# Temperature-dependence of plasticity and fracture in an Al-Cu-Li alloy

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#### ABSTRACT

The microstructure of an Al-Cu-Li alloy sheet is characterised in the solution treated, underaged, and peak aged tempers. Its mechanical response under uniaxial tension is measured at 90K, 173K, and 300K along  $0^{\circ}$ ,  $45^{\circ}$ , and  $90^{\circ}$  to the rolling direction. The anisotropic stress-strain curves are interpreted using a polycrystal plasticity model, which implements three hardening modes suggested by the microstructure, viz., matrix hardening, hardening due to isotropic precipitates, and hardening due to anisotropic precipitates. Phenomenological activation theory based analysis of the work hardening suggests that the physical mechanism underlying work hardening due to anisotropic precipitates remains constant over the temperature range studied, while the mechanism underlying matrix hardening varies strongly with temperature.

#### **KEYWORDS**

aluminium-lithium; precipitate hardening; flow stress; work hardening; phenomenological model

# 1. Introduction

The higher specific strength, higher stiffness, excellent corrosion resistance, good combination of strength and fracture toughness at ambient and cryogenic temperatures, combined with resistance to fatigue crack growth makes the third generation Al-Cu-Li alloys the preferred choice in aerospace structural applications. In these alloys,  $T_1$ (Al<sub>2</sub>CuLi) precipitates, which form on  $\{111\}_{Al}$  habit planes, are the major strengthening phase. Strengthening may also derive from isotropic precipitates, and from solute hardening of the matrix [1].

In aerospace applications, Al-Cu-Li alloys are subjected to a wide range of temperatures. In sheet form, the present Al-Cu-Li alloy finds application in stage III rocket cryogenic tanks. These tanks are to be used for storing liquid oxygen at 90K, and liquid hydrogen at 20K. The tanks must withstand proof testing to 40% above service pressure. Design codes also stipulate that they must plastically elongate at least 5% over the service temperature range under uniaxial tension applied in any in-plane direction [2, 3]. Satisfying these requirements with a limited testing program calls for understanding the temperature-dependence of the plasticity and fracture response of the material. Also, it may not be feasible to perform component-level tests at very low service temperatures, such as liquid hydrogen temperature. In this case, reliably bounding the mechanical response at service conditions using test data at higher temperatures requires conservative extrapolation schemes. Obtaining such schemes requires quantitative understanding of the variation of the plasticity and fracture characteristics with temperature.

Much work has focussed on studying the anisotropic yielding characteristics of Al-Cu-Li alloys [1, 3–9]. However, their flow response, particularly, anisotropic work hardening, is not as well-studied. Macroscopic plastic flow in these alloys is accommodated at the microscale by dislocation motion. This entails (i) the rearrangement of solute atom clusters in the matrix [10], and (ii) bypassing or shearing of the precipitates [3, 7, 11]. Nayan et al. [12] have developed a phenomenological hardening model, which accounts for the simultaneous action of three hardening modes: (i) matrix hardening, (ii) hardening due to isotropic precipitates, and (iii) hardening due to anisotropic precipitates such as  $T_1$ . The hardening in each mode is described by the empirical Voce [13, 14] law. This model could capture the experimentally measured anisotropic stress-strain curves at room temperature in three material tempers.

Nayan et al. [12] did not explore the temperature-dependence of the macroscopic flow properties of the Al-Cu-Li material. This aspect is the focus of the present work. Presently, the precipitate microstructure and texture of an Al-Cu-Li alloy in three temper conditions are characterised, and the mechanical stress-strain curves are measured at 90K, 173K, and 300K. The model of Nayan et al. [12] is employed to determine the grain level hardening parameters corresponding to the three hardening modes at each of the three temperatures. The variation of the modal hardening parameters with temperature is analysed using the phenomenological procedure due to Kocks et al. [15] and Kocks and Mecking [16]. It will be shown that the hardening modes differ in their variation with temperature, which suggests differences in the temperature dependence of their underlying mechanisms. The fracture surfaces are also analysed at the different temperatures. It is found that the fracture mode is highly correlated with slip planarity in the grains at over the entire temperature range studied.

## 2. Materials and experiments

Si	Fe	Cu	Mg	Mn	Zn	Ti
0.08	0.10	2.90 - 3.50	0.25 - 0.80	0.50	0.35	0.10
	$\operatorname{Zr}$	Ag	Li	Al		
	0.04 - 0.18	0.10 - 0.50	0.80 - 1.10	balance		

Table 1.: Chemical composition of the present AA 2198 alloy, in weight-%, according to the manufacturer's specification.

# 2.1. Material

The alloy used in the present study is a third generation Al-Cu-Li-Mg-Zr alloy (AA2198) processed by M/s Constellium<sup>TM</sup>. It is supplied in the form of 2 mm thick sheets in the

T8 temper (solution treatment, quenching, pre-deformation under tension, and peak aging) condition. The deformation prior to aging to impart T8 temper was done by stretching the sheets to 4%. Table 1 gives the design chemical composition limits of this alloy. The as-received temper condition, hereafter referred to as the peak-aged (PA) temper, was used as the starting material for subsequent heat treatments. Different heat treatments for achieving various temper conditions were carried out on three batches of samples. For the first batch, the PA samples were re-solutionised at 505°C for 45 minutes and water quenched to produce solution treated (ST) condition. Aging of the ST material at 155°C for 10 hrs resulted in underaged (UA) temper material, comprising the second batch. The as-received PA material itself comprises the third batch.

# 2.2. Microscopy

Specimens were prepared for metallographic examination by conventional polishing on a series of emery papers followed by disc polishing using alumina and one micron diamond paste. The freshly prepared specimens were etched with Keller's reagent. Submicroscopic characterization to determine the presence of the various precipitates in different temper condition was done by transmission electron microscopy (TEM). Thin foils for TEM were prepared using discs 3 mm in diameter which were spark cut from mechanically ground strips of 0.2 mm thickness. TEM was carried out on a FEI TECH-NAI 20 TEM operating at 200 kV.

## 2.3. Texture

Bulk texture measurements for the samples were carried out by the Schulz reflection method, using an X-ray texture goniometer with Co  $K_{\alpha}$  radiation (D8 Discover, Bruker Germany). Four incomplete pole figures ( $\alpha = 0^{\circ}$  to 85°) from the {111}, {200}, {220} and {311} peaks were measured at mid thickness section parallel to the rolling plane. Defocussing correction for the measured data was performed using a random aluminium powder sample. The three dimensional orientation distribution function (ODF) was calculated from the measured pole figures using the free Matlab toolbox M-Tex software [17].

# 2.4. Mechanical testing

Flat tensile samples of dimension  $110 \times 10 \times 2$  mm and gauge length of 25 mm were extracted from 2 mm thick sheet in various orientation from the rolling direction in different temper conditions for tensile testing at ambient temperature. Tensile testing was performed at room temperature, T = 300K, in an MTS servo-hydraulic test machine operating in stroke control mode as per ASTM: E8-M-08 standard with displacement rate of 2 mm/min ( $\dot{\epsilon} \approx 1.3 \times 10^{-3}$  s<sup>-1</sup>). The strain measurement was done by 25 mm gauge length extensioneter.

Tensile tests were also performed at temperatures of 90K and 173K, as per ASTM E1450-09 with displacement rates of 1 mm/min ( $\dot{\epsilon} \approx 0.6 \times 10^{-3} \text{ s}^{-1}$ ) on tensile specimens having dimensions  $72.5 \times 15 \times 2$  mm with a gauge length of 22.5 mm. Testing was performed using servo hydraulic Universal testing machine (UTM) with a cryostat attached. The cryostat is a vacuum insulated casing containing heat exchanger, copper radiation shield and a central sample space designed to accept the load string. Liquid nitrogen was used to cool the specimen to 173K and 90K. Rhodium-Iron resistance

temperature detector (RTD) was used to measure the temperature of the specimen. The temperature of the cryostat was maintained to an accuracy of  $\pm 1^{\circ}$ C with the help of regularization of tank pressure of the container and flow rate of cryogens. Both are dynamically controlled to maintain the required test temperature. At the two cryogenic temperatures, it was not possible to measure the strain using a gauge extension crosshead displacement readings were therefore used to infer strains.

## 3. Modelling

Rigid-plastic rate-independent polycrystal plasticity simulations [18] are used to model the mechanical response of the material. The measured initial texture is discretised into 1024 orientations. These define the initial orientations of the model grains. The model grains are assumed to accommodate plastic deformation using  $\{111\}\langle 110\rangle$  slip.

A three-mode hardening model, developed by Nayan et al. [12], is used to evolve the critical resolved shear stresses in the slip systems of each grain. The critical resolved shear stress,  $\tau_s$  of slip system s, is obtained as a weighted average of that of the modes:

$$\tau_s = \sum_{m=1}^3 f^{(m)} \tau_s^{(m)},\tag{1}$$

where,  $f^{(m)}$  denotes the volume fraction of the grain over which each mode is activated. The physical volumes over which each of the three modes activate may overlap, i.e.,  $\sum_{m=1}^{3} f^{(m)} \geq 1$ . The hardening of slip system s in mode  $(m), m \in \{1, 2, 3\}$  is assumed to obey the Voce law [19]:

$$\tau_s^{(m)}(\Gamma_s^{(m)}) = \tau_0^{(m)} + \tau_1^{(m)} \left\{ 1 - \exp\left(-\frac{\Gamma_s^{(m)}\theta_0^{(m)}}{\tau_1^{(m)}}\right) \right\},\tag{2}$$

where,  $\tau_0^{(m)}$ ,  $\tau_1^{(m)}$ , and  $\theta_0^{(m)}$  represent material parameters associated with mode (m).  $\Gamma_s^{(m)}$ , is the effective accumulated slip in slip system in slip system s, or mode m. The initial hardness of slip systems, and their interaction during slip, are represented through initiation, and evolution equations for  $\Gamma_s^{(m)}$ , as detailed in Ref. [12].

It follows from Eq. (2) that the hardening of a slip system,  $\Theta^{(m)}(\Gamma_s^{(m)})$ , is given by

$$\Theta^{(m)}(\Gamma_s^{(m)}) = \frac{d\tau_s^{(m)}}{d\Gamma_s^{(m)}} = \frac{\theta_0^{(m)}\tau_V^{(m)}}{\tau_1^{(m)}} \left(1 - \frac{\tau_s^{(m)}}{\tau_V^{(m)}}\right),\tag{3}$$

where the saturation stress, or Voce stress is:

$$\tau_{\rm V}^{(m)} = \tau_0^{(m)} + \tau_1^{(m)}.\tag{4}$$

The linear decrease of  $\Theta^{(m)}(\Gamma_s^{(m)})$  given by Eq. (3) obeys the scaling observed by Voce [13, 14].

## 4. Results and discussion

#### 4.1. Microstructure and texture

Figure 1 (a) shows the three-dimensional (3D) micrographs of 2 mm thick sheet in the as-received peak aged (PA) temper condition. A pan-cake like microstructure, typical of rolled sheets was observed. The grains are significantly flattened along the normal direction, and elongated in rolling direction. Figure 1 (b) shows the optical microstructure of the alloy in the ST, UA and PA temper conditions. The grains are relatively coarser in ST and UA samples as compared to PA, due to re-solutionizing of PA samples during the heat treatment given to impart ST and UA temper. The average grain size in PA samples is 250–300  $\mu$ m.

The phases present in the alloy in different temper conditions have been identified with the help of diffraction patterns in three different zone axes:  $[100]_{AI}$ ,  $[110]_{AI}$  and  $[112]_{AI}$ . Figure 2 (a) provides schematics of the diffraction pattern along various zone axes consisting of streaks and spots expected for most of the phases in the AA2198 alloy. Figures 2 (b), (c) and (d) show the bright field transmission electron micrographs for ST samples, along the  $\langle 100 \rangle_{AI}$ ,  $\langle 111 \rangle_{AI}$ , and  $\langle 112 \rangle_{AI}$  zone axes, respectively. The corresponding selected area electron diffraction pattern (SAEDP) is also shown in the inset. In the ST sample, the mottled microstructure as well as presence of streaks along the [100] orientation in SAEDP corresponds to GP/GPB zones [20].

Also, dislocation loops (shown by white double arrow) and helices (shown by black double arrow) in the ST samples (solution treated and unavoidably natural aged) suggest that not all the vacancies are entrapped by Li and Mg atoms in the solid solution. This in turn indicates that the 1 wt% of Li in this alloy is not sufficient to inhibit nucleation of G-P and G-P-B zones because of deficiency of free vacancies. Figures 2 (c), and (d) also show wavy and unevenly arranged dislocations. The faint streak along [100] orientation in SAEDP of Fig. 2 (c) also indicates solute clusters resembling G-P/G-P-B zones, as in Ma et al. [21]. The Al<sub>3</sub>Zr phase with Ll<sub>2</sub> crystal structure is present non-uniformly in all temper conditions as it forms during the solidification of the alloy. The superlattice spot corresponding to Al<sub>3</sub>Zr coincides with that of Al<sub>3</sub>Li ( $\delta'$ ). However, since the Li content of the present alloy is only 1wt%, the superlattice spot may be attributed predominantly to Al<sub>3</sub>Zr. This conclusion is consistent with that of Deschamps et al. [22] in the same alloy. The Al<sub>3</sub>Zr phases act as heterogeneous nucleation sites for the precipitation of other phases.

Figures 2 (e), (f), and (g) show the bright field transmission electron micrograph of underaged sample along the  $[100]_{Al}$ ,  $[110]_{Al}$  and  $[112]_{Al}$  zone axes, respectively. The corresponding SAEDP is also shown in the inset. Since UA condition was generated by aging of the ST sample at 155°C for 10 hours without any prior stretching, the nucleation of precipitates on the dislocations is observed rarely. In UA samples, it can be noticed that  $\theta'$  phase nucleates at Al<sub>3</sub>Zr dispersoids (a few are marked by white single arrows in the micrographs). The faint streaks in the SAEDP (shown by the dotted white arrow) patterns corresponding to G-P/G-P-B zones observed in the ST condition become more prominent in UA condition due to artificial aging. Also, the regions seen in the ST samples as mottled structure develop into a more discrete microstructure in UA samples. A uniform distribution of dark spots become more distinct. These dark spots in Fig. 2 (e) represent the end on G-P-B rods aligned along the  $\langle 100 \rangle_{Al}$  orientation. This is consistent with the observation of faint streaks along the  $\langle 100 \rangle_{Al}$  direction in the corresponding SAEDP (shown by dotted arrow).

The nucleation of some phases, particularly,  $\theta'$  and  $T_1$ , can be seen in the UA temper





(b)

Figure 1.: (a) Pseudo-3D grain micrographs (representative triplanar EBSD structures) of the AA2198-T8 2 mm-thick sheet. in TS, LS, and LT planes. (b) Optical microstructures of solution treated (ST), underaged (UA) and peakaged (PA) temper conditions in rolling direction.















Figure 2.: (a) Scheme of the spots and streaks expected from the main precipitate phases in the  $\langle 100 \rangle_{Al}$ ,  $\langle 110 \rangle_{Al}$ , and  $\langle 112 \rangle_{Al}$  zone axes of Al. Bright field (BF) transmission electron micrographs (TEM) along  $\langle 100 \rangle_{Al}$ ,  $\langle 110 \rangle_{Al}$ , and  $\langle 112 \rangle_{Al}$  zone axes, respectively, for samples in (b, c, d) solution treated (ST) condition; (e, f, g) under aged (UA) condition; and (h, i, j) peak aged (PA) condition. The respective selected area diffraction patterns are also shown in the insets.

condition. Some  $\theta'$  and  $T_1$  precipitates are indicated by thick white and black single arrows, respectively, in Fig. 2 (e), (f), and (g). Figure 2 (f) shows the dark field transmission electron micrograph taken along  $\langle 110 \rangle_{A1}$  zone axis. Two variants of  $T_1$ , consistent with the corresponding SAEDP observation, are marked in this micrograph. Figure 2 (d), a bright field TEM micrograph obtained for the UA sample along the  $\langle 112 \rangle_{A1}$  zone axis further identifies only one variant of  $T_1$ , which appears edge on. This is indicated by the black arrow in the bright field TEM image. Here again, the  $T_1$  precipitates with high aspect ratio are distributed homogeneously.

Figures 2 (h), (i), and (j) show the bright field transmission electron micrographs obtained in the  $\langle 100 \rangle_{Al}$ ,  $\langle 110 \rangle_{Al}$  and  $\langle 112 \rangle_{Al}$  zone axes with corresponding selected area electron diffraction pattern (SAEDP) of PA samples, respectively. Figure 2 (h) shows the widespread presence of T<sub>1</sub>, a few of which are marked by black single arrows, and G-P-B zones viewed end on as dark dots. Here, some of the dark dots would have grown into the S phase due to peak aging of the samples. The  $\theta'$  phase can also be noticed but with a smaller number density. The SAEDP shown in the inset presents the electron diffraction patterns indicating the presence of  $\beta'$  (Al<sub>3</sub>Zr) precipitates (marked by dotted arrow), T<sub>1</sub> precipitates (marked by black single arrow), and G-P-B/S rods (marked by dotted white arrow) as streaks along  $\langle 100 \rangle_{Al}$  direction. The T<sub>1</sub> precipitate has the {111} habit plane and therefore can be best viewed in  $\langle 110 \rangle_{Al}$  zone axis in PA samples.

The dominance of  $T_1$  precipitate is further confirmed by SAEDP along the  $\langle 110 \rangle_{AI}$ zone axis in Fig. 2 (i). It shows intense diffraction spots and streaks (marked by white and black single arrow) corresponding to different variants of T1. Figure 2 (j) shows the bright field TEM micrograph with its corresponding SAEDP obtained in the  $\langle 112 \rangle_{AI}$ orientation for the PA sample. In the SAEDP, the streaks along the  $\langle 111 \rangle_{AI}$  direction (dotted single white arrow) corresponds to T<sub>1</sub> plate-shaped precipitates, which are marked in the micrograph by single black arrows. Also, the T<sub>1</sub> precipitates can be identified by characteristic diffraction spots at  $1/3\langle 220 \rangle_{AI}$  and  $2/3\langle 220 \rangle_{AI}$  positions, shown by single white arrow. The G-P-B zones have converted to S phase during peak ageing, and can be seen in the bright field TEM micrographs (shown by long single white arrow) in Fig. 2 (j).

The orientation distribution functions (ODF) corresponding to the  $\varphi_2 = 0^\circ$ , 45° and 65° sections obtained from X-ray diffraction are shown in Fig. 3, for the three tempers. The positions of the ideal orientations are also shown in Fig. 3 (a). The texture in all three tempers has Goss {110}(001), copper {112}(112), and S {123}(634) components, with the Goss and S components being the most and least dominant, respectively.

TI values in the figure refer to the texture index, defined as:

$$\Gamma \mathbf{I} = \int_{g} f^{2}(g) \, dg,\tag{5}$$

where g denotes the continuous variable running over the crystallographic orientation space, and f(g) represents the orientation distribution function. The random texture corresponds to a TI value of unity, and a strong texture to a large TI value. The TI in all the temper conditions of the present sample is quite low, which indicates a weak textures. This is further corroborated by the maximum texture intensities, which are 20, 15, and 20 for the ST, UA, and PA samples. The weak texture, and resulting small plastic anisotropy is characteristic of third generation AlLi alloys [23].

# 4.2. Mechanical response

Strains in the 90K and 173K mechanical tests are inferred from the crosshead displacement, as it is not possible to mount extensometers in the low-temperature specimen. Strain measurement from crosshead displacements must be corrected for testing machine compliance. The measured stress-strain curve, the compliance corrected curve, the true stress-strain curve, and the smoothed true-stress vs. plastic strain curves for the PA specimen at 173K are shown in Fig. 4, for the case wherein the loading direction is aligned with the rolling direction.

Fig. 5 (a) shows a comparison of the engineering stress-strain curve obtained from crosshead displacements with that obtained from a mounted extensioneter at room temperature. As expected, the comparison is poor at small strains in the linear elastic regime. However, it is reasonably good in the plastic domain. Fig. 5 (b) compares the true stress vs. plastic strain deduced from Fig. 5 (a). Upto the ultimate tensile strength of the material, the two measurements deviate from each other by not more than 6 MPa, or about 1%. This demonstrates that the true stress-plastic strain response deduced by duly compliance correcting the crosshead-based measurement is accurate.

#### 4.2.1. Hardening parameters

The yield stress anisotropy is the variation in the yield stress with loading direction in the plane of the sheet. This variation is plotted for the three temper conditions, ST, UA, and PA in Figs. 6 (a), (b), and (c), respectively, at the three temperatures studied. It is seen that the yield stress increases with decreasing temperature, and with increasing precipitation, as expected. Also, in all the tempers, the yield stress anisotropy is small, compared to the anisotropy reported in previous studies on second generation Al-Li alloys [8, 24]. This is a general feature of third generation Al-Li alloys with limited Li content [23].

Figs. 7, 8, and 9 show the measured true stress vs. plastic strain curves at 90K, 173K, and 300K, respectively, in the ST, UA, and PA tempers. These figures also





Figure 3.: (a) Locations of ideal orientations in the orientation distribution function sections at  $\varphi_2 = 0^\circ$ , 45° and 65°. X-ray diffraction based measurements of the  $\varphi_2 = 0^\circ$ , 45° and 65° of the (b) ST, (c) UA, and (d) PA materials.



Figure 4.: Data processing applied to the raw data obtained for the PA material at 173K, to obtain the true stress vs. plastic strain plots. The intermediate steps are described in the text. The square dot represents the yield point, and the dotted line indicates the linear elastic loading curve offset 0.2%.

Temperature	angle	Mode 1		Mode 2			Mode 3			
		$ au_{0}^{(1)}$	$ au_{1}^{(1)}$	$ heta_0^{(1)}$	$ au_{0}^{(2)}$	$ au_{1}^{(2)}$	$\theta_0^{(2)}$	$ au_{0}^{(3)}$	$ au_{1}^{(3)}$	$ heta_0^{(3)}$
		(MPa)		(MPa)		(MPa)				
	0°	133	530	700	25	0	0	0	270	5800
90K	$45^{\circ}$	130	530	530	25	0	0	0	285	4000
	$90^{\circ}$	120	530	580	0	0	0	0	305	5300
	0°	103	180	660	0	0	0	0	255	5700
173K	$45^{\circ}$	91	180	630	10	0	0	0	270	4000
	$90^{\circ}$	95	170	650	10	0	0	0	240	5200
	0°	60	100	1300	55	0	0	0	180	4200
$300 \mathrm{K}$	$45^{\circ}$	65	105	1000	45	0	0	0	168	5200
	90°	70	100	890	50	0	0	0	190	3500

Table 2.: Voce hardening parameters (in MPa) for each of the three hardening modes at each of the three temperatures, 90K, 173K, and 300K.



Figure 5.: Comparison of (a) engineering stress-engineering strain, and (b) true-stress vs. plastic strain curves obtained from crosshead displacement, and extensioneter measurements at 300 K. The comparison shown corresponds to a PA specimen tensioned along the sheet rolling direction.



Figure 6.: Yield stress anisotropy. The abscissa corresponds to the angle between the uniaxial tensile direction, and the rolling direction of the sheet.



Figure 7.: Experimentally measured true stress vs. plastic strain curves (dots) at 90K compared with model predictions (lines) for the (a) ST, (b) UA, and (c) PA tempers. Model predictions attempt to fit the initial portion of the experimental measurements.



Figure 8.: Experimentally measured true stress vs. plastic strain curves (dots) at 173K compared with model predictions (lines) for the (a) ST, (b) UA, and (c) PA tempers. Model predictions attempt to fit the initial portion of the experimental measurements.



Figure 9.: Experimentally measured true stress vs. plastic strain curves (dots) at 300K compared with model predictions (lines) for the (a) ST, (b) UA, and (c) PA tempers. Model predictions attempt to fit the initial portion of the experimental measurements.

show the predicted stress-strain curves obtained from polycrystal plasticity simulations implementing the hardening model of Sec. 3. Prior to imposing uniaxial extension on the model PA temper polycrystal, pre-deformation, in the form of uniaxial stretching to 4% true strain along the rolling direction, is imposed upon the model polycrystal. The activated slip systems are identified, and the initial density of isotropic and anisotropic precipitates is determined, as detailed in Ref. [12]. The present material, AA2198, is identical to that studied in Nayan et al. [12]. Therefore, the parameters describing the precipitates are assigned the values given in Nayan et al. [12, Table 3]. Uniaxial tensile deformation along various loading directions is then imposed upon the polycrystal. Simulation of uniaxial tension of the UA temper proceeds similarly, except that predeformation stretching to 4% is skipped. This is because the effects of stretching are expected to be eliminated during re-solutionising. For the ST temper, both stretching to 4% and determination of the effective accumulated slip due to ageing are skipped, as precipitates are absent.

The hardening parameters for the three hardening modes are fit as described in Ref. [12]: first the mode 1, or matrix hardening parameters are fit to the ST stress-strain curves. Keeping the mode 1 parameters fixed, the mode 2, or isotropic precipitate hardening parameters are fit to the UA data. Keeping the parameters of both modes 1 and 2 fixed, the mode 3, or anisotropic precipitate hardening parameters are fit to match the experimental PA stress-strain curves. The hardening law, Eq. (2), cannot account for softening due to e.g., shearing of precipitates, which may occur at larger strains. Therefore, the fitting aims primarily to capture the experimental hardening at smaller plastic strains.

Separate parameter sets are fit to match the experimental stress-strain curves measured at loading angles 0°, 45°, and 90°, corresponding to each mode. The fits obtained are shown in Figs. 7, 8, and 9. The parameter values are listed in Table 2. It is seen in Table 2 that the model parameters,  $\tau_1^{(2)} = \theta_0^{(2)} = 0$ . This indicates that isotropic hardening contributes toward increasing the yield stress of the material, but not to its subsequent hardening.

The polycrystal plasticity model accounts for the crystallographic texture, and also for anisotropic T<sub>1</sub> precipitation in the {111} planes. Thereby, it accounts for the variation of the mechanical response with loading angle. In principle, the present model must therefore be able to capture the flow stress evolution for all loading angles, with a single set of model parameters,  $\tau_0^{(m)}$ ,  $\tau_1^{(m)}$ , and  $\theta_0^{(m)}$ , for fixed temperature and temper conditions. However, the model parameters vary with loading angle. This suggests that the simple hardening model of Sec. 3 does not fully capture the interactions between the various hardening mechanisms. For example, presently only the self hardening of slip systems is accounted for, while neglecting the latent hardening between slip systems. Also, any interaction between the obstacles to dislocation motion corresponding to different gardening model.

#### 4.2.2. Activation theory based analysis

Phenomenological theories have been proposed in the literature to explain the temperature and strain-rate dependence of the flow curves. We presently use the phenomenological theory, due to Kocks et al. [15], and Kocks and Mecking [16] developed for single phase fcc materials. This phenomenological theory attempts to compact the flow curves experimentally obtained over a range of temperature and strain-rates into two master



Figure 10.: Scaling of the Voce saturation stress,  $\tau_{\rm V}$  with temperature, T, both scaled by the temperature-dependent shear modulus  $\mu(T)$ . The dotted line represents the best fit straight line corresponding to the data points of mode 3.

curves. The first master curve describes the hardening as a linearly decreasing function of flow stress, following the empirical observation of Voce [13, 14]. The flow stress corresponding to zero hardening is termed the Voce stress, and denoted  $\tau_{\rm V}$ . A simple relation, Eq. (4), relates the Voce stress,  $\tau_{\rm V}^{(m)}$  corresponding to mode m, to the hardening parameters of that mode.

The second master curve seeks to relate the Voce stress non-dimensionalised by the temperature-dependent shear modulus,  $\mu(T)$ ,  $\tau_V/\mu(T)$  to the temperature Tand strain-rate,  $\dot{\epsilon}$ . The non-dimensional group involving these factors takes the form  $kT\log(\dot{\epsilon}_0/\dot{\epsilon})/(\mu b^3)$ . Here,  $k = 1.38 \times 10^{-23}$  JK<sup>-1</sup> denotes the Boltzmann factor,  $b = 2.86 \times 10^{-10}$  m denotes the magnitude of the Burgers vector of Al, and  $\dot{\epsilon}_0$  denotes a reference strain-rate, about  $10^7$  s<sup>-1</sup>. Kocks and Mecking [16] have proposed several simple algebraic forms for the second master curve, including a linear relation between  $\log(\tau_V/\mu(T))$  and  $kT\log(\dot{\epsilon}_0/\dot{\epsilon})/(\mu(T)b^3)$ .

The phenomenological scaling is usually applied only to single phase materials. The present material has multiple phases, which prevents its direct application. However, the analysis of Sec. 4.2.1 decomposes the hardening of the material into that of the three hardening modes. It is meaningful to apply the present analysis to each of the three hardening modes independently.

Fig. 10 shows the variation of non-dimensional saturation hardness,  $\tau_V^{(m)}/\mu$  for modes  $m \in \{1, 2, 3\}$  with non-dimensionalised temperature,  $kT \log(\dot{\epsilon}_0/\dot{\epsilon})/(\mu b^3)$ . The temperature dependence of  $\mu$  for the present alloy is assumed to be identical to that of a similar alloy, Weldalite, reported by Ruschau and Jata [25]. Mean values of the Voce stress, averaged over the three tests at loading angles 0°, 45°, and 90° for each mode at each temperature are plotted; their standard deviation is indicated as error bars in Fig. 10.

It is seen in Fig. 10 that  $\tau_{\rm V}^{(3)}$ , corresponding to anisotropic precipitate hardening) plots as a linear function of  $kT \log(\dot{\epsilon}_0/\dot{\epsilon})/(\mu b^3)$ . It satisfies the equation:

$$\frac{kT}{\mu b^3} \log\left(\frac{\dot{\epsilon_0}}{\dot{\epsilon}}\right) = \frac{\Delta F}{\mu b^3} \left[1 - \frac{\tau_{\rm V}/\mu}{\tau_{\rm V_0}/\mu_0}\right],\tag{6}$$

with  $\Delta F/(\mu b^3) \approx 0.26$ , and  $\tau_{V_0}/\mu_0 \approx 0.012$ . In the classification of Frost and Ashby [26, Table 2.1, p. 8], this puts the T<sub>1</sub> precipitates within the range of medium strength obstacles. Following Frost and Ashby [26], letting

$$\frac{\mu b}{\ell} = \frac{\tau_{\rm V_0}}{\mu_0},\tag{7}$$

and assuming an athermal shear modulus  $\mu_0 = 80$  GPa, results in a T<sub>1</sub> precipitate spacing of  $\ell \approx 24$  nm. This spacing is reasonable when compared with the microstructures shown in Fig. 2 (e), and (f).

Nie and Mulder [27], and Dorin et al. [11] have proposed a mechanism for the shearing of  $T_1$  precipitates, and experimentally tested it at room temperature. That Eq. (6) describes the temperature dependence of the hardness of this mode, implies that (i) the mechanism proposed prevails in the temperature range from 90K to 300K, and (ii) the shape of the obstacle offered by a  $T_1$  precipitate closely approximates a step function [15, Fig. 43-7, p. 142].

On the other hand, the Voce saturation stress of mode 1 (matrix hardening) has a rapidly increasing slope with decreasing temperature in Fig. 10. Attempts to fit this



Figure 11.: Fractographs of AA2198 ST samples tensile tested at (a–c) 173K and (d–f) 300K.

curve to the form

$$\frac{kT}{\mu b^3} \log\left(\frac{\dot{\epsilon_0}}{\dot{\epsilon}}\right) = \frac{\Delta F}{\mu b^3} \left[1 - \left(\frac{\tau_{\rm V}/\mu}{\tau_{\rm V_0}/\mu_0}\right)^p\right]^q,\tag{8}$$

for any  $0 , and <math>1 \le q \le 2$ , as suggested by Kocks et al. [15], prove unsuccessful. It appears essential to let  $p \to 0$ , which implies that the free enthalpy of activation, given by the right side of Eq. (8) has a very weak dependence on the stress,  $\tau_{\rm V}$ , and is almost wholly dependent on the temperature, T.

It is known that clusters comprised of solute elements, microalloying elements, and vacancies resist dislocation motion in the matrix [10], and that dislocation motion proceeds by rearrangement of these clusters. The present result suggests that the mechanism of rearrangement depends strongly on temperature, and only weakly on the stress level, at least between 90K, and 173K.

The saturation stress due to mode 2 (isotropic precipitate hardening) is very small compared to the other modes. It is also non-monotonic. Such a variation cannot be accounted for by Eq. (8). In other words, it is not possible to draw any conclusions about the temperature dependence of mode 2 hardening due to isotropic precipitates from the present data.

# 4.3. Fracture

Figure 11 shows the fracture surfaces of the ST tensile specimens with loading angles  $0^{\circ}$ ,  $45^{\circ}$ , and  $90^{\circ}$  at 173K, and 300K. At both temperatures, it is seen that the fracture mode is transgranular ductile. The dimples are oriented due to the slanted geometry of the fracture surface. Figure 12 shows the fracture surfaces of the PA tensile specimens with loading angles  $0^{\circ}$ ,  $45^{\circ}$ , and  $90^{\circ}$  at 173K, and 300K. At both temperatures, the fracture surface appears as multiple fine steps, with smooth walls between them. The fracture surface is fibrous with friction marks and involves failure at grain boundaries. These fractographs resemble those obtained in similar conditions by Chen [28] and Steglich et



Figure 12.: Fractographs of AA2198 PA samples tensile tested at (a–c) 173K and (d–f) 300K.



Figure 13.: Slip lines on the polished fracture faces of the AA2198 PA samples tensile tested at 300K. The tensile axes are inclined (a)  $\alpha = 0^{\circ}$ , 45°, and 90°, to the rolling direction.



Figure 14.: Slip planarity, as characterised by the number of grains with a pair of active coplanar slip systems at 90K, 173K, and 300K, for three inclinations of the tensile axis to the rolling direction. Note that slip planarity is nearly independent of temperature, but highly dependent on loading direction.

al. [29], and are characteristic of a fracture controlled by intergranular decohesion with a heavily elongated grain structure in the loading direction.

An interesting feature of Fig. 12 is the similarity of the 0° and 90° fractographs (Figs. 12a,c,d,f), and their dissimilarity with the 45° fractograph (Figs 12b,d) at both 90K, and 173K. While clear signs of brittle decohesion nucleated at the grain boundaries are visible in the former, the latter shows a quasi-ductile fracture mode, with fewer areas of brittle decohesion. To investigate this difference further, the fracture surfaces of the PA specimen have been polished. Figure 13 shows the slip steps that are revealed. It is seen that the slip steps in the 45° specimen (Fig. 13b) are more uniformly distributed than those in the 0°, and 90° specimen (Figs. 13a,c). This suggests that slip-uniformity is key to determining whether the fracture mode will be quasi-brittle, or quasi-ductile.

The fraction of grains in which a pair of coplanar slip systems is activated in the simulations was studied by Nayan et al. [12] at room temperature. The polycrystal plasticity calculation yields the slip rates in all the slip systems, in all the grains. Two to four slip systems are found to activate in most grains. Focussing attention at the instant of yielding (zero plastic strain), it is straightforward to determine if any pair of the activated slip systems is coplanar, by comparing the normals to the respective slip planes. The fraction of grains in which coplanar slip systems activate can then be readily determined.

The fraction of grains in which coplanar slip is activated is presently evaluated at 90K, 173K, and 300K, for the three directions of uniaxial testing. The results obtained for the PA material is shown in Fig. 14. It is seen that the fraction is sensitive to the loading direction, and has a minimum at  $45^{\circ}$ . At  $0^{\circ}$ , and  $90^{\circ}$ , the slip planarity is comparable. The fraction of the grains in which coplanar slip is activated is insensitive to temperature.

Comparing Fig. 14 with Figs. 12, and 13 suggests a correlation between the fracture mode, and slip-coplanarity. Large slip coplanarity appears to be correlated with quasi-brittle fracture, and small values with quasi-ductile fracture. Furthermore, the temperature insensitivity of slip coplanarity, observed in Fig. 14, agrees with the observed temperature insensitivity of the fracture mode also, as seen in Fig. 12.

## 5. Conclusion

Micrographs of the microstructure revealing the strengthening phases of a precipitate strengthened alloy, AA2198 in three temper conditions have been obtained. Mechanical data in uniaxial tension has also been obtained for the three temper conditions at three temperatures: 90K, 173K, and 300K. The data is interpreted using a polycrystal plasticity model that accounts for three hardening modes: matrix hardening, hardening due to isotropic precipitates, and hardening due to anisotropic precipitates. The decomposition of the overall hardening into three modes allows activation theory based phenomenological hardening analysis to be performed individually on the modes. The main conclusions are as follows:

- (1) G-P, and G-P-B zones,  $\beta'$  (Al<sub>3</sub>Zr) dispersoids,  $\theta'$ , and T<sub>1</sub> precipitates are observed in the microstructure of the AA2198 alloy.
- (2) It is possible to explain the mechanical flow response of the present material through the superposition of contributions from matrix hardening, isotropic precipitates, and anisotropic precipitates.

- (3) Isotropic precipitates contribute meagerly to the yield strength, and negligibly to hardening.
- (4) The microscopic mechanism underlying precipitate hardening due to anisotropic  $T_1$  precipitates prevails at low temperatures. The saturation stress due to anisotropic precipitates scales with temperature following activation theory.
- (5) The microscopic mechanism underlying matrix hardening is qualitatively different at 90K, and 300K. The corresponding saturation stress does not scale following activation theory.
- (6) The fracture mode of the specimen, quasi-ductile, or quasi-brittle, is correlated with slip planarity, and is insensitive to temperature.

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