European Journal of Orthodontics 29 (2007) 8–13
doi:10.1093/ejo/cjl038
Advance Access publication 5 September 2006

Improvement of pseudoelasticity and ductility of Beta III titanium alloy—application to orthodontic wires

P. Laheurte*, A. Eberhardt**, M. J. Philippe* and L. Deblock***
*LETAM (CNRS—UMR 7078), Université de Metz, **LPMM (CNRS—UMR 7554), ENIM Metz, and
***Private practice, Vandœuvre-les-Nancy, France

SUMMARY The pseudoelasticity of metastable Beta III titanium alloy (TMA™) used for orthodontic applications is obtained by cold wiredrawing. This wire has higher rigidity than cold-drawn NiTi (Nitinol™, superelastic NiTi SE) and lower recoverable deformation. The low ductility value of Beta III is due to the deformation imposed by wiredrawing. The aim of this research was to improve the behaviour of this alloy by modifying the microstructural parameters to decrease the rigidity and increase the recoverable deformation and ductility of the alloy. The effects of second phase precipitate, grain size, and deformation on the wire mechanical properties were also examined.

The isothermal precipitation of alpha (α) or omega (ω_{isoth}) phases precludes the expression of the pseudoelastic effect. The presence of an ω_{isoth} phase considerably increases fracture strength, whereas the α phase strongly decreases the ductility and adversely affects the strain recovery (ε_r).

To control the grain size, the growth of the recrystallized grains was studied by considering several parameters, which are known to have an influence on grain size, including the cold rolled strain, the temperature, the time of annealing, and the initial grain size. A structure with coarse grains, quenched from a temperature higher than the beta transus (T_β), associated with a plastic pre-deformation, contributed to an improved pseudoelastic behaviour, due to the presence of a reversible martensite phase (α'') induced by the pre-deformation.

Introduction

The desirable properties of an orthodontic wire depend on the requirements set by mechanotherapy at a specific treatment stage, and thus, selection of the appropriate wire is often complex. Criteria such as the degree of torque control, the desired load-deflection ratio, and the need for a specified elastic or plastic zone must be taken into account in the process.

In general, three factors determine the rigidity of the wire: the length, the cross-section, and the elastic modulus of the alloy. The traditional method to modify the rigidity consists of varying the dimensions of the wire by engaging incrementally larger cross-section wires in the slot, or adjusting the configuration of the loops. The use of materials with a low elastic modulus, such as titanium alloys, was introduced as an alternative way to advance treatment, namely, 'variable modulus orthodontics' (Goldberg *et al.*, 1977; Burstone and Goldberg, 1979, 1980; Goldberg and Burstone, 1979; Burstone, 1981). The use of alloys with different rigidities provides a range of force during deactivation, which is independent of the wire cross-section.

Figure 1 illustrates the behaviour of an 'ideal' archwire providing a constant force throughout treatment, which coincides with the concept of optimal tooth movement. The behaviour of archwire A (Figure 1) during deactivation shows a variation of force which requires reactivation of the archwire in order to reach the desired tooth position. A compromise between the two extreme situations consists of using a lower rigidity wire (wire B, Figure 1). The variation of force however is lower and thus, the archwire requires less frequent activation.

To meet clinical needs, various alloys have been introduced, presenting a force-deflection curve depicted in Figure 2. Stainless steel, which constitutes the most common alloy, possesses a high rigidity, and is characterized by an apparent elasticity modulus of 210 GPa. The titanium alloys for use in orthodontics include the cold-drawn Beta III developed by Burstone and Goldberg (1980, TMA™) and nickel-titanium (NiTi). The latter are also used in a strong, cold work-hardened form (Nitinol™) and in austenitic form, which provide superelasticity. NiTi wires, and especially NiTi superelastic (SE), present a substantial recovery strain (Table 1), where the unloading secant modulus (E_s) is 20 GPa and its behaviour resembles that of bone during loading. NiTi wires are not weldable, have minimal ductility, and are used in pre-formed arches. However, the presence of the high Ni concentration in NiTi alloys raises some concerns about potential allergencity or toxicity of this wire. Therefore, the introduction of a nickel-free, low-stiffness pseudoelastic wire, with improved formability that can be joined with other wires or auxiliaries, would be of great value to the practising orthodontist.

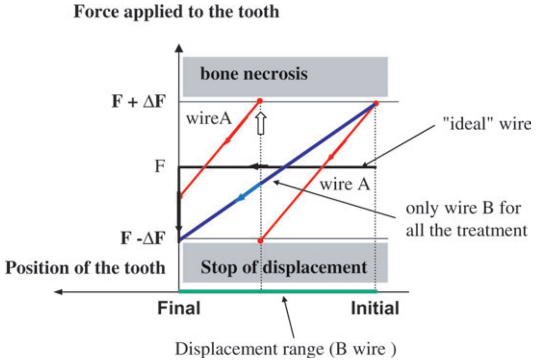

Figure 1 Wire rigidity on dental displacement—'ideal' wire with constant force (A) and with two different rigidities (B).

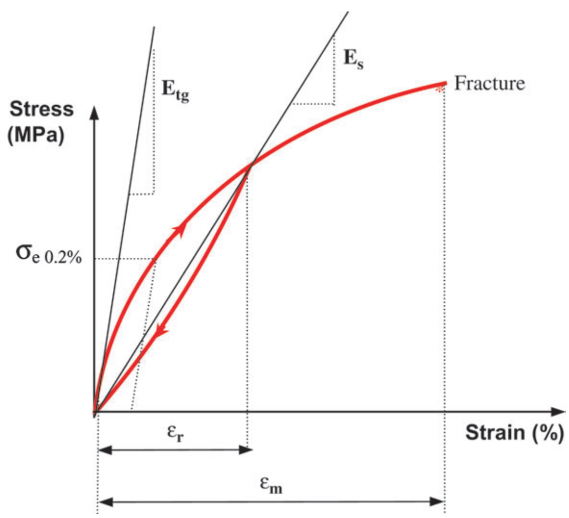

Figure 2 Tensile diagram showing the apparent modulus (E_{tg}), unloading secant modulus (E_s), recoverable strain (ε_r), and strain to fracture (ε_m).

Table 1 Qualitative evaluation of NiTi and Beta III wire for orthodontic use.

	Unloading secant modulus E_s (GPa)	Recovery strain e_r (%)	Workabilty (%)	Weldability	Biocompatibility
NiTi	30–12	4–5	No	No	Allowed
Beta III	40	3	Yes	Yes	Yes (nickel free)

While Beta III alloy does not contain nickel and has good weldability, its performance for recoverable deformation is inferior and it has a higher unloading E_s compared with NiTi. The pseudoelasticity of Beta III titanium alloy obtained by cold working (wiredrawing) as shown in Figure 2 corresponds to a mechanism of selective reorientation of the martensite variant (Duerig and Zadno, 1990; Laheurte *et al.*, 2005). For orthodontic applications, a significant recoverable deformation and a low unloading E_s are desirable.

The purpose of this study was to modify the wire properties and obtain a beta titanium wire with improved pseudoelasticity through the variation of the phases of the wire alloy.

Materials and methods

As-received Beta III alloy was wiredrawn to segments of 0.43 × 0.63 mm. Treatment solutions and isothermal precipitation of the α or ω phases were carried out in vacuum quartz tube. Heat treatment for 1 hour at 800°C [above the beta transus (T_β) = 760°C] was followed by quenching with water. The average grain size (range 6–100 µm) of wires was obtained by thermomechanical treatment. To control the grain size in the wire, the normal growth of the recrystallized grains was studied by taking into account several parameters known to have an influence on grain size: the cold rolled strain, the temperature and time of annealing, and the initial grain size. The influence of these parameters was evaluated using an experimental design presented previously (Laheurte *et al.*, 2004b). The tensile tests were carried out on a tensile testing machine (Zwick France sarl, Roissy Airport Ch. de Gaulle Cedex, France) in the form of loading–unloading cycles with increasing deformations to fracture. The resultant strain was measured by an extensometer (Zwick France sarl) with a gauge length of 20 mm at a strain rate of 4×10^{-3} s^{-1}.

Characterization in tension

The apparent tangent modulus (E_{tg}), the yield strength $\sigma_{e0.2\%}$, and the ductility (elongation to fracture) are represented in Figure 3. In the relevant literature (Shastry and Goldberg, 1983; Goldberg and Shastry, 1984), the yield strength/modulus of elasticity ratio ($\sigma_{e\ 0.2\%}/E_{tg}$ as shown in Figure 2) is often used as an index of the use of an orthodontic wire. However, this elastic strain does not sufficiently account for the action of the wire. It has been proposed (Laheurte *et al.*, 2004a) that the wire should be characterized by E_s and the recoverable strain (ε_r) on the unloading curve (Figure 2).

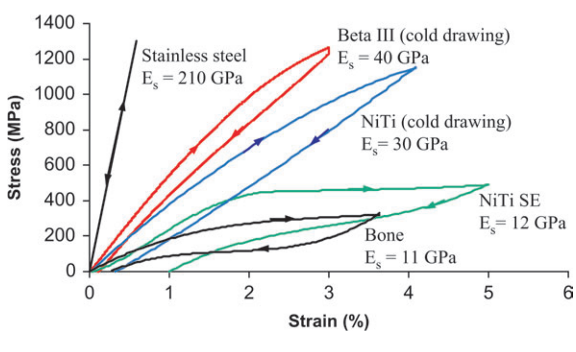

Figure 3 Tensile diagrams of principal orthodontic wires, and compared with bone E_s = secant modulus.

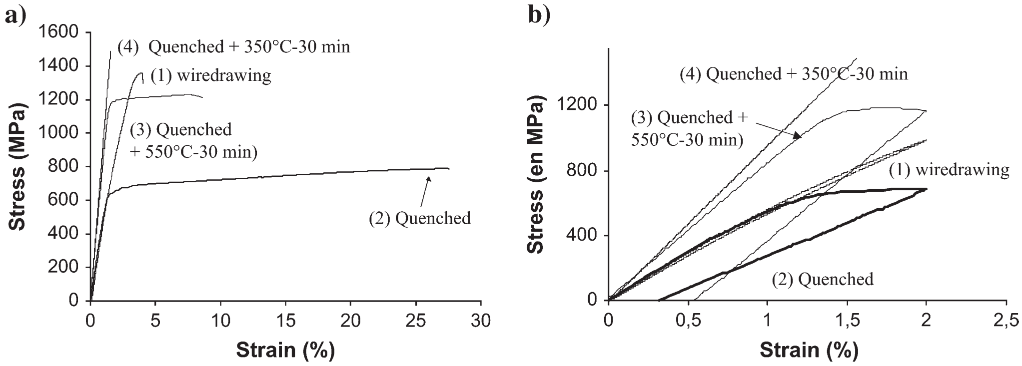

Figure 4 Influence of heat treatment on (a) ductility and (b) the recoverable strain and unloading secant modulus.

Table 2 Mechanical characteristics after imposed deformation of 2 per cent or after fracture (average of three tests and standard deviation).

Treatment	Strain recovery (%)	Unloading secant modulus E_s (GPa)	Strain to fracture (%)	UTS (MPa)
Wiredrawn (1)	2 ± 0.1	47 ± 2.5	4 ± 0.5	1370 ± 27
Quenching 800°C—1 hour (2)	1.7 ± 0.1	37 ± 1.8	26 ± 2.3	790 ± 19
Quenching + ageing 550°C—30 minutes (3)	1.55 ± 0.1	79 ± 2	8.5 ± 1.7	1227 ± 22
Quenching + ageing 350°C—30 minutes (4)	Failure	96 ± 2.7	1.6 ± 0.3	1495 ± 31

Table 3 Optimized structure of Beta III—characteristic of bending test: unloading forces for three deformations (wire 0.17 × 0.25 inch, distance between the two bracket 14 mm).

Deflexion (mm)	Beta III—optimized structure—unloading force (N)	
	Cold drawing	Optimized structure
0.5	4.7	2.3
1.0	10.2	6.9
1.5	21.5	14

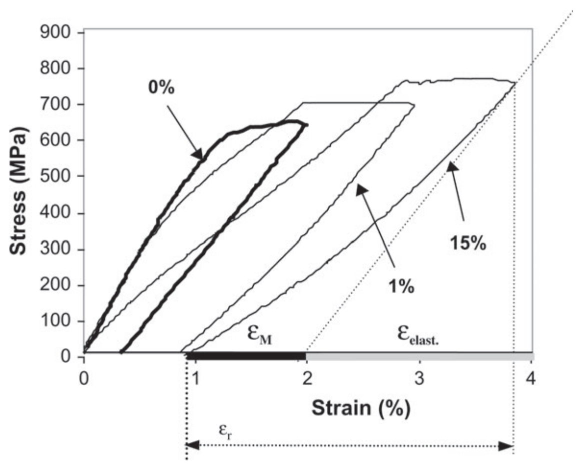

Figure 5 Influence of plastic pre-deformation (0 1, and 15 per cent) on the recoverable strain $\varepsilon_r = \varepsilon_M + \varepsilon_{elast}$ and on the unloading secant modulus (rectangular wires 0.63 × 0.43 mm).

Results

Effect of precipitation treatment on pseudoelastic behaviour

The tensile test to fracture (Figure 4a) and at 2 per cent with unloading (Figure 4b) carried out for various heat treatments were compared with the drawn state (1). State (2) is obtained after water quenching from 800°C, while states (3) and (4) correspond to an ageing of 30 minutes at 550°C and 350°C, respectively. This process results in a precipitation of 40 per cent of α phase and 50 per cent of ω_{isoth} phase (Laheurte, 2003). In the quenched state, the ductility was 25–27 per cent, while both the unloading E_s and the recoverable deformation were lower relative to the as-received specimens (Table 2). The precipitation of the ω_{isoth} and α phases during ageing, and states 3 and 4, considerably increased the ultimate tensile strength. The very low ductility, which was present in state 3, confirmed the brittleness due to the ω_{isoth} phase. The α phase strongly decreased ductility and did not improve the ε_r compared with the as-received cold drawn. The unloading E_s was higher than that of the cold-drawn state.

Effect of uniaxial pre-deformation on pseudoelastic behaviour

After quenching, the recrystallized beta grains had an average size of 22 μm. When the deformation increased from 0 to 15 per cent, the E_s decreased from 37 to 25 GPa

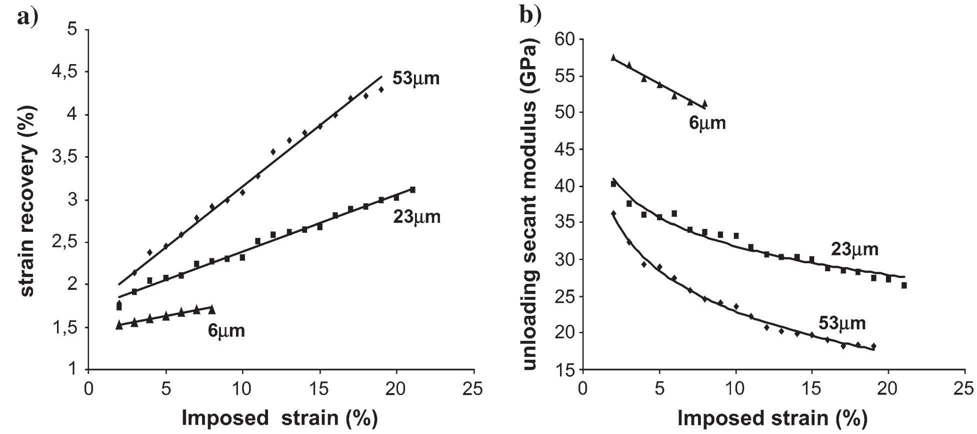

Figure 6 Influence of grain size on (a) recoverable strain and (b) unloading secant modulus.

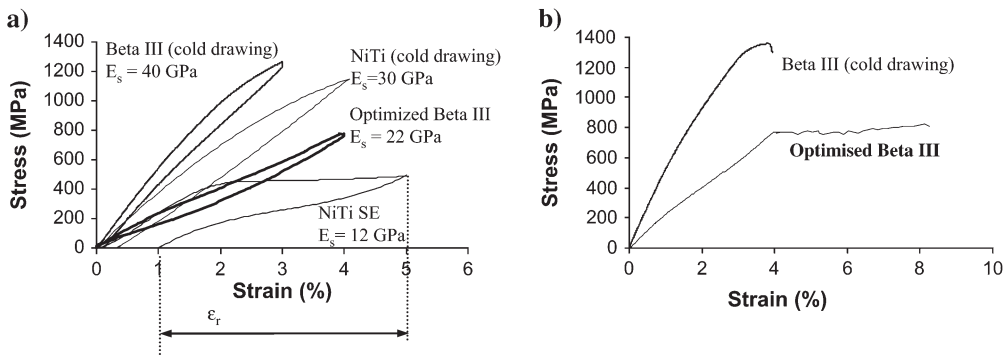

Figure 7 Comparison of unloading secant modulus (E_s) and recoverable strain (ε_r) of principal alloys with optimized Beta III: (a) ε_r and E_s modulus and (b) strain to fracture.

(Figure 5). The unloading curve showed an elastic pattern for the first cycle but deviated from linearity for the following cycles. ε_r increased with the imposed deformation. This variable is the sum of the elastic strain (ε_{elast}) and the deformation (ε_M) due to the variant reorientation of the martensite.

Effect of the grain size

ε_r and the reduction of the E_s increased with grain size (Figure 6a,b). For small grain sizes, no hysteresis was apparent and the ε_r was purely elastic. The grain boundaries constituted obstacles for growth and propagation of the martensite variants and their mobility during unloading.

Comparison of a structure optimized with a cold work-hardening structure

By combining grain growth and plastic deformation, a significant decrease in the E_s and an increase of ε_r were obtained. Figure 7a shows the optimized structure of Beta III wire with the other wires and, in particular, the Beta III cold work-hardened wire. The unloading E_s varied from 40 to 21 GPa and the maximum ε_r from 3 to 4 per cent leading to increased ductility in the order of 100 per cent (Figure 7b), relative to the as-received, cold-drawn form (Laheurte *et al.*, 2003). Scanning electron micrographs (Figure 8a,b) illustrate the greater aptitude for the formation of loops with low curvature radii for the optimized structure compared with the cold work-hardening structure (presence of cracks). The bending test (Figure 9) with a configuration resembling that of clinical conditions showed that the unloading E_s was decreased by 40 per cent, corresponding to a maximum deflection of 1.5 mm (Table 3).

Discussion

The precipitation of the α phase involves enrichment in β-stabilizing elements of the β phase. The latter, which is more mechanically stable, deforms without β to α″ (reversible martensitic transformation). These results are in agreement with Shastry and Goldberg (1983) and Goldberg and Shastry (1984), who studied Beta III wire alloys for orthodontic application.

For the 800°C heat treatment regimen, the increase of the average grain size and pre-deformation lead to a higher ε_r and a lower unloading E_s. X-ray diffraction studies of wires

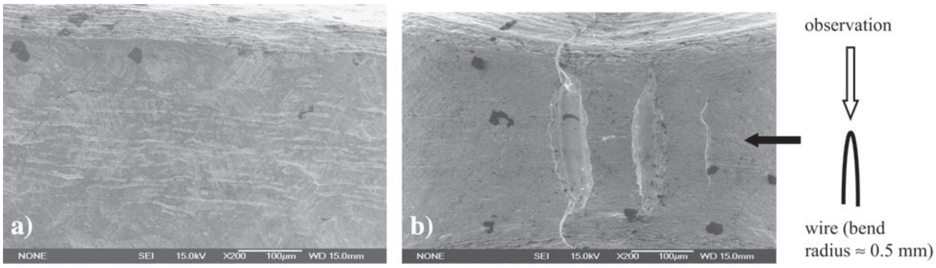

Figure 8 Scanning electron micrograph of Beta III alloy (×200) after bending at 180 degrees: (a) optimized structure and (b) cold wiredrawing. Note the presence of cracks (arrow).

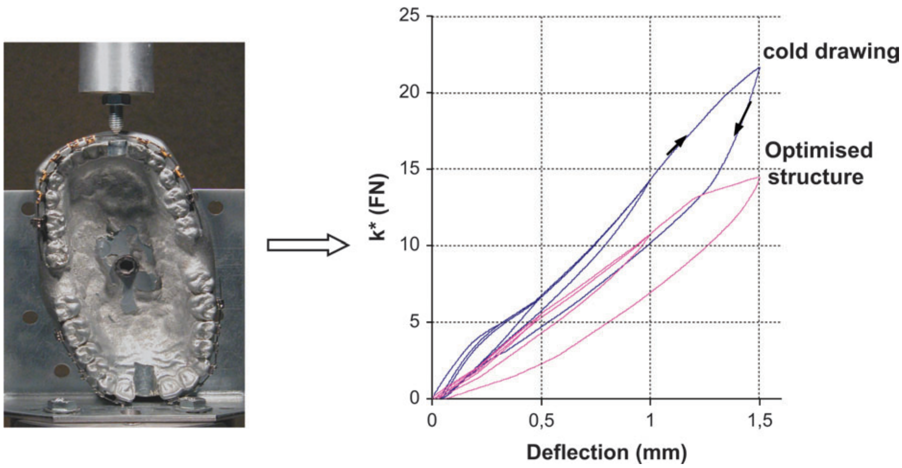

Figure 9 Beta III characteristic of bending test: loading–unloading cycles for three imposed deflexions at 0.5, 1, and 1.5 mm (wire 0.17 × 0.25 inch, distance between the two brackets 14 mm).

with coarse grains, plastically deformed by 12 per cent in tension, have revealed the presence of the stress-induced orthorhombic martensitic α″ phase, retained by the plastic deformation. In these alloys, the grain boundaries act as obstacles to martensite variant growth and decrease their mobility during unloading (Laheurte *et al.*, 2005).

Moreover, the 'slip' deformation is generally accompanied by twinning in the structures with small grains and by twinning and martensitic transformation in structures with coarse grains. The reduction of the unloading E_s is due to an increase in the ε_r, which is the sum of elastic strain and variant reorientation of martensite induced by deformation. When the imposed deformation increases, the elastic strain remains effectively constant, whereas the ε_r attributed to variant reorientation increases. This is in accordance with previous research indicating that the volume fraction of martensite does not change in an unloading–loading loop (Laheurte, 2003). However, for grains sizes obtained after heat treatment at 900°C, oxidation (residual oxygen and reduction of tube SiO_2 by Ti) is responsible for the loss of ductility, the hardening of the alloy, and pseudoelastic degradation. The pseudoelastic degradation of the behaviour and ductility is really the result of a chemical modification of the material at 900°C treatment.

Conclusions

The findings show that the presence of an α or ω_{isoth} second phase, resulting from isothermal treatment or an insufficient cooling rate, decreases the ductility and pseudoelastic behaviour of beta titanium alloy. For an alloy quenched at a higher temperature than the T_β, the plastic deformation and grain size growth results in an increase in the ε_r and a decrease in the unloading E_s, leading to a significant improvement in the mechanical properties of the alloy.

Address for correspondence

Dr P. Laheurte
LETAM/ISGMP
Université de Metz
Ile du Saulcy
57 000 Metz
France
E-mail: laheurte@letam.sciences.univ-metz.fr

References

Burstone C J 1981 Variable modulus orthodontics. American Journal of Orthodontics 83: 374–381

Burstone C J, Goldberg A J 1979 Patent No 7906380, USA

Burstone C J, Goldberg A J 1980 Beta titanium: a new orthodontic alloy. American Journal of Orthodontics 77: 121–132

Duerig T W, Zadno R 1990 An engineer's perspectives of pseudoelasticity. In: Duerig T W (ed.) Engineering aspect of SMA. Butterworth-Heinemann Publishers, London, pp. 369–393

Goldberg A J, Burstone C J 1979 An evaluation of beta titanium alloys for use in orthodontic appliances. Journal of Dental Research 58: 593–599

Goldberg A J, Shastry C V 1984 Age hardening of orthodontic beta titanium alloys. Journal of Biomedical Materials Research 18: 155–163

Goldberg A J, Vanderby R, Burstone C J 1977 Reduction in the modulus of elasticity in orthodontic wires. Journal of Dental Research 56: 1227–1231

Laheurte P 2003 Contribution à l'étude du comportement pseudoélastique d'un alliage de titane bêta métastable—applications aux arcs orthodontiques. Thesis, University of Metz, France

Laheurte P, Eberhardt A, Philippe M J, Deblock L 2003 Fil en alliage de titane bêta pour l'orthodontie et procédé d'obtention d'un tel fil. Patent No 03 15094, University of Metz

Laheurte P, Eberhardt A, Philippe M J, Deblock L 2004a Caractéristiques comparées des fils orthodontiques de Bêta III: proposition d'un nouveau critère de choix. Revue d' Orthopédie Dento-Faciale 38: 205–220

Laheurte P, Eberhardt A, Philippe M J, Deblock L 2004b Etude de la croissance des grains recristallisés dans l'alliage de titane Bêta III. La Revue de Métallurgie 12: 1021–1026

Laheurte P, Eberhardt A, Philippe M J 2005 Influence of the microstructure on the pseudoelasticity of a metastable beta titanium alloys. Materials Science and Engineering A 396: 223–230

Shastry C V, Goldberg A J 1983 The influence of drawing parameters on the mechanical properties of two beta-titanium alloys. Journal of Dental Research 62: 1092–1097