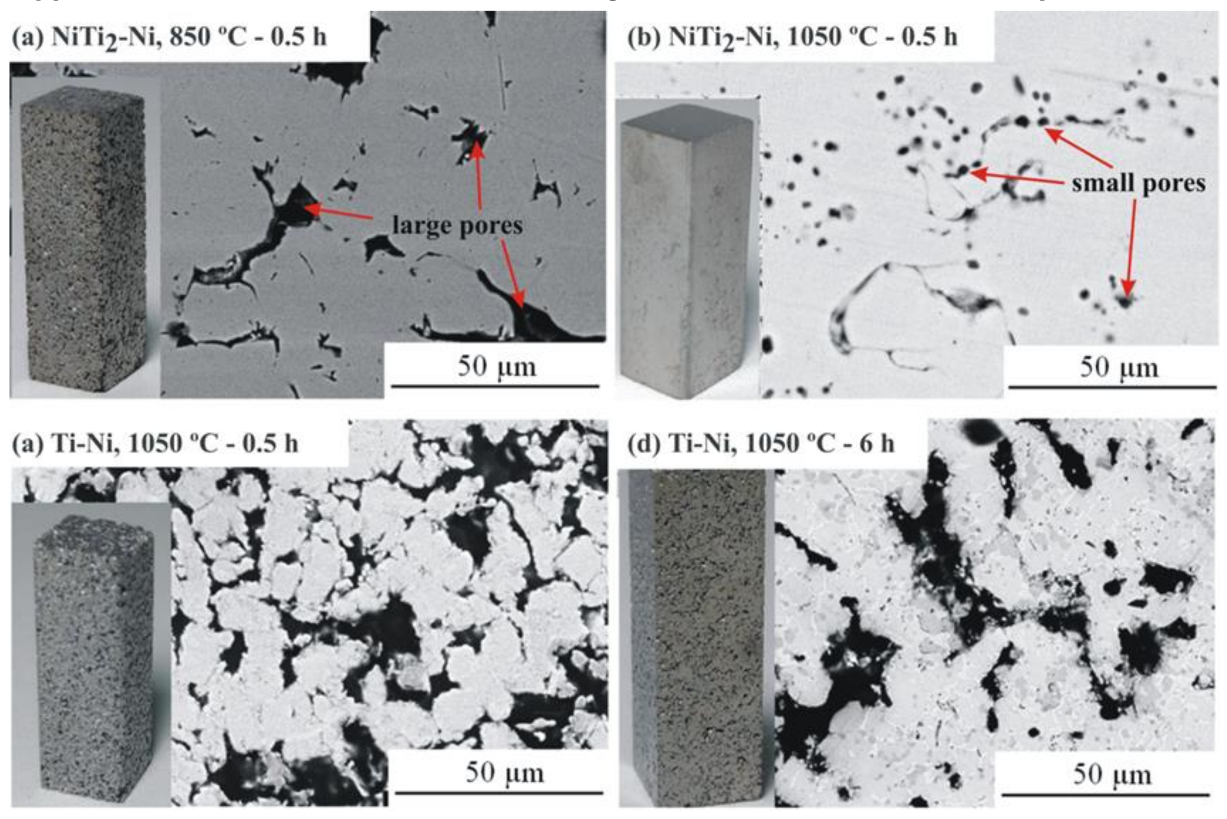
XRD patterns of the mechanically activated powders and vacuum sintered specimens for the (a) ($NiTi_2$-Ni) and (b) (Ti-Ni) systems



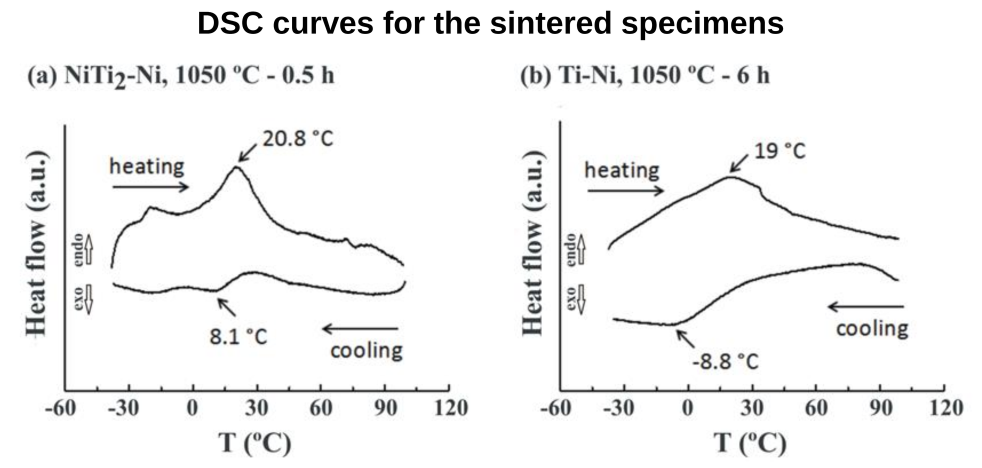
DTA curves of the mechanically activated powders



Typical macro and microstructures images of the vacuum sintered specimens



DSC curves for the sintered specimens



Conclusions

- An innovative approach for improving the synthesis of NiTi alloys through powder metallurgy is proposed. This approach involves the substitution of elemental Ti powder by pre-alloyed $NiTi_2$ powders.
- The mechanical activated ($NiTi_2$-Ni) powder mixtures showed higher reactivity, which was responsible for the better sintering behavior of those powders when compared to the mechanical activated (Ti-Ni) powder mixtures.
- The reduction in the total processing time achieved for the ($NiTi_2$-Ni) powder mixtures vs the (Ti-Ni) powder mixtures, in particular when considering the mechanical activation step, is a real improvement due to the potentiality of mitigating all possible contaminations sources.
- For the ($NiTi_2$-Ni) powder mixtures, an amorphous powder was formed with the mechanical activation step shortly after 2 h at 300 rpm. NiTi specimens were obtained after vacuum sintering at 1050 ºC for 0.5 h.
- For the (Ti-Ni) powder mixtures, Ni and Ti phases were still present after 16 h of milling and a NiTi specimen was obtain only after 16 h at 1050 ºC.

Reference

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