Ultrasonic characterization of phase transformation in NiTi wire during thermomechanical loading

Michal Landa[a], Petr Sedlák[ac], František Maršík[a], Petr Šittner[b], Václav Novák[b]

[a]*Institute of Thermomechanics, ASCR, Dolejškova 5, Prague 8, 182 21, Czech Republic*

[b]*Institute of Physics, ASCR, Na Slovance 2, 182 21, Czech Republic*

[c]*Faculty of Nuclear Sciences and Physical Engineering, CTU in Prague, Břehová 7, Prague 1, 110 00, Czech Republic*

Summary *Evaluation of thermo-mechanical properties of NiTi wires intended for stent applications is carried out by combination of tensile tests at constant temperature with in-situ ultrasonic measurements (wave speed, attenuation) and electrical resistivity measurements. It is found that the mechanical and electric resistance results are mainly sensitive to R-B19' stress induced martensitic transformation, but the ultrasonic wave speed and attenuation vary most significantly when the R-phase reorientation or distortion take place.*

INTRODUCTION

Biocompatibility and high recoverable strain properties of superelastic nickel-titanium alloy make it an interesting material for medical application like vascular support wire-structures (self-expanding stents) [1]. Detailed knowledge of phase transition mechanisms is essential for utilization of the unique properties (shape memory effect and superelasticity) of the NiTi alloys in optimal design of the implants.

The high temperature parent phase of NiTi alloys has B2 type ordered cubic structure that transforms to B19' monoclinic martensite phase by cooling or applied stress. Following suitable termomechanical treatments, the martensitic transformation in NiTi may proceed as two step B2-R-B19' [2]. The intermediate R-phase has rhombohedral structure differing only slightly from the B2 by rhombohedral distortion. The B2-R transformation exhibits very small transformation strain and hysteresis compared to the B2-B19' martensitic transformation. The activity B2-R transformation is rather difficult to recognize in mechanical test on NiTi wires, although it might be responsible for significant part of inelastic strains [3], particularly if loading is limited to small strains (<2%). In this research, an in-situ ultrasonic technique was applied during tensile test on NiTi wire showing R-phase upon cooling slightly below the room temperature. The aim was to check whether the acoustic method can detect activity of the various transformation processes taking place during the tensile tests at various temperatures.

EXPERIMENT

Evaluation of thermo-mechanical properties of commercial NiTi wires (Ni55.75%wt, Fort Wayne Metals Corp. , 0.18mm in diam.) was carried out by strain controlled tensile tests with the ultrasonic pulse-transition in-situ measurement. The temperature of the specimen was kept constant during the load cycle. The wire was heated by electrical current, impedance of the electrical supply was matched to the wire impedance, thus constant heating condition was ensured. The pair of miniature piezoelectric transducers, used as ultrasonic emitter and receiver, were placed at the gripped ends of the wire (Fig.1). At constant time period, the transmitted pulses along the wire were recorded and longitudinal wave velocity c_L and amplitude A were evaluated. The instantaneous Young modulus E is given by

$$E = \rho . c_L^2$$

and wave attenuation α is determined by

$$\alpha = -\frac{1}{H} 20 \log(A / A_{ref}) ,$$

where ρ is mass density and H is the instantaneous wire length. The attenuation is related to the initial value α_o .

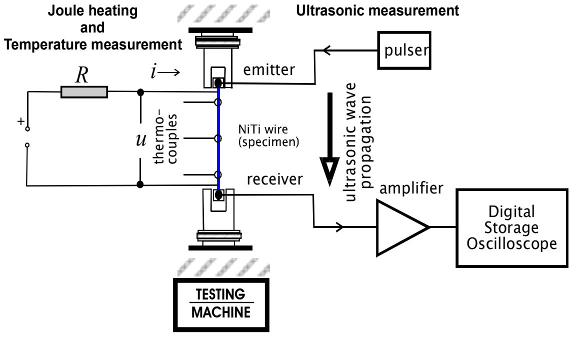

Figure1: Experimental arrangement

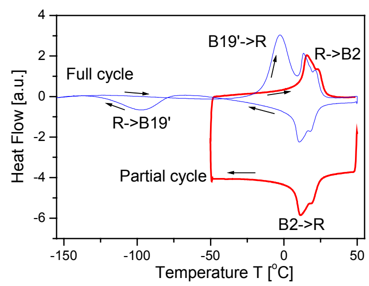

Figure 2: DSC record during full and partial cooling-heating cycles showing temperature ranges where individual transformation events take place.

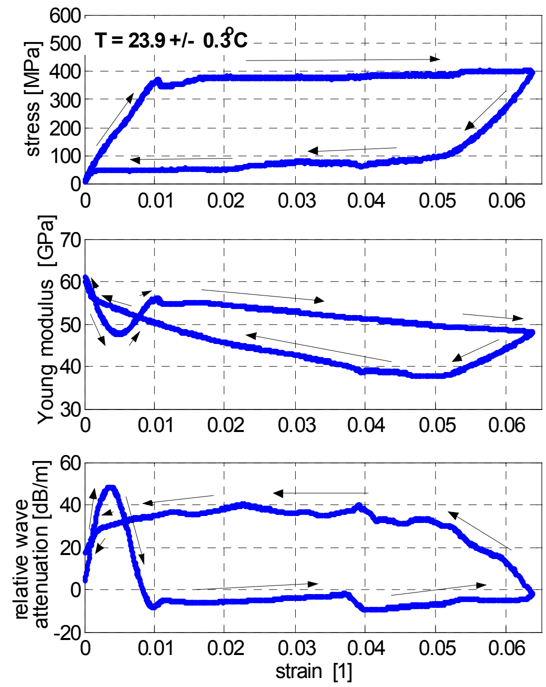

Figure 3: Stress and quantities, determined by ultrasound, during virgin loading of the tested NiTi wire.

RESULTS

Prior the mechanical tests, differential scanning calorimetry measurement was carried out (Fig.2) . It is clear, particularly from the partial cycle scan, that the alloy may exist at room temperature (T=24 °C) either in R-phase or in the B2 phase state depending on the history. Since the mechanical test (Fig.3) was carried out after cooling to approx. 10 °C and subsequent heating to room temperature, the specimen was in the R-phase state prior the start of the test. Stress induced transformation to the B19' martensite phase proceeds at approximately constant stress level 400MPa between 1%-6% strain most likely in Luders-like localization mode. It is very interesting to note that significant changes of wave speed and attenuation are not detected during the R-B19' transformation to martensite but in the preceding quasilinear elastic range. In that range, reorientation of R-phase variants takes most likely place at first (strain <0.5%) and later on elastic deformation (or possibly further distortion) of R-phase structure occurs (0.5%< strain <1%) [3]. Clearly, the ultrasonic quantities seem to be much more sensitive to the R-phase phenomena than to the martensitic transformation itself.

CONCLUSION

Combined ultrasonic–mechanical-electrical technique was applied to investigate stress induced martensitic transformation in commercial NiTi wire intended for stent applications. While the mechanical and electric resistance results are mainly sensitive to R-B19' martensitic transformation, the ultrasonic wave speed and attenuation varies significantly when the R-phase reorientation or distortion take place.

Acknowledgement

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