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Influence of ageing treatment on the thermophysical characteristics and mechanical properties of forging wire Ni-rich NiTi alloy for superelastic applications

P.F. Rodrigues[a,e,*], F.M.B. Fernandes[a], E.N. Texeira[b], S. Baptista[c], A.S. Paula[c,d], J.P. Oliveira[a]

[a]CENIMAT, Materials Science Dept, Faculdade de Ciências e Tecnologia, Universidade NOVA de Lisboa, Portugal
[b]Centro Universitário de Volta Redonda - UNIFOA, Brazil
[c]Metallurgical Engineering Pos-graduation Program - PPGEM, Universidade Federal de Fluminense – UFF, Brazil
[d]Mechanical and Materials Engineering Department - SE-4, Instituto Militar de Engenharia – IME, Brazil
[e]CAPES – Foundation Ministry of Education of Brazil, Brasília – DF – Brazil

Abstract

NiTi alloys used for orthodontic applications need to show superelastic characteristics at room and oral temperatures. The ideal scenario is that where the material has a final austenitic phase transformation value below the room temperature. This study aims at understanding the influence of the ageing treatments in the austenitic structure at room temperature on a wire of a Ni-rich NiTi alloy produced by rotary forging by the evaluation of the phase transformation temperatures and mechanical behaviour in order to promote the superelastic behaviour at room temperature. The investigation was conducted using DSC (Differential Scanning Calorimetry) analysis and instrumented ultramicrohardness. The solubilisation at 950°C for 120 min with water quenching showed a satisfactory amount of B2 phase at room temperature when compared to the sample after forging. After solubilisation, ageing treatment at 350°C for 30 min gave a relatively higher hardness value and an A_f temperature below the room temperature, ensuring the presence of austenitic phase at room and oral temperatures.

Key words: Ni-Ti alloys; solubilisation; orthodontic applications; differential scanning calorimetry; ultramicrohardness.

1. Introduction

To properly achieve the special characteristics of shape memory effect and superelasticity in the development of NiTi, a strict control during the manufacturing processing steps of melting, refining, thermal and mechanical's treatment must be carried out [1]. Thermal and/or thermo-mechanical treatments, Ni and Ti content, the addition of alloying elements and presence of impurities (C and/or O) are factors that can influence the structure, shape memory effect and superelasticity at a given working condition.

As the presence of undesired contaminants contribute to the decrease in transformation temperatures due to formation of fragile phases, when these are massively produced, they can promote premature failure. Therefore, follow-up of the chemical composition and the thermo-mechanical process variables is mandatory [2,3]. The products made from NiTi need to have hot and cold forming in the same process, since the effects produced by the cold forming processes and heat treatments are of great importance for the properties exhibited by materials. This cold processing aims to provide suitable surface finishing, refinement of the microstructure and mechanical properties of the material [4]. Thus, when it is desired to obtain distinct properties, such as superelasticity and shape memory, these alloys require an adequate thermal treatment

* Corresponding author.

E-mail address: pf.rodrigues@campus.fct.unl.pt (P. Rodrigues)

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after the forming step. The phase transformation temperature adjustment in Ni-rich alloys is done by thermal ageing treatments. This study aimed at understanding the influence of the ageing treatment to obtain austenitic structure at room and oral temperatures (35-37ºC) on a wire made with a Ni-rich NiTi alloy, produced by rotary forging, and to evaluate the transformation temperature phase and mechanical behaviour, to promote the superelastic behaviour at room temperature.

2. Material and Methods

2.1. Material

The sample supplier describes that the alloys in the present work are Ni-rich and were produced by melting in a vacuum induction furnace [3]. A slice of the ingot, designated as VIM74, was used in this study, which has approximately 49.2 at.% Ti and 50.8 at.% Ni. Subsequently, the ingot was subdivided into smaller samples, about 90 g each, using electroerosion technic in order to further recast in the arc melting furnace. After being remelted, it was submitted to the thermomechanical rotary forging process, starting with hot mechanical forming sequence, followed by cold work interspersed by steps of heat treatment, as shown in Fig. 1 – resulting in After Forged (AF) sample with 3.31 mm.
To achieve the proposed objective, the thermophysical characteristics and mechanical properties of the alloy were studied after solubilisation at 950°C for 120 min (SOL sample), followed by comparative aging steps at 350ºC, 400ºC and 450ºC for 30 min (Fig. 2).

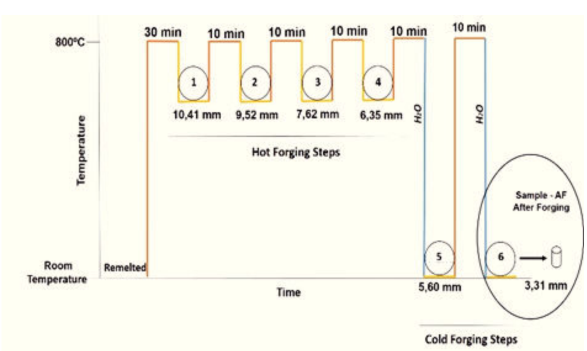

Fig. 1. Thermo-mechanical process.

2.2. Methods

The characterization was done by Differential Scanning Calorimetry (DSC) and analysis of instrumented nanoindentation. For the DSC analysis a DSC 204 F1 Phoenix was used, with thermal cycles from -150ºC to 150°C and heating and cooling rate of 10°C/min.

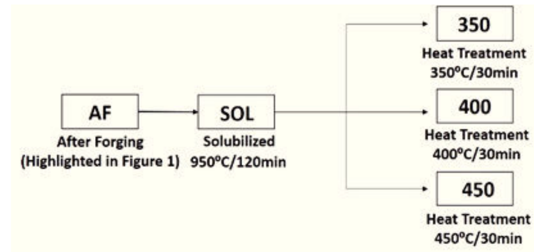

Fig. 2. Samples' processing scheme.

The start and finish temperatures of the phase transformations were determined after each heat treatment to assess their influence on the transformation behaviour of the processed material.
The DSC curves are represented in graphs which show heat flow per unit mass (mW/mg) versus temperature (°C). The ultramicrohardness measurements were performed using an instrumented nanoindentator (Shimadzu, DUH-211S model). The tests were conducted with maximum load at 250 mN (25 gf), with 60 increments on load/unload and 0.5s between increments. The creep time on maximum load was 20s.

3. Results and Discussion

Fig. 3 shows the DSC curves of the samples after forging (AF) and after solubilisation at 950°C for 120 minutes (SOL). The solubilisation treatment promotes a decrease of A_f temperature. The homogenization resulting from the solubilisation treatment may be considered as an efficient preliminarily treatment for the following aging steps.
Fig. 4 shows the DSC curves for the samples: after forging (AF), solubilized (SOL) and aged (350, 400 and 450ºC/30 min).
The austenite presence at room temperature is highlighted in the DSC plots (Fig. 4). Table 1 shows the values of the A_f temperatures for the different conditions tested on this work. It is possible to verify that the A_f for the sample aged at 350°C/30 min is below room temperature, which ensures the superelastic condition at the working temperatures.
It can be seen that the samples aged at 400°C and 450°C have A_f values higher than room temperature. This may be related to the presence of the precipitates Ni_4Ti_3.
By observing the routes chosen for ageing heat treatment (350°C, 400°C and 450°C for 30 min with subsequent cooling in water at room temperature), it can be observed that the treatment at 350°C showed a

relatively higher hardness value (Fig. 5). Increasing the ageing temperature, the hardness decreased as a result of a decreasing amount of austenite present at room temperature.

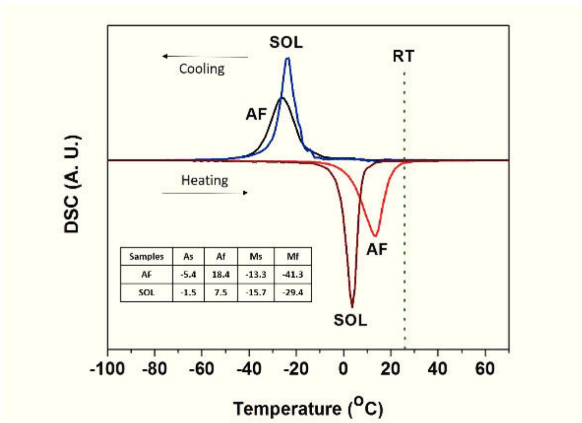

Fig. 3. DSC test (AF = After Forging; SOL = Solution Heat Treatment).

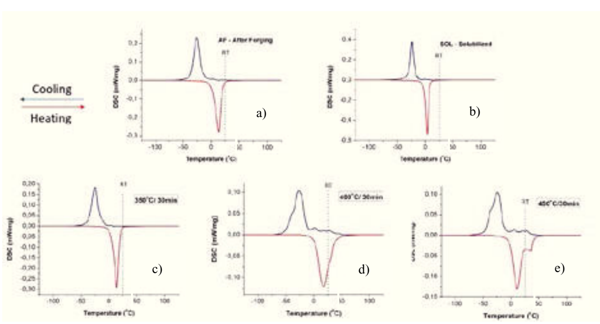

Fig. 4. DSC curves – a) AF - After forging and b) SOL - Solubilized at 950ºC for 120 min. Ageing temperature (30 min soaking time): c) 350ºC, d) 400ºC and e) 450ºC.

Table 1. Values of the austenitic transformation final temperature.

Samples	A_f(ºC)
After forging (AF)	18.4
Solubilized (SOL)	7.5
350ºC/30 min	20.0
400ºC/30 min	37.5
450ºC/30 min	39.6

Fig. 5 shows the relationship existing between A_f and the hardness values for the samples in different conditions. The room temperature is marked on the graph to provide a better understanding of the hardness values measured at room temperature for the different ageing conditions.

The ageing temperature is increased, the A_f temperature increases and the hardness decreases, possibly due to the formation of precipitates in the matrix that reduces the amount of Ni content and corresponding Ti enhancement [6].

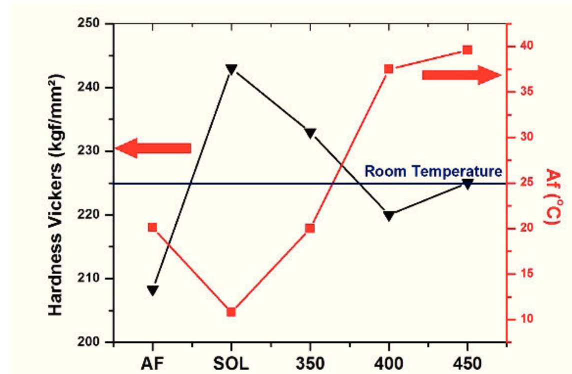

Fig. 5. Comparative Vickers hardness and final austenitic transformation temperature at room temperature.

4. Conclusions

It can be concluded that the increase of the ageing temperature and time in the ageing heat treatments proposed probably promoted increasing precipitation of Ni_4Ti_3, thus increasing Ti content and giving increased A_f temperature.

The ageing proposed at 350°C/30 min was the best route among the conditions tested in this work since, at this condition, A_f is below room temperature and, on the other hand, the hardness is higher than the values obtained for 400 and 450ºC.

This route satisfies the goal proposed once the wires must exhibit superelastic characteristics at room and oral temperatures.

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