Effect of short-time direct current heating on phase transformation and superelasticity of Ti–50.8at.%Ni alloy

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1. Introduction

Superelastic TiNi wires have the ability to change their shapes, thus to output mechanical work, in response to temperature variations as well as stress changes [1]. Owing to this unique property, these wires have been used in a wide range of practical applications, most notably as orthodontic arch wires in biomedical area [2–4]. TiNi arch wires have the most mildly force delivery and are highly popular in clinical use [5]. However, this kind of arch wires cannot be easily deformed because of their excellent superelasticity, so, in such application, electrical heating is often used as the means for shape setting [6]. Near-equiaxotmic TiNi alloys have high electrical resistivities, typically 80 μΩ cm for the martensite phase and 100 μΩ cm for the austenite phase, and thus can be easily and rapidly heated to activation temperatures [7].

Whereas electrical heating provides easy and practical means for processing TiNi materials and for operation of TiNi components in designs, it has the problem of causing irreversible structural variations, such as anneal to cold worked structures [8] or ageing to cause formation or overgrowth of precipitates [9], due to overheating. These changes in microstructure inevitably alter the transformation behaviour and functional properties of the alloys [10,11]. Wang et al. investigated the effect of electrothermal heating on the transformation behaviour of a cold worked Ti–49.8at.%Ni alloy wire and found that the heating is effective in shifting the B2 → R phase transformation to lower temperatures and the R → B1′ transformation to higher temperatures, demonstrating the effectiveness of direct electrical heating for local anneal [12].

In clinical practice, heating time for orthodontic wire setting is typically several seconds. Knowledge of the effects of such short-time electrical heating is critical for the design and application of such wires. However, such information is not available in the literature. This study investigated the effect of short-time DC heating on the transformation and mechanical behaviour of Ti–50.8at.%Ni, a typical industrial alloy for superelastic applications.

2. Experimental procedure

The material used was a Ti–50.8at.%Ni alloy sheet. The as-received material was in 15% cold-rolled condition with 230 μm in thickness. The sheet was cut into 4 mm × 105 mm strips by means of electric-discharge machining. Some strips were heat treated at 673 K for 1.8 ks and then quenched in water, denoted the aged condition. Some strips were heat treated at 973 K for 1.8 ks and then quenched in water, denoted the annealed condition. All samples were mechanically polished on both sides. The thickness of the polished samples was ~210 μm. DC heating was performed using a Shinwa Bender Soarer-II equipment (Shinwa Corporation, Tokyo, Japan), which is designed for TiNi orthodontic arch wire shape setting. The heating gauge length was 105 mm and the switch used was No. 9 (power level), which gave a voltage of 1.6 V and a current of 13.5 A during heating. The surface temperature
was determined using a K-type thermocouple attached to the center of the sample. Fig. 1 shows the surface temperature of the sample as a function of DC heating time in still air. It is seen that the heating was very rapid, reaching a steady state of 700 K after 20 s.

Transformation behaviour of the samples was characterized by means of differential scanning calorimetry (DSC) using a PerkinElmer Diamond calorimeter with a cooling/heating rate of 20 K/min in nitrogen atmosphere. X-ray diffraction for phase identification was conducted using a Phillips X’Pert diffractometer with a Cu Kα radiation (λ = 0.1540598 nm) at room temperature. Tensile tests were carried out using an Instron 3365 testing machine at a strain rate of $2 \times 10^{-4}$/s at room temperature (300 K).

3. Results and discussion

Fig. 2 shows DSC measurement of the transformation behaviour of the samples after short-time DC heating for various durations. The as-received samples, as shown in Fig. 2(a), showed a single step $A \rightarrow M$ transformation. DC heating did not have obvious influence on the transformation behaviour of the samples. The annealed samples, as shown in Fig. 2(b), also showed a single step

![Fig. 1. Surface temperature of sample during DC heating.](image)

![Fig. 2. DSC curves of Ti–50.8 at.%Ni alloy samples treated by the short-time DC heating for different durations. (a) As-received samples, (b) annealed samples, (c) aged samples.](image)
A→M transformation behaviour, similar to the as-received samples. The samples also exhibited negligible response to DC heating, as expected for the annealed condition.

In contrast, the aged samples, as shown in Fig. 2(c), showed sensitive response to DC heating. The samples initially exhibited an A→R→M two-stage transformation behaviour. DC heating for short periods caused a progressive increase of the temperature of the R→M transformation. After 8 s of heating, the alloy exhibited quite different transformation behaviour. All transformation peaks have become suppressed in intensity. The A→R and R→M transformations have obviously moved to lower temperatures and the M→R and R→A transformations have merged. With further DC heating, the temperatures of the R→M (and A→M) and M→A transformations remained little changed whereas the A→R transformation diminished after 15 s of heating, resulting in a single-stage A→M transformation.

Fig. 3 shows the effect of DC heating time on the transformation temperatures for all samples. The as-received and the annealed samples showed little variation with their transformation temperatures over time during DC heating. The aged samples, on the other hand, showed drastic changes between 6 and 10 s of heating. The aged samples, on the other hand, showed drastic changes between 6 and 10 s of heating. The as-received and the annealed samples exhibited quite different transformation behaviour. All transformation peaks have become suppressed in intensity. The A→R and R→M transformations have obviously moved to lower temperatures and the M→R and R→A transformations have merged. With further DC heating, the temperatures of the R→M (and A→M) and M→A transformations remained little changed whereas the A→R transformation diminished after 15 s of heating, resulting in a single-stage A→M transformation.

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Fig. 4 shows tensile stress–strain curves of Ti–50.8at.%Ni alloy treated by the short-time DC heating for different durations. (a) As-received samples, (b) annealed samples and (c) aged samples.

Fig. 5 shows XRD spectra of the samples at room temperature. The as-received alloy (Fig. 5(a)) was predominantly of the B2 phase, with trace amount of the B19′ phase. DC heating did not cause any obvious change to the phase formation. The annealed samples (Fig. 5(b)) were practically the same as the as-received alloy. The aged samples, as shown in Fig. 5(c), exhibited a complex mixing of various phases, with the R and the B2 being the main phases and small amounts of B19′ and Ti3Ni4. DC heating for up to 7 s did not cause noticeable changes. Heating for 8 s and longer led to the B2 parent phase becoming the main phase and the disappearance of Ti3Ni4.

It is seen in Fig. 1 that the surface temperature reached a steady 700 K during DC heating. The interior temperature is expected to be higher. Based on consideration of the geometry and thermal conductivity of the alloy, the interior temperature could be estimated to be approaching 900 K. Such temperatures are sufficient to cause anneal to cold worked NiTi and precipitation or changes to precipitate structures of Ni-rich alloys.

Considering the shape memory effect, the nominal 15% cold-rolling of the as-received material is only slightly beyond the limit of transformation strain, thus is expected to produce very low levels of structural defects. This is consistent with the observation that the transformation behaviour of the as-received samples was practically identical to the anneal samples, except at a lower temperatures. Short-time DC heating caused very little change to the phase transformation behaviour or the superelasticity even though the temperature may have reached the recrystallization point (873 K).

For the annealed samples, it is evident that DC heating did not cause any noticeable change to the transformation and deformation properties. Furthermore, XRD analysis also indicates that no precipitates were formed in the matrix. These observations imply that at the DC heating temperature the alloy is within the solubility limit of Ni for Ti3Ni4 in the B2 phase, consistent with a previous study (833 K for a Ti–50.8at.% alloy) [13].

For the aged samples, the original ageing at 673 K for 1.8 ks induced coherent Ti3Ni4 precipitates from the TiNi matrix, as evidenced by the XRD results shown in Fig. 5(c) and the occurrence of the R-phase transformation (Fig. 2(c)). DC heating for as short as 8 s is effective in causing drastic changes to the transformation and mechanical behaviour. The change of the transformation sequence from A→R→M to A→M is indicative of the disappearance of the

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**Fig. 3.** Effect of DC heating time on transformation temperatures of Ti–50.8at.%Ni alloy.

**Fig. 4.** Tensile stress–strain curves of Ti–50.8at.%Ni alloy treated by the short-time DC heating for different durations. (a) As-received samples, (b) annealed samples and (c) aged samples.
effect of Ti₃Ni₄ precipitates. This could be a consequence of either of two possibilities: dissolution of Ti₃Ni₄ into the matrix or destruction of the coherency of Ti₃Ni₄ with the B2 matrix. The XRD profiles shown in Fig. 5(c) reveal the disappearance of Ti₃Ni₄ after heating. The DSC results shown in Fig. 2(c) demonstrate that the transformations of the aged samples after heating were practically the same as those of the annealed samples, suggesting similar Ni contents in the matrix. In addition, the conclusion above from the annealed samples that the heating temperature was within the solvus of Ni for Ti₃Ni₄ also supports the Ti₃Ni₄ dissolution scenario. All these evidences seem to suggest that short-time DC heating (∼15 s) is sufficient to cause dissolution of Ti₃Ni₄ in Ti–50.8at.%Ni.

The progressive increase of the critical stress for inducing the A → M transformation and the realization of the superelasticity at the room temperature (Fig. 4(c)) with increasing heating time is obviously related to the progressive decrease of Tₐ↔M. Because the diminishing of Ti₃Ni₄ particles caused by DC heating, made the Ni content in the matrix gradually increased. So the Aᵣ temperature (M → A finishing temperature) decreased and when Aᵣ temperature was lower than the tensile testing temperature (RT in the present study), the superelasticity would appear.

4. Conclusions

In summary, it is evident that DC heating is sufficient to cause marked changes to the transformation and mechanical properties of aged Ti–50.8at.%Ni alloy, even after very short times in the order of several seconds. For applications where such conditions apply, e.g., orthodontic arch wires, caution should be taken when applying DC heating. Short-time DC heating within a few seconds (e.g., <6 s) may be safe without changing the original properties. Longer DC heating (>10 s) may improve the superelasticity of the orthodontic wires and increase the alignment forces. Prolonged DC heating (e.g., >20 s) may deteriorate the superelasticity.

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References