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**NiTi WIRES FOR ORTHODONTIC APPLICATION**

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**Abstract** – In order to characterize NiTi orthodontic wires microstructure, transformation temperatures, and deformation behaviour have been investigated. Various states of dislocation density were established by additional heat treatment of a work hardened material. The as recieved wire and the change in microstructure due to the chosen heat treatment were described by optical lighth microscopy. The stress induced transformation behaviour is related with the experimental results in transformation characteristic measurements and microstructural observations.

**1. Introduction**

NiTi wires, especially those in the pseudoelastic state, have been introduced to medical treatment of dental malalignment /1–3/. A large working range, a constant force, and a small modulus of elasticity make them well suited for orthodontic application. Concerning the use of NiTi in dentistry, orthopedics, and other medical devices, biocompatibility was tested by in vivo investigations as well as by in vitro studies of fibroblast reproduction rates /4/.

Characteristic features of several orthodontic NiTi wires have been described in terms of thermal and mechanical analysis /5/. There are two reasons to apply a heat treatment on a NiTi wire in the as drawn or as recieved condition. On one hand a dramatical improve in pseudoelastic properties is available by additional heat treatment /6/. On the other hand there is a need to prepare defined shaped devices, whereby, the fixation of the desired shape has to be carried out by the application of a specific amount of heat in a constrained condition /7/.

The effect of heat treatment after cold work on the thermal transformation signal in differential scanning analysis (DSC) has been described /8/. Thermal analysis (DSC) in combination with mechanical testing was first presented in a thermomechanical study of orthodontic NiTi alloys /9/. This paper is part of an investigation on orthodontic NiTi wires. It is intended to give a first description on the microstructural changes and the variation in thermal and mechanical transformation behaviour for one special orthodontic NiTi wire, when it has been heat treated after cold work.

**2. Experimental Procedure**

Orthodontic NiTi wires (Nitinol SE, Unitek Corporation) were obtained by commercial sources. Specimens were cut from the wire in the as recieved condition and then annealed between 260 °C and 500 °C for one hour followed by air cooling.

For optical microscopy, in polarized light illumination (PLM), the specimen were polished electrolytically in a solution of 8 vol% perchloric acid, 12 vol% distilled water, 10 vol% butylglycol

rest ethylic alcohol. The surface was finally etched after Beraha I /10/. The etching solution was 10 ml hydrochloric acid, 50 ml distilled water, 1,2 g ammonium bifluoride, and 8 g potassium metabisulphite.

The thermal transformation behaviour was determined by differential scanning analysis (DSC). Samples of 0,05 g – 0,06 g were cut from the orthodontic wires. The heating/cooling rate was 10 °C/min.

Uniaxial tensile tests were carried out in a Zwick 1387 universal testing machine. All tests were performed at a temperature of 37 °C and at a strain rate of 0,033 mm/sec.

### 3. Results

The microstructure in polarized light illumination is presented in Fig. 1 and Fig. 2 in the as recieved condition and after a heat treatment at 360 °C/1 h and 440 °C/1 h respectively. Subgrains became visible in the as recieved wire. A preferential orientation has been built up by the drawing direction (Fig. 1a). At a higher magnification (Fig. 1b) the presence of a perfectly deformed microstructur is revealed.

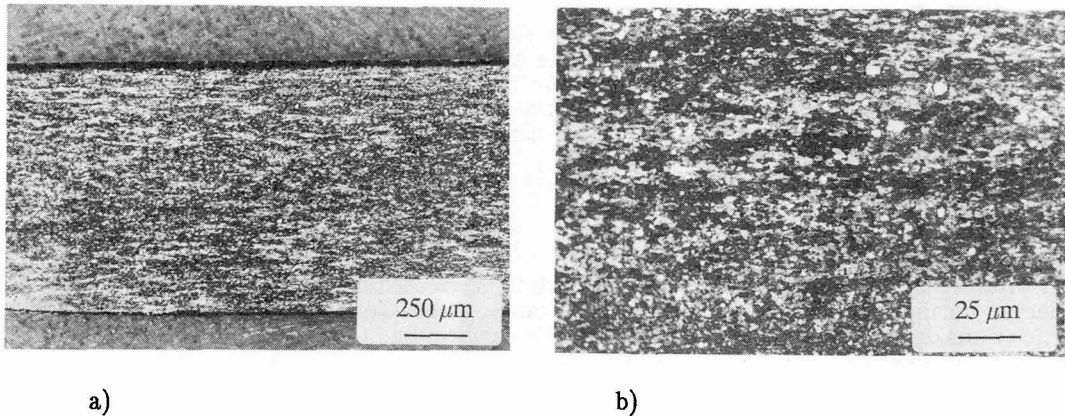


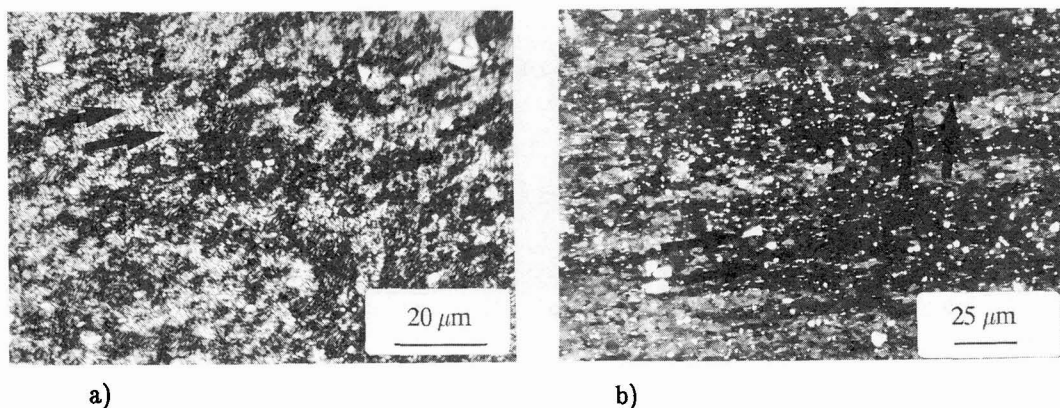
Fig. 1: Microstructure of work hardened orthodontic NiTi wire in the as recieved condition. The surface was observed in polarized light illumination.

Two significant changes in microstructure became observable when a wire had been heat treated. After an annealing for 360 °C/1 h fine striations, parallel orientated in each subgrain, could be found (Fig. 2a) in a still all over deformed matrix. When the heat treatment was carried out at 440 °C, obviously a partial recrystallization has taken place (Fig. 2b). Furthermore, presumed hard particles and small striations, parallel to the drawing direction, could be seen. The striations and the particles were both white coloured.

The signal from thermal analysis (DSC) was almost the same for a heat treatment between 260 °C and 440 °C (Fig. 3a). Martensitic transformation became detectable in thermal analysis when the annealing was carried out at 500 °C/1 h (Fig. 3b).

The deformation behaviour was characterized by a perfect pseudoelasticity for a heat treatment up to 440 °C/1 h (Fig. 4a). No pseudoelastic recovery took place after a heat treatment at 500 °C/1 h (Fig. 4b).

The enthalpy change for R-phase transformation during cooling and the enthalpy change due to the sum of R-phase – and austenite transformation in the heating cycle is shown in Fig. 5. An obvious influence in thermal transformation behaviour was revealed for an annealing temperature higher than 400 °C (one hour). The reverse stress of reformation to martensite and the strain hysteresis due to pseudoelastic formation and reformation of martensite (elastic deformation was



**Fig. 2:** Micrograph of orthodontic NiTi when heat treated at 360 °C/1 h (a) and 440 °C/1 h (b).

taken out) are given in Fig. 6. When the annealing temperature was increased the strain hysteresis became larger and a considerable decrease in the stress level of reverse transformation to austenite was found.

## 4. Discussion

### 4.1 Transformation behaviour

#### Microstructure and transformation behaviour

In the as received condition the orthodontic NiTi wire is of entirely plastically deformed microstructure (Fig. 1). As already shown, a signal from thermal transformation, whether it was taken by DSC or by electrical resistivity measurement (ER), could be perfectly depressed by cold work [11]. Microstructural observations, dealing with the effect of heat treatment on a plastically deformed NiTi alloy, will be the point of interest in the following. In addition, changes in thermal and mechanical transformation behaviour will be discussed.

An obvious influence on microstructure was observed for a 360 °C/1 h heat treatment. Fine striations, parallelly orientated, became visible in each subgrain (Fig. 2a). No consequence on the thermal transformation behaviour could be found due to this heat treatment (Fig. 3a, Fig. Fig. 5). In addition, no significant change in deformation behaviour could be detected (Fig. 4a).

Recrystallization started when the work hardened NiTi wire was annealed at 440 °C/1 h. Furtheron, though there was no principal change in thermal transformation behaviour, the heat of transformation started to change quantitatively. The signal belonging to R-phase transformation decreased while that from the sum of R-phase- and austenite transformation started to increase (Fig. 5). The deformation behaviour has changed by a dramatical decrease in reverse flow stress and an increase in strain hysteresis from 3 % up to 6 % (Fig. 6). On one hand, there was still no martensitic transformation to be detected (DSC) in a partially recrystallized microstructure while on the other hand an obvious change in pseudoelastic deformation behaviour took place.

When the orthodontic wire was annealed at 500 °C/1 h, the change in thermal transformation behaviour was marked by the occurrence of martensitic transformation signal in DSC scanning. Parallel to this behaviour, the increase in the reverse transformation enthalpy was documented (Fig. 5). The enthalpy change, taken from the sum of R-phase- and austenite transformation, has increased from about 5 J/g to 20 J/g. Due to the placement of transformation temperature range in this alloy as well as the environmental temperature, there was no more pseudoelastic recovery to be observed (Fig. 4b). Strain, produced after a 500 °C/1 h heat treatment, will be recovered only after heating up above the finish temperature of reverse transformation to austenite.

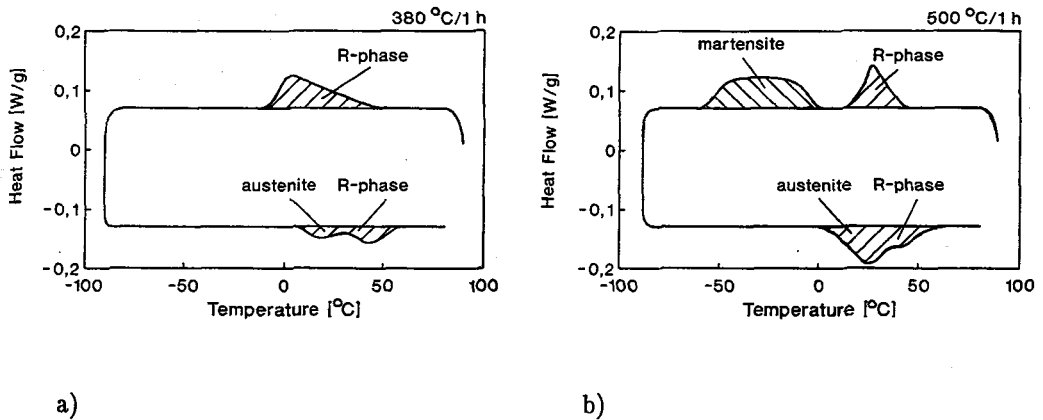


Fig. 3: DSC signal of work hardened orthodontic NiTi wire when annealed at 380 °C/1 h (a) and 500 °C/1 h (b).

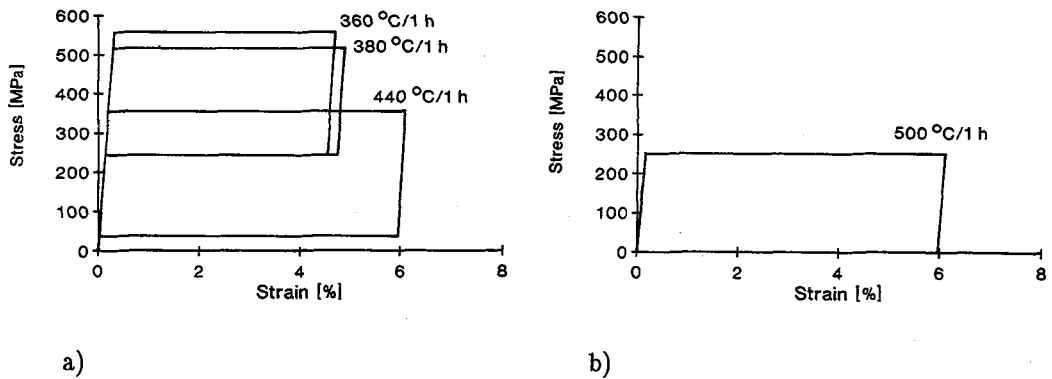


Fig. 4: Reversible stress strain behaviour of work hardened orthodontic NiTi after heat treatment at 360-, 380-, 440 °C/1 h (a) and quasiplastic behaviour after 500 °C/1 h annealing (b)

Thermal and stress induced transformation

The heat of transformation, detectable for heat treatment temperatures lower than 500 °C (one hour) are shown principally in Fig. 3a and summarized quantitatively in Fig. 5. Martensitic transformation in thermal cycling is suppressed by cold work, although a double peak, indicating R-phase- and reverse martensite to austenite transformation, was found in each DSC scan. However, the quantity of thermal martensite to austenite transformation is quite small. The sum of R-phase- and austenite transformation kept a constant level of about 5 J/g for heat treatment temperatures lower than 400 °C.

Although there was no or at least a very small amount of martensitic transformation detectable in thermal analysis (DSC), considerable values of stress induced formation of martensite were recorded during uniaxial tensile testing. For heat treatment temperatures lower than 400 °C/1 h a strain hysteresis of about 4 % was measured.

Thermal transformation to martensite seems to be suppressed (Fig. 3a) due to plastic deformation (Fig. 1), whereas perfect pseudoelasticity, presumably based on reversible stress induced formation of martensite (SIM), was detected during uniaxial tensile testing (Fig. 4a).

## 4.2 Orthodontic application

The characteristic features (Fig. 6), which are of interest to the orthodontists are the driving force for the tooth correction i.e. the reverse transformation stress level and the range of working (strain hysteresis, SIM).

An additional heat treatment of the as drawn wire might be necessary to achieve a defined shape as well as to use the chance of correct tailoring of the transformation behaviour. As visualized in

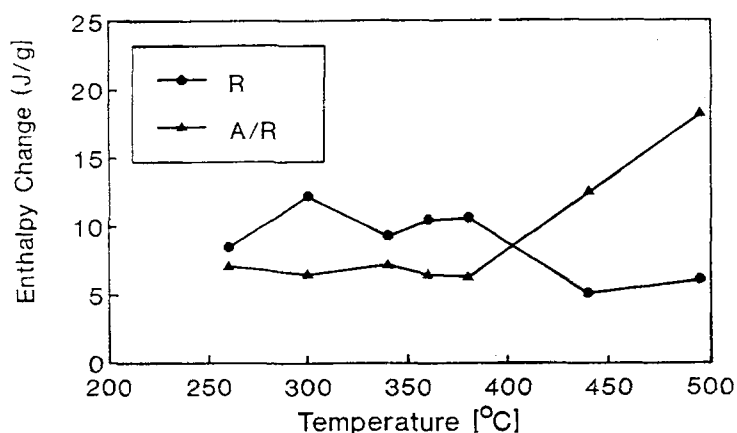


Fig. 5: Enthalpy change from the R-phase transformation (R) and from the sum of the R-phase and austenite transformation (A/R) during cooling and heating cycle in DSC scanning. Data were recorded for 260 °C/1 h up to 500 °C/1 h annealing.

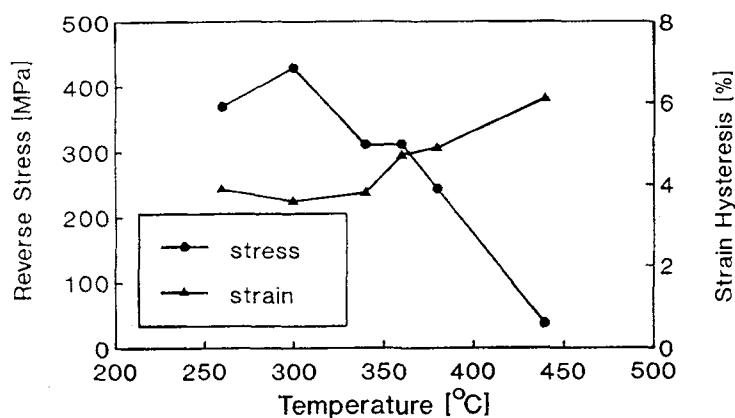


Fig. 6: Strain hysteresis (strain) and reverse transformation stress level (stress) for pseudoelastic orthodontic NiTi wire when heat treated between 260 °C/1 h and 440 °C/1 h

Fig. 6, the heat treatment at various temperatures is always a compromise between an enlargement of the strain hysteresis and the reduction of reverse transformation stress level. Applied on orthodontic needs, there is the chance to get a larger range of working for corrective tooth movement and it became possible to put the driving force during tooth movement on a physiological level.

It has to be pointed out that information about fatigue effects, according to the pseudoelastic behaviour described here, can not be drawn out of these investigations.

## 5. Conclusions

A work hardened orthodontic NiTi wire (Nitinol SE) has been heat treated at several temperatures. The description of the microstructural changes and relating variations in thermal and mechanical transformation behaviour have been part of the investigation. The order of appearance of phenomena which have been detected was found to be:

Fine striations were observed in an all over deformed matrix when the annealing was carried out at 360 °C/1 h.

A quantitative change in the enthalpy of transformation becomes detectable when the heat treatment is done at about 400 °C/1 h. The signal due to the sum of R-phase- and austenite transformation started to increase. Parallel to this, the reverse transformation stress started to decrease.

A partially recrystallized microstructure became visible after a heat treatment at 440 °C/1 h. Pseudoelasticity was still observed.

When the annealing was carried out at 500 °C/1 h, martensitic transformation became detectable in thermal analysis. No more pseudoelastic behaviour could be measured.

The application of heat treatment on an orthodontic NiTi wire involves the chance of correct tailoring of the transformation behaviour. The working range for corrective tooth movement will be enlarged and the driving force during tooth movement can be put on a physiological level. Furtheron, heat treatment at an appropriate temperature provides the possibility to create defined shaped devices.

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