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Characterization and modeling of forged Ti-6Al-4V Titanium alloy with microstructural considerations during quenching process

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Abstract

The present investigation proposes an experimental device able to assess the thermo-mechanical behavior of Ti-6Al-4V Titanium alloy throughout the die-forging operation. Constitutive equations are developed to assess the influence of the process (die-forging temperature, cooling rate) and the microstructure parameters on the mechanical response of the alloy. For this purpose, a non-unified behavior model formulation is implemented, which defines two main mechanisms related to α and β phases and allows the prediction of hardening, strain rate sensitivity and temperature, combined with the phase evolution that is dependent on the cooling conditions and which can greatly affect the mechanical behavior. This identification strategy is then applied for die-forging temperatures below the β -transus temperature, which requires microstructural information provided by SEM (Scanning Electron Microscopy) observations and image analysis. Finally, the approach is extended to die-forging temperatures above the β -transus temperature.

Keywords: Behavior modeling, Microstructural evolution, Heat treatment, Forged Titanium alloy

1. Introduction

Titanium alloys are widely used in the aerospace industry for their well-known high mechanical strength/weight ratio [1]. They can be used as forged semi-finished products in many industrial applications. These products are transformed into final parts by subsequent thermo-mechanical heat treatments and machining operations. Depending on the temperature of the thermo-mechanical processing (TMP) and the heat treatments (HT), various complex microstructures can be achieved. Titanium alloys can be heat-treated above or below the β -transus temperature depending upon the specific micro-structural aspects required in terms of grain size and morphology, as well as the existence of phases and mechanical strength requested by the end-users. HT generally consists in an isothermal dwell for a certain period of time

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12 following quenching. To fulfil the microstructural characteristics and mechanical
 13 performance requirements and according to the size of the semi-finished products,
 14 an appropriate quenching environment (e.g., air, oil, water, etc.) is selected. The
 15 dimensions of the product and the quenching conditions can drastically influence
 16 the spatial quenching rates, specifically the time-temperature history, in any points
 17 in the product. In fact, quenching operations generate transient time-temperature
 18 histories in a part, moving inwards from the surface to the bulk. One of the major
 19 concerns, particularly for products with large dimensions, is to guarantee a homo-
 20 geneous microstructure through all regions of the product. Another major technical
 21 concern is to avoid any excessive distortions during quenching and also hot tearing,
 22 which result in internal defect initiation, such as micro- and/or mesoscopic crack-
 23 ing. While the post-quenching surface cracking can be eliminated by subsequent
 24 machining, the undetectable internal cracking becomes a major parameter that can
 25 drastically disqualify a semi-finished product for reasons of nonconformity. More-
 26 over, these operations induce important residual stresses in the part, which makes
 27 the final milling stage difficult. Predicting the internal residual strains/stresses has
 28 become mandatory in industrial practice. The relevant constitutive laws therefore
 29 need to be developed. However, one important problem is that during HT and
 30 quenching the microstructure and phases in titanium alloys evolve depending on the
 31 kinetics of the phase evolutions, in that they are time-temperature rate dependent.
 32 Furthermore, titanium alloys have a complex mechanical behavior, exhibiting strain
 33 rate sensitivity effects [2, 3], which can be reproduced through viscosity laws [4–6].
 34 These effects becomes significant for temperatures greater than 500°C [7, 8]. De-
 35 pending on the test temperature, this phenomenon can be combined with hardening
 36 effects induced by dislocation motions and resulting from a competition between
 37 storage and annihilation terms [9, 10]. At very high temperatures, other mecha-
 38 nisms are induced, such as grain boundary sliding [11, 12]. Moreover, important
 39 microstructural changes can occur, such as grain growth, or phase fraction evolu-
 40 tions, which themselves greatly influence the mechanical behavior, and therefore
 41 need to be introduced in the model formulation [13–15]. Indeed, these evolutions
 42 can involve strain hardening [5, 16] or softening due to dynamic recrystallization [17]
 43 under large deformation conditions. Moreover, when the β -phase is predominant,
 44 particular phenomena, caused by the pinning-depinning effect of the dislocation,
 45 can affect the mechanical behavior (*Yield point effect*). This effect was investigated
 46 at the scale of the single crystal [18, 19] then generalized at the scale of the poly-
 47 crystal [11, 20–22]. Hence, non-unified approaches can be implemented in order to
 48 define several inelastic mechanisms associated with each phase evolution [14, 23–25].
 49 Such approaches can translate grain-boundary strengthening caused by a lamellar
 50 microstructure [26–29].

51 The present study proposes a behavior model that is able to faithfully predict the
 52 strain-stress response of the material during the quenching operation. Considering
 53 all the aspects described above, a laboratory experiment testing facility has been
 54 developed to conduct in-situ heat-treatment at temperatures beyond or lower than
 55 β -transus temperature on a cylindrical specimen [30]. Then, tensile tests were com-
 56 bined in order to assess the behavior of the alloy under transient thermo-mechanical
 57 loadings. The microstructural evolutions are greatly influenced by the cooling rate

and can exhibit several phases (primary and secondary α phase and β phase), the proportions of which were assessed by SEM observations and image analysis. In such complex conditions, the mechanical behavior was characterized for several temperature levels and cooling rates. From these experiments, non-unified constitutive equations were implemented to define several mechanisms related to each phase. They consider a rule of mixture between phases depending on the cooling conditions. This rule plays an important role on the activated mechanisms influencing the mechanical behavior. The model formulation can take account of the strain rate sensitivity and the hardening over a wide temperature range (from the die-forging temperature to the ambient temperature) and several cooling rates connected to various microstructural states. This behavior model was identified for die-forging temperatures below the β -transus temperature. Finally, the approach was successfully extended to die-forging temperatures above the β -transus temperature.

2. Experimental procedures

2.1. Material and device

Industrial thermo-mechanical heat treatment consists in 3 main operations, as shown in Fig. 1:

- forging at 940°C
- die-forging at 950°C
- tempering at 730°C .

Figure 1: Thermo-mechanical industrial process

The material studied in the present work was supplied by Aubert & Duval as a billet of Ti-6Al-4V Titanium alloy after the forging operation. Cylindrical specimens were machined from this billet. At this stage, an equi-axed microstructure was observed, which included α primary nodules, decorated at grain boundaries by the β phase, as shown in Fig. 2a. Depending on the microstructure needs (equiaxed, duplex or lamellar morphology), the die-forging operation can be carried out at different temperatures that are higher or lower than the β -transus temperature ($T_\beta = 1000^\circ\text{C}$). In the present case, the die-forging temperature considered is 950°C (i.e. below the β -transus temperature). During the cooling/quenching from

87 this temperature, the β phase transforms into a lamellar microstructure consisting of
 88 colonies of secondary α in the β phase (labelled β_t). At this die-forging temperature,
 89 the phase transformation is not entirely completed and a duplex microstructure is
 90 obtained, regardless of the cooling conditions (Fig. 2b at $60^\circ\text{C}/\text{min}$).

Figure 2: Starting microstructure of Ti-6Al-4V : (a) after Forging, (b) after time-temperature heat treatment corresponding to the Die-Forging (cooling rate: $60^\circ\text{C}/\text{min}$) [Kroll Reagent | SEM | $\times 2000$]

91 The cooling rate greatly influences the induced microstructure. Indeed, the thick-
 92 ness of the secondary (lamellar) α -phase diminishes as the cooling rate increases. In
 93 order to characterize the mechanical behavior during the quenching operation after
 94 die-forging, an experimental device was specially developed to reproduce the
 95 industrial heat treatment. It is based on a *Schenck hydropuls* hydraulic tensile test
 96 machine with a nominal force up to 250 kN. It allows an in-situ heat treatment
 97 by using an induction coil, which ensures a homogeneous temperature at the center
 98 of the sample. The specimens were heated by a 2 kW Celes generator. A cylin-
 99 drical specimen was first instrumented by 9 spot-welded thermocouples to control
 100 the longitudinal and circumferential temperature gradients. In the gauge length,
 101 thermal gradients were about $1^\circ\text{C}/\text{mm}$ and $1^\circ\text{C}/120^\circ$ angle respectively in longitu-
 102 dinal and circumferential directions. The strain was measured by high temperature
 103 extensometer with ceramic rods and a 10 mm gauge length. The system allows fast
 104 heating and cooling steps. During each test, the samples were heated up to 950°C
 105 with a dwell time of 2 hours at this temperature (die-forging temperature). Then the
 106 samples were cooled down to different temperatures (ranging from 950°C to room
 107 temperature) before starting the mechanical loading. Thus, after only a few seconds
 108 of dwell time, the tensile tests were performed in air atmosphere by maintaining the
 109 temperature constant. The tensile test consisted in a first mechanical loading with
 110 a constant strain rate and a maximal strain of 1%, followed by a tensile dwell time
 111 of 10 min and a second loading to reach a total strain of 2%. At the end of the
 112 mechanical test, air spraying was applied at the surface of the sample to prevent

an evolution of the microstructure (mainly the growth of the phases). A schematic view of the tests performed is shown in Fig. 3.

Figure 3: Test procedure (a) In-situ heat treatment; (b) Isothermal loading path

2.2. Tensile tests

The test conditions (see Fig. 3) were selected in order to accurately reproduce the thermo-mechanical loadings induced in the billet during the die-forging step. This analysis led to three cooling conditions being considered ($\dot{\theta} = \{5, 60, 200\}^{\circ}\text{C}/\text{min}$) and several strain rates ($10^{-4}\text{s}^{-1} \leq \dot{\epsilon} \leq 10^{-2}\text{s}^{-1}$). Moreover, three temperature domains were investigated:

- from 950°C to 800°C
- from 800°C to 500°C
- from 500°C to 20°C

In the following section, the effects of the cooling rate, strain rate and test temperature on the stress-strain response are discussed. Interpretations are based on microstructural evolution analysis (fraction and size of α_I nodules, α_{II} lamellae or β phase).

2.2.1. Influence of the cooling rate

As shown by many research works [6, 31] on the topic of microstructure evolution during cooling from temperatures above 950°C , while the phase transformation ($\beta \leftrightarrow \alpha$) mainly depends on the temperature, the cooling rate mainly affects the size of the primary α nodules as well as the initiation and growth of the α_{II} phase, leading to different sizes and morphologies. Five tensile tests at different temperature levels ($\theta = \{950, 800, 700, 500, 300, 20\}^{\circ}\text{C}$) were conducted with a constant strain rate of 10^{-2}s^{-1} . The present study shows that, during cooling, an important phase transformation of β into α_{II} occurs, mainly between 950°C and 900°C . This result is confirmed by other research works [32, 33] and is illustrated in more detail in the

138 next section (section 2.2.2). Thus, for the tensile tests carried out at and above
 139 800°C , it can be considered that the microstructure observed at room temperature
 140 is the one generated at the test temperature, as shown by Fig. 4 for tests conducted
 141 at 20°C and 700°C under various cooling conditions. For the rapid cooling rates
 142 ($\dot{\theta} = \{60, 200\}^{\circ}\text{C}/\text{min}$), many thin α_{II} lamellae can be observed in the transformed
 143 β grains. At a slower cooling rate ($\dot{\theta} = 5^{\circ}\text{C}/\text{min}$), the nucleation of α_{II} lamellae
 144 takes a longer time to grow, leading to lamellar coarsening. Hence, the morphology
 145 of these lamellae is quite similar to that of the α_I nodules.

Figure 4: SEM observations for tensile tests performed at 700°C and 20°C for different cooling rates: $5^{\circ}\text{C}/\text{min}$ (left), $60^{\circ}\text{C}/\text{min}$ (center), $200^{\circ}\text{C}/\text{min}$ (right) [Kroll Reagent | SEM | $\times 2000$]

146 The corresponding tensile tests (see Fig. 5) show a significant hardening with
 147 the increase in the cooling rate. This feature can be related to the decrease in
 148 the α_{II} -lamellar thickness with the cooling rate. This induces a higher number
 149 of α/β boundaries that can act as more obstacles to the dislocation movements.
 150 This hardening is thus linked to the plasticity of the α phase (α_I nodules and α_{II}
 151 lamellae) as the evolution of the lamellar thickness can be related to the yield stress
 152 [31, 34, 35] or to the hardness or the ductility of the material [27, 35]. Image analysis
 153 was conducted to determine the α_{II} lamellar thickness L ($\dot{\theta} = \{60, 200\}^{\circ}\text{C}/\text{min}$) and
 154 the α_I nodule size ($\dot{\theta} = 5^{\circ}\text{C}/\text{min}$). Fig. 6 illustrates the evolution of α_{II} -lamellae
 155 thickness L for different cooling rates. As shown, this trend can be aligned to a power
 156 law. By plotting the curve given by Eq. 1 in a bi-logarithmic diagram, parameter
 157 B can be determined from the value of the slope. Regarding the α_I nodules, the
 158 observations do not exhibit a significant evolution, regardless of the test conditions.
 159 The average size considered next is thus $15\mu\text{m}$.

Figure 5: Stress-Strain response for different cooling rates at 20°C(a); and 700°C(b) and a constant strain rate of $10^{-2} s^{-1}$

Figure 6: Evolution of the α_{II} -lamellae thickness with the cooling rate

$$L = B \times \dot{\theta}^{-1} \quad (1)$$

2.2.2. Time effects: temperature and strain rate

As mentioned previously, the phase transformation, in terms of fraction of phase, no longer evolves below 800°C, as the transformation of β into mainly α_{II} lamellae occurs between 950°C and 800°C, as shown in Fig. 7.

At 950°C, the β fraction can be deduced from the SEM images by analyzing the fraction of β matrix β_{HT} transformed through the fast cooling (air spraying) operated at the end of the mechanical test. At 900°C, the measurements are complex, as the cooling time (from 950°C to 900°C) is not long enough to clearly distinguish the transformed β phase induced by the controlled cooling rate and the fast cooling from 900°C to 20°C. Therefore, this temperature level will not be deeply investigated in

Figure 7: SEM observations for tensile tests performed at 950°C(a); and 800°C(b) for a cooling rate of 60°C/min[Kroll Reagent | SEM | × 2000]

the sequel. From these measurements, the evolution of the β fraction can be assessed (see Fig. 8).

Figure 8: Evolution of the β -phase fraction with the temperature

These results are very similar to those provided in the work of Elmer [32] showing, in a Ti-6Al-4V alloy, the evolution with the temperature of the β phase amounts measured by in situ X-ray diffraction techniques. As in our present study, it seems that most of the $\beta \leftrightarrow \alpha$ phase transformation occurs at a temperature in the 800°C – T_β range. This therefore confirms that, during cooling, most of the β phase had completed its transformation into α_{II} phase around 800°C. Regarding the mechanical behavior, as expected, the stress-strain curves show a decrease in the flow stress with the temperature (see Fig. 9). Moreover, a significant stress relaxation occurs during the tensile dwell time for the test temperature above 500°C, involving considerable viscous stress, whereas it is considerably reduced below this temperature.

182 Lastly, at 950°C , a yield point phenomenon is observed. It is probably due to a
 183 pinning-depinning process of the dislocations in a *Cottrell atmosphere* [36, 37], as
 184 observed in BCC metals. Indeed, in BCC metals (as the β phase), the dislocations
 185 can be pinned by interstitials, in which case a higher force is required in order to
 186 leave such dislocations away from their *Cottrell atmosphere*. Thus, during the first
 187 loading at 950°C , this higher force is responsible for the upper yield point (stress
 188 peak). After unpinning, the dislocations can move easily at a lower stress leading
 189 to a slight stress softening. During the dwell time, the initial Cottrell atmosphere
 190 is recovered (static recovery) involving a new stress peak after the second loading.
 191 This phenomenon is predominant at 950°C where the mechanisms related to the β
 192 phase play an important role, but vanishes at lower temperatures where the plastic
 193 deformation is mainly governed by the α phase.

Figure 9: Stress-Strain response for constant strain (10^{-2} s^{-1}) and cooling ($60^{\circ}\text{C}/\text{min}$) rates at different temperatures $\theta \leq 600^{\circ}\text{C}$ (a); $\theta \geq 700^{\circ}\text{C}$ (b)

194 3. Behavior modeling

195 The previous analysis leads to the definition of 3 mechanisms acting on the
 196 mechanical behavior. The first one is related to the β phase, whereas the two others
 197 are related to the α phase through the nodular part (α_I) and lamellar part (α_{II}).

198 3.1. Non-unified Constitutive Equations

199 A homogeneous deformation (Eq. (2)) is assumed in each phase [14] and a strain
 200 partition of the total strain into elastic and plastic parts is considered (Eq. (3)).

$$\underline{\varepsilon}^t = \underline{\varepsilon}_{\alpha_I}^t = \underline{\varepsilon}_{\alpha_{II}}^t = \underline{\varepsilon}_{\beta}^t \quad (2)$$

$$\underline{\varepsilon}_{\phi}^t = \underline{\varepsilon}_{\phi}^e + \underline{\varepsilon}_{\phi}^p \quad \forall \phi = \alpha_I, \alpha_{II}, \beta \quad (3)$$

201 Hooke's law is given by Eq. 4 for each phase. And each strain component can
 202 be related to a phase ratio Z_{ϕ} (Eq. 5).

$$\underline{\sigma}_\phi = \underline{\underline{C}}_\phi (\underline{\varepsilon}_\phi^t - \underline{\varepsilon}_\phi^p) \quad \forall \phi \quad (4)$$

$$\underline{\varepsilon}^e = \sum_\phi Z_\phi \underline{\varepsilon}_\phi^e; \quad \underline{\varepsilon}^p = \sum_\phi Z_\phi \underline{\varepsilon}_\phi^p \quad \text{with:} \quad \sum_\phi Z_\phi = 1 \quad \forall \phi \quad (5)$$

203 A von Mises yield surface is assumed for each phase, as shown by Eq. 6. Its
204 evolution is defined through an isotropic hardening variable R_ϕ .

$$f_\phi = \sigma_\phi^{eq} - R_\phi - \sigma_\phi^0 = 0 \quad \forall \phi \quad (6)$$

205 σ_ϕ^{eq} and σ_ϕ^0 , are respectively the equivalent stress and the elasticity limit related
206 to the phase ϕ .

207 This approach is in agreement with the thermodynamics of the irreversible pro-
208 cess defined by two potentials, the Helmotz free energy ψ and the dissipation po-
209 tential Ω .

210 The free energy can be partitioned into elastic and inelastic parts $\psi = \psi^e + \psi^{in}$
211 and, in the present study, formulated for each phase (Eq. 7).

$$\varrho \psi^e = \frac{1}{2} \sum_\phi Z_\phi^2 \underline{\underline{C}}_\phi \underline{\varepsilon}_\phi^e : \underline{\varepsilon}_\phi^e; \quad \varrho \psi^{in} = \frac{1}{2} \sum_\phi Z_\phi^2 b_\phi Q_\phi r_\phi^2 \quad \forall \phi \quad (7)$$

212 The state laws giving the Cauchy stress and the macroscopic isotropic hardening
213 variable derive from this potential (Eq. 8).

$$\underline{\sigma} = \varrho \frac{\partial \psi^e}{\partial \underline{\varepsilon}^e} = \sum_\phi Z_\phi \underline{\sigma}_\phi; \quad R = \varrho \frac{\partial \psi^{in}}{\partial r} = \sum_\phi Z_\phi R_\phi = \sum_\phi Z_\phi b_\phi Q_\phi r_\phi \quad (8)$$

214 with r_ϕ the internal variable associated to the isotropic hardening. b_ϕ and Q_ϕ
215 are temperature-dependent coefficients.

216 The dissipation potential Ω allows definition of the evolution of the internal
217 variables (Eq. 9). It includes, first, a static recovery part (Eq. 10) and a classical
218 viscoplastic potential formulated in the form of a power law. However, its expression
219 differs from one phase to another. Indeed, a similar form is used to describe the
220 primary and secondary alpha phase (Eq. 11), whereas a particular form is considered
221 for the β phase (Eq. 12) so as to reproduce the yield point phenomenon.

$$\Omega = \sum_\phi Z_\phi^2 (\Omega_\phi^p + \Omega_\phi^r) \quad \forall \phi \quad (9)$$

$$\Omega_\phi^r = \frac{a_\phi R_\phi^2}{2 b_\phi Q_\phi} \quad \forall \phi \quad (10)$$

$$\Omega_\alpha^p = \frac{K_\alpha}{n_\alpha + 1} \left\langle \frac{f_\alpha}{K_\alpha} \right\rangle^{n_\alpha + 1} \quad \forall \alpha = \alpha_I, \alpha_{II} \quad (11)$$

$$\Omega_\beta^p = \frac{b_\rho \rho_m}{M} \frac{D}{n_\beta + 1} \left\langle \frac{f_\beta}{D} \right\rangle^{n_\beta + 1} \quad (12)$$

223 K_α and n_α are temperature-dependent parameters of the phase $\alpha = (\alpha_I, \alpha_{II})$. a_ϕ
 224 defines the static recovery term in the hardening variables of each phase. Especially
 225 for the β -phase, D is a material parameter, b_ρ is the Burgers vector, ρ_m the density
 226 of mobile dislocations and M the Taylor factor.

227 The viscoplastic flow derives from this potential (Eq. 13).

$$\underline{\dot{\varepsilon}}^p = \partial\Omega/\partial\underline{\sigma} = \sum_{\phi} Z_{\phi} \frac{3}{2} \frac{S_{\phi}}{\sigma_{\phi}^{eq}} \dot{p}_{\phi} = \sum_{\phi} Z_{\phi} \underline{\dot{\varepsilon}}_{\phi}^p \quad \forall \phi \quad (13)$$

228 where S_{ϕ} is the deviatoric part of $\underline{\sigma}_{\phi}$.

229 The cumulative plastic strain for each phase is given by Eq. 14 for the α phase
 230 and Eq. 15 for the β phase.

$$\dot{p}_{\alpha} = \Omega'_{\alpha}(f_{\alpha}) = \left\langle \frac{f_{\alpha}}{K_{\alpha}} \right\rangle^{n_{\alpha}} \quad \forall \alpha = \alpha_I, \alpha_{II} \quad (14)$$

231

$$\dot{p}_{\beta} = \Omega'_{\beta}(f_{\beta}) = \frac{b_{\rho}\rho_m}{M} \left\langle \frac{f_{\beta}}{D} \right\rangle^{n_{\beta}} \quad (15)$$

232 Lastly, the evolution equation related to the isotropic hardening for each phase
 233 is determined from Eq. 16.

$$\dot{r} = -\partial\Omega/\partial R = \sum_{\phi} Z_{\phi} \dot{r}_{\phi} \quad \text{with: } \dot{r}_{\phi} = \dot{p}_{\phi} (1 - b_{\phi} r_{\phi}) - a_{\phi} r_{\phi} \quad \forall \phi \quad (16)$$

234 The positivity of intrinsic dissipation $\mathcal{D}$ ensures good agreement of the model
 235 formulation with thermodynamic principles. It can be expressed by Eq. 17.

$$\mathcal{D} = \sum_{\phi} \underline{\sigma}_{\phi} : \underline{\dot{\varepsilon}}_{\phi}^p - \sum_{\phi} R_{\phi} \dot{r}_{\phi} \quad (17)$$

236 The positivity of $\mathcal{D}$ can be proved by Eq. 18.

$$\mathcal{D} = \sum_{\phi} \left(f_{\phi} + R_{\phi} + \frac{R_{\phi}^2}{Q_{\phi}} \right) \dot{p}_{\phi} + \sum_{\phi} \frac{a_{\phi}}{b_{\phi}} \left(\frac{R_{\phi}}{Q_{\phi}} \right)^2 \geq 0 \quad (18)$$

237 3.2. Introduction of the microstructural parameters

238 The viscoplastic flows (Eq. 14 and 15) require the identification of material pa-
 239 rameters for each phase, which were determined by using SEM observations and
 240 image analysis. Thus, the influence of the microstructural evolutions related to the
 241 cooling rate during the quenching stage can be introduced into the model formula-
 242 tion.

243 3.2.1. α_I phase

244 The proposed model acts on the coefficient K_{α_I} and establishes a relationship
 245 between this parameter and the average size of the primary α nodules d_{α_I} , as shown
 246 by Eq. 19 following the Hall-Petch law.

$$K_{\alpha_I} = K_1 d_{\alpha_I}^{-n_d} \quad (19)$$

with K_1 a temperature-dependent material parameter and n_d the Hall-Petch coefficient, $n_d = 0.5$.

3.2.2. α_{II} phase

Similarly, the proposed law introduces a relationship, given by Eq. 20, between $K_{\alpha_{II}}$ and the thickness of the α_{II} lamellae L .

$$K_{\alpha_{II}} = K_2 L^{-n_L} \quad (20)$$

K_2 and n_L are material parameters. L depends on the cooling rate $\dot{\theta}$ (see Eq. 1).

3.2.3. β phase

The mobile dislocations are at the root of the yield point phenomenon [24] and the density of these dislocations ρ_m is a part f_m of the density of the total dislocations ρ_t (Equation 21). Moreover, this part evolves between a starting value f_{m0} and an asymptotic value f_{ma} [38]. Finally, an empirical law is used to define the relation between the densities of the total dislocations and the cumulative plastic strain.

$$\rho_m = f_m \rho_t; \quad \rho_t = \rho_0 + C_\rho p_\beta \quad (21)$$

where C_ρ and a_ρ are material parameters.

The strain rate had a significant influence on the yield point phenomenon, therefore, the following time evolution of f_m is assumed (Equation 22).

$$\dot{f}_m = -\lambda \dot{p}_\beta^\kappa (f_m - f_{ma}) \quad (22)$$

with: $f_m(t=0) = f_{m0}$ and λ and κ are material parameters.

This equation differs from the literature [14]. Indeed, in the present study, the yield point phenomenon increases with the strain rate, which is an effect that has not been observed on steels [24, 25] or on metastable β -titanium alloys [21, 22].

As shown previously, the tensile dwell time induces a stress relaxation which can be reproduced by introducing a static recovery term into the isotropic hardening component. This phenomenon involves a dislocations rearrangement with a decrease in the dislocation density [39–41]. Moreover, during this dwell time at high temperature, some interstitial atoms can diffuse back around the dislocations, leading to the re-pinning of dislocations in the Cottrell atmosphere. Therefore, the yield point phenomenon is again observed during the second loading. In order to account for this effect in the model formulation, Eq. 22 is modified by Eq. 23 and a static recovery term is added, describing the decrease in the density of the mobile dislocations during dwell time.

$$\dot{f}_m = -\lambda \dot{p}_\beta^\kappa (f_m - f_{ma}) - \mu f_m^\delta \quad (23)$$

where μ and δ are material parameters.

All the constitutive equations are given in Appendix A (table A.1).

4. Results

4.1. Identification Strategy

4.1.1. Young's Modulus

The evolution of Young's modulus with the temperature is obtained by using the relationship given by Eq. 24.

$$E = (Z_{\alpha_I} + Z_{\alpha_{II}})E_{\alpha} + Z_{\beta}E_{\beta} \quad (24)$$

The same values are assumed for α_I nodules and α_{II} lamellae. A tensile test at 1030°C was performed to determine the modulus of the β -treated alloy. A literature review [15, 42–45] allows Young's modulus evolution to be determined at lower temperatures. Then, knowing the phase fraction Z_{ϕ} , the Young's modulus values of the α phase are deduced from Eq. 24. The results obtained are in a good agreement with the values found in the literature, as shown in Fig. 10.

Figure 10: Temperature evolution of Young's modulus for α and β phases: (a) literature (b) values used in this study

4.1.2. Time-dependent parameters

The tensile tests performed with a cooling rate of $60^{\circ}\text{C}/\text{min}$ were used to identify the viscous parameters K_{ϕ} and n_{ϕ} . The stress relaxation curves $\sigma - \sigma_i = f(\text{time})$ were plotted in a bi-logarithmic diagram to determine these parameters for temperature level, where σ_i is the non-viscous stress corresponding to the stabilized stress value at the end of the relaxation time. The static recovery term a_{ϕ} of the isotropic hardening variable allows a better description of the relaxation curve, as shown in Fig. 11. This term $a = a_{\phi}$ is assumed to be equal for each phase and is obtained by an optimization procedure for each temperature level.

The curve gives the value of $n = n_{\phi}$, which is assumed to be the same for each phase. The K_{ϕ} parameter depends on the phase, as shown in Eq. 19 and 20. Assuming $K_{\alpha_I} = K_{\alpha_{II}}$ given by the bi-logarithmic curve, L (provided by Eq. 1) and d_{α_I} , obtained by image analysis, one can determine, on the one hand, K_1 for the α_I nodules, and on the other hand, K_2 and n_L for the α_{II} lamellae. It can be observed that Eq. 19 corresponds to a Hall-Petch law and the coefficient

Figure 11: Comparison between the relaxation range provided by the model and the experiment for a cooling rate of $60^\circ\text{C}/\text{min}$ and a temperature of 700°C

n_d is equal to 0.5. Moreover, the D parameter for the β phase is obtained by taking $Z_\beta = 1$. Then, a rule of mixtures between phases gives a weighting of the parameters related to each phase. To consider the yield point phenomenon in the model formulation induced at high temperature, some additional parameters need to be identified. The values of the Burgers vector b_ρ and of the Taylor factor M come from the work of Wang for a titanium alloy [21]. Moreover, the parameters related to the dislocation densities ρ_0 , C_ρ and f_{m0} are also found in the literature [22]. Lastly, the parameter a_ρ is identified from the tests performed at 950°C and 900°C , its value is usually between 0.7 and 1.5 [24, 37, 39]. The parameters λ , δ , κ and μ related to the volume fraction of mobile dislocation are also identified at 950°C and 900°C where the yield point phenomenon is observed, they are selected to fit the stress-strain curve and are determined by an optimization procedure. The volume fraction of mobile dislocations f_m evolves between an initial value f_{m0} and an asymptotic one f_{ma} [24, 25]. f_{m0} is constant with the temperature, whereas f_{ma} decreases in order to take into account the decrease in the mobile dislocations density at lower temperatures. This evolution will only be activated at 950°C and 900°C in order to consider the yield point phenomenon. For the other temperature levels, this phenomenon vanishes by taking $f_{ma} = f_{m0}$.

4.1.3. Hardening parameters

The parameters $Q = Q_\phi$ and $b = b_\phi$ of the isotropic hardening variable and the elasticity limit $\sigma^0 = \sigma_\phi^0$ are assumed to be equal for each phase. An optimization procedure is used for a cooling rate of $60^\circ\text{C}/\text{min}$ considering the whole database.

4.2. Results

4.2.1. Model prediction on $(\alpha + \beta)$ Ti-6Al-4V

Fig. 12 illustrates a comparison between simulation and experiment at 950°C . At this temperature, the mechanism related to the β phase is predominant and the

333 modified version of the Yoshida model (Eq. 15, 21, 22) presented in the previous
 334 section allows a good description of the behavior at different strain rates.

Figure 12: Yield point prediction at $950^{\circ}C$ and several strain rates (*line*: simulation, *marker*: experiment)

335 For a cooling rate of $60^{\circ}C/min$ the model response is in a good agreement with
 336 the experiment at lower temperatures and different strain rates, as shown in Fig.
 337 13a at $800^{\circ}C$ and in Fig. 13b at $700^{\circ}C$.

Figure 13: Computed Strain-Stress data (*line*) compared to Experimental results (*marker*) for several strain rates and at $\theta = 800^{\circ}C$ (a); $\theta = 700^{\circ}C$ (b).

338 Lastly, the model predictions for several cooling rates are illustrated in Fig. 14
 339 at $\theta = 500^{\circ}C$ (a) and at $\theta = 20^{\circ}C$ (b).

340 All the values of the model parameters are given in Appendix B (Tables B.2-B.6).

341 4.2.2. Model extension on β -treated Ti-6Al-4V

342 In this section, the model is extended to a β -treated alloy. For this purpose,
 343 an experimental test campaign similar to the previous one was performed. It used
 344 the same starting samples obtained after the Forging operation (see Fig. 1) but
 345 the experimental procedure was changed by considering a solution annealing at

Figure 14: Computed Strain-Stress data (*line*) compared to Experimental results (*marker*) for several cooling rates and at $\theta = 500^\circ C$ (a); $\theta = 20^\circ C$ (b).

1030°C above the β -transus temperature ($T_\beta = 1000^\circ C$). In this case, all the model parameters related to the β -phase are identified at 1030°C (see table C.7). At this temperature, the parameters of the mechanical model are α -phase independent, therefore $Z_{\alpha I} = Z_{\alpha II} = 0$. For the other temperature levels, the model prediction was made by maintaining the values of almost all the model parameters identified for the $(\alpha + \beta)$ heat treatment (Tables B.2-B.6). Only the microstructural parameters (Table C.8) related to the phase fractions Z_Φ and the evolution of lamellae morphology L with the cooling rate needed to be identified again from SEM image analysis in order to investigate the β -heat-treated alloy. The mechanism related to the α_I nodules vanished in this case ($Z_{\alpha I} = 0$), as during the solid solution annealing at 1030°C where all the α_I nodules were transformed into β . Then, during cooling below the β -transus temperature, the β phase was finally transformed into a fully lamellar $(\alpha + \beta)$ microstructure.

Figure 15: Computed Strain-Stress data (*line*) compared to Experimental results (*marker*) at $\theta = 950^\circ C$ and several strain rates (a); at $\theta = 700^\circ C$ and several cooling rates (b).

Figure 16: Computed Strain-Stress data (*line*) compared to Experimental results (*marker*) for several cooling rates and at $\theta = 500^{\circ}\text{C}$ (a); $\theta = 20^{\circ}\text{C}$ (b).

Fig. 15 and Fig. 16 illustrate the comparison between the computed Strain-Stress data and experimental results in the case to the β -heat treated Ti-6Al-4V, at 950°C and for several strain rates (Fig. 15a), at intermediate temperatures ($T=\{20, 500, 700\}^{\circ}\text{C}$) and for several cooling rates (Fig. 15b and 16a-b). Aside from the temperature of 950°C where the stress levels are very low, discrepancies between the model response and the experiment were less than 15% for the other test conditions. This is quite acceptable since the only parameters that have to be changed concern microstructural features of the β -heat treated alloy.

4.3. Discussion

The quenching of industrial parts generates transient temperature variations that induce plastic straining due to thermal self-constraining. The degree of self-constraining depends on the dimensions of a part and on the heat transfer mechanisms between the part and the quenching environment (for example oil or water quenching ...) that control the mean global cooling rate [46]. In general the residual stresses are investigated at room temperature, and can be measured through X-Ray Diffraction or hole drilling methods. In-situ measurements of the residual stresses are not possible and the only rational manner is to use advanced thermo-mechanical modeling and numerical simulations analysis. Therefore relevant and reliable constitutive laws have to be developed. However, alloys such as Ti-6Al-4V alloy are very much prone to microstructural evolution at high temperature or during transient temperature-time conditions. In many approaches, the reliability of the constitutive laws is examined by laboratory testing of heat-treated alloys. The high temperature assessments are thus run on specimens heated up again to a prescribed temperature for mechanical testing. In the present investigation, the main objective was to assess and to model the thermo-mechanical behavior of Ti-6Al-4V alloy, through stepwise-temperature mechanical testing by first conducting an in-situ heat-treatment and then by quenching with a controlled rate to a prescribed temperature and finally by conducting the mechanical testing at this temperature. Such combining of time-temperature-mechanical scenarios are not commonly reported in the literature and often absent in the open literature. Although, many investigations deal with the

mechanical behavior of Titanium alloys especially Ti-6Al-4V alloy, most of them consider the thermodynamic equilibrium conditions. However, some comparisons can be made with the present study considering the influence of the microstructure on the mechanical response of the material.

First, the phase analysis (Fig. 8) illustrates a drastic decrease of the β phase from 950 to 800°C with a β phase around 78% at 950°C. Similar results are found in the literature at this temperature [32, 33]. This means that the role of the β phase cannot be neglected and the plasticity induced has to be linked to this phase. The yield point phenomenon illustrated in Fig. 12 is observed on many Body-Centered Cubic (BCC) materials [39, 41] such as the β phase for titanium alloys. It can be related to the dislocations locked by the solute atoms then broken away from the pinning points at a high stress level. It is also associated with discontinuous yielding with the increase of new mobile dislocations generated from the grain boundary [20, 21, 47, 48]. The behavior model implemented in the present study is based on a rule of mixture between two mechanisms, one related to the α phase (divided in primary and secondary phases), the second to the β phase. At high temperatures, the constitutive equations associated with the β phase are predominant, and the formulation is based on the works performed by Yoshida et al on BCC materials [21, 22, 24, 25]. As discussed previously, these equations are modified to take into account some particular effects observed, such as the increase of the yield point with the strain rate and the dislocations rearrangement with a decrease in the dislocation density during dwell times (see Eq. 23).

Secondly, at lower temperatures, the effect of the α phase increases while that of the β phase decreases. Moreover, Fig. 4 shows a decrease of the α_{II} -lamellar thickness with the cooling rate involving an increase of the flow stress (Fig. 5). This result is induced by an increase of the α/β boundaries acting as obstacles to the dislocation movements. These results are also shown in several investigations [28, 35, 49]. This effect is described in the constitutive equations through the viscous flow where the K_Φ ($\Phi = \{\alpha_I, \alpha_{II}\}$) parameter evolves with the thickness of the α_{II} lamellae L (see Eq. 20), itself related to the cooling rate, or with the average size of the primary α nodules d_{α_I} (Eq. 19).

Finally, the strain rate sensitivity is reduced for temperatures inferior to 500°C [8] involving a significant hardening effect which is assumed similar for each phase. On the other hand, Fig. 12 and 13 show an important strain rate effect for the temperatures exceeding 600°C as mentioned in [3] compared to the hardening effect.

5. Conclusions

In the present work, an experimental device was developed to reproduce the mechanical behavior of Ti-6Al-4V throughout the die-forging operation. A non-unified behavior model was implemented and the following conclusions can be drawn.

- For a $(\alpha + \beta)$ dual-phase alloy, the phase transformation is greatly influenced by the cooling rate conditions, which themselves play an important role in the strain-stress response of the material.
- The non-unified behavior model is able to predict the mechanical behavior, assuming an initial phase proportion (α_I, α_{II} or β).

- 433 • Depending on the test temperature, the model gives a good prediction of the
434 strain rate and hardening effects. Moreover, based on a modified Yoshida
435 model formulation, it can describe the yield point phenomenon observed at
436 high temperature and the static recovery effect exhibited during the dwell
437 times.
- 438 • Lastly, the model was successfully extended to the behavior prediction of a
439 β -treated Ti-6Al-4V alloy.

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445 **Appendix A. Model summary**

446 The table A.1 summarizes the model formulation.

Table A.1: non-unified model formulation

	Multi-axial formulation	Uni-axial formulation
Yield criterion	$f_\phi = \sigma_\phi^{eq} - R_\phi - \sigma_\phi^0 \quad \forall \phi$	$f_\phi = \sigma_\phi - R_\phi - \sigma_\phi^0 \quad \forall \phi$
Hooke's law	$\underline{\sigma}_\phi = \underline{C}_\phi \left(\underline{\varepsilon}_\phi^t - \underline{\varepsilon}_\phi^p \right) \quad \forall \phi$	$\sigma_\phi = E_\phi \left(\varepsilon_\phi^t - \varepsilon_\phi^p \right) \quad \forall \phi$
Flow rules	$\underline{\dot{\varepsilon}}^p = \sum_\phi Z_\phi^2 \frac{3}{2} \frac{\underline{\sigma}_\phi}{\sigma_\phi^{eq}} \dot{p}_\phi \quad \forall \phi$ with $\dot{p}_\alpha = \left\langle \frac{f_\alpha}{K_\alpha} \right\rangle^{n_\alpha} \quad \forall \alpha = \alpha_I, \alpha_{II}$ and $\dot{p}_\beta = \frac{b_\rho \rho_m}{M} \left\langle \frac{f_\beta}{D} \right\rangle^{n_\beta}$	$\dot{\varepsilon}^p = \sum_\phi Z_\phi \dot{p}_\phi \text{sign}(\sigma_\phi) \quad \forall \phi$ with $\dot{p}_\alpha = \left\langle \frac{f_\alpha}{K_\alpha} \right\rangle^{n_\alpha}$ and $\dot{p}_\beta = \frac{b_\rho \rho_m}{M} \left\langle \frac{f_\beta}{D} \right\rangle^{n_\beta}$
Microstructural parameters	$K_{\alpha_I} = K_1 d_{\alpha_I}^{-n_d}$ $K_{\alpha_{II}} = K_2 L^{-n_L} \quad \text{with } L = B \dot{\theta}^{-1}$	
Metallurgical parameters	$\rho_m = f_m \rho_t; \quad \rho_t = \rho_0 + C_\rho p_\beta$ $\dot{f}_m = -\lambda \dot{p}_\beta^\kappa (f_m - f_{ma}) - \mu f_m^\delta; \quad f_m(0) = f_{m0}$	
Isotropic hardening	$\dot{r} = -\sum_\phi Z_\phi \dot{r}_\phi \quad \text{with } \dot{r}_\phi = \dot{p}_\phi (1 - b_\phi r_\phi) - a_\phi r_\phi$ and $R_\phi = b_\phi Q_\phi r_\phi \quad \forall \phi$	

447 Appendix B. Model coefficients for $\alpha + \beta$ treated Ti-6Al-4V

448 Table B.2 illustrates the phase-independent model parameters, tables B.3 and B.4
 449 the α -phase dependent model parameters and tables B.5 and B.6 β -phase dependent
 450 model parameters

Table B.2: Phase independent and temperature dependent model parameters

θ [$^{\circ}C$]	950	900	800	700	600	500	400	300	20
σ^0 [MPa]	1	1	3	10	34	145	327	386	650
Q [MPa]	1	2	55	85	92	97	100	101	103
b	1	5	190	317	400	425	433	434	435
a [s^{-1}]	$5 \cdot 10^{-1}$	$2.5 \cdot 10^{-1}$	$8 \cdot 10^{-2}$	$2 \cdot 10^{-2}$	$8 \cdot 10^{-3}$	$3 \cdot 10^{-3}$	$2 \cdot 10^{-4}$	$1.4 \cdot 10^{-4}$	$4 \cdot 10^{-5}$
n	3.3	3.4	3.6	4.9	7.8	9.8	11.2	11.7	12

Table B.3: α -phase and temperature dependent model parameters (1)

θ [$^{\circ}C$]	950	900	800	700	600	500	400	300	20
E_{α} [GPa]	35	47	60.7	70	77.4	86	85.1	84.1	109.5
$Z_{\alpha_{II}}$	0	0.22	0.44						
K_1	23.7	24	62.8	77.3	73.5	51.5	20.8	20.2	32.3
K_2	95	96	251	309	294	208	83	81	131

Table B.4: α -phase dependent model parameters (2)

Z_{α_I}	d_{α_I} [mm]	n_d	n_L	B
0.22	$15 \cdot 10^{-3}$	0.5	0.105	$66.7 \cdot 10^{-3}$

Table B.5: β -phase and temperature dependent model parameters (1)

θ [$^{\circ}C$]	950	900	800	700	600	500	400	300	20
E_{β} [GPa]	39.3	48.2	60.7	69	74	81	79	78	90
D	181	182	370	472	493	372	160	148	290
Z_{β}	0.78	0.56	0.18						
f_{ma}	$5 \cdot 10^{-3}$	$1 \cdot 10^{-3}$	$5.5 \cdot 10^{-4}$	$4.5 \cdot 10^{-4}$	$4 \cdot 10^{-4}$				

Table B.6: β -phase dependent model parameters (2)

b_{ρ} [cm]	M	ρ_0 [cm^{-2}]	C_{ρ} [cm^2]	f_{m0}	λ	κ	μ	δ
$2.5 \cdot 10^{-8}$	2.76	$6.4 \cdot 10^{10}$	$2.1 \cdot 10^{11}$	$4 \cdot 10^{-4}$	$18 \cdot 10^3$	2	1.25	2

451 **Appendix C. Model coefficients updated for β treated Ti-6Al-4V**

452 Table C.7 gives the values of the model parameters at 1030° C and table C.8 the
 453 temperature evolution of the microstructural parameters identified for the β -heat
 454 treated titanium alloy.

Table C.7: Temperature model parameters at 1030° C

σ^0 [MPa]	Q [MPa]	b	a [s^{-1}]	n	E_β [GPa]	D [MPa]	f_{ma}
0	1	1	1.1	3	35	85	$1 \cdot 10^{-1}$

Table C.8: Updated microstructural parameters for the β -treated Ti-6Al-4V

θ [$^{\circ}C$]	1030	950	900	800	700	600	500	400	300	20
$Z_{\alpha I}$	0									
$Z_{\alpha II}$	0	0.22	0.44	0.82						
Z_{β}	1	0.78	0.56	0.18						
B	$80.6 \cdot 10^{-3}$									

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