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Anelastic relaxation associated to phase transformations and interstitial atoms in the Ti–35Nb–7Zr alloy

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ABSTRACT

Ti–35Nb–7Zr (wt.%) alloy was analyzed by mechanical spectroscopy. The results showed a relaxation peak, involving components of phase transformations and matrix–interstitial interactions and substitutional–interstitial interactions. The phase transformation β → ω in the first thermal cycle was identified by increases in elastic modulus and Vickers hardness. In the subsequent thermal cycles, a fall in the strength of relaxation and stabilization of the dynamical elastic modulus were associated with variation in the fraction of ω-phase formed and nucleation of the α-phase, as indicated by DSC, XRD and SEM–EDX results. In addition, once the strength of relaxation was stabilized shifts of the relaxation peak to higher temperatures were observed with increasing oscillating frequency. This frequency dependence of the peak was accompanied by a change in the oscillation frequency that is characteristic of a Snoek-type relaxation. Thus, this behavior can be related to stress-induced reorientation of interstitial oxygen atoms with matrix (Ti) and substitutional (Nb) atoms in the bcc structure of the β-phase.

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1. Introduction

It is well known that titanium and titanium alloys are widely used as structural biomaterials, owing to a favorable combination of properties, such as high corrosion resistance, good biocompatibility, high strength to weight ratio and low elastic modulus [1,2], which depends directly on the microstructure as well as on the phases present.

Pure titanium exhibits an allotropic phase transformation at 1155 K, changing from a body-centered cubic (bcc) crystal structure (β-phase) at higher temperatures to a hexagonal close-packed (hcp) crystal structure (α-phase) at lower temperatures. This transformation temperature is strongly influenced by the concentration of interstitial and substitutional elements [3]. Thereby by means of alloying and adequate heat treatment a particular microstructure can be obtained with specific mechanical properties [4].

Many studies have been focusing on β-titanium binary alloys, with the addition of elements such as Nb, Ta and Mo [5–8], since these do not lead to biocompatibility problems, in contrast to the elements V and Al used in the current Ti–6Al–4V alloy [9]. Metastable phases such as hexagonal α′ martensite, orthorhombic α″ martensite and ω-phase (thermal or athermal) of hexagonal structure are formed during quenching, aging or deformation of the β-titanium alloys [10]. The control of the crystalline phases is of great importance in the design new biomaterials alloys, as it is known that ω-phase has the highest elastic modulus, while martensite α″-phase has a lower modulus than the α′-phase and β-phase has the lowest modulus of any of phases in most Ti alloys [6].

Thus, ternary alloys such as the Ti–Nb–Zr system are being investigated, since the addition of Nb stabilizes the β-phase at room temperature and Zr is a neutral element that can help to suppress the ω-phase and in many compositions may have special properties such as shape memory effect and super-elasticity [11–13].

The aim of this investigation was to study the anelastic behavior of the alloy Ti–35Nb–7Zr (wt.%) by measuring the dynamical elastic modulus (E) and internal friction (Q^{-1}), during successive thermal cycles.

2. Experimental procedures

The Ti–35Nb–7Zr (wt.%) alloys were produced from commercially pure materials (Ti, Nb and Zr) by arc melting under an argon atmosphere in a water-cooled copper hearth. The ingots obtained were remelted and homogenized above the β-transition temperature (β-field at 1170 K), then cold worked by swaging (80% plastic deformation) followed by a further recrystallization heat treatment at 1273 K for 2 h. The sample after recrystallization treatment was called TNZ alloy. The interstitial elements content (O, N) was estimated using a LECO TC-436

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nitrogen/oxygen analyzer. Differential scanning calorimetry (DSC) was carried out in Netzch STA 404 equipment during heating at a constant rate of 20 K/min, from room temperature (RT) to 850 K, to predict the phase transformations behavior.

Dynamical variations in the elastic (E) and shear (G) moduli can be measured by means of flexural and torsional vibration, using the relationships ($E \propto f^2$) and ($G \propto f^2$), where f is the vibration frequency of the specimen. The variations in elastic (E) and shear (G) modulus and internal friction (Q^{-1}) of the material with temperature were observed in an AE-102 Acoustic Elastometer System (Vibran Technology®) from the first tone of flexural vibration in the clamped-free mode in a kilohertz bandwidth and torsional vibration of the samples in a Ke-type Torsion Pendulum operating in a hertz bandwidth. The principle of detection of these devices is described in [14].

Elastic modulus (E) for flexural vibration in clamped free configuration can be obtained from [15]

$$f_1 = 0.1615 \frac{h}{l^2}\sqrt{\frac{E}{\rho}}$$

where h is the thickness, l is the length and ρ is the density of the sample.

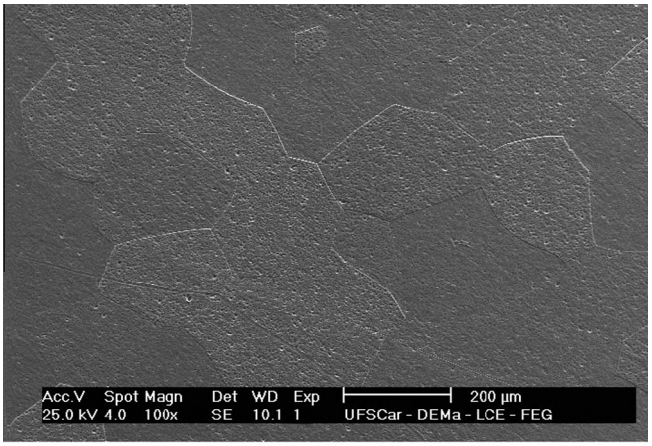
For the anelastic spectroscopy studies, the Ti–35Nb–7Zr specimens were cut as bars, of dimensions $(20 \times 5 \times 0.5)\,mm^3$ for use in flexural apparatus and $(40 \times 2 \times 2)\,mm^3$ for use in torsional apparatus. Experimental spectra of anelastic relaxation were collected over the temperature range from RT to 700 K at a heating rate of 1 K/min under a pressure of 10^{-5} Torr, in both devices. The crystalline structure was first analyzed by X-ray diffraction (XRD), using Cu Kα radiation in a Siemens D5005 X-ray Diffractometer. Besides, specimens were characterized microstructurally by scanning electron microscopy (SEM) in a Philips XL-30 FEG in combination with EDX (Energy Dispersive X-ray spectroscopy), the samples being prepared metallographically by grinding, polishing and etching with Kroll reagent. Additionally, Vickers hardness measurements were carried out on microhardness Stiefelmayer KL2 equipment. The values were obtained from mean value of six indentations with load of 200 gf during 15 s.

3. Results and discussion

3.1. Structural and thermal characterization

SEM results in combination with compositional analysis EDX for TNZ alloy are shown in Fig. 1, in which well-defined grain boundaries corresponding to a predominant β matrix were observed. This observation is in accordance with the XRD pattern (Fig. 2), where typical peaks corresponding to the β-phase were identified [10,16,17].

In order to study the phase transformations behavior, a DSC measurement was carried out on the TNZ alloy. In the DSC curve (Fig. 3) there is a first endothermic process around 500 K extending over a broad temperature range, which can be associated with transformations of metastable phases such as martensite α″ and



Sample	Phase	Element (wt. %)		
		Ti	Nb	Zr
Ti-35Nb-7Zr	β	57.74	33.94	8.32

Fig. 1. SEM–EDX of the TNZ alloy.

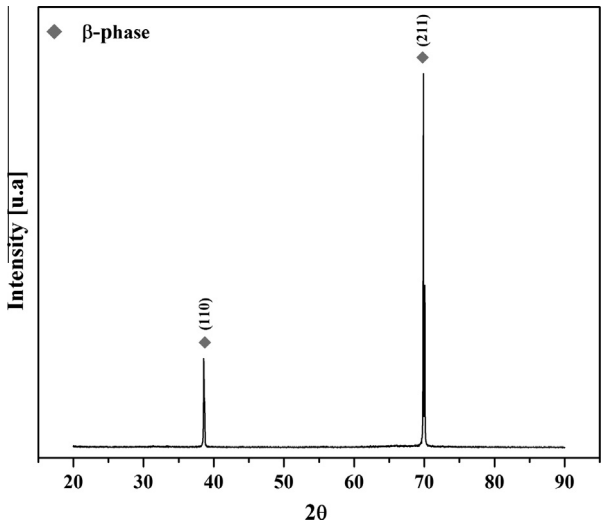


Fig. 2. X-ray diffraction pattern of the TNZ alloy.

ω-phase, which were not revealed by XRD. However, from the extended stability diagram for titanium alloys proposed by Abdel and co-authors [18] based on averages of the electronic parameters, bond order (B_o) and d-orbital energy level (M_d), Ti–35Nb–7Zr alloy is locates between β + ω and α″ phase boundaries. Thus, the endothermic peak may include α″ decomposition, precipitation of the ω-phase and nucleation of the α-phase, as reported by other authors [10,16,19]. The other endothermic peak, around 700 K, has been related directly to the allotropic β-transus temperature.

3.2. Anelastic behavior of the Ti–35Nb–7Zr

The relaxation processes observed can be related to phase transformations, besides matrix-solute interactions and substitutional–interstitial interactions in the TNZ alloy, during cyclic heat treatment (heating and cooling) at a constant heating rate of 1 K/min. Typical anelastic spectra are shown in Fig. 4. The elastic modulus obtained from Eq. (1) for the flexural mode at room temperature was 54 ± 4 GPa, in the first cycle of measurements.

The anelastic spectra in Fig 4 shows that the internal friction (Q^{-1}) curve during the first heating cycle increases sharply to an anelastic relaxation peak with a maximum value around 583 K; this process is accompanied by a rise in the elastic modulus to its maximum (35%) at ~640 K. In the subsequent cooling, the internal friction decreases, whereas the elastic modulus continues to rise up to 50% higher at room temperature, as shown from the start of the second heating. This behavior may be associated firstly with the precipitation of isothermal ω-phase, which is known to increase the values of hardness and elastic modulus [4] and can be favored by the low heating rate.

The subsequent heat treatment cycles showed a continuous fall in the internal friction values, which were stabilized only after the sixth cycle. However, the dynamical elastic modulus values falls up to the fourth measurement cycle and after a slight increase at the seventh measure is maintained constant, with a final change of 30% in the elastic modulus.

In the torsional mode, where ($f^2 \propto G$), the anelastic spectra (Fig. 5) showed a similar behavior in the first heating cycle, with a continuous rise in the shear modulus (G) up to ~500 K, the temperature at which the anelastic relaxation peak reaches a maximum. Above this temperature, there is a sharp increase in the shear modulus (G), to a maximum change of ~4% at a temperature of ~640 K. As observed in the flexural mode, this process may be

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