PREFACE

Physics based deformation models play a crucial role in the safe life estimation of creep exposed components and for providing insights about hot working processes. Examining the creep and flow response of the metals employing constitutive models pertaining microstructural-based input parameters and variables helps in understanding of deformation characteristics that in turn influence the response of the material during creep and metal forming operations. Modeling and simulation can be a proficient alternative technique that provides the solutions and insights about the various issues. Creep phenomena and metal hot working both involves the movements of dislocations. Therefore, the mechanisms of substructure evolution involve similar physics for both the cases. Since creep research focuses on limiting strain and strain rates at high temperatures, hot working studies aim to at relatively higher strain rates between 10^{-2} and 10^{2}/s. In either creep or hot deformation, the dislocations are the mostly responsible for accommodating plastic deformation. Therefore, while capturing their interactions dislocation density-based models can be developed that provide the anatomy of the induced microstructure with ongoing creep as well as during hot working. In this direction, dislocation density based physical modeling is a decent choice, as it elucidates the complex deformation behaviours as well as provides the insights about the ongoing substructure evolution. The main objective of this research work done in this thesis is to develop microstructure-based creep and flow stress models to reveal the substructure evolution with ongoing deformation. In each of the chapter, an improved model is presented that fills the gap in the literature to some extent.

Chapter 01. Considering the physics-based approaches flow stress models [4, 5, 34, 37, 63] are represented as,

$$\sigma = f(\sigma_0, \rho, \lambda_{mean}) \tag{1.1}$$

where σ_0 refers to the contribution from short range barriers, ρ is the average dislocation density and λ_{mean} being the mean free path of the dislocations. Following issues were observed with respect to flow stress models.

- A physical based model which also incorporates annealing twin density effect and explains the microstructural evolution during plastic deformation of low stacking fault energy material was not found.
- Most of the flow stress models predicts the microstructure evolution along with flow stress response but the velocity of dislocations and influence on recrystallization phenomena was not observed.
- All the experimental curves are fitted with the models, limiting the prediction capabilities.
- Individual contribution of different type of strengthening was rarely addressed.

Similarly, creep rate is modelled as,

$$\dot{\varepsilon} = f\left(\rho_m, \rho_b, \rho_{dip}, R_{sub}, T, \sigma_{appl}, N_v, r_p, D_{cav}, D_{ppt}\right), \tag{1.2}$$

where, ρ_m is mobile dislocation density, ρ_b is boundary dislocation density, ρ_{dip} is dipole dislocation density, R_{sub} is the subgrain radius, N_v is the number density of the precipitate, T is temperature, σ_{app} is applied stress, D_{cav} is the cavitation damage and D_{ppt} is damage due to coarsening of precipitates.

The problem lies in a way that, if models address complete creep curve then microstructural evolution and all other important parameters are not demonstrated [14, 43, 66]. Impact of all

internal variables during the creep exposure is very important and rarely reported. Following issues were observed with existing creep models:

- Slip systems were not incorporated in rate equations of microstructural evolution.
- Influence of stacking fault energy on dislocation velocities was not considered.
- Complex dislocation climb velocity models were used.
- Temperature dependent shear modulus was not incorporated.
- Evolution of some variables was ignored in the literature, that are boundary dislocation spacing, subgrain boundary mobility, dipole capture spacing, climb stress, mean radius of precipitates
- Evolution of damage due to different types of precipitates was not demonstrated separately.
- Temperature and time dependent mobility model for subgrain boundary migration was not implemented.

In light of these issues, physics based flow stress and creep models were developed in the subsequent chapters to address these issues.

Chapter 02. As the flow stress is a crucial parameter in understanding thermomechanical processes such as extrusion, rolling, forging, and other manufacturing processes, its modelling depends on complex microstructures that enable the definition of internal variables. In order to get more insights about the microstructure evolutions during the hot deformation a physical-based model for DDRX that validates the simulation results in terms of flow curves, dislocation density, DDRX fraction, and the predicts the dislocation velocities is a need. In this chapter a flow stress model was developed and validated with the experimental data of IN 718WP.

The key points of this new model is given below,

$$\sigma_{deform} = \underbrace{\sigma_{dis} + \sigma_{HP}}_{athermal\ stress} + \underbrace{\sigma_{ss} + \sigma_{gen}}_{thermal\ stress} \quad (2.1)$$

In Equation (2.1), where σ_{dis}, $\sigma_{HP,}$ and σ_{ss} are the strength contributions due to dislocations, grain size and solid solutions, respectively. σ_{gen} represents strength contribution due to interaction among orthogonal dislocations. The estimation of v_{gl} can be done using the following approach [107],

$$v_{gl} = \lambda_{mean} v_0 exp\left(-\frac{\Delta G}{k_B T}\right) sign(\sigma_{SR_{eff}}) \quad (2.2)$$

In Equation (2.2), λ_{mean} is the mean free path can v_0 is attempt frequency, ΔG is the average activation energy needed to encounter the obstacles, k_B is the Boltzmann constant and $\sigma_{SR_{eff}}$ is the difference between the total flow stress and athermal stress as orthogonal dislocations.The mean free path can be estimated by considering the Pythagorean summation and taking all the possibilities as [113],

$$\frac{C_k}{\lambda_{mean}} = \frac{1}{h_m} + \frac{1}{s} + \frac{1}{g_{avg}} \quad (2.3)$$

Herein, C_k is the dislocation interaction coefficient, s is dislocation substructure size, g_{avg} is the average grain size and h_m is mobile dislocation spacing. The total flow stress ($\sigma_{total,flow}$) response during the hot deformation is estimated using the rule of the mixture of non-recrystallized and recrystallized areas as [116],

$$\sigma_{total,flow} = (1 - X_{DDRX})\sigma_{deform} + X_{DDRX}\sigma_{DDRX} \quad (2.4)$$

Where X_{DDRX} is recrystallization fraction, σ_{DDRX} is the flow stress contribution from DDRX region and σ_{deform} is the flow stress response from unrecrytallized region. The dislocation density-reliant flow stress model was developed by considering the physics behind DDRX phenomena to address the complete flow stress curve as well as microstructure evolution for the

IN718WP superalloy. Model capabilities for microstructure prediction was validated by comparing the results obtained with EBSD data in terms of dislocation density and DDRX fraction. The important conclusions of this work are,

- Model predicts the flow stress response with a single set of calibrated parameters for the wide range of temperature and strain rate.
- The average grain size was observed to be decreasing and approaching to steady state easily at low strain rates or higher temperatures as compared to that of higher strain rates or lower temperatures.

Chapter 03. In this chapter a flow stress model was developed that includes the addition of twin boundaries as a strengthening elements as a new addition to previous developed models [34]. The model was validated with respect to flow curves of IN718 [4]. Total flow stress response can be expressed as,

$$\sigma_{flow} = \sigma_{dis} + \sigma_{HP} + \sigma_{ss} \, , \tag{3.1}$$

Herein, the strengthening contribution from the dislocation is given by Taylor hardening relations,

$$\sigma_{dis} = \alpha M G_{sm} b \sqrt{\rho_i} \, , \tag{3.2}$$

Herein, α is a parameter that is dependent on the dislocation interactions, ρ_i is immobile dislocation density, M is the Taylor factor, b is magnitude of Burgers vector and G_{sm} is shear modulus. Furthermore, by incorporating the twin boundary concept [137], the grain boundary strengthening contribution is calculated using the modified Hall-Petch contribution and is represented as [34],

$$\sigma_{HP} = \frac{K_{HP} G_{sm}(T)}{\sqrt{g_{eff}} G_{RT}} = \frac{K_{HP} \; G_{sm}(T)}{\sqrt{g} G_{RT}} \left[1 + 2K_t ln\left(\frac{g}{g_0}\right)\right]^{\frac{1}{2}} . \tag{3.3}$$

Herein K_{HP} is the Hall-Petch strengthening coefficient, $G_{sm}(T)$ is temperature dependent G_{sm}, G_{RT} is room temperature G_{sm} and K_t is a constant, g is the grain size before annealing and g_0 is the grain size after annealing. The short range contribution provided by the solid solutions is estimated through the following expression [138],

$$\sigma_{ss} = \left[\sum_{i=0}^{n} Z_L G(T)^{\frac{3}{2}} \left(\alpha_s \delta_i + \acute{\eta}(T)\right)^2 c_i\right]^{\frac{2}{3}} \quad , \tag{3.4}$$

Herein Z_L is a material parameter, c_i is solute concentration α_s is a dimensionless constant and $\acute{\eta}(T)$ is the temperature-dependent misfit of G_{sm}. The major outputs of the model are discussed below,

- The contributions of dislocation strengthening and Hall-Petch strengthening dominates over others with respect to the overall σ_{flow}.
- The predictions of flow responses are not good and there is significant deviation in experimental and predicted flow curves if twins are not considered and the traditional Hall-Petch equation is used.

Chapter 04. The anatomy of the induced microstructure with ongoing creep plays a crucial role in the safe life estimation of components and for the new alloys development programs. In this chapter a creep model was developed and validated with the experimental data of RAFM steel. The novelty of the model lies with [47, 57]:

- Implementation of temperature and time dependent mobility model for subgrain boundary migration as,

$$M_{sb} = \underbrace{\frac{2\pi\eta D_s \Omega}{bk_B T}}_{\text{Core mobility}} + \underbrace{\frac{2\pi b D_{vp} \Omega}{h_b^2 k_B T}}_{\text{Lattice diffusion mobility}} \quad (4.1)$$

Herein, η is the transfer coefficient of dislocation core to jogs and h_b is boundary dislocation spacing, D_{vp} is pipe diffusion coefficient and D_s is the lattice self-diffusion coefficient.

- A more simplified method is presented to evaluate the climb velocity and associated equations are presented in form of variables N_1, N_2 and N_3 to obtain the climb stress and climb velocity.

Equation for the dislocation climb velocity can be given as,

$$v_{cl} = \frac{D_s \Omega \sigma_c}{bk_B T} \quad (4.2)$$

Combined equation for climb velocity and climb stress can be given as,

$$\sigma_{cl} + \frac{v_{cl}}{B_{vis}} = \frac{G_T b}{2\pi(1-\upsilon)} \frac{2}{d_{spon} + d_{dip}} \quad (4.3)$$

$$\sigma_{cl} - v_{\mathrm{cl}} * N_1 = 0, where\ N_1 = \frac{bk_B T}{D_s \Omega} \quad (4.4)$$

$$\sigma_{cl} + v_{cl} * N_2 = N_3, where\ N_2 = \frac{1}{B_{vis}}, N_3 = \frac{G_T b}{2\pi(1-\upsilon)} \frac{2}{d_{spon} + d_{dip}} \quad (4.5)$$

$$\sigma_{cl} = \frac{N_3 * N_1}{(N_2 + N_1)} \quad (4.6)$$

- Temperature-dependent shear modulus is incorporated.
- Evolution of additional variables that were not discussed in the literature, those are boundary dislocation spacing, subgrain boundary mobility, dipole capture spacing, climb stress, mean radius of MX (V and Ta based carbonitrides) precipitates, mean radius of $M_{23}C_6$ carbides is presented and discussed thoroughly.
- Evolution of damage due to two different types of precipitates is demonstrated separately.

The following observation can be noted from the investigated conditions:

- Mobile and dipole dislocation density decrease with ongoing creep due to glide and climb recovery. In the beginning of creep exposure, boundary dislocation density is found to be increasing and afterward decreases due to dominance of subgrain growth.
- Radius of subgrain is found to coarsen with ongoing creep. The both dislocation velocities were found to be increasing with creep.
- The computed internal stress was observed to be decreasing with creep. The effective stress on dislocations was observed to be increasing with creep.

Chapter 05. Existing models LSFE materials either do not provide information about the microstructural changes occurring during creep or employ too many adjustable parameters. Thus, a sound model that considers the influence of SFE on dislocation velocities, explains the evolution of the internal variable along with creep curves, and works with a single set of calibration parameters is a need. In this chapter a creep model was developed and validated with the experimental data of IN718. The new equations that were implemented in this work are,

- Glide velocity*:*

$$v_{gl} = a_1 M\Gamma \left(\frac{1}{br_p}\right) v_{p,i} L_p C_j D_s \, sinh\left(\frac{\sigma_{effe} b^2 L_p}{M k_B T}\right), \quad (5.1)$$

In Equation (5.1), $v_{p,i}$ is the phase fraction of the precipitates, a_1 is a prefactor for the gliding velocity, Γ is the stacking fault factor, X is the magnitude of the SFE, L_p is the mean spacing between all types of precipitates, σ_{effe} is effective stress and C_j is the jog density.

- Climb velocity

$$v_{cl} = \frac{\Omega D_{vp} \sigma_{cl}}{b K_B T} . \Gamma, \quad (5.2)$$

- Rate equation of dislocation densities incorporated slip system information, for example,

$$\dot{\rho}_m = \overbrace{\underbrace{\left(\frac{v_{gl}}{\lambda_{mean}}\rho_m\right)}_{production}}^{\rho_m^+} - \overbrace{\underbrace{\left(\frac{v_{gl}}{s}\rho_m\right)\left(1-\frac{1}{n_{slip}}\right)}_{immoblization} - \underbrace{d_{spon}\rho_m v_{gl}(\rho_m+\rho_i)\left(\frac{1}{n_{slip}}\right)}_{glide\ recovery}}^{\rho_m^-}, \quad (5.3)$$

In Equation (5.3) ρ_i is immobile dislocation density, d_{spon} is the spontaneous annihilation distance, n_{slip} is the number of active glide systems and $s = k_c/(\rho_{imob})^{0.5}$ is the dislocation substructure size, where k_c is the calibration parameter. Herein, we assumed that the total number of active glide systems is $n_{slip,}$ and an equal number of dislocations are available on each system. The normalisation will be $1/n_{slip}$, for any dislocation reaction. Furthermore, since the reaction partners are available on all glide systems, we replaced $1/n_{slip}$ with $(n_{slip}-1)/n_{slip}$ [113].

Experimental creep curves were modelled and validated up to the secondary stage. In addition to this, the model was shown to be capable of addressing the evolution of the internal variables such as ρ_m, ρ_i, λ_{mean}, v_{gl}, σ_{int}, σ_{effe} and σ_{cl} with creep time. To get more confidence about the model predictions, the boundary dislocation density estimated from the EBSD characterization was compared with the dislocation density predicted by the model. From the explored conditions following conclusions can be drawn,

- The model suggests that both the dislocation densities, internal stress and climb stress increase over the period of creep. Whereas the mean free path, glide velocity and effective stress have a decreasing trend.
- The proposed model is capable of addressing the effect of the stress and temperature on the primary to steady state creep regime using a single set of parameters.

Chapter 06. In this chapter a creep model was developed and validated with the experimental data of steel 316 LN.

This work presents some improvements compared to existing literature in the sense that,

- A more generalized mobility model is assimilated that considers temperature dependent maximum back stress.

$$M_{cg} = \frac{D_0 b}{k_B T}\, e^{\frac{\sigma_{eff} b^3}{k_B T}}\, e^{-\frac{Q}{RT}\left[1-\left(\frac{\sigma_{eff}}{\sigma_{max}}\right)^2\right]}. \quad (6.1)$$

where, σ_{max} = 1.5×σ_{UTS}, and Q being the activation energy for self-diffusion.

In order to make the equation (6.1) more physical, temperature dependent ultimate tensile strength is incorporated to find the maximum back stress as σ_{max} = 1.5×$\sigma_{UTS\,(T)}$

$$\sigma_{UTS\,(T)} = -1.1241\,T + 1411.8000 \quad (6.2)$$

- Effective stress was considered in place of applied stress for estimating dislocation glide velocity

$$v_{cl} = M_{cg} b \sigma_{effe}\,, \quad (6.3)$$

- The temperature dependent shear modulus is incorporated

Outcome of this work is delineated in brief as,

- Mobile and forest dislocation density increase and average dislocation glide distance decreases with ongoing creep. The Internal stress is found to be increasing whereas the effective stress decreasing with ongoing creep.
- The velocity and mobility of dislocations decrease with accumulated strain. They were observed between 2.37 ×10^{-12} - 1.45 ×10^{-11} m/s and 1.61 ×10^{-10} - 3.41 ×10^{-10} $mPa^{-1}s^{-1}$, respectively.
- The damage caused by the carbonitrides precipitate (MX-type) and carbides precipitate ($M_{23}C_6$-type) are increasing with ongoing creep. Cavitation damage rate was found to be increasing with creep time.

References section provides the list of relevant references (258) cited in Chapter 1-6 of the thesis.