Superplasticity in Ti–6Al–4V: Characterisation, modelling and applications

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Abstract
The processing regime relevant to superplasticity in the Ti–6Al–4V alloy is identified. The effect is found to be potent in the range 850–900 °C at strain rates between 0.001/s and 0.0001/s. Within this regime, mechanical behaviour is characterised by steady-state grain size and negligible cavity formation; electron backscatter diffraction studies confirm a random texture, leaving grain-boundary sliding as the overarching deformation mechanism. Outside of the superplastic regime, grain size refinement involving recrystallisation and the formation of voids and cavities cause macroscopic softening; low ductility results. Stress hardening is correlated to grain growth and accumulation of dislocations. The findings are used to construct a processing map, on which the dominant deformation mechanisms are identified. Physically-based constitutive equations are presented which are faithful to the observed deformation mechanisms. Internal state variables are used to represent the evolution of grain size, dislocation density and void fraction. Material constants are determined using genetic-algorithm optimisation techniques. Finally, the deformation behaviour of this material in an industrially relevant problem is simulated: the inflation of diffusion-bonded material for the manufacture of hollow, lightweight structures.

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1. Introduction

Superplasticity [1] can be exploited for the production of components of complex architecture. Examples exist in the aerospace, defence, biomedical, sports and automotive sectors [2]. Although usually found in metallic systems, for example those based upon aluminium [3], magnesium [4] and zinc [5], superplasticity has also been reported in some ceramics [6] and geological materials [7]. However, it is for titanium alloys of the type studied in this paper that the effect has the greatest technological relevance.

Nevertheless, to exploit the superplastic effect in an effective manner more fundamental research is necessary. First, a greater understanding of the underlying physical mechanisms is needed, for example to elucidate the microstructural effects which cause the superplastic effect to arise. Second, for design purposes, this understanding needs to be translated into validated material laws which are accurate and which capture the relevant phenomena. Third, computer-based simulations of the relevant manufacturing operations are needed, probably operating across a range of length scales and with a multi-physics interdisciplinary flavour.

The research reported in this paper was carried out with the above in mind. Superplasticity in the Ti–6Al–4V material is studied under constant strain-rate conditions; this has allowed the regime of superplasticity to be pinpointed. Next, microstructurally explicit material laws are proposed based upon the micromechanical modes of deformation which are shown to be operating. In the final part of the paper, modelling is used to simulate an industrially-relevant manufacturing process which is important for the construction of hollow, lightweight structures which are of significant practical importance.

2. Background

Since its discovery over 80 years ago, an abundance of papers regarding superplasticity have emerged in the literature. Experimentalists have worked on the establishment of rules that enhance the superplastic behaviour of materials. Two basic requirements are found to optimise the effect: a very fine grain size and a high-forming temperature [1]. However, these requirements are often considered to be incompatible, because of grain growth at elevated temperatures; hence a compromise is needed. Theorists have tried to elucidate the mechanisms of superplasticity, but with varying degrees of success. The role of grain boundary sliding is
often acknowledged. But whether grain boundary sliding is diffusion-accommodated [8–11], dislocation-accommodated [12,13], or a combination of both [14] remains unclear; moreover, the role of cooperative grain boundary sliding [15] or the effect of cavity formation [16] remains controversial. It seems reasonable to believe that more than one mechanism may be operating and that processing conditions and material characteristics will have a great effect on which ones are active.

In the case of titanium alloy Ti–6Al–4V, it is known that the two-phase microstructure plays an important role during superplasticity. For example, assisting the grain boundary sliding process [17], influencing the growth of cavities [18] or enhancing the superplastic effect by reducing – but not inhibiting – grain growth at high-temperatures. Regarding the influence of microstructure on macroscopic behaviour, it is accepted that stress hardening during high-temperature superplasticity is related to grain growth [19]. However, the cause of flow softening at higher strain rates and lower temperatures is not so well understood. Some researchers relate flow softening to adiabatic heating during compression tests [20], globularisation of lamellar secondary \( \alpha \) phase in transformed microstructures [21,22] and dynamic recrystallisation of the \( \beta \) phase above the \( \beta \)-transus temperature [23]. However, none of these phenomena explain the flow softening which arises in equiaxed, superplastically-tested materials.

Traditionally, material models for the constitutive modelling of metals have been divided into empirical and physical models [24]. Physically-based internal state variable models (ISVs) fall into the second category [25]. This method describes the macroscopic behaviour of a material using a number of dynamic equations that represent its underlying deformation mechanisms. A number of relevant papers – modelling different kinds of metals – can be found for both single-phase [19,26–30] and multi-phase materials [31–35]. This kind of approach is adopted here.

Regarding the study of the industrial manufacturing of superplastic forming processes – primarily using finite element methods – a series of papers are available in the literature. For example, the simulation of the blowing process of thin sheets [36] and the inflation of multi-sheet components [37,38]. These papers describe accurately the industrial processes and can be used for the optimisation of some manufacturing conditions. However, most of them use simple ‘power-law’ material expressions which do not contain any information about the microstructure and its deformation path. The construction of a simulation capability for the superplastic forming process using material laws which describe the underlying mechanisms of superplasticity – as function of some microstructure evolution parameters as illustrated in Fig. 1 – is an overarching aim of the present work.

### 3. Experimental methods

Typical chemical composition for Ti–6Al–4V titanium alloy used in this study is presented in Table 1. Starting microstructure consists of equiaxed \( \alpha \) phase grains with small amounts of intergranular \( \beta \) phase. Mean grain size of received material was 10 \( \mu \)m. Although ultra-fined grained (UFG) materials have been proved to enhance superplastic properties [39], high price and material high sensitivity to grain growth makes them unsuitable for certain applications, for example, those where high temperature pre-processing is necessary prior SPF (i.e. diffusion bonding and forming of multi-sheet parts) [40].

#### 3.1. High temperature tensile testing

Superplasticity in the Ti–6Al–4V alloy was evaluated using specimens of 25 mm gauge length, 6 mm width and 5 mm thickness [41]; these were first electro-discharge machined from plate and then surface ground to ensure the very best surface finish. Custom-made grip boxes were manufactured following E2448 ASTM standard directions using nickel superalloy Inconel 718. These allowed for the insertion of thermocouples so that temperature could be measured accurately on the samples during testing. Isothermal, constant strain-rate tensile tests were carried out using a Zwick Roell Z250 electro-mechanical testing machine. Temperatures ranged from 700 to 950 °C with 50 °C intervals between tests. Constant true strain rate was ensured during the

![Fig. 1. Microstructural changes in Ti–6Al–4V during a hot forming process, and their effect on a typical stress–strain curve.](image-url)
test at \(10^{-2}, 10^{-3}, 10^{-4}\) and \(10^{-5}/s\). The specimens were heated up at a rate of approximately 5 °C/s and then held for 15 min to reach uniform temperature before straining commenced. A three-zone conventional furnace was used to guarantee the uniformity and control of the temperature. Tests were performed in air using a proprietary glaze coating to minimise oxidation. Deformation was held until rupture or until a maximum elongation of 300% strain was reached.

3.2. Microstructural characterisation

Electron backscatter diffraction (EBSD) was used to characterise the grain size both before and after testing. This allowed a quantitative measure of the mean grain size for both \(\alpha\) and \(\beta\) phases. Local true strain on tested samples was analytically determined for each point of the gauge following the expression \(\varepsilon = \ln(A_0/A)\), where \(A\) is the sectioned cross-sectional area and \(A_0\) is the initial area. The specimens were sectioned and marked at different points in length, prepared for metallographic studies and analysed using EBSD. Measurements were taken using a Zeiss EVO scanning electron microscope equipped with an electron dispersion X-ray (EDAX) EBSD camera. Results were analysed using the OIM data analysis software. Grain size distributions were obtained as a function of strain, strain rate and temperature. Table 2 shows the value of parameters specified by the standard for grain measurements using EBSD [42]. Texture measurements of the \(\alpha\) phase in the 0001 plane were taken, identifying the direction and intensity of the basal poles.

Longitudinal sections of the specimens were also characterised to estimate the degree of cavitation. Optical microscopy in combination with imaging processing techniques were used for this purpose. Different measurements along the centre line of the specimen were taken. Results were used to create a cavitation map as function of strain, strain-rate and temperature. For image acquisition, an optical microscope was used at \(\times 10\) magnification. The captured images were processed using digital image processing software to determine the area fraction of voids and cavities. The sample surface was left unetched, to facilitate the using software ImageJ to determine the area fraction of voids and cavities. The captured images were processed using digital image processing software to determine the area fraction of voids and cavities. The sample surface was left unetched, to facilitate the acquisition, an optical microscope was used at \(\times 10\) magnification.

4. Modelling methods

The overarching aim is to model the superplasticity in titanium alloy Ti–6Al–4V using an approach which is physically faithful to the underlying degradation mechanisms which are operative. The material model is calibrated to the experimentally obtained data acquired here, which are in the form of stress–strain curves, grain size measurements and cavitation-strain data sets. To illustrate the power of the model developed for analysis across the length scales, it has been incorporated into finite element analysis software and used to simulate the practical example of superplasticity in diffusion-bonded components which have significant industrial significance.

4.1. Assumptions used for model construction

A series of evolutionary equations – employing internal state variables – have been formulated to consider dynamic changes in the material. These equations are based in a series of physical parameters and phenomena such as grain size, dislocation density, recrystallisation fraction or formation of cavities. In order to avoid the overuse of material parameters, an optimisation route is proposed, see Fig. 2, in which a series of equations are activated or otherwise based on the fitting quality of the global model. This approach allows for the elimination of redundant parameters but more importantly has provided physical insight into the deformation modes which are operative.

4.2. Unified viscoplastic model

The rate of superplastic flow – which is considered a regime of high temperature creep – is expressed in terms of steady-state strain \(\dot{\varepsilon}\), as

\[
\dot{\varepsilon} = \frac{ADGb}{kT} \left(\frac{b}{\sigma}\right)^p \left(\frac{\varepsilon}{\varepsilon_e}\right)^n
\]

(1)

where \(D\) is the diffusion coefficient, \(G\) is the shear modulus, \(b\) is the Burgers vector, \(k\) is the Boltzmann constant, \(T\) is the temperature, \(\sigma\) is the stress, \(d\) is the grain size, \(\varepsilon\) is a material constant and \(p\) and \(n\) are the grain size and stress exponents. Nevertheless, one needs to account for the probability of different deformation conditions being predominant in different deformation regimes. In order to account for this, a hyperbolic sine relationship following [19] has been used. When Ti–6Al–4V is deformed at superplastic temperatures, it is believed that grain boundary sliding (GBS) is the main deformation mechanism. However, an accommodation mechanism (i.e. dislocation or diffusional creep) is necessary in order to achieve continuity at the grain boundaries. At low stress, the material is expected to be diffusion controlled; hence consistent with the hyperbolic relationship a linear stress response is appropriate. For high stress values and strain-rates – where deformation is probably controlled by the motion of dislocations – the relationship becomes non-linear. A unified viscoplastic model is then

\[
\dot{\varepsilon}_{\text{VP}} = \dot{\varepsilon}_{\text{DP}} + \dot{\varepsilon}_{\text{GRS}}
\]

(2)

where \(\dot{\varepsilon}_{\text{DP}}\) is the viscoplastic strain rate, \(\dot{\varepsilon}_{\text{GRS}}\) is the stress, \(d\) is the grain size and \(x\) and \(\beta\) are temperature-dependent material parameters. At high temperatures, a two-phase microstructure is found. The \(\alpha\) phase is harder than the \(\beta\) phase; therefore the strain contribution of each phase should be different. Using the iso-strain condition, the total plastic strain rate can be written as [22]

\[
\dot{\varepsilon}_{\text{GRS}} = \dot{\varepsilon}_{\text{GRS}}(1 - f_\beta) + \dot{\varepsilon}_{\text{GRS}} f_\beta
\]

(3)

where \(\dot{\varepsilon}_{\text{GRS}}\) and \(\dot{\varepsilon}_{\text{GRS}}\) are the strain rate contribution from \(x\) and \(\beta\) phases respectively. The volume fraction of \(\beta\) phase \(f_\beta\) has been assumed to vary with temperature according to [34]

\[
f_\beta = \left(\frac{T}{1270}\right)^{10}
\]

(4)

where \(T\) is temperature in Kelvin. For temperatures above 1270 K it has been assumed that the 100% \(\beta\) phase predominates.

4.2.1. Grain growth

Experimental studies have shown that the ratio of \(x\) and \(\beta\) mean grain size is close to 1 for temperatures near 900 °C [43]. Thus in terms of grain size, both phases are treated equally in this work.

A kinematic grain size model which accounts for the effect of grain growth and dynamic grain growth has been used, consistent with

\[
d = d_{\text{static}} + d_{\text{dynamic}}
\]

(5)
The static grain growth rate is considered an atomic diffusion process affected by temperature related to grain boundary mobility \((M)\) and grain boundary energy density \((\gamma_{\text{surf}})\) [44]. Following [19], static grain growth is modelled as
\[
d_{\text{static}} = \frac{a_1}{C_0} \frac{d}{c_1} \quad \text{for} \quad d = 0
\]
where \(a_1 = (M \cdot \gamma_{\text{surf}})\) and \(c_1\) are temperature-dependent material constants. Dynamic grain growth is represented in the second term of Eq. (5) as
\[
d_{\text{dynamic}} = \frac{a_2}{C_0} \frac{d}{c_2} \quad \text{for} \quad d > 0
\]
where \(a_2\); \(c_2\) are material constants.

### 4.2.2. Dynamic recrystallisation

During high temperature deformation, continuous dynamic recrystallisation is generally associated with the storage of dislocations. When a critical dislocation density is reached, new grains will nucleate in the grain boundaries or areas with a high density of defects. A minimum, critical dislocation density is necessary for dynamic recrystallisation to start, which can be described by [45]
\[
d_{\text{crit}} = x_3 \rho_0 d^{\gamma_1}
\]
where \(x_3\) \((\sigma_{\text{crit}})\) and \(\gamma_1\) are temperature-dependent material constants. Dynamic grain growth is represented in the second term of Eq. (5) as
\[
d_{\text{dynamic}} = x_2 \rho d^{\gamma_2}
\]
where \(x_2\); \(\gamma_2\) are material constants.

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\[
\rho_{\text{crit}} = \left( \frac{10 \gamma_1 \dot{\rho}}{3 b M \tau^2} \right)^{1/3}
\]
where \(\gamma_1\) is the grain boundary energy, \(M\) is the grain boundary mobility, \(\tau\) is the dislocation mean free path \((\tau = c_1 \mu b^2)\). Parameters \(\sigma\), \(\mu\), \(b\), \(c_1\) and \(c_2\) represent stress, shear modulus, Burger vector and two material parameters respectively.

The effect of dislocation density on grain refinement was modelled following [33]. Grain rate change due to dynamic recrystallisation is accounted for in the third term of Eq. (5) as
\[
d_{\text{DRX}} = x_3 S d^{\gamma_3}
\]
where \(x_3\); \(\gamma_3\); \(\gamma_4\) are material constants. \(x_3\) has been assumed temperature-dependent in the current study and \(S\) is the volume fraction of recrystallised material. The dynamic recrystallisation rate is characterised as [44]
\[
\dot{S} = \frac{\nu \gamma d}{d}
\]
where \(\nu\) is the evolving grain boundary area. The parameter \(\nu\) is the moving grain boundary velocity \((\nu = M \tau \rho)\) and \(\rho\) is a material constant. Eq. (10) can be expressed as
\[
\dot{S} = \frac{q_1 \gamma d}{d}
\]
where \(q_1\) is a material parameter. The parameter \(\gamma\) is related to the fraction of mobile boundaries and can be described as [46]
\[
\gamma = (0.1 + S^b) (1 - S) \rho
\]
where \(q_2\) is a material constant.

The dislocation density rate was treated as an internal variable. Mecking and Kocks [47] developed a phenomenological model (the KM model) to characterise the rate of dislocation density accumulation as function of the storage and annihilation of dislocations. A number of dislocation density rate models have arisen since then [27,48,28,49]. The evolution of dislocation density \(\dot{\rho}\) can be expressed as [50]
\[ \frac{d\rho}{d\varepsilon} = K_1\sqrt{\rho} - K_2\rho \]  
(13)

where \( K_1 \) and \( K_2 \) are material constants. The first term in Eq. (13) is related to the athermal process of dislocation storage as a result of plastic straining. The second term models the reduction of dislocation density due to dynamic recovery. A third term is introduced to consider the change in dislocation density caused by static recovery as [44]

\[ \dot{\rho} = -2M\tau \rho^2 \]  
(14)

where \( M \) is the grain boundary mobility and \( \tau \) is the energy per unit length of dislocation. Eq. (14) can be described in a phenomenological form as [27]

\[ \dot{\rho} = -k_2\rho^3 \]  
(15)

where \( k_2 \) and \( k_3 \) are material constants.

Finally, a fourth term to account for the reduction of dislocation density due to recrystallisation may be needed. Recrystallisation annihilates dislocation networks when new dislocation-free grains are created. This phenomenon translates into a variation of the dislocation density expressed as

\[ \dot{\rho} = -k_4\rho \frac{\dot{S}}{1-S} \]  
(16)

where \( k_4 \) is a temperature-dependent material constant and \( S \) is the recrystallised fraction.

Combining Eqs. (13)–(16) and considering the normalised dislocation density concept introduced by Lin et al. [27], a constitutive equation to model the evolution of the normalised dislocation density evolution is defined as

\[ \dot{\rho} = k_1(1 - \rho)\dot{\varepsilon}_p - k_2\rho^3 - k_4\rho \frac{\dot{S}}{1-S} \]  
(17)

where \( k_1, k_2, k_3 \) and \( k_4 \) are material constants. The normalised dislocation density \( \rho \) can take a value from 0 (initial state) to 1 (saturated state).

4.2.3. Cavity and void damage rate

Voided materials are considered to have a reduction in the effective stress following the general expression proposed by Perzyna [51] and further developed by Khaleel et al. [52] for superplastic materials; thus

\[ \sigma = \sigma_m(1 - n_1\dot{\varepsilon}_p^n) \quad \text{and} \quad \dot{\varepsilon} = \sigma / \sigma \]  
(18)

\[ \xi = -A_p / \sigma \]  
(19)

where \( \xi \) is the void fraction parameter defined as function of the area of voids \( A_v \) and the total area of interest \( A \). The terms \( n_1, n_2 \) and \( n_3 \) are the void geometry constants and can be fitted to the experimental stress–strain data.

The rate of change in the void fraction parameter can be partitioned into the nucleation of cavities and the growth of voids so that

\[ \dot{\xi} = \dot{\xi}_{\text{growth}} + \dot{\xi}_{\text{nucleation}} \]  
(20)

The evolution of cavitation growth can then be written as

\[ \dot{\xi}_{\text{growth}} = \eta(1 - \xi)\dot{\varepsilon}_p \]  
(21)

where \( \eta \) is a material constant function of the strain rate \( (\propto \dot{\varepsilon}_p^3) \).

The amount of cavitation due to nucleation can be expressed as

\[ \dot{\xi}_{\text{nucleation}} = \frac{F_0}{(1 - \xi)}\sigma \dot{\varepsilon}_p \]  
(22)

where \( F \) and \( \sigma \) must be determined using experimental cavitation measurements. The parameter \( F \) is assumed proportional to the strain rate \( (\propto \dot{\varepsilon}_p^4) \).

Several studies have found that interactions between hard \( \alpha-\alpha \) phase boundaries facilitate the nucleation of voids and cavities compared to \( \alpha-\beta \) and \( \beta-\beta \) phase boundary interactions. Here, it has been assumed that the damage due to cavitation is proportional to the amount of \( \alpha \) phase and therefore that it alone affects the value of strain contributed by the \( \alpha \) phase.

4.3. A unified viscoplastic model

The mechanism-based unified viscoplastic equations in uniaxial form are presented in Fig. 2. This set of equations has been used to define the mechanical behaviour of Ti–6Al–4V working under high temperature conditions. The physically-based evolutionary equations \( R, d, \dot{\rho}, S \) and \( \xi \) represent kinematic hardening, grain size evolution rate, normalised dislocation density rate, recrystallised fraction rate and void nucleation rate respectively. Each one of the state variables represents a physical phenomenon of the material during superplastic deformation.

4.4. Temperature dependence of the material parameters

Temperature dependent material constants were defined using Arrhenius relationships according to

\[ (C_i)_j = c_i \exp \left( \frac{Q_i}{RT_j} \right) \quad i = 1, 2, \ldots; j = 1, 2, \ldots, J \]  
(23)

where \( C_i \) are the temperature compensated constants, \( c_i \) is a temperature independent parameter, \( Q_i \) is the ith activation energy (J mol\(^{-1}\)), \( I \) is the number of constants to be determined, \( R \) is the gas constant (8.3145 J mol\(^{-1}\) K\(^{-1}\)) and \( T_j \) is the jth temperature in Kelvin.

4.5. Optimisation of material constants

The introduced material constants were determined based on the experimentally obtained data. Genetic algorithms (GA) employing optimisation techniques were used for this purpose. This method minimises the residual error of a given objective function. In this study, a universal objective function developed following [53] was used. The error function was defined as

\[ f(x) = f_{\sigma} + f_{d} + f_{\xi} \]  
(24)

where \( f_{\sigma}, f_{d} \) and \( f_{\xi} \) are the residual error for stress, grain size and void fraction respectively. Each of these residuals can be expressed as function of computed results and experimental measurements as

\[ f_{\sigma,d,\xi} = \frac{1}{M} \sum_{j=1}^{M} \left( \frac{1}{N} \sum_{i=1}^{N} (\sigma_{\text{comp},j,i} - \sigma_{\text{exp},j,i}) \right) \]  
(25)

4.6. Finite element model of a superplastic forming process

To illustrate the advantages and power of the material model which is proposed, it has been incorporated into a numerical treatment of the deformation of a multi-sheet superplastic forming structure using finite element analysis (FEA). The process model conditions follow Wood et al. [54]. The FEA software ABAQUS was employed, with the proposed material model being introduced using a ‘CREEP’ subroutine in which the defined strain rate potential was written as function of von Mises equivalent deviatoric stress and a series of solution-dependent variables or evolutionary equations, solved using the forward Euler method. This subroutine
was called at each material point at the beginning and end of each time increment. The difference in strain rate was compared to a defined stability creep increment ($< 10^{-6}$/s) to judge the accuracy of the integration scheme.

For illustrative purposes, the superplastic forming process model was applied to the practical example of the optimisation of the inflation cycle (pressure) of a hollow, honeycomb-like structure, with the aim of maximising the constant rate of deformation in the internal membrane. This was accomplished using an Abaqus built-in algorithm. This algorithm compares the ratio, $r = \dot{e}_{\text{max}}/\dot{e}_{\text{tar}}$, between the maximum equivalent strain rate, $\dot{e}_{\text{max}}$, and a target creep strain rate, $\dot{e}_{\text{tar}}$ which adjusts the applied pressure following a series of rules.

If the ratio is between 0.2 and 3.0, the increment is accepted and the pressure is adjusted as follows:

1. If $0.2 \leq r < 0.5$ then $P_{\text{new}} = 1.5P_{\text{old}}$
2. If $0.5 \leq r < 0.8$ then $P_{\text{new}} = 1.2P_{\text{old}}$
3. If $0.8 \leq r < 1.5$ then $P_{\text{new}} = P_{\text{old}}$
4. If $1.5 \leq r < 3.0$ then $P_{\text{new}} = 0.83P_{\text{old}}$

where $P_{\text{new}}$ is the updated pressure and $P_{\text{old}}$ is the previous increment pressure magnitude. However, if $r$ is not between 0.2 and 3.0, the increment is restarted as:

1. If $r < 0.2$ then $P_{\text{new}} = 2P_{\text{old}}$
2. If $r > 3.0$ then $P_{\text{new}} = 0.5P_{\text{old}}$

This algorithm is able to generate forming profiles as function of a constant target strain-rate. However, it has been shown that the optimal target strain rate varies through the forming process. Superior optimisation approaches have been developed to overcome this limitation, for example, a method which changes the target strain rate to maximise the strain rate sensitivity [55] or a method that optimises the pressure as function of a flow localisation factor [56–58]. Here, for the purpose of studying the effect of microstructure, the basic built-in algorithm is chosen.

Since one wants to evaluate the advantage of the material model and for the purpose of offering a point of comparison, the material behaviour was also modelled using a simple strain-rate dependent power law according to

$$\sigma = k\dot{e}^m$$

(26)

where $k$ is a material constant and $m$ is the strain-rate sensitivity parameter. This kind of model has been widely used in FEA models for simulation of superplastic forming processes [36], but is phenomenological and only captures steady-state behaviour. The parameters $k$ and $m$ were fitted to represent the steady-state behaviour of the proposed viscoplastic internal state variable (ISV) model with purpose of comparing the effect that microstructural changes – which are included in the ISV model – can have in the superplastic forming process.

Fig. 3. Ti–6Al–4V testpieces after superplastic testing at various temperatures and strain rates. Figure shows elongation-to-failure in terms of engineering strain for each testpiece.

Fig. 4. Stress–strain rate curves for strain values from 0.1 to 0.5, at 0.1 increments.

Fig. 5. Map of strain rate sensitivity exponent for 0.1 strain value, as a function of temperature.
Fig. 6. Electron backscatter diffraction analysis and estimated texture for the α phase at 800 °C for (a) initial microstructure; (b) strain of 0.5 and strain rate $10^{-2}$/s; (c) strain of 1 and strain rate $10^{-3}$/s; (d) strain of 1.25 and strain rate $10^{-4}$/s; (e) strain of 0.5 and strain rate $10^{-5}$/s and at 900 °C for (f) initial microstructure; (g) strain of 0.75 and strain rate $10^{-2}$/s; (h) strain of 1 and strain rate $10^{-3}$/s; (i) strain of 1.25 and strain rate $10^{-4}$/s; (j) strain of 0.5 and strain rate $10^{-5}$/s.
5. Results

5.1. Identification of strain rate/temperature regime for superplastic effect

Results obtained using constant strain rate tests at temperatures relevant to the superplastic forming processes were used to quantify the effect. The full range of testpieces after superplastic testing are presented in Fig. 3. Recorded force–displacement conditions were processed to obtain true stress-true strain curves. Fig. 4 presents the true stress–strain rate curves for 5 different values of true strain, from 0.1 to 0.5. A morphologic analysis of these curves is performed to identify and isolate deformation mechanisms in terms of changes in the microstructure. The following claims are made; (i) At relatively high strain rates (>\(5 \times 10^{-2}/\text{s}\)) and low-medium temperatures (<850 °C) the curves show continuous flow softening. Dynamic recrystallisation and growth of cavities are believed responsible. Further drop of flow stress at high values of strain is related to cavitation reaching catastrophic values. (ii) At medium and high temperatures (>750 °C) and low strain rates (10^{-4}/\text{s}), steady-state flow stress might indicate that work hardening mechanisms such as grain growth or dislocation pile-up are fast enough to compensate the flow softening. (iii) At low strain rates (<10^{-3}/\text{s}), only work hardening is reported. Such behaviour indicates that longer exposure at high temperatures is causing grain growth.

The strain rate sensitivity parameter \(m\) can be expressed as a function of logarithmic values of stress \(\sigma\) and strain rate \(\dot{\varepsilon}\) given in Fig. 4 as

\[
m = \frac{\partial \ln \sigma}{\partial \ln \dot{\varepsilon}}. 
\]

The derived strain rate sensitivity parameter contour map for a given value of 0.1 strain is shown in Fig. 5. The contour map was verified and completed using stress-relaxation tests to determine the strain rate sensitivity parameter \(m\) as described in [59]. This map is used to identify conditions where the superplastic effect is stronger. A maximum \(m\) value is found for temperatures between 800 and 900 °C and strain rates between \(10^{-4}/\text{s}\) and \(5 \times 10^{-4}/\text{s}\). The strain rate sensitivity is linked to the values of elongation to failure during superplasticity. As already reported for similar alloys, the strain rate sensitivity (Fig. 5) and hence, the ductility of the material (Fig. 3) tends to have a sigmoidal shape function of both temperature and strain-rate, producing peaks of strain rate sensitivity [60]. Testing under these ‘hot-zones’ produced elongations in excess of 300% engineering strain with no observable necking (Fig. 3). Comparison of Figs. 4 and 5 shows that steady-state behaviour is necessary in order to obtain optimal superplastic properties (\(m > 0.36\)).
5.2 Deduction of microstructure degradation mechanisms causing superplasticity

Experimental proof of the degradation mechanisms which arise during – and outside – superplasticity are here presented. Electron backscatter diffraction (EBSD) inverse pole figure maps and texture analysis for samples deformed at 800 and 900 °C are presented in Fig. 6. For all tested conditions, the randomness of the crystal orientation increases thus the texture weakens as the superplastic strain accumulates. This effect supports the claim that grain boundary sliding is the main deformation contributor. At 900 °C and $10^{-3}$/s, where the maximum strain rate sensitivity, $m = 0.51$, is achieved, the original grain shape and size remains essentially identical for most of the accumulated strain. At lower strain rates, grain growth is observed. At faster strain rates, grain refinement is reported. Fig. 6(g) and (h) shows partially recrystallised microstructures. At 800 °C, recrystallisation is reported at every rate of deformation. Fig. 6(d) shows a fully recrystallised microstructure after 1.25 true strain. Under these conditions, mean grain size is reduced from 10 to 6 μm. Fig. 6(b) and (c) shows new
grains nucleated around the grain boundaries, however, the strain is not large enough to obtain a fully recrystallised microstructure. Grain refinement facilitates the sliding of grains which reduces the effective stress. Therefore, grain refinement due to dynamic recrystallisation is one of the causes of flow softening.

Fig. 7 shows the unetched surfaces of the testpieces after testing at temperatures of 800 and 900 °C and strain rates of 10^{-2} and 10^{-4}/s. The micrographs were taken at points of maximum strain in each one of the samples. Fig. 7 proves that cavitation is the cause of the low material ductility at high strain rates, reaching up to 10% of voided area before fracture at 800 °C and 10^{-2}/s. For superplastic conditions, cavitation is still present. At 900 °C and 10^{-4}/s the shape of the cavities is rounded, small, widespread and not large enough to cause premature fracture. Therefore, a small amount of cavitation is needed in order to accommodate the sliding of grains at optimal superplastic conditions.

5.3. Determination of material parameters for constitutive law

The series of material parameters found in the proposed constitutive equations need to be determined based on experimental measurements. An optimisation path is defined for this purpose as given in Fig. 2. This step-by-step approach allows the identification of governing mechanisms based on the overall fitness satisfaction of each individual stage. As no microstructural changes are expected at low strain, stress softening and hardening are neglected during the first optimisation step. The first set of equations presented in Fig. 2 were optimised to the yield strength of each curve. Computed curves obtained after the optimisation of parameters (z, k, k_0, μ, k_y, k_p, k_0, μ) are able to capture the yield strength as function of temperature and strain rate as shown in Fig. 8, but generate unsatisfactory results for non-steady state conditions.

For the second and third optimisation steps, changes in grain size are introduced in terms of grain growth and grain refinement respectively. Electron backscatter diffraction maps are statistically processed to obtain the mean grain size at temperatures of 700, 800 and 900 °C and strain rate values between 10^{-2} and 10^{-4}/s. Measured mean grain size data, given as symbols in Fig. 9, are used for the determination of parameters z_1, x_2, y_1, y_2, k_1, k_2, k_3, q_1, and q_2. Solid lines in Fig. 9 represent the computed values of grain size for each temperature and strain rate.

The fourth optimisation step accounts for microstructural degradation in terms of evolution of cavities. Symbols given in Fig. 10 represent the experimentally measured surface fraction of cavities at 800 and 900 °C for strain rates of 10^{-2}, 10^{-3} and 10^{-4}/s. These measurements are used to calibrate the parameters F, p and η, related to the growth and nucleation of cavities. Results are presented as solid lines in Fig. 10. Each internal state variable equation is then reintroduced in the global constitutive law.

The value of each one of the fitted material parameters is shown in Fig. 11 as function of temperature. The material model results are presented with solid lines in Fig. 12 along with the experimental stress–strain curves. The optimised ‘power law’ material parameters k and m for temperatures of 800 and 900 °C and strain-rates between 10^{-3} and 10^{-4}/s are also presented in Fig. 11. The ‘power law’ model stress–strain response is illustrated with dashed lines in Fig. 12(c) and (e).

6. Discussion

6.1. Deformation mechanisms

A deformation map for Ti–6Al–4V has been constructed on the basis of the dominant modes of deformation identified using the optimisation technique previously explained. Fig. 13 presents the different regimes identified for the range of tested temperatures and strain rates. For strain rate sensitivity value greater than m = 0.3, grain boundary sliding is dominant. Microstructure is shown stable in terms of shape and size even at large values of strain. Under these conditions, the first set of constitutive equations presented in Fig. 2 give a satisfactory fit. At lower strain rates, hardening is related to grain growth. In this case, the second set of equations is successfully fitted. At higher strain rates and lower temperatures, dynamic recrystallisation is assumed. After calibrating the third set of equations in Fig. 2 to the experimental grain size measurements, the material model is able to track the flow softening observed at low and moderate strains (ε < 0.5). However, further drop in stress – especially at low temperatures – cannot be explained by this phenomenon. Low ductility is believed to be caused by cavitation formation reaching catastrophic values. The fourth set of equations is introduced and calibrated. Fig. 12 shows how the whole curve morphology can be explained using these four set of microstructurally-based equations.

Given the highly non-linear behaviour, the response of the material is highly sensitive to its deformation path. The approach here adopted – internal state variable model – has shown successful when used to link variations in flow stress to microstructure degradation mechanisms. Although a good correlation between computed and experimental results over a wide range of temperatures and strain rates is shown, the accuracy of the predictions is subjected to the accuracy of the experimental data to which the material parameters are fitted. Physically based equations assure a mathematical and physical foundation of the model but its use should be limited to the range of experimentally obtained data.
Fig. 12. Computed stress-strain data compared to experimental results for temperature of (a) 700 °C; (b) 750 °C; (c) 800 °C; (d) 850 °C; (e) 900 °C and (f) 950 °C.
The fitness quality between model and experiments is considered satisfactory for the main purpose of this work – to correlate the flow stress variation with strain to microstructure evolution mechanisms – however, some disagreement is observed in terms of stress, grain size and cavitation predictions. This discrepancy can be attributed to errors introduced during the experimental and statistical determination of the mean grain size and the area of voids and to errors introduced when assuming that constants are temperature dependent following the Arrhenius equation. A potential improvement is to assume that the material parameters are isothermal. In this case, the material constants must be independently optimised for each given temperatures of interest. Using this approach, the correlation between model and experiments is improved but at expense of having more than a single set of parameters for the range of tested temperatures.

6.2. Superplastic forming

To illustrate the importance of the elucidated deformation map and the relevant regimes of deformation in Ti–6Al–4V, a representative industrial application has been modelled. The material law is used to model the superplastic forming of a multi-sheet part. Geometry and predicted strains at different time points during the inflation process are shown in Fig. 14. This model is used to predict optimal inflation cycles while targeting different conditions shown in Fig. 13 so that different deformation regimes can be compared. Optimised pressure cycles for target strain rates of $10^{-3}$/s and $10^{-4}$/s at temperatures of 800 and 900 °C are presented in Fig. 15. The influence of the proposed material model is studied. A comparison between a ‘power law’ creep model and the internal state variable model was performed to study the effect that microstructural changes have on the optimisation of pressure profiles. Results are presented in Fig. 15. When forming inside the dynamic recrystallisation regime, grain size refinement produces a decrease of stress.

![Deformation map for Ti–6Al–4V](image1)

**Fig. 13.** Deformation map for Ti–6Al–4V, with the dominant modes of deformation in the different regimes identified.

![Optimised pressure cycles](image2)

**Fig. 15.** Optimised pressure cycles for target strain rates of (a) $10^{-3}$/s and (b) $10^{-4}$/s.

![Simulations of superplastic forming](image3)

**Fig. 14.** Simulations of superplastic forming of diffusion-bonded multi-sheet part.
The optimisation tool recognises an increase in the velocity of deformation due to the decrease of stress and regulates the inflation pressure to assure a constant strain-rate. Therefore, the optimisation of the forming process is proved sensitive to microstructure degradation mechanisms. For conditions where the microstructure is stable (900°C and 10−4/s) the predicted pressure cycle is almost identical for each one of the models. Under these conditions, a ‘power law’ model offers significant advantages. For example, this kind of model is already implemented into the majority of commercial FE codes and the identification of material parameters does not require the use of specialised tools – such as genetic algorithms.

The effect that processing conditions have on the evolution of grain size is given in Fig. 16. Dynamic recrystallisation is shown to have a relevant effect on the way that the structure is formed. Localised reduction of mean grain size translates into non-uniform strain distribution along the superplastic membrane. This partially softened structure leads to necking localisation as shown in Fig. 16(a). It is therefore important to control the rate of deformation of the process in order to avoid partial recrystallisation. Fig. 17 illustrates the effect of forming conditions in the formation of cavities. The difference between two given typical processing conditions is illustrated. At 800°C, the softening due to dynamic recrystallisation and evolution of cavities leads to rapid strain increase which would cause the membrane to break due to accumulation of damage. It is demonstrated that the degradation of microstructure has an important effect on the superplastic forming of multi-sheet parts.

One of the main challenges in the SPF industry is to increase the efficiency of production. The goal is to minimise the forming time while assuring the structural integrity of the component. In order to achieve this, criterion techniques have been developed for the evaluation and design of processing parameters. Two main approaches are here discussed. The first one consists of the use

![Fig. 16. Effect of forming temperature (between 800 and 900°C) and strain rate (10−3 and 10−4/s) on mean grain size at the end of the inflation cycle.](image)

![Fig. 17. Effect of temperature and target strain rate on the formation of voids and localisation of damage for (a) 800°C and εtar = 10−3/s and (b) 900°C and εtar = 5·10−4/s.](image)
1. The regime of superplasticity in the Ti–6Al–4V alloy has been pinpointed, using constant strain rate tests in tension. A strain rate sensitivity map is proposed which illustrates the areas where the superplastic effect is optimal. It is prevalent in the range 850–900 °C at strain rates between 0.001/s and 0.0001/s.

2. EBSD analysis indicates that the microstructure remains equiaxed even after large deformations; moreover, the texture remains random. It would appear that grain boundary sliding is responsible for deformation in the superplastic regime but also beyond it.

3. Between 700 and 950 °C, dynamic recrystallisation (DRX) and grain growth are observed. DRX at lower temperatures and higher strain rates is linked to stress softening. At lower strain rates and higher temperatures the stress hardening is associated with grain growth. The elongation to failure is dependent upon the kinetics of cavity/void formation.

4. Microstructural changes during deformation have been modelled using a rate-dependent internal state variable (ISV) model. Changes in dislocation density, grain size, phase fraction and cavity/void formation are included explicitly. Material constants have been determined using genetic algorithms.

5. Prediction fitness has been used to assess the quality of the model and to support the elucidation of the governing mechanisms. It is shown that the capturing of the stress–strain behaviour requires the use of historically-dependent microstructural variables. Implementation into a finite element (FE) model and comparison with test results confirms the accuracy of our modelling.

6. The inflation of a hollow, lightweight box structure of practical relevance is analysed. This demonstrates the advantages of the modelling approach used.

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