Rate Dependency During Relaxation of Superelastic Orthodontic NiTi Alloys After Hydrogen Charging

Wissem Elkhail Letaief¹ · Tarek Hassine¹ · Fehmi Gamaoun¹

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Abstract The relaxation behavior under tensile loading of a superelastic NiTi alloy was investigated after hydrogen charging with respect to aging from one to 77 days in air at room temperature. The specimens were immersed for 3 h in a 0.9 % NaCl aqueous solution and then relaxed with an imposed strain of 4.8 %—which results in half of the martensite transformation—for different strain rates of 10^{-4} , 10^{-3} , and $5 \times 10^{-3} \text{ s}^{-1}$. For the non-charged specimens, the relaxed stress at the beginning exhibited a temporary dependence on the strain rates and then reached the same equilibrium stress after 2.5 h. After hydrogen charging, this equilibrium stress did not vary for the as-charged specimen. Nevertheless, the greater the aging period is the greater the equilibrium stress is. This behavior can be attributed to the diffusion of hydrogen into the entire specimen, which hinders the relaxation mechanism of the martensite bands.

Keywords NiTi · Hydrogen · Relaxation · Aging · Strain rate

Introduction

When they are fixed to teeth, NiTi orthodontic wires have a problematic situation since they may suddenly fracture due to hydrogen embrittlement. This phenomenon can be excited by a galvanic current between different materials located in the oral cavity and by the presence of hydrogen impurities that often reside in toothpastes and other oral hygiene products.

In this regard, studies have been performed to explain the hydrogen embrittlement effect on the mechanical properties of NiTi alloys that are characterized by the reduction of the ductility after hydrogen diffusion [1]. This behavior has been studied for different amounts of absorbed hydrogen, and it was reported that for an amount between 50 and 300 ppm, hydrogen was highly mobile; whereas at a greater concentration, it was trapped at interfaces and dislocations [2]. Moreover, it was shown that for an amount greater than 50–200 ppm, fracture occurred in a brittle manner at an earlier stage of plastic deformation [3]. In addition, in our previous work [4], we noticed that the occurrence of embrittlement was detectable, in particular, during the martensite transformation. This embrittlement was related to the distribution of hydrogen that diffused from the sub-surface into the bulk volume during aging. In addition, we found that after aging for 24 h, the NiTi alloy became brittle at low strain rates rather than for high strain rates [5]. This phenomenon was attributed to the interaction between the diffused hydrogen and the martensite band interfaces that nucleated and grew under the applied strain rate [6]. Furthermore, we showed that the increase in the current density had the same effect as the growth of the charging duration on the mechanical properties of the NiTi alloy to cause embrittlement [7]. Yokoyama et al. [8, 9] mentioned that the amount of

✉ Wissem Elkhail Letaief
wissem.khal@gmail.com
Tarek Hassine
tarekhassine007@gmail.com
Fehmi Gamaoun
fehmi.gamaoun@insat.rnu.tn

¹ Laboratory of Mechanics of Sousse, National Engineering School of Sousse, University of Sousse, Hammam Maarouf, 4054 Sousse, Tunisia

hydrogen absorption and the distance of hydrogen diffusion rose in the martensitic phase more than the austenitic one. Moreover, Tomita et al. [10] reported that after 240 h of aging in air at room temperature, hydrogen did not desorb. However, Pelton et al. [11] confirmed that hydrogen desorbs from the NiTi for a temperature greater than 400 °C.

Cheng et al. [12] performed X-ray diffraction and optical microscopy on a charged NiTi alloy and monitored hydride nucleation and growth at grain boundaries. In another work [13], it was noticed that the NiTiH-type hydrides developed after hydrogen charging at a current density of 10 A m^{-2} in <4 h, then decomposed and disappeared after aging 240 h at ambient conditions. In addition, Zhao et al. [14] indicated, in a recent study, that the hydrogen-induced martensite reduced with the formation and growth of the $\text{Ti}_2\text{NiH}_{0.5}$ -type hydrides that presented pinning points that increased sharply the internal friction. Also, Biscarini et al. [15] confirmed this increase in internal friction by performing a dynamic study at different temperatures and frequencies on a NiTi alloy charged with hydrogen and explained this behavior by activating a new relaxation process after hydrogen absorption [16, 17].

For the case of an as-received NiTi alloy, the relaxation stress has been investigated in various studies. It was shown that the strain rate has an important influence on the material's hardening during the austenite–martensite transformation. This influence affects directly the relaxation stress behavior by varying the initial relaxation stress. In return, the strain rate does not affect the stress equilibrium [18]. Helm et al. [19] assumed that strain rate dependency could be explained by the viscoelastic behavior of the material. On the other hand, most researchers have assumed that the rise in temperature, caused by latent heat during transformation, has been the primary cause of stress growth [20, 21]. Thus, Brinson et al. [22] controlled the specimen temperature during relaxation and noticed that the structure's temperature and the relaxation stress had the same rate of decline and reached the equilibrium stress at the same time. Furthermore, Grab et al. [23] performed relaxation experiments by controlling the structure's temperature in order to understand the isothermal relaxation behavior and noted no drop in stress during the imposed strain, even for the high strain rate.

The aim of this present research was to study the hydrogen charging and aging effects on a NiTi orthodontic alloy during the relaxation stress at different strain rates. This paper is divided into four sections beginning with an introduction and ending with conclusions. The second section details the experimental procedure while the third section presents the results of the relaxation and tensile experiments.

Experimental Methods

Commercial orthodontic wires with a chemical composition of 50.8 at.% Ni and 49.2 at.% Ti have been investigated in the present study. Table 1 contains the different mechanical properties and the transformation temperatures of the material. M_s (the martensite transformation start temperature), M_f (the martensite transformation finish temperature), A_s (the austenite transformation start temperature), and A_f (the austenite transformation finish temperature) were measured by a differential scanning calorimetry (DSC).

Wire-shaped specimens were performed with a gage length of 20 mm and an area of $0.43 \times 0.64 \text{ mm}^2$. The tensile and relaxation tests were done at room temperature in air with different strain rates of 10^{-4} , 10^{-3} , and $5 \times 10^{-3} \text{ s}^{-1}$ using a 10 kN uniaxial tensile machine. To study the material's retarded response, the relaxation tests were conducted for 12 h and 21 h at an imposed strain of 4.8 %, which corresponds to half of the martensite transformation.

In order to study the effect of hydrogen on the relaxation mechanism of the NiTi alloy, the wires were charged electrolytically with hydrogen in a 0.9 % NaCl (0.15 NaCl) aqueous solution for 3 h with a current density of 10 A m^{-2} . The tensile and relaxation tests were conducted immediately after immersion in a NaCl solution and after aging, from one to 77 days, so as to study the effect of hydrogen diffusivity on the mechanical proprieties of this NiTi alloy.

Results and Discussion

Strain Rate Effect

Figure 1 shows a typical stress–strain curve of an as-received superelastic NiTi alloy conducted at room temperature with a $5 \times 10^{-3} \text{ s}^{-1}$ strain rate. It shows that the tensile curve is composed by an elastic deformation of the austenite phase until reaching a critical stress of about $330 \pm 5 \text{ MPa}$, which presents the stress required to trigger mechanically the martensite transformation. This critical stress can be determined by the intersection of a linear curve fit of the transformation's plateau and the tangent of the austenite's elastic curve. The second part is called the upper “plateau”. This zone presents the martensitic transformation in which the martensite bands nucleate and grow within the austenite phase. This plateau presents a number of fluctuations that are related to the number and size of martensite bands. After the plateau, practically all the structure becomes transformed to the martensite phase and so the material retakes the elastic behavior of the

Table 1 Mechanical properties and transformation temperatures of material

Wire alloy	Chemical composition	Critical stress (MPa)	Tensile strength (MPa)	Transformation temperatures (°C)			
				A _f	A _s	M _s	M _f
NiTi	Nickel 50.8 at.% Titanium 49.2 at.%	330 ± 5	1230 ± 10	−3	−11	−9	−32

Fig. 1 Typical stress–strain curve of as-received superelastic NiTi alloy at strain rate of $5 \times 10^{-3} \text{ s}^{-1}$

martensite phase with the Young's modulus less than that of the austenite phase. The material deforms plastically in the martensitic phase before fracture occurs.

The relaxation curves of the as-received NiTi alloy at an imposed strain of 4.8 % and at different strain rates of 10^{-4} , 10^{-3} , and $5 \times 10^{-3} \text{ s}^{-1}$ are shown in Fig. 2. Initially, the strain rate dependency appears clearly with an increase in the “plateau” slope during transformation and then rises in the stress-induced martensite transformation. This growth can be explained by the viscoelastic behavior of the material due to the friction between the martensite band interfaces. Contrary to Tobuchi and Lim [20, 21], the strain rate dependency was explained by the thermomechanical NiTi behavior since the martensite transformation was exothermic. Due to its limited thermal conductivity, when we increase the strain rate, the latent heat will be retained in the material and the structure's temperature will be affected. This rise in temperature results in an increase in the stress required to complete the martensite transformation, hence the plateau's slope and the amount of stress-induced martensite will increase.

During the plateau of the austenite–martensite transformation, we notice that fluctuations intensify at the

Fig. 2 Relaxation curves of as-received superelastic NiTi alloy at imposed strain of 4.8 % for strain rates of 10^{-4} , 10^{-3} , and $5 \times 10^{-3} \text{ s}^{-1}$

greater strain rates. These fluctuations are related directly to the feature of nucleated martensite bands. The number of martensite bands grows with the increase in the strain rate, in contrast to its size that gets thinner. Moreover, He et al. [24] confirmed that the drop of stress during the plateau austenite–martensite transformation was exhibited directly after the nucleation of a new martensite band or after merging two bands.

Regarding the relaxation behavior of the NiTi alloy, we remark that at the first stage, the stress promptly drops immediately from the initial stress to a stabilized equilibrium stress. Furthermore, it is noticeable that the initial stress shows a high dependency on the strain rate due to the increase in the plateau's slope. However, all the relaxation curves converge to the same equilibrium stress regardless the strain rate since this stress presents the delayed response of materials. It is the same response of the quasi-static test at this imposed strain. The decrease in the relaxation stress could be related to the viscous effect in material that is presented by the friction caused by the martensite band interfaces that become numerous at a high strain rate compared with a lower strain rate even at the

same martensite volume fraction. Therefore, the viscoelasticity behavior of the NiTi alloys delays the martensite transformation, thus inducing a hardening in the material in order to nucleate new martensite bands instead of growing in size [25]. Furthermore, during the imposing strain, Brinson et al. [22] observed a redistribution of martensite plates within the grain. Added to this, Takeda et al. [26] noticed merging martensite bands during relaxation. Another mechanism could explain the relaxation behavior of the NiTi alloys. In fact, the fall in relaxation stress can be related to the decline in the structure's temperature, during the imposing strain, before reaching room temperature that coincides with the equilibrium stress. The latter approach appears to be more exact after verification by several studies [23].

Hydrogen Effect

Figure 3 presents typical stress–strain curves of the NiTi that are for as-received material and immediately after hydrogen charging, aging for 1 day, and aged for 7 days. All these latter curves are obtained by tensile testing at the same strain rate. For the as-charged specimen, we notice that the fracture stress has occurred suddenly at the end of the martensite transformation without any plasticity

appearance. Nevertheless, after one-day aging, the fracture stress and strain increased, respectively, by ~ 200 MPa and 5 % from the as-charged specimen. In contrast to the as-charged results, before a fracture occurs, a decrease in hardening behavior appears, which results in plasticity of the structure. Since the specimens were aged for more than 7 days, the material tended to recover its tensile properties; for instance, the fracture occurred practically at the same stress and strain of the as-received specimen.

In fact, the variation of the fracture stress returns principally due to the hydrogen concentration and distribution that develops during aging. Immediately after hydrogen charging, the NiTi alloy adsorbs hydrogen, thus it concentrates at the surface. This latter becomes brittle and more susceptible to fracture. During aging, a part of the adsorbed hydrogen diffuses into the material and the other part desorbs [9, 10]. On the one hand, the desorbed hydrogen decreases the hydrogen concentration at the surface and results in the material recovering its tensile properties. Therefore, the fracture stress and strain increase until reaching their maximum when the hydrogen distribution achieves its equilibrium. On the other hand, part of the diffused hydrogen raises the stress-induced martensite transformation and induces embrittlement of the material during transformation.

Fig. 3 Stress–strain curves of as-received, as-charged and aged (for 1 day and 7 days) superelastic NiTi alloy, at strain rate of $5 \times 10^{-3} \text{ s}^{-1}$

The superposition of the relaxation curves of the NiTi alloy, immediately after hydrogen charging at an imposed strain of 4.8 % for the different strain rates 10^{-4} , 10^{-3} , and $5 \times 10^{-3} \text{ s}^{-1}$, is shown in Fig. 4. It is remarkable, that after hydrogen charging, the stress-induced martensite transformation increases by approximately $15 \pm 5 \text{ MPa}$ for the different strain rates. This observation is explained by the presence of hydrogen that blocks the motion of the Ni and the Ti. Consequently, they retard the occurrence of the martensite transformation and contribute to its hindrance [4, 27]. Moreover, it is observed that during the plateau, the fluctuation has been heightened. This supplemental fluctuation may be due to the diffusion of hydrogen into the interior of the material. In addition, it is noticed that the relaxation of the as-charged NiTi alloy has a particular behavior. At the first stage, regardless of the strain rate, the relaxation curves drop at a high strain rate. After 2 h, they converge and still decrease in a fixed rate ($-0.63 \pm 0.04 \text{ MPa h}^{-1}$) until reaching the equilibrium after 18 h (Fig. 5). The supplemental stress decline is caused by desorbing hydrogen that has been adsorbed after hydrogen charging. On top of that, it is remarked that the equilibrium stress of the as-charged NiTi alloy (reached after 18 h) has been essentially the same compared with the equilibrium stress of the as-received specimen (reached after 2.5 h). Therefore, this is in agreement with the fact that hydrogen desorbs from the surface of the NiTi after 18 h of relaxation. Besides, we can consider that the hydrogen desorption rate can be characterized by the rate of the relaxation stress decrease after 2.5 h. Another approach could be considered in that. The supplemental

Fig. 4 Relaxation curves of as-charged superelastic NiTi alloy, at imposed strain 4.8 %, after loading with strain rates of 10^{-4} , 10^{-3} , and $5 \times 10^{-3} \text{ s}^{-1}$

Fig. 5 Relaxation curves of superelastic NiTi alloys: as-received, as-charged and aged for 1, 7, and 77 days, at imposed 4.8 % strain, after loading with strain rate of 10^{-4} s^{-1}

decrease in stress is caused by the high rate of hydrogen diffusion in different sites, which is created in the martensite phases so as to minimize the internal energy.

Table 2 shows the variation of the initial relaxation stress of the NiTi (as-received, as-charged, and aged for 1, 7, 11, and 77 days), obtained by a tensile test at a strain rate of 10^{-4} , 10^{-3} , and $5 \times 10^{-3} \text{ s}^{-1}$. After hydrogen charging, this stress rises by about 20 MPa for the different strain rates. Whenever the specimens are aged, the initial stress grows until reaching stabilization ($36 \pm 5 \text{ MPa}$) compared with the as-received initial stress. This result is due to the diffusion of hydrogen from the surface to the specimen's center to concentrate around more Ni and Ti atoms. This fact intensifies the obstruction of martensite transformation, so the stress-induced martensite transformation and the initial relaxation stress increase until the end of diffusion. In addition, it is noticed that the gaps of the initial

Table 2 Initial relaxation stress (MPa)

	Strain rate (s^{-1})		
	10^{-4}	10^{-3}	5×10^{-3}
As-received	331 ± 4	365 ± 8	411 ± 10
As-charged	350 ± 4	383 ± 8	434 ± 10
Charged and aged (day)			
1	356 ± 4	396 ± 8	436 ± 10
7	362 ± 4	404 ± 8	445 ± 10
11	363 ± 4	406 ± 8	450 ± 10
77	365 ± 4	400 ± 8	443 ± 10

relaxation stress from a 10^{-4} s^{-1} strain rate have been kept the same at $38 \pm 5 \text{ MPa}$ and $82 \pm 5 \text{ MPa}$, respectively, for 10^{-3} and $5 \times 10^{-3} \text{ s}^{-1}$. Therefore, the strain rate dependency on the NiTi alloy has not been affected by hydrogen charging and aging despite their effects on the hardening behavior during martensite transformation.

The relaxation curves of the as-received, as-charged and aged NiTi, at an imposed strain of 4.8 % and a strain rate of $5 \times 10^{-3} \text{ s}^{-1}$, are shown in Fig. 5. It is remarked that after aging, the relaxation curves retake the behavior of the as-received NiTi to achieve an equilibrium stress essentially at the same time (2.5 h). Indeed, after aging for 1 day, the hydrogen diffuses from the surface to the interior of the specimen. Thus, during relaxation, the hydrogen desorption becomes less pronounced and so the supplemental fall in the relaxation stress is eliminated as seen with the as-charged specimen.

Moreover, it is noticed that after hydrogen charging and aging, the equilibrium stress increase by $\sim 17 \pm 2 \text{ MPa}$ after 1 day and stabilizes after 7 days to $\sim 22 \pm 2 \text{ MPa}$, compared to the equilibrium stress of the as-received specimen. Furthermore, the variation of the equilibrium stress is practically the same as that of the initial relaxation stress (Table 2), and both stresses explain the increase in the hardening behavior after charging and aging. Yet, it is remarkable that the equilibrium stress is independent from the strain rate since this stress presents a delayed response in materials. Therefore, it is the same response of the quasi-static test at 4.8 % strain. In addition, the relaxation amount, after hydrogen charging and aging, has the same variation regardless of the strain rate. After immediate hydrogen charging, the relaxation amount has promptly increased. In return, after aging of 1 day, it has declined and has become stabilized above the as-received one.

In the work of Biscarini et al. [15], they noticed the appearance of two peaks on the internal friction curve and explained them by the activation of the relaxation process caused by the pinning of twin boundaries by hydrogen. In another explanation, Fan et al. [17] assumed that the dislocations had a twofold opposite role in the relaxation behavior. They obstructed the movement of the twin boundary and at the same time they liberated hydrogen from the twin boundary to be trapped in a dislocation site. In contrast, in the Zhao study they assumed that the hydrides, formed after charging, presented pinning points that increased the internal friction [14]. On the other hand, during aging, the hydrides tended to be decomposed and thus they would affect the decrease in internal friction [13]. In addition, Runciman et al. [28] showed that the latent heat, that was responsible for the relaxation behavior of as-received NiTi, decreased with the increase in the hydrogen amount.

In the present work, it is suggested from the variation of the relaxation amount that, after hydrogen charging and aging for more than 1 day, another relaxation process has been added due to the presence of hydrogen, which can intensify the internal friction. Otherwise, the as-charged specimens have greater relaxation than the aged ones. It can be considered that the hydrides present an additional friction for the as-charged NiTi before its decomposition during aging. Hence, it is proposed from the results of the relaxation experiments that hydrogen has a twofold role on the NiTi alloys. The first is the hydrogen-damping dependency, where an increase in the relaxation amount after hydrogen charging is noticed. This damping is the result of friction between the twin boundary, hydrides, and hydrogen. The second role is the hydrogen-hardening dependency. As it is shown in the relaxation curves, the increase in the initial relaxation and the equilibrium stresses of the charged and aged NiTi explains the growth of hardening. We suggest that this hardening is the result of the dislocation's hindrance caused by hydrogen, which may contribute to the hindrance of the martensite transformation.

Conclusions

The relaxation and tensile mechanisms of the superelastic NiTi alloy have been investigated after undergoing hydrogen charging for 3 h in a 0.9 % NaCl aqueous solution with a current density of 10 A m^{-2} and aging for different durations at room temperature. It has been shown that immediately after hydrogen charging, the fracture stress and strain clearly decrease in contrast to the stress-induced transformation. Moreover, it has been noticed that the relaxation stress reaches its equilibrium after 18 h more than the as-received one (2.5 h). During aging, the fracture stress and the strain increase with the rise of aging duration. Contrary to the as-charged specimen, the relaxation stress of the aged specimens reaches its equilibrium practically at the same time of the as-received one. In fact, the greater the aging period is the greater the equilibrium stress will be. In addition, regardless the aging duration, the strain rate affects mainly the initial relaxation stress and has no effect on the equilibrium stress. It has been found also that the relaxation amount rises after hydrogen charging and is still constant during aging.

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