Influences of Reduction Ratio on Mechanical Properties and Transformation Temperature of NiTi Drawn Wires.

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Abstracts

NiTi alloy wires are widely used in many applications for examples, orthodontic arch wires, mini screw implant, sensors, etc. Usually, wire drawing process is chosen to produce arch wires because of its high productivity as well as superior surface quality of the drawn wire surface. In the case of NiTi arch wire, the cold work occurred during wire drawing operation also plays important role to control mechanical properties and transformation behavior of the wires. Thus, the purpose of this work is to study the influences of reduction ratio of cross sectional area and annealing temperature on transformation and mechanical behavior of the drawn wires. The wire materials used in the experiment are Ni₅₁.₄Ti₄₈.₆ (atomic percentage) with initial wire diameter of 0.64 mm. The die tip is made of tungsten carbide which is inserted into the steel case made of tool steel. The reduction ratios of cross sectional area (%Re) are varied at three different levels, i.e., 10, 20 and 30%, respectively. In order to study the influences of post heat treatment temperature, the drawn wires are annealed for 3,600 sec at two different levels; 400 and 600°C. The lubricant used in the experiments is sodium stearate powder. From the results, drawing force and surface roughness of the drawn wire strongly depend on %Re. In addition; tensile properties and phase transformation temperature noticeably depend on heat treatment temperature.

Keyword: NiTi arch wire, Wire drawing, Reduction ratio, Heat treatment temperature, Phase Transformation temperature, Shape memory alloy
1. Introduction

Shape memory alloys (SMA) are in the class of smart materials that have two unique properties, the shape memory effect (SME) and superelasticity (SE)[1]. SME is the ability of a material to recover to its original shape under heating, to a specific temperature after plastically deformed. Superelasticity is the ability to recover very high amounts of strain upon unloading [2]. NiTi alloys which are known to exhibit superior ductility, high strength, good corrosion resistance and biocompatibility, have resulted in many applications, among which use in the manufacture of wire forms figures clearly[3-5]. In wire applications, TiNi SMAs have been successfully applied as biomaterials such as orthodontic arch wires, guide wires and stent, in addition to many engineering applications. The successful application of NiTi wires requires control of both its transformational and mechanical properties [7]. Some papers have reported the properties of NiTi SMA can be manipulated through cold work, heat treatment or a combination of both [8]. The roadblocks to their development are caused by difficulties in the manufacturing process [5]. Therefore, the understanding of drawn wire properties is important for NiTi SMA applications [6]. However, there is little systematic investigation of the drawing properties of NiTi SMA has been reported. The purpose of this study is to investigate the NiTi Drawn Wires properties in wire drawing process under various reduction ratios of cross sectional area and annealing temperatures. The influence of degrees of reduction ratio and heat treatment temperatures will be discussed.

2. Experimental procedure

The material used in this experiment is commercial Ni_{51.4}Ti_{48.6} (at %) round wire having initial diameter of 0.64 mm. Before wire drawing residual stress was removed by annealing at 600 °C for 3600 s.

The die tip made of tungsten carbide with an approach angle (2α) of 20 degree. The wires were drawn at the reduction ratios of cross sectional area of 10%, 20% and 30% respectively. The lubricant used in the experiments is sodium stearate powder.

The experimental apparatus is shown in Figure1. The drawing speed was controlled at 30 mm/min. The universal testing machine is used and applied in order to monitor the drawing force on real-time. After drawing process, the specimens were annealed at 400 °C and 600 °C for 3600 s. Transformation temperatures were tested by using Differential Scanning Calorimeter (DSC). During the test temperature was varied in the range of -50°C to 100°C with cooling and heating rate of 10°C/min. Tensile properties were determined in accordance with JIS Z2201 with strain rate of 5 mm/min.
3. Results and Discussion

3.1 Drawing force

Figure 2. Drawing force versus reduction ratio of cross-section area of drawn wires.

Figure 2 shows the results of drawing force for the NiTi wires with various reduction ratios tested at room temperature. It is found that the drawing force increases with increasing degree of reduction ratio because the drawing force strongly depend on the amount of deformation during the drawing process. Moreover, it can be also clearly understood that the friction coefficient and adhesion between wire and die increase with increasing degree of reduction ratio.

3.2 The surface roughness

Figure 3. Average surface roughness values versus reduction ratio of cross-section area.

Figure 3 shows average surface roughness (Ra) of conventional NiTi SMAs wire annealed at 600 °C followed by wire drawing process with various reduction ratios. The roughness is measured in circumferential direction in order to detect the scratches, which occurs in drawing direction. The surface qualities of the drawn wires improve considerably when increasing degree of reduction ratio. However, quality of the drawn wires obtained is lower than those of the initial wire. Since the thin film oxide was created by annealing, which used...
as lubricant during drawing process. This oxide film will damage on the surface during drawing process and affect on the wires surface quality [5,11].

### 3.3 Tensile properties

Figure 4 and 5 show the results from tensile test of the drawn wires with various reduction ratio after heat treatment at 400 °C and 600 °C respectively. The increasing of degree of reduction ratio increased the tensile strength and yield strength but decreased the percentage of elongation after heat treatment at 400 °C. Since cold work in the wire drawing process is expected to increase dislocations density resulting in higher strength [8]. However, there was no significant difference in ultimate tensile strength and Yield strength after heat treatment at 600 °C. Since this temperature is higher than recrystallization temperature which is about 526 °C [10].

**Figure 4.** Ultimate tensile strength and Yield strength versus Reduction ratio of cross-section area of NiTi drawn wires after heat treatment at 400 and 600 °C

**Figure 5.** Percentage elongation versus Reduction ratio of cross-section area of NiTi drawn wires after heat treatment at 400 and 600 °C
3.4 Transformation temperature

Figure 6 shows the effect of reduction ratio on phase transformation temperatures of the NiTi drawn wires after heat treatment at 400°C. The peaks of the DSC curve represent for the phase transformation taken place by temperature change. The peak R, M and A represent the transformations of rhombohedral (R-phase). Martensite phase (B19) and Austenite phase (B2). It indicates that the martensite and the R-phase transformation occurs in the cooling curves, while only reverse transformation to Austenite phase can be confirmed in the heating curves. It is known that R-phase transformation can be induced by wire drawing followed by 300-450 °C interannealing [5] and easily found in Ni-rich NiTi alloys [9]. Austenite temperature decreases with increasing reduction ratio because the transformation is suppressed by internal stress due to cold work. In other words, the internal structure of the work-hardened is composed of multiple dislocations that hinder the phase transformation [11]. However, for specimen heat-treated at 600 °C, no significant effect from reduction ratio on transformation temperature can be observed. R-phase transition disappears in cooling curve, Since temperature at 600°C is higher than recrystallization temperature of NiTi alloys which remove all dislocation in the structure that result unaffected of cold work with various reduction ratios.

**Figure 6.** DSC curves of NiTi drawn wires cold rolled at 10%, 20% and 30% reduction ratio followed by heat treatment at 400 °C for 3,600 s

4. Conclusions

This study investigates the effect of reduction ratio on mechanical properties and transformation temperature of NiTi wires in wire drawing process. The following conclusions are drawn from this study.

1) In wire drawing process of NiTi alloy, the amount of drawing force strongly depend on the amount of cold work at various reduction ratios. Moreover, surface quality of drawn wires improves after drawing process.

2) Mechanical properties and transformation temperatures strongly depends on reduction ratios after heat-treatment at 400 °C which is the temperature under recrystallization temperature.
3) No significant difference in mechanical properties and phase transformation temperatures was found after heat treatment at 600 °C irrespective of reduction ratio, since dislocations from cold work were removed by recrystallization.

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