



POLISH ACADEMY OF SCIENCES - COMMITTEE OF MATERIALS SCIENCE
SILESIA UNIVERSITY OF TECHNOLOGY OF GLIWICE
INSTITUTE OF ENGINEERING MATERIALS AND BIOMATERIALS
ASSOCIATION OF ALUMNI OF SILESIA UNIVERSITY OF TECHNOLOGY

Conference
Proceedings

12th INTERNATIONAL SCIENTIFIC CONFERENCE
ACHIEVEMENTS IN MECHANICAL & MATERIALS ENGINEERING

Structure and properties of NiTi and TiNiCo shape memory wires for maxillofacial surgery

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X-ray diffraction and DSC methods for studies of the structure and courses of phase transitions and their characteristic temperatures after thermomechanical treatments of superelastic NiTi and of memory TiNiCo wires were used. At room temperature the NiTi wires have a parent phase structure, whereas TiNiCo wires have a martensitic structure. In the alloy with cobalt addition after treatment the transition sequences $B2 \leftrightarrow R \leftrightarrow B19'$ during cooling and reheating were observed. After select treatments the wires with desired temperature range of shape recovery near body temperature and between 40 to 55°C were obtained. The stress-strain curves of NiTi superelastic wires of various diameters during cyclic extension in the Instron machine shows a characteristic hysteresis loop and constant stress in a wide displacement range. The superelastic behaviour of NiTi wires was observed also during deformation by 3-point bending tests which were carried out during stretching and unloading at a specially constructed measuring device, equipped with the Hottinger force converter and linear variable differential transducer with a recorder connected with a computer. From wires with the desired properties the clamps for mandible osteosynthesis and expansible springs for elongation of mandible bones were prepared.

1. INTRODUCTION

In recent years NiTi shape memory alloys have been widely applied as implants in medicine. They have good mechanical properties, corrosion resistance and biocompatibility. The orthodontic archwires, endosurgical stents, Simone filters and orthopaedic staples are especially widespread. This application uses superelastic properties and shape recovery effects [1-3]. The phenomenon of superelasticity can be observed when the material undergoes deformation in the parent phase in the vicinity of temperature A_f . The stress martensite that is formed during stretching disappears when the loading is removed and material goes back to its original shape in a superelastic way. When the effect of shape memory is used, the element deformed in martensitic state below temperature M_f goes back by itself to its original shape by heating in the temperature range of the reverse transition from A_s to A_f temperature. In both cases large forces are generated during transition.

This paper presents a fragment of structure and properties studies of a superelastic NiTi and of memory TiNiCo wires which were used for the preparation of clamps for human mandible osteosynthesis and prototypes of distractors for experimental elongation of mandible bones [4]. It was necessary to select the alloys and thermomechanical treatments for obtaining the wires with the suitable properties.

2. MATERIALS AND EXPERIMENTS

Superelastic NiTi wires with diameters of 0.8 mm, 1.0 mm and 1.2 mm from SMATEC (Belgium) and our own TiNiCo alloy smelted and cast in a vacuum induction furnace were used. The ingot after the homogenizing treatment was processed by hot profiling into rods which after rotary forging were hot drawn into wires with diameters from 2 to 1 mm. In the final phase some of the wires underwent cold drawing. Quenching from the temperatures 800-700°C and the annealing in the temperature range 200-600°C were carried out in a resistance furnace, in the argon atmosphere. The temperature and angle of shape recovery were measured by bend and free recovery tests. After heat treatment the wires were dipped in ethanol cooled to about -60°C and bent to the angle of about 90°, and heated up to 70°C while the shape recovery angle and the temperature were measured. Conditions for the heat treatment were chosen and the wires were prepared which act at body temperature or in the temperature range of shape recovery from 40 to 55°C. X-ray diffraction phase analysis of wires was carried out using X-Pert Philips diffractometer with $\text{CuK}\alpha$ radiation. Diffraction patterns were recorded at an angle from 37 to 47° (2 θ). The X-ray temperature experiments were carried out on a self made attachment in which the electric heater and vapour nitrogen for heating and cooling were used. The DSC measurements during cooling and heating with the rate of 10 deg/min over the temperature range from -100 to +100°C were carried out using the TA-2100 calorimeter. Cyclic stretching of the superelastic wires was done in an Instron tensile test machine. Three-point bending tests were carried out at a specially constructed measuring point, equipped with an extensometer Hottinger force converter and Peltron transformer linear displacement indicator with an MC201 recorder connected with a computer.

2. RESULTS AND DISCUSSION

At room temperature the superelastic NiTi wires had the parent phase B2 structure, while TiNiCo wires after select heat treatment showed the martensite B19' structure (Fig. 1). By X-ray temperature diffraction the reversible $\text{B2} \leftrightarrow \text{R} \leftrightarrow \text{B19}'$ transition was recorded. The sequences of transitions changes in dependence of thermomechanical treatment (Fig. 2). DSC measurements shows that in TiNiCo alloy after quenching the phase transitions took place below room temperature. A slight increase of transition temperatures occurred after annealing (Fig. 3).

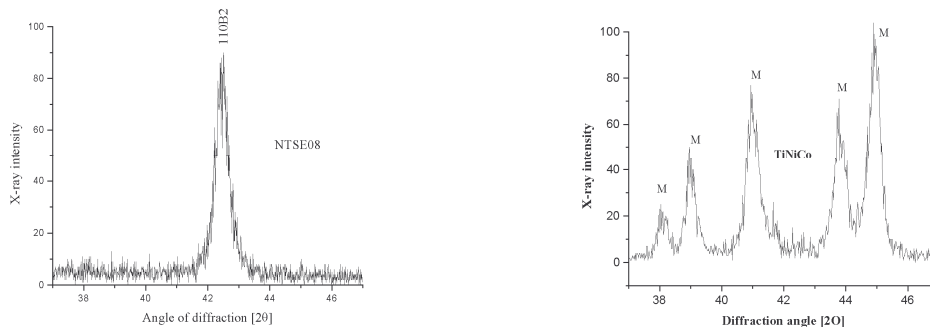


Fig. 1. Diffraction patterns recorded at room temperature. NiTi superelastic wire with the diameter of 0.8 mm in the parent phase structure - (on the left), TiNiCo shape memory wire with the diameter of 1.5 mm in the martensitic state - (on the right).

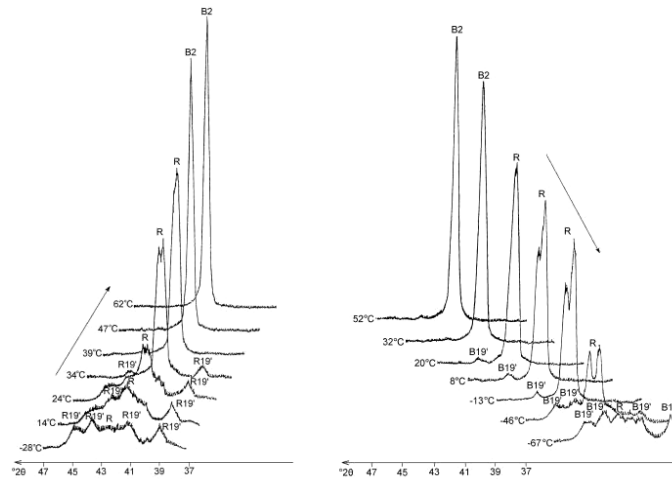


Fig. 2. Courses of phase transitions during heating and cooling in TiNiCo alloy after annealing at 400°C recorded by X-ray temperature diffraction.

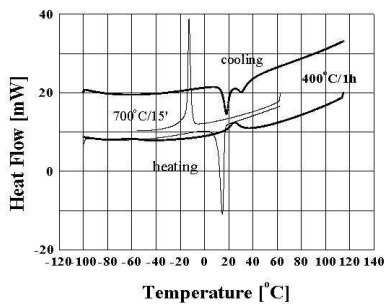


Fig. 3. DSC curves of TiNiCo samples after quenching from 700°C and annealing at 400°C.

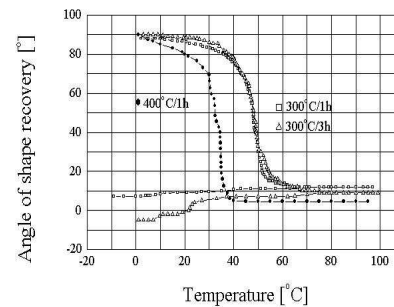


Fig. 4. Selection of heat treatment for obtaining wires with shape recovery temperature at body temperature and in the range 40 to 55°C.

Shift of the temperature range to body temperature and over the temperature of 40°C for the wires needed to make the implants activated by body heat or by current heating was achieved by annealing after cold drawing (Fig. 4). It was important that for implants heated by electrical current the reverse transition was ended at harmless temperature below 55°C. In tension and bending tests were proved that provided NiTi wires had the superelastic properties (Fig. 5, 6).

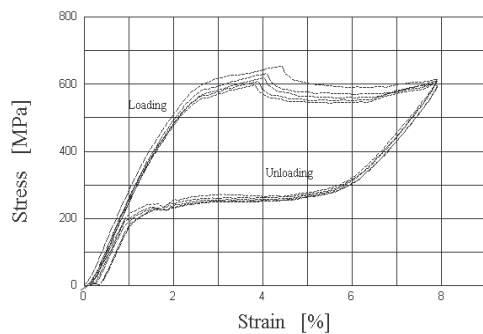


Fig. 5. Superelastic behaviour during cyclic tension of NiTi wire with diameter 0.8 mm.

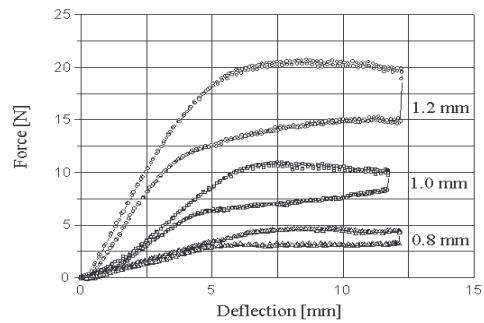


Fig. 6. Force - deflection curves for superelastic wires recorded by 3-point bending tests.

4. CONCLUSIONS

- X-ray diffraction analysis show that the superelastic NiTi wires at room temperature have a B2 structure, whereas TiNiCo wires have the martensitic B19' structure. During cooling and heating the reversible $B2 \leftrightarrow R \leftrightarrow B19'$ transitions were observed.
- By suitable processing and thermomechanical treatment it was possible to obtain wires with desirable shape recovery at body temperature or above 40°C.
- Studied NiTi wires have a desired superelastic properties for prepare expansible springs which acts with constant force in a wide range deformation.

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