

## INTERNAL FRICTION AND MICROSTRUCTURE OF Ti AND Ti-Mo ALLOYS CONTAINING OXYGEN

Ti-Mo alloys are promising materials for use as biomaterials, because these alloys have excellent corrosion resistance and a good combination of mechanical properties such as fatigue, low elastic modulus, hardness, and wear resistance. The objective of this paper was to study the effect of heavy interstitial atoms on anelastic properties of Ti-Mo alloys using mechanical spectroscopy. The internal friction and Young's modulus were measured as a function of temperature using dynamic mechanical analyser. The internal friction spectra were brought about by relaxation processes attributed to short-range stress induced reorientation of interstitial and substitutional complexes in solid solution. It is suggested that the nature of the relaxing entities can be worked out in further research on Ti-Mo single crystals.

*Keywords:* Mechanical spectroscopy, anelasticity, internal friction, Ti-Mo alloys, interstitials, biomaterials

## 1. Introduction

Combining mechanical strength and density, as well as high corrosion resistance, titanium alloys are excellent materials for applications in the aeronautical and automobile industry, in addition to often being used as a biomaterial due to biocompatibility and high biocorrosion resistance [1-3]. The addition of heavy interstitial atoms O, C, and N in the hcp metals leads to strong changes in their mechanical properties, causing, for example, softening or hardening [4,5].

Mechanical spectroscopy [6-14] provides important information about the behavior of interstitial atoms in binary and ternary alloys such as the concentration of dissolved interstitial atoms in solid solution, diffusivity, solubility limit [6,14-21], precipitation, and their interaction phenomena caused by interaction between relaxing entities [22-30], short- and long-range interaction of mobile point defects with dislocations [31-36,37-41], and strain aging. In general, the time-invariant harmonic stress field imposed on a sample containing interstitial atoms dissolved in solid solution yields splitting of free-energy levels of different elastic dipoles' orientations [17]. This phenomenon gives rise to the classic relaxation process, which manifests (in bcc lattice) as internal friction peak known as Snoek peak [6-7,13,15-17,28]. However, the relaxation mechanism of point defects in the hcp metals [6,42-48] differs from the classic Snoek relaxation observed in the bcc metals [6,15]. Stated another way, the Snoek relaxation is the stress induced redistribution of elastic dipoles whose symmetry is lower than that of the surroundings. Considering mechanical external harmonic perturbation, the energies of the positions available for elastic dipoles are time-dependent leading to redistribution of the elastic dipoles toward the orientation with the lowest energy level. This means that there occurs a phase difference between mechanical polarization induced by

reorientation of the elastic dipoles and an imposed external harmonic stress field. The reorientation of the elastic dipoles proceeds by thermally activated jumps between different positions over a potential barrier DH (activation enthalpy) with the microscopic relaxation time  $\tau$ .

Ti-6Al-4V is the alloy most frequently used for orthopedic implants, although aluminum causes neurological disorders, and vanadium is associated with enzymatic disturbances, among other problems [49,50]. Thus, many researchers have been seeking alternatives to these titanium alloys in an attempt to obtain materials with similar (or higher) mechanical strength, lower elastic modulus, and better biocompatibility. The most promising alternatives are the ones that use niobium [51], zirconium [52], and molybdenum [53] as alloying elements added to titanium.

Ti-Mo alloys integrate a new class of titanium-based alloys, without aluminum and vanadium, and with low elastic modulus (around 90 GPa), which makes these alloys very attractive for use as biomaterials. This alloy system has been thoroughly studied with the main emphasis put on the microstructure and mechanical properties [54,55]. It should be pointed out, though, that information about the effects of interstitial atoms such as oxygen and nitrogen is limited.

Dynamical mechanical analyser (DMA) was used to study internal friction in Ti-Mo alloys containing 5, 10, 15, 20, 25, and 30 wt.% molybdenum. The internal friction spectra were resolved into constituent Debye peaks corresponding to different relaxation processes. The analysis of secondary features of internal friction peaks caused by the interaction between interstitial atoms and substitutional-interstitial pairs [22-30] as well as the anisotropy effect of Snoek relaxation [18-21], and the type of stress applied are outside the scope of the present study. Notwithstanding, it is now generally recognized that the secondary features are responsible for the

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broadening of Snoek peak, the variation in peak's temperature [21-30] and peak's height [18-21].

## 2. Experimental

The Ti-Mo alloys were produced by arc-melting under argon atmosphere, using commercially pure titanium (99.7% purity, Sandinox, Brazil) and molybdenum (99.5% purity, Interteknik, Brazil) [53,56]. The chemical composition of the samples was obtained with optical emission spectroscopy (Spectra, SPECTROMAX) and the thermoconductivity difference (LECO, TC-400). Table 1 shows the chemical analysis of the commercially pure titanium (cp-Ti) and Ti-Mo alloys after melting.

The ingots were subjected to a homogenization heat treatment at 1273 K for 24 h, in a vacuum of  $10^{-6}$  Torr, followed by slow cooling. Subsequently, hot rolling was performed at 1273 K, followed by air cooling. Finally, the ingots were reheated to relieve residual stresses, under the same conditions as those used in the first step. After the stress relief heat treatment, the samples underwent a gas doping process, carried out in a quartz tube under a vacuum of  $10^{-7}$  Torr, a heating rate of 10 K/min, and an isothermal holding at 973 K during 2.0 h, and cooling in water to room temperature.

Oxygen was introduced into the quartz tube ( $1.0 \times 10^{-1}$  Torr) during the isothermal holding. To measure the amount of oxygen in the samples, gas analysis was performed with the thermoconductivity difference using LECO instrument, model TC-400. Table 2 shows the content of oxygen and nitrogen. The relatively low strength cp-Ti sample was used as a reference standard.

The structure of investigated samples was analyzed with X-ray diffraction (XRD) in a Rigaku D/Max 2100/PC diffractometer using the powder method with Cu-K $\alpha$  ( $\lambda = 1.544$  Å) radiation in fixed time mode with a step of  $0.02^\circ$ , a fixed step time of 1.6 s, and a scan from 20 to  $80^\circ$ . Microstructural analysis was performed with scanning electron microscopy using Phillips-FEG and Zeiss EVO-LS15 equipment.

The internal friction and frequency of forced oscillations were measured as a function of temperature in the dynamical mechanical analyzer (AREVA, DM-25) in the temperature range from 300 to 700 K. A 1.0 K/min heating rate and the frequency range from 1 to 10 Hz were used.

## 3. Results and discussion

The X-ray diffraction patterns of the alloys after the stress relief heat treatment are shown in Fig. 1. The microstructure of the alloys is sensitive to the molybdenum concentration in solid solution. For the alloy with 5 wt.% molybdenum, peaks that are characteristic of the  $\alpha'$  phase (with an hcp structure) and the  $\alpha''$  phase (with an orthorhombic structure) were observed, while in the alloy with 10 wt.% molybdenum, peaks that are characteristic of the  $\beta$  phase (bcc structure) were observed, in addition to peaks characteristic of the  $\alpha''$  phase. The alloys with 15 to 30 wt.% molybdenum presented a diffraction pattern formed only by peaks that characterize the  $\beta$  phase. Ho et al. [54] observed the action  $\beta$ -stabilizer of the molybdenum in the binary Ti-Mo system, since the  $\beta$  phase began to be observed when the alloy contained 9 wt.% molybdenum.

Figure 2 shows micrographs of the investigated alloys after the stress relief heat treatment. In accordance with the X-ray diffraction results illustrated in Fig. 1, the microstructure of Ti-Mo alloys depends on the molybdenum concentration. The  $\alpha''$  phase predominated in the Ti-5Mo alloy, as described in the literature [57]. It should be noted, however, that the  $\alpha'$  phase was also present. The  $\alpha'$ ,  $\alpha''$ , and  $\beta$  phases coexisted in the Ti-10Mo alloy. In the Ti-15Mo alloy, the  $\beta$  phase predominated, with the presence of the  $\alpha'$  phase, which arises from the grain boundaries, while the Ti-20Mo alloy was comprised entirely of the  $\beta$  phase.

Mechanical spectroscopy was used to measure variation of Young's modulus with temperature in the processed alloys. Figure 3 illustrates Young's modulus of the cp-Ti and Ti-Mo alloys compared to other materials and Ti-based alloys used as biomedical materials [2]. All processed alloys show

Chemical compositions of the cp-Ti and Ti-Mo alloys used in this study

Element (wt.%)	cp-Ti	Ti-5Mo	Ti-10Mo	Ti-15Mo	Ti-20Mo	Ti-25Mo	Ti-30Mo
Ni	0.009	0.004	0.005	0.007	0.007	0.007	0.008
Mn	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Fe	0.050	0.031	0.032	0.026	0.022	0.052	0.010
Cr	0.003	0.005	0.007	0.009	0.008	0.003	0.006
Al	0.004	0.002	0.005	0.004	0.005	0.003	0.005
Zr	0.004	<0.001	0.002	<0.001	<0.001	<0.001	<0.001
Mo	0.002	5.10	9.95	14.90	20.63	25.68	29.23
Ti	balance	balance	balance	balance	balance	balance	balance

TABLE 1

Oxygen and nitrogen content in the cp-Ti and Ti-Mo alloys

	cp-Ti		Ti-5Mo		Ti-10Mo		Ti-15Mo		Ti-20Mo		Ti-25Mo		Ti-30Mo	
(wt.%)	O	N	O	N	O	N	O	N	O	N	O	N	O	N
#3	0.14	0.02	0.24	0.02	0.21	0.02	0.15	0.01	0.23	0.01	0.25	0.01	0.24	0.02

TABLE 2

Young's modulus below the value of the cp-Ti. Thus, it is clearly demonstrated that Young's modulus depends on the molybdenum content and the phases present in the material.

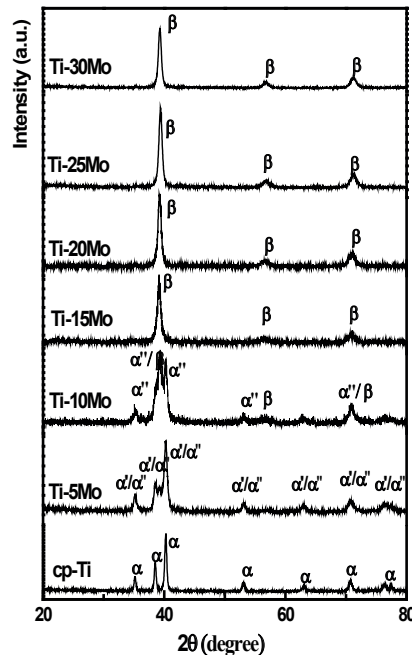


Fig. 1. X-ray diffraction pattern for the cp-Ti and Ti-Mo alloys after the stress relief heat treatment

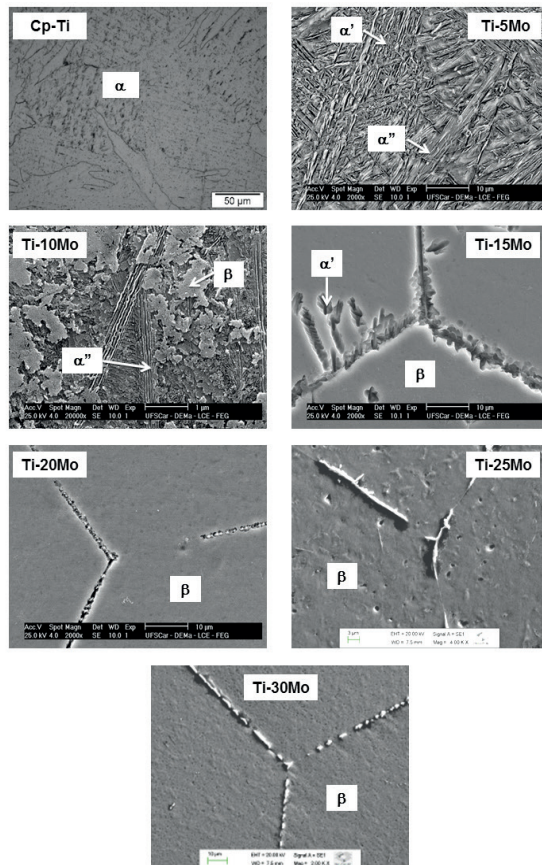


Fig. 2. Microstructure of the cp-Ti and Ti-Mo alloys after the stress relief heat treatment

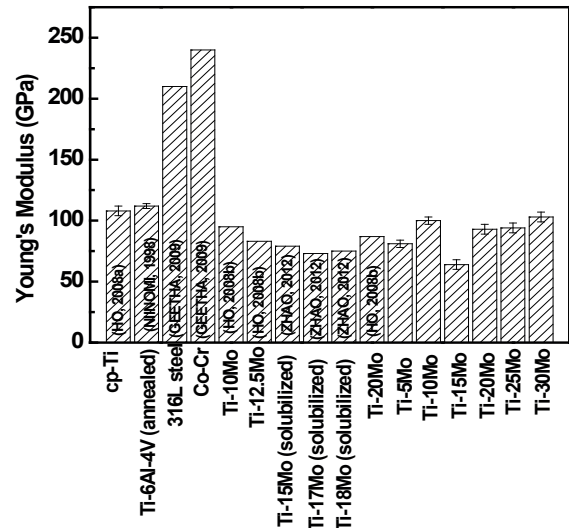


Fig. 3. Young's modulus of the reference cp-Ti and Ti-Mo alloys, and selected data reported in the literature

According to the X-ray diffraction results (Fig. 1) and the micrographs shown in Fig. 2, the  $\alpha''$  phase is predominant in the Ti-5Mo alloy, the  $\alpha''$  and  $\beta$  phases coexist in the Ti-10Mo alloy, the Ti-15Mo alloy presents a small quantity of the  $\alpha'$  phase, and from the Ti-20Mo alloy, the  $\beta$ -phase is predominant, as described in the literature [54,55,57]. In Ref. [58], the modulus of  $\beta$ -type Ti-Mo alloys is lower as compared to the alloys of  $\alpha$ - and  $(\alpha + \beta)$ -type, and  $\alpha''$  phase has smaller modulus values as compared to the  $\alpha$ -type and  $(\alpha + \beta)$ -type, but higher when compared with the  $\beta$ -type [54]. Alloys that contain an athermal  $\omega$  phase have high value of Young's modulus. It should be noted that the Ti-15Mo alloy presented the smallest value of Young's modulus among the studied alloys (64 GPa). This unexpected result contradicts the results found in the literature for the  $\beta$ -type alloys with the  $\alpha'$  phase. A possible explanation is that the Ti-15Mo alloy has the lowest amount of oxygen [59].

Figures 4 and 5 show the mechanical loss angle (internal friction),  $\tan \delta$ , and Young's modulus measured as a function of temperature for the cp-Ti and Ti-5Mo alloy, at three different frequencies: 1, 5, and 10 Hz, after subtraction of an exponential high-temperature background. The content of interstitial oxygen in the samples was 0.14 wt.% for the cp-Ti and 0.24 wt.% for the Ti-5Mo alloy.

Figure 4 shows thermally activated internal friction peak at about 525 K and the corresponding variation of Young's modulus. The peak at 525 K is a relaxation internal friction peak attributed to stress induced short-range ordering of interstitial atoms in solid solution [6]. Variation of peak's temperature and Young's modulus as a function of frequency yields the activation energy and pre-exponential factor. The activation energy and pre-exponential factor of the 525 K peak were  $H = 1.23 \pm 0.04$  eV and  $\tau_0 = 1.02 \pm 0.01 \times 10^{-14}$  s. Another method for analyzing an internal friction peak, assumes that Snoek peak can be estimated with Debye's equation [6,60]. Thus, internal friction peaks, shown in Fig. 4, were analyzed using a nonlinear regression method. The activation energy



and the pre-exponential factor were  $H = 1.23 \pm 0.02$  eV and  $\tau_0 = 1.32 \pm 0.02 \times 10^{-14}$  s.

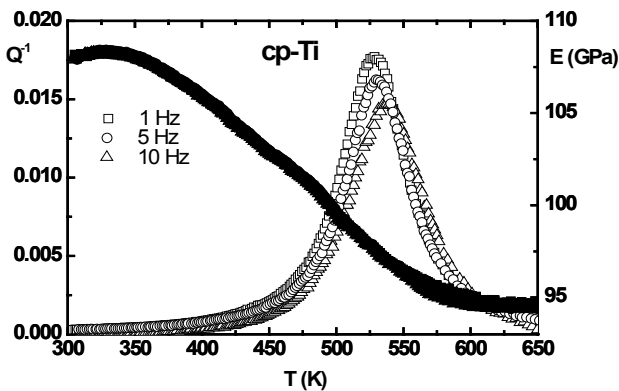


Fig. 4. Internal friction spectra and variation of Young's modulus with temperature for the commercially pure titanium, cp-Ti, sample measured at 1, 5, and 10 Hz

Old results on internal friction in Ti-O alloys report internal friction peak with peak's temperature around 697-754 K, for frequency around 1 Hz, using free decay vibrations (see ref. [10]). These results were obtained on titanium of lower purity and such samples contain more substitutional impurities that the samples used in this study. Thus other complexes might have been involved.

Grandini et al. studied the stress induced ordering of oxygen atoms in Nb-Ti [61-65] and Ti-based alloys [66,67] and reported the activation energy of 1.23 eV for the stress induced ordering of oxygen atoms around titanium atoms. The chemical and gas analysis show (Tables 1 and 2) that the samples contain mainly titanium and small quantities of impurities, and a certain amount of oxygen (0.14 wt.%) and nitrogen (0.02 wt.%). Thus, it is suggested that the 525 K peak is due to the stress induced short-range ordering of oxygen complexes around titanium atoms in the metallic matrix.

Figure 5 shows two thermally activated internal friction peaks, at about 440 and 525 K. Both peaks were subjected to the same analysis as the cp-Ti reference sample, that is, the Arrhenius plot and the Debye peak fitting procedure. It is tacitly assumed here, that the internal friction peaks might be estimated as single relaxation processes. Nonetheless, more detailed analysis should take into account the interaction between interstitial atoms [22-30,48], the anisotropy effect of Snoek peak [18-21], and grain size.

Further study of the interactions between relaxing species requires advanced deconvolution of internal friction spectra measured with sufficiently high precision to obtain satisfactory resolution in the lognormal distribution of relaxation times. In this study, we confined ourselves to the preliminary estimation of the relaxation parameters in ternary Ti-Mo-O alloys to demonstrate potential applications of mechanical spectroscopy to study Ti-Mo-based biomaterials.

The activation energy and pre-exponential factor of the internal friction peak observed at around 525 K were  $H = 1.23 \pm 0.02$  eV and  $\tau_0 = 1.12 \pm 0.01 \times 10^{-14}$  s (Arrhenius plot), and  $H = 1.23 \pm 0.02$  eV and  $\tau_0 = 1.23 \pm 0.03 \times 10^{-14}$  s (Debye

fitting). The peak is the same as observed in the cp-Ti (Fig. 4), and thus, is due to the stress induced short-range reordering of oxygen complexes around the titanium atoms in hexagonal lattice [6,42-48].

The activation energy and pre-exponential factor of the 460 K peak were  $H = 1.17 \pm 0.02$  eV and  $\tau_0 = 3.82 \pm 0.01 \times 10^{-14}$  s (Arrhenius plot), and  $H = 1.17 \pm 0.02$  eV and  $\tau_0 = 4.00 \pm 0.02 \times 10^{-14}$  s (Debye fitting). The chemical and gas analysis showed (Tables 1 and 2) that the samples contained mainly titanium and molybdenum, small quantities of impurities, 0.24 wt.% of oxygen, and a low quantity of nitrogen (0.02 wt.%) in solid solution.

In ternary Ti-X-O solid solutions the interaction between the oxygen atoms and the substitutional atoms is stronger (varies for different substitutional atoms) than the oxygen-oxygen interaction, and, thus, significantly affects the short-range order of interstitial atoms, displacement of the host atom, and, thus, internal friction spectra in the hcp metals [48]. The 460 K peak may be attributed to the stress induced short-range ordering of oxygen atoms around the solute molybdenum atoms. Yet, the relaxing entities are still not resolved. Further research is needed regarding how to determine the relaxation mechanism of the 460 K peak and the complex elastic dipole involved in the relaxation process, which can be gained only by the investigation of Ti-Mo-O single crystals. The data available to answer this question are very limited, but sufficient to suggest that the situation is similar to that in the other hcp metals such as Zr and Hf. We have not pursued this line of investigation in the current work, since we confined our attention to polycrystalline cp-Ti and Ti-Mo alloys.

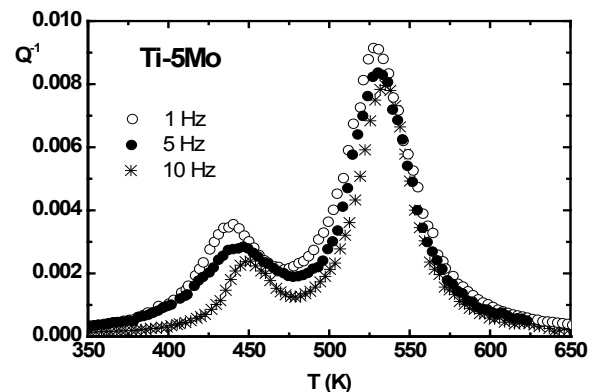


Fig. 5. Internal friction spectra for the Ti-5Mo sample measured at different frequencies: 1, 5, and 10 Hz

#### 4. Conclusions

Samples of Ti-Mo alloys were melted using an arc furnace and characterized with chemical analysis, X-ray diffraction, scanning electron microscopy, and mechanical spectroscopy. Young's modulus depends on the molybdenum and oxygen content, and the phases present in the alloys. The Ti-15Mo alloy which comprises low oxygen content is the Ti-Mo alloy with the lowest value of Young's modulus

(64 GPa). Internal friction results indicate that, in the first approximation, the peak observed in the cp-Ti, can be described by nearly a single Debye relaxation peak, which stems from stress induced short-range ordering of oxygen pairs in solid solution. Two internal friction peaks were observed in the Ti-5Mo alloy. These peaks are attributed to the stress induced short-range ordering of oxygen atoms around the titanium and molybdenum atoms.

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