Materials Science Forum Vols. 738-739 (2013) pp 306-310
© (2013) Trans Tech Publications, Switzerland
doi:10.4028/www.scientific.net/MSF.738-739.306

Effect of Annealing on the Transformation Behavior and Mechanical Properties of Two Nanostructured Ti-50.8at.%Ni Thin Wires Produced by Different Methods

Xiebin Wang[1, a], Behnam Amin-Ahmadi[2, b], Dominique Schryvers[2, c], Bert Verlinden[1, d] and Jan Van Humbeeck[1, e]

[1]Department MTM, KU Leuven, Kasteelpark Arenberg 44, B-3001 Leuven, Belgium

[2]EMAT, University of Antwerp, Groenenborgerlaan 171, B-2020 Antwerp, Belgium

[a]Xiebin.Wang@mtm.kuleuven.be, [b]Behnam.Amin-Ahmadi@ua.ac.be, [c]Nick.Schryvers@ua.ac.be,

[d]Bert.Verlinden@mtm.kuleuven.be, [e]Jan.VanHumbeeck@mtm.kuleuven.be

Keywords: NiTi, Annealing, Nano-structure, Thin wires.

Abstract. A Ti-50.8at.%Ni wire produced using a co-drawing method and a commercial Ti-50.8at.%Ni wire were annealed at different temperatures between 450°C and 700°C. Grains with diameter less than 100nm were revealed by transmission electron microscopy for both wires before annealing treatment. However, the microstructural heterogeneity of the co-drawn wire is more obvious than that of the commercial wire. Multi-stage martensitic transformation was observed in the co-drawn wire, compared with the one-stage A↔M transformation in the commercial wire after annealing at 600°C for 30min. The differences of total elongation, plateau strain and pseudoelastic recoverable strain between the commercial wire and the co-drawn wire were also observed. The differences of the transformation behavior and mechanical properties between the commercial wire and the co-drawn wire are attributed to the microstructural difference between these two wires.

Introduction

The transformation behavior and mechanical properties of Ni-rich NiTi shape memory alloys (SMAs) are sensitive to the thermomechanical treatment, such as annealing after cold deformation [1,2] and ageing after solution treatment [3]. Cold deformation can improve the materials strength. With increase of yield strength, the plastic deformation, which undermines the shape memory effect (SME) and pseudoelastic effect (PE), can be reduced during thermoelastic martensitic transformation. However, the defects, mainly dislocations, introduced by cold deformation can also suppress the martensitic transformation in NiTi alloys. During the post-deformation annealing, recovery of the deformation microstructure or even recrystallization can occur [4]. Many researchers have investigated the variation of transformation behavior and mechanical properties of NiTi alloys with respect to annealing temperatures. However, the effect of initial microstructure before annealing has not been dealt with in depth. As different production methods can lead to different microstructures, the effect of the initial microstructure on NiTi alloys after annealing is worth studying. In this study, two NiTi thin wires with identical composition produced by different methods were studied. Particular attention is taken to link the initial microstructure to the differences of transformation behavior and mechanical properties between these two wires after annealing.

Experimental Procedure

In this work, two Ti-50.8at.%Ni wires with diameter of 50μm were studied. One is a commercial wire produced by Memory-Metalle GmbH (Weil am Rhein, Germany), the other wire is produced using a co-drawing method. In this method, several wires with diameter of 500μm were embedded in a copper matrix enclosed in a stainless steel tube and further cold drawn to get the wires with diameter of 50μm. Both the co-drawn wire and the commercial wire have a cold deformation of about 35%. Both the wires were annealed at different temperatures between 450°C and 700°C for 30min in argon atmosphere, followed by quenching in water to room temperature. The microstructure was observed using a JEOL 3000 transmission electron microscope (TEM) operating at an accelerating voltage of 300kV. The specimens for TEM were prepared by focused ion beam (FIB) in an FEI Helios Nanolab 650 SEM/FIB instrument with the lift-out procedure and selected from different locations inside the wire. The FIB cuts were performed along the long axis of the wires. The transformation characteristics were measured using a TA Q2000 differential scanning calorimeter (DSC) at the temperature range of 150°C to -150°C with a cooling/heating rate of 10°C /min. Every sample was subjected twice to the cooling/heating cycle. The results obtained in the second cycle were taken as the transformation temperatures. The mechanical properties were tested using a TA Q800 dynamic mechanical analyzer (DMA) at room temperature (25°C). The stress-strain curves were measured at Controlled Force mode with a force ramp rate of 0.2N/min. The recoverable strain was measured at a strain rate of 1%/min. The lengths of the specimens for DMA measurements were 10-11mm.

Results and Discussions

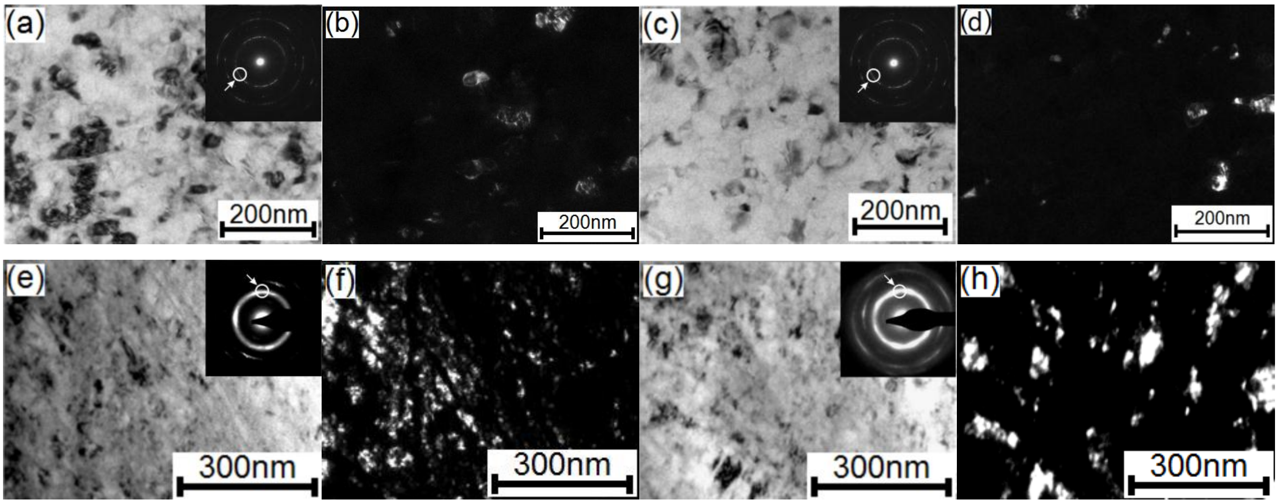

Fig. 1. TEM micrographs of the commercial wire (a-d) and the co-drawn wire (e-h). (a) and (b) are bright field image (BF), inset diffraction pattern (DP) and dark field image (DF) taken from the region close to the surface of the commercial wire; (c) and (d) are BF, inset DP and DF taken from the region close to the central axis of the commercial wire; (e) and (f) are BF, inset DP and DF taken from the region close to the surface of the co-drawn wire; (g) and (h) are BF, inset DP and DF taken from the region close to the central axis of the co-drawn wire.

The TEM results (Fig. 1) show that in both the commercial and the co-drawn wires, the electron diffraction reveals ring patterns indicating nanostructured NiTi B2 austenite. In the commercial wire no martensite rings were observed, while in the center of the co-drawn wire some indications of martensite were found. The region close to the surface of the commercial wire contains round grains of 30-50nm in diameter while in the co-drawn wire this region shows elongated grains of 30-70nm, as measured from both bright and dark field images. The centers of both wires show a non-directional morphology of grains.

Fig. 2 shows the DSC curves of both the commercial and the co-drawn wires annealed at different temperatures. Fig. 3 shows that the peak transformation temperatures of A→R, R→M and A→M transformations are higher in the co-drawn wire than in the commercial wire under the same annealing conditions. Because the chemical composition of these two wires is the same, therefore, the differences of transformation temperatures are attributed to the difference of microstructure between the co-drawn wire and the commercial wire. As shown in Fig. 1, a multi-stage martensitic transformation is observed in the co-drawn wire when annealed at 600°C, while the commercial wire shows only one-stage A↔M transformation. Moreover, as shown in Fig. 4, two-stage stress-induced martensite transformation is observed in the co-drawn wire when annealed at 600°C, while the commercial wire shows only one stress plateau during the stress-induced martensite transformation. The chemical composition of these two wires is the same, while also the grain size at different regions for both wires is very close (in the range of 20-90nm), as revealed by TEM. Therefore, the multi-stage martensitic transformation and two-stage stress-induced martensite transformation in the co-drawn wire are attributed to the inhomogeneous microstructure in the co-drawn wire as compared with the commercial wire.

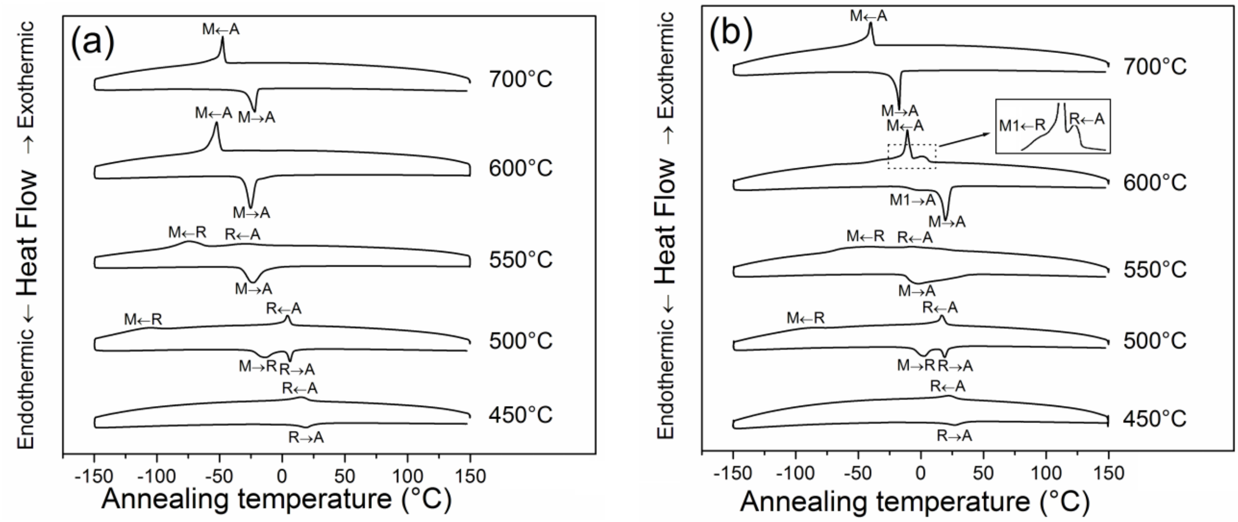

Fig. 2. Effect of annealing at different temperatures for 30min on the transformation behavior of (a) the commercial wire and (b) the co-drawn wire.

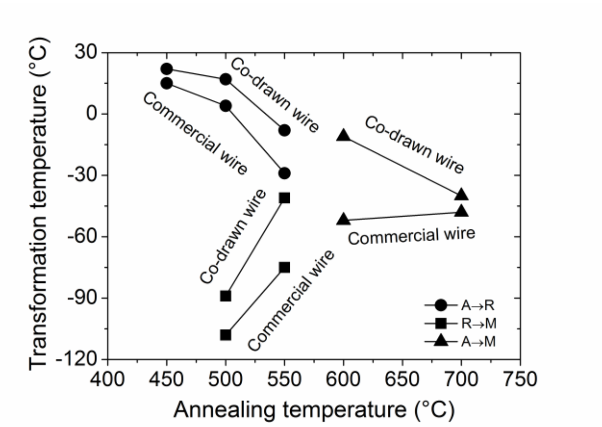

Fig. 3 Effect of annealing temperature on the peak transformation temperature during cooling.

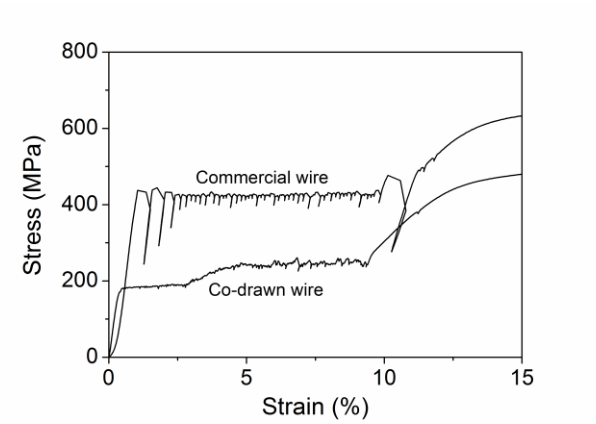

Fig. 4 Stress-strain curves of the commercial wire and the co-drawn wire after annealing at 600°C for 30min. The stress-strain curves were measured at strain controlled mode with a strain rate of 1%/min at 25°C.

For both the commercial wire and the co-drawn wire, the ultimate tensile stress decreases and the total elongation increases with increase of annealing temperature, as shown in Fig. 5. The ultimate strength of the co-drawn wire is lower than that of the commercial wire under the same annealing conditions, indicating that the inhomogeneous microstructure in the co-drawn wire undermines the strength of the wire as compared with the commercial wire. As shown in Fig. 5b, when annealed at 600°C, both wires show a significant increase of total elongation, which is attributed to the occurrence of recrystallization in these samples [5].

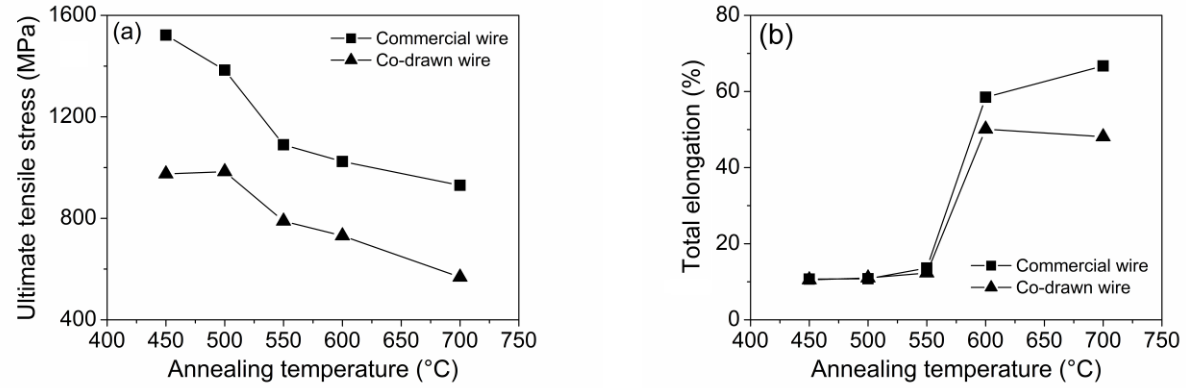

Fig. 5. Effect of annealing at different temperatures for 30min on (a) ultimate tensile stress and (b) total elongation of the commercial wire and the co-drawn wire.

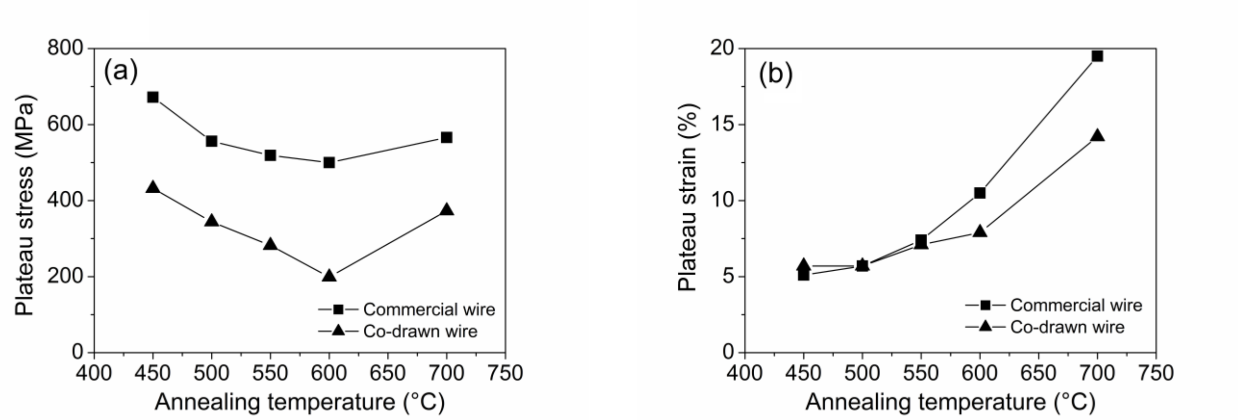

Fig. 6. Effect of annealing at different temperatures for 30min on (a) plateau stress and (b) plateau strain of stress induced martensite transformation at 25°C.

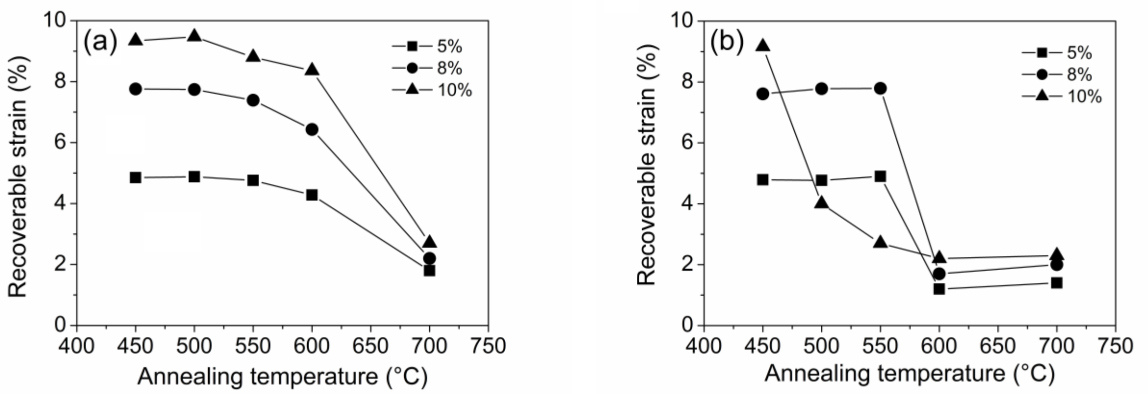

Fig. 7. Effect of annealing at different temperatures for 30min on the pseudoelastic recoverable strain of (a) commercial wire and (b) co-drawn wire at 25°C. The pre-strain is 5%, 8% and 10%, respectively.

The plateau stress and plateau strain for stress-induced martensite transformation of both the commercial wire and the co-drawn wire as a function of annealing temperature are shown in Fig. 6. For both wires, the plateau stress decreases and the plateau strain increases with increasing annealing temperature, when annealed at temperatures below 600°C. At all annealing temperatures, the plateau stress of the co-drawn wire is lower than that of the commercial wire, which is due to the higher martensite transformation temperatures in the co-drawn wire, as shown in Fig. 2 and Fig. 3. When annealing at 700°C, both the commercial wire and the co-drawn wire show a large plateau strain of

19.5% and 14.2%, respectively. This is because the yield stress of austenite and the critical stress for inducing martensite transformation are at the same level, therefore, the large plateau strain is a combination of the transformation strain of stress-induced martensite transformation and the plastic strain of austenite and/or martensite.

Fig. 7 shows the effect of annealing temperature on the pseudoelastic recovery at room temperature of both wires with pre-strain of 5%, 8% and 10%. The recoverable strain is measured as the pre-strain minus the strain which cannot spontaneously recover at 25°C. When annealed at 500°C and 550°C, both the commercial wire and the co-drawn wire show good pseudoelasticity with pre-strain of 5% and 8%. With pre-strain of 10%, the commercial wire shows a recoverable strain of 9.4% and 8.8% when annealed at 500°C and 550°C, respectively. However, a recoverable strain of 4% and 2.7% is observed in the co-drawn wire when annealed at 500°C and 550°C, respectively. The sharp decrease of the recoverable strain in the co-drawn wire indicates that the inhomogeneous microstructure in the co-drawn wire is easier to trigger plastic deformation, as compared with the commercial wire.

As shown in Fig. 2b, when annealed at 600°C, the reverse transformation finish (Af) temperature of the co-drawn wire is 24°C, which is just below the testing temperature (25°C), therefore, resulting in the decrease of recoverable strain. The sharp decrease of recoverable strain of both wires after annealing at 700°C is attributed to the fact that the plastic deformation of austenite phase occurs before the stress-induced martensite transformation.

Conclusions

Two nano-structured Ti-50.8at.%Ni thin wires produced by different methods were annealed at different temperatures between 450°C and 700°C. The transformation behavior and mechanical properties of these two wires were investigated in this study. As compared with the commercial wire, the more inhomogeneous microstructure in the co-drawn wire was revealed by TEM. The different microstructure results in a different transformation behavior and mechanical properties between the commercial wire and the co-drawn wire after annealing treatment. When annealed at 700°C, both the commercial wire and the co-drawn wire show a large plateau strain of 19.5% and 14.2%, respectively, which results from the combination of austenite and/or martensite plastic deformation and stress-induced martensite transformation in both wires when annealed at 700°C.

References

[1] X. Huang, Y. Liu, Effect of annealing on the transformation behavior and superelasticity of NiTi shape memory alloy, Scr. Mater. 45 (2001) 153-160.

[2] Z.G. Wang, X.T. Zu, X.D. Feng, S. Zhu, J.M. Zhou, L.M. Wang, Annealing-induced evolution of transformation characteristics in TiNi shape memory alloys, Physica B. 353 (2004) 9-14.

[3] K. Otsuka, X. Ren, Physical metallurgy of Ti-Ni-based shape memory alloys, Prog. Mater. Sci. 50 (2005) 511-678.

[4] R. Delville, B. Malard, J. Pilch, P. Sittner, D. Schryvers, Transmission electron microscopy investigation of dislocation slip during superelastic cycling of Ni-Ti wires, Int. J. Plast. 27 (2011) 282-297.

[5] S. Miyazaki, Y. kohiyama, K. Otsuka, T.W. Duerig, Effect of several factors on the ductility of the Ti-Ni alloy, Mater. Sci. Forum. 56-58 (1990) 765-770.