

1 Forging and Heat Treatment Conditions That Produce Visible Grains in a 2 Nickel Alloy

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12 13 Abstract

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15 Experiments were undertaken to understand forging and heat treatment conditions that give rise to
16 large, visible grains in a new nickel alloy after super-solvus heat treatment. Such grains are
17 undesirable as they reduce strength and low cycle fatigue performance. The information that is
18 reported is required to design a forging practice to manufacture closed die forgings, intended for
19 disk rotors that are used in gas turbine engines. The alloy is a development composition, which
20 contains about 51 % gamma prime and has been produced by powder metallurgy. Compression
21 tests were conducted to specified upsets on right circular cylinder and double cone test pieces.
22 Segments of double cones were heat treated and examined to characterise grain size. The results of
23 these experiments are understood having reviewed starting microstructure, the results of process
24 simulations, and the results of electron backscattered diffraction on forged material.

25 26 Introduction

27 High bypass ratio turbofan aircraft engines and operating cycles are continuously evolving to provide
28 improved efficiencies for reduced fuel consumption and emissions [1, 2]. However, whilst
29 propulsive and aerodynamic optimizations of aircraft engines are possible, the increased demands
30 upon superalloys, which are used in the hot section parts, limit the thermal efficiency improvements
31 that can be achieved. The requirements for reduced engine core sizes and increased temperatures
32 and stresses pose a complex set of seemingly conflicting property requirements for the materials
33 considered for safety-critical disk rotor applications. Specifically, materials with higher strength
34 levels are needed to reduce the size and weight of components. Whilst this necessitates the
35 development of compositions with increased amounts of the gamma prime (γ') phase, further
36 optimization is possible by using a fine grain size. Yet such grain structures produce less appealing
37 time dependent crack growth behaviour [4], which may limit the design life of the component or the
38 interval between inspections. This is more relevant in today's engines as high climb rates are
39 increasingly required by commercial airlines to move aircraft more quickly to altitude to reduce fuel
40 burn [5]. Therefore, acceptable strength is required from coarse grain microstructures, which
41 demands effective precipitation strengthening from alloy design [6] and control of grain size in near
42 net shaped forgings.

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44 Inevitably, this is only possible using powder metallurgy to minimize elemental segregation to length
 45 scales of a micron (μm) or less for these complex, multi-component alloys with high levels of reactive
 46 elements (Al, Ti, Ta *etc.*) [7-9]. Subsequent hot deformation of consolidated powder compacts
 47 produces billet material with extremely fine grains, which enables superplastic flow of the work
 48 piece during isothermal forging at high temperatures and low strain rates, to make the desired near
 49 net disc shapes [7-9]. A uniform average grain size of 20-40 μm can then be created by super-solvus
 50 solution heat treatment. This microstructure produces an ideal balance in material properties
 51 between tensile strength and resistance to time dependent crack growth.

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53 Closed die forgings, from which disc rotors are produced, often show significant variations in forging
 54 strain due to changes in geometry, notably for drive arms. Factors that control grain size in these
 55 large complex forgings, after solution heat treatment above the γ' solvus temperature (T_{solvus}), have
 56 been discussed by the authors in an earlier publication [10]. These include, (i) the size of grains and
 57 γ' microstructure in the billet material or forging stock, (ii) forging temperatures and strain rates that
 58 promote super plastic forming, (iii) the effects of low forging strains and relevant strain rates for
 59 isothermal forgings, (iv) heating rates employed for solution heat treatment, (v) the role of grain
 60 boundary primary γ' precipitates and other pinning particles such as primary MC carbides. This
 61 understanding has guided the compression testing and heat treatment experiments that are
 62 reported in this paper.

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64 The earlier publication also discussed studies in which electron backscattered diffraction (EBSD) has
 65 been used to understand the mechanisms that give rise to visible grains. Bozzolo *et al.* [11, 12]
 66 observed that large visible grains have low values of stored energy and concluded that stored energy
 67 is the driving force for overcoming the Smith-Zener pinning pressure that is provided by pinning
 68 particles such as primary γ' . It was proposed that selective grain growth is due to the activation of a
 69 few nuclei (from low forging strains) that have sufficient stored energy to exceed a critical value,
 70 which appears to decrease with increasing heating time for super-solvus solution heat treatment.
 71 This suggests that the observed behaviour is a recrystallisation phenomenon that requires a critical
 72 strain or stored energy. Miller *et al.* [13] have also shown that the low grain growth front velocities,
 73 less than 1 μm per s, that have been cited for abnormal grain growth in other alloys, were not found
 74 in René 88DT. Much higher values were reported, suggesting that the critical process is not
 75 abnormal grain growth but abnormal recrystallization with a low density of nuclei.

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77 Table 1 Alloy composition [14].

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wt.%	Ni	Co	Cr	Fe	Mn	Mo	W	Al	Ti	Ta	Nb	Si	C	B	Zr	Hf
min	Bal.	14.6	11.5	0.8	0.2	2.0	3.3	2.9	2.6	3.6	1.2	0.1	0.02	0.01	0.05	0.000
max	Bal.	15.9	13.0	1.2	0.6	2.4	3.7	3.3	3.1	5.1	1.8	0.6	0.06	0.03	0.11	0.045

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81 Initial microstructure

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83 Compression tests were undertaken on a new alloy composition (Table 1), which has been specified
 84 in [14] and reported in [6]. Differential scanning calorimetry and metallographic examination of
 85 small samples, heated using rates of at least 10°C/min., indicate that the alloy T_{solvus} is about 1160 to

1165°C. Two batches of starting material have been considered in this study. In Table 2, these are referred to as Microstructure 1 and 2. Both batches were produced from argon gas atomised powder that had been screened to -270 mesh (53 µm), consolidated by hot isostatic pressure, and then subjected to hot deformation.

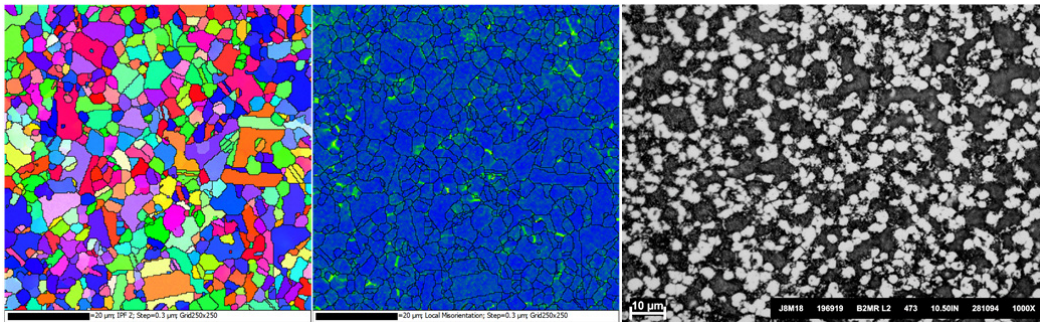
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Table 2 Microstructure and test piece details. The grain size ratings were established from optical microscopy, with samples etched using Kalling’s Reagent. RCC refers to right circular cylinder test pieces.

Microstructure	Grain size ASTM E112	RCC dims. (mm)	Soak time
1	90 % ASTM 15, 10 % 8.5 - 9, ALA 6.5 - 6	φ10 x 13.3 high	4 or 8 h
2	more than 99.9 % less than ASTM 12	φ12 x 16 high	15-30 mins

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Images of samples from Microstructure 2 are shown in Fig. 1. The inverse pole figure (IPF) image, from electron backscattered diffraction (EBSD), provides better resolution than those of etched samples from optical microscopy, from which the grain size ratings in Table 2 were derived. The step size used for EBSD was 0.3 µm and the area examined was 250 x 250 µm. Note that EBSD does not differentiate gamma (γ) grains from primary γ’ precipitates. There is a large volume fraction of primary γ’ precipitates in the starting material of the development alloy, about 32-37% in the optical microscope image in Fig. 1 (right). These are the white particles in the sample, which was electrolytically etched with 10% phosphoric acid (H₃PO₄) solution. When expressed as an equivalent circular diameter, primary γ’ precipitates are typically 1 – 2 µm in size. The larger γ grains in the IPF image, Fig. 1 (left), show twins from hot deformation and appear to be greater than ASTM 12 (5.6 µm). A Kernel Average Misorientation (KAM) image, calculated from EBSD data, is also provided in Fig. 1 (centre). This is the average misorientation between a point and its nearest neighbours and is a useful measure of retained strain, as shown by the bright green areas in the KAM image that are for rotations of 5°. These bright green locations are often at the interface between γ grains or the interface between γ grains and primary γ’ particles.



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Fig. 1 Images of Microstructure #2, from electron backscatter diffraction (EBSD), (left) inverse pole figure (IPF), (centre) kernal average misorientation (KAM), with green indicating 5° rotations. Optical micrograph (right) shows primary γ’ precipitates (white), electrolytically etched with 10 % H₃PO₄ solution. Circa 32-37 % primary γ’ precipitates.

120 **Experimental Work**

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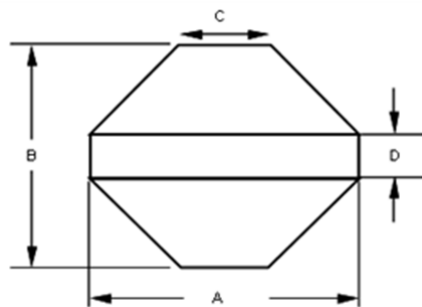
Initially isothermal compression tests were conducted on right circular cylinder (RCC) test pieces, for Microstructure 1, at Swansea University to understand the effect of forging conditions

124 (temperatures of 1050, 1075, 1100 and 1125°C and strain rates of 0.1, 0.01 and 0.001 strain per s)
125 on flow stress. They were conducted under constant strain rate control using a servo-hydraulic test
126 frame fitted with Mar-M002 flat die platens. A split box resistive elements furnace placed around
127 the platens provided a temperature capability of up to 1200°C where temperature control was
128 provided by two N-type thermocouples attached to the bottom platen. Temperature control was
129 specified to be within $\pm 3^\circ\text{C}$ of the target test temperature. All test pieces were given a single coat of
130 boron nitride prior to upset to minimise friction effects and inhibit the test piece from sticking to the
131 platens on compression. Test pieces were pre-soaked to either 4 or 8 hours, as indicated in Table 2.
132 Test pieces were pre-soaked using a bench-top resistive elements furnace for a period of 3.5 and 7.5
133 hours with the final 30 minutes being applied on the test frame prior to upset. Raw data was
134 provided in the form of platen position (in mm) versus load (in kN), from which the true stress and
135 true strain values were calculated. Friction and adiabatic heating effects were considered during
136 data analysis using software proprietary to Swansea University.

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138 Isothermal compression tests for Microstructure 2 were conducted on RCC test pieces (Table 2) at
139 the Illinois Institute of Technology at a constant strain rate of 0.2 strain per min. (about 0.003 strain
140 per s) using a servo-hydraulic test frame and loading bars fitted with Si_3N_4 anvils. Temperature
141 control was provided by K-type thermocouples, which were attached to test pieces. Boron nitride
142 coating was applied to both test pieces and anvils. Unlike the test pieces from Microstructure 1, test
143 pieces from Microstructure 2 were not soaked for 4 or 8 hours at the forging temperature prior to
144 compression testing. This was an error and not intentional. As such, differences in flow stress
145 behaviour between Microstructures 1 and 2 were expected due to differences in starting grain size
146 (Table 2) and secondary γ' % and size.

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148 To understand the effect of forging conditions on grain size, isothermal compression tests were
149 conducted on 2-inch (Microstructure 1) and 2.5-inch (Microstructure 2) double cone (DC) test pieces
150 (Fig. 2) at ATI Forged Products, Cudahy using a laboratory isothermal press. Test pieces were soaked
151 for 8 hours at the forging temperature prior to upset or loss of height of about 50%. Forging
152 temperatures of 1050, 1075, 1100 and 1125°C were examined for Microstructure 1, with a constant
153 strain rate of 0.2 per minute. The results of work on Microstructure 1 informed the subsequent
154 tests on Microstructure 2, which were undertaken at forging temperatures of 1030, 1045, 1060 and
155 1075°C. In addition to a strain rate of 0.2 strain per min., DC test pieces from Microstructure 2 were
156 also forged at 0.05 and 0.5 strain per min. at 1060°C. After forging, DC test pieces were cut into
157 quarters for solution heat treatment. To simulate large forgings, the DC quarters were subjected to
158 the following heating profile to a solution heat treatment (SHT) temperature of 1185°C: 900 to
159 1115°C at a rate of 10°C/min., 1115 to 1155°C at 4°C/min., 1155 to 1175°C at 1°C/min. and 1175 to
160 1185°C at 0.5°C/min. On reaching 1185°C, they were soaked for 1 hour.

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164 Fig. 2 Sketch of a double cone (DC) compression test piece. For 2-inch DC test pieces, A = 50.8
165 mm, B = 42.2 mm, C = 17 mm and D = 8.4 mm. For 2.5-inch DC test pieces, A = 63.5 mm, B
166 = 52.75 mm, C = 21.25 mm and D = 10.5 mm.

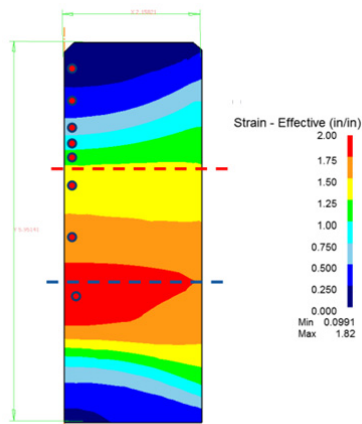
167 Grain size in the DC quarters was assessed from back scattered electron (BSE) images of polished
168 surfaces, either visually to confirm the presence of large visible grains or to determine mean grain
169 size using lineal intercept method, according to ASTM E112.

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171 To complement DC experiments, samples were cut from a 12 mm diameter bar, which was wire
172 electro-discharge machined from the centre section (55 mm in diameter x 151 mm high) of a large
173 forging, produced from Microstructure 2 (Fig. 3). A fraction of each sample was cut for heat
174 treatment trials. The aim was to examine the effect of heating rate on grain size. Four heating rates
175 were considered from 1000°C to the solution heat treatment temperature of 1185°C: 60°C/min.
176 (duration of about 3 mins.), 5°C/min. (duration of about 37 mins.), 2.1°C/min. (duration of about 88
177 mins.) and 1.3°C/min. (duration of about 142 mins.). Samples were soaked for 1 hour at 1185°C and
178 then air cooled. Grain size was rated according to ASTM E112 and E930 from optical microscope
179 images of surfaces etched with Kalling's Reagent. Polished surfaces were also assessed using EBSD.

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183 Fig. 3 Half section of a forging bore centre (55 mm in diameter x 151 mm) showing the location
184 of samples taken from a 12 mm diameter bar. The coloured contours indicate effective
185 strain values from a DEFORM forging simulation.

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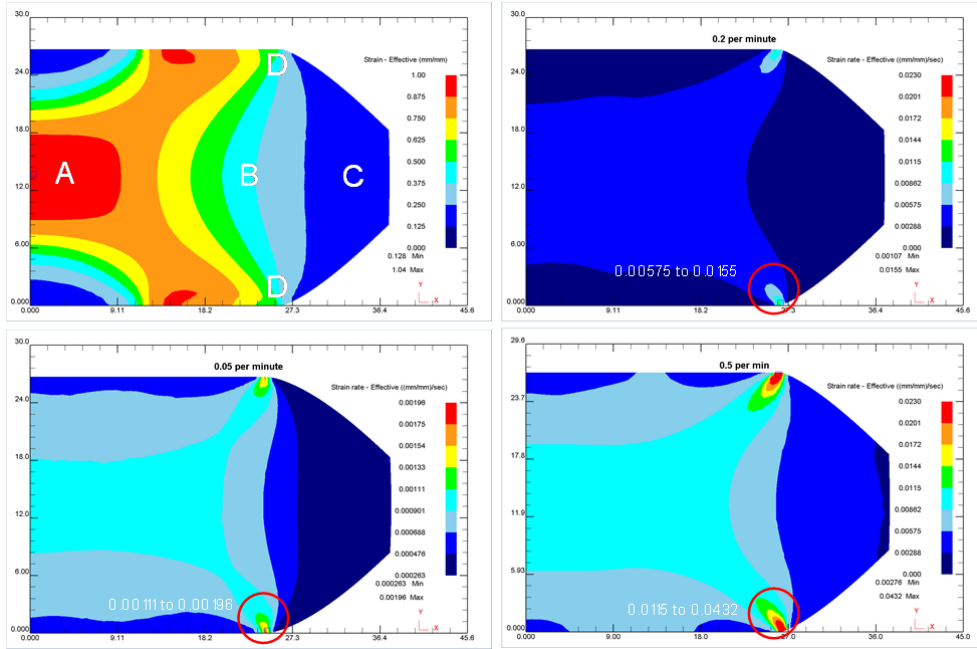
188 Process Models

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190 Forging simulations were undertaken using commercial finite element package DEFORM®. Half of
191 the model domain was created using axisymmetric elements with automatic remeshing and
192 increased size control at highly curved locations. An optimised Coulomb friction coefficient of 0.07
193 was used after correlating the final workpiece geometry with the deformed model geometry. The
194 simulation was stopped once a 50 % reduction in original height was achieved. Contour plots of
195 effective strain and maximum instantaneous strain rate (at the end of the forging step) were
196 extracted and are presented in this paper. The die or crosshead speed was set to reduce
197 logarithmically versus the die displacement to simulate as close as possible constant strain rate
198 conditions.

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203 Fig. 4 Results of DEFORM forging simulations on the 2-inch double cone test piece after 50%
204 reduction in height. Top left image shows predicted effective strain contours, noting
205 locations A, B, C and D. The other 3 images show predicted maximum instantaneous strain
206 contours values for a double cone forged at 0.2 per min. (top left), 0.05 per min.
207 (lower left) and 0.5 per min. (lower right).

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209 Results from Experimental Work

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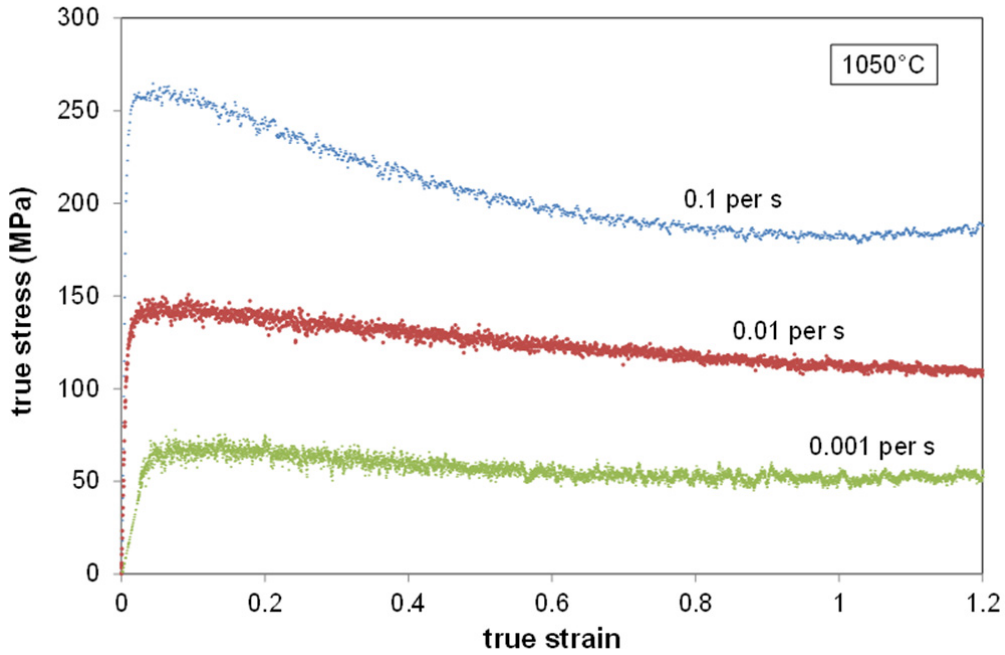
211 (i) Flow stress data

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213 An example of the true stress versus true strain data from compression tests on Microstructure 1 is
214 presented in Fig. 5. As expected, the lower strain rates of 0.01 and 0.001 per s produced lower true
215 stress values, which did not change significantly with increasing true strain unlike the true stress
216 values for 0.1 per s that diminished after a strain of 0.1, suggesting grain refinement through
217 dynamic recrystallisation (DRX). Figure 6 shows the influence of temperature on flow stress data for
218 Microstructure 2. The true stress, true strain data follow the expected behaviour, with less change
219 in true stress with increasing true strain for the higher forging temperatures. Forging at 1090°C is
220 associated with a low peak stress value, which changed very little with increasing strain.
221 Considerably higher flow stresses were recorded at forging temperatures of, or below, 1060°C; the
222 flow stress behaviour is similar to that shown in Fig. 5 for high strain rates, in which true stress
223 continues to diminish with increasing true strain, which is indicative of DRX.

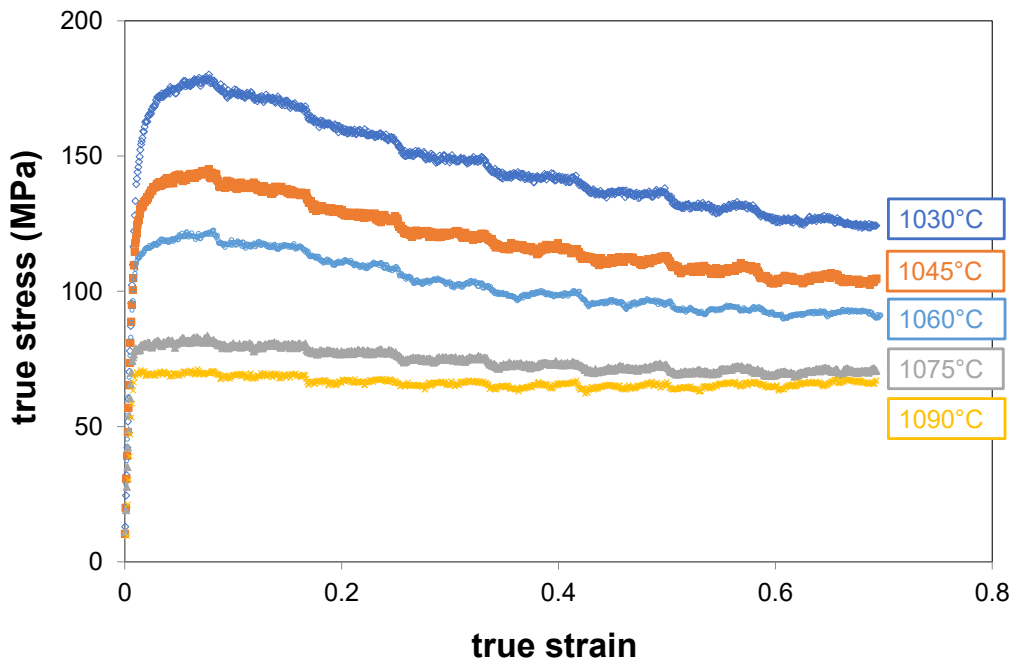
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225 Figure 7 correlates peak true stress data with strain rate for both Microstructure 1 and 2. For most
226 test conditions, there is little difference in peak true stresses for Microstructure 1 material that was
227 soaked for 4 or 8 hours; the variation in data for 1050°C/0.1 per s and 1100°C/0.001 per s is likely to
228 be test-to-test scatter. Whilst direct comparisons are not possible, except for 1075°C, peak stress
229 values for unsoaked Microstructure 2 are consistently higher than those for Microstructure 1 despite
230 the greater fraction of coarse grains (Table 2), which is expected to increase flow stress at these
231 forging temperatures.



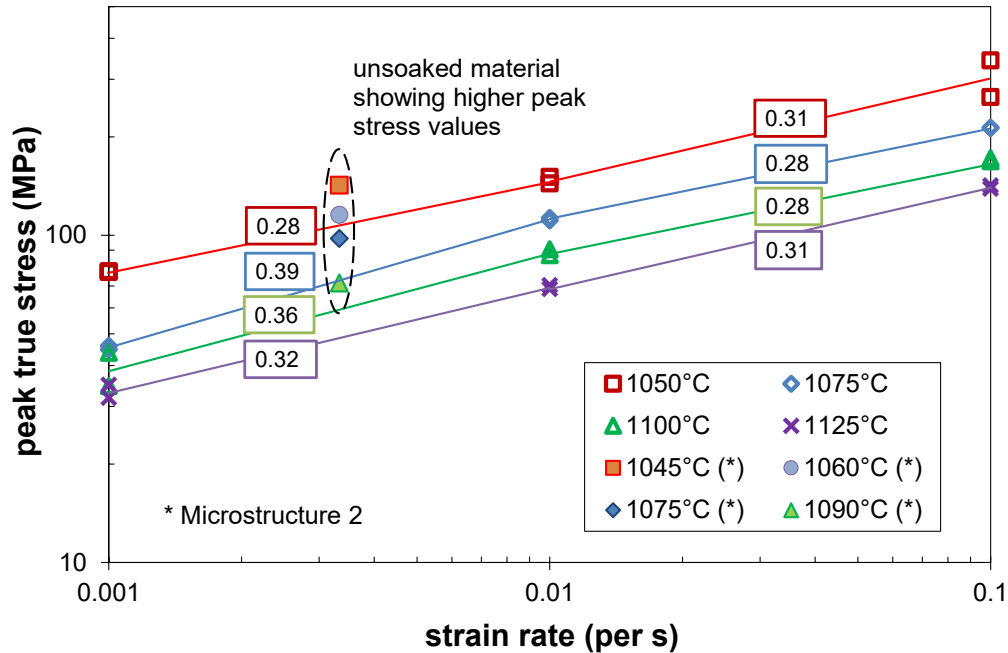
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Fig. 5 True stress, true strain data from compression tests on RCC test pieces for Microstructure 1 at 1050°C and strain rates of 0.1, 0.01 and 0.001 strain per s. Test pieces were soaked for 8 hours.



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Fig. 6 True stress, true strain data from compression tests on RCC test pieces for Microstructure 2 at a strain rate of 0.2 strain per min. and temperatures of 1030, 1045, 1060, 1075 and 1090°C. test pieces were soak for 15 minutes.



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247 Fig. 7 Peak true stress versus strain rate data for Microstructure 1 (1050, 1075, 1100 & 1125°C)
248 and Microstructure 2 (1045, 1060, 1075 & 1090°C) showing strain rate sensitivity index *m*
249 values (text in coloured boxes). Straight lines connect data from Microstructure 1 test
250 pieces, which were soaked for 4 and 8 hours.

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253 The strain rate sensitivity index *m* for describing the power law dependence of flow stress (σ) on
254 strain rate ($d\varepsilon/dt$) is from

$$\sigma = K \left(\frac{d\varepsilon}{dt} \right)^m \quad [1]$$

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257 where *K* is a material constant [15]. As the graph axes in Fig. 7 show the logarithm of peak true
258 stress and strain rate, values of *m* can be determined from the gradients of straight lines that are
259 fitted between data points. These are between 0.28 and 0.39, and are within 0.3 and 0.5, which
260 indicates superplastic deformation.

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261 Table 3 A summary of grain size in forged Microstructure 1 material after solution heat treatment
262 (SHT) at 1185°C for 1 hour. See Fig. 4 for locations A, B and C.

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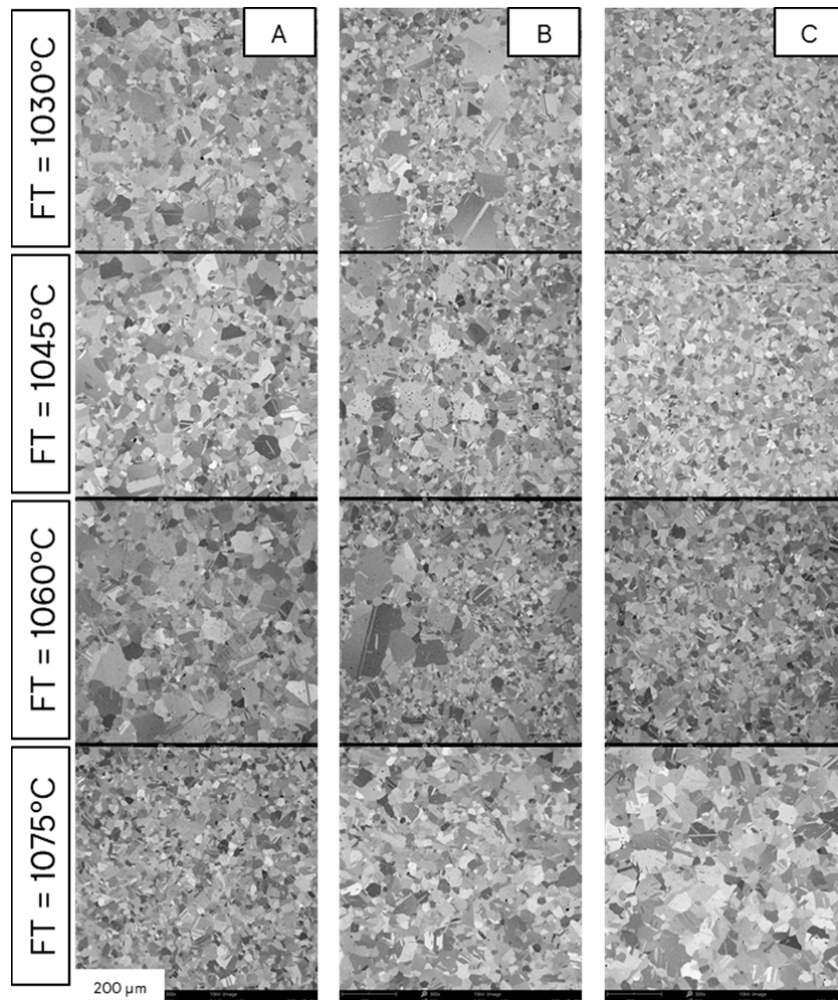
Forge T (°C)	Mean grain size (μm)			ALA grain size (μm)	Location
	A	B	C		
1125	69	53	74	444	B
1100	26	39	29	306	B/C
1075	26	24	33	107	C
1050	25	29	31	127	B/C

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265 (ii) Grain size

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267 The results of the solution heat treat experiments on forged Microstructure 1 material are
 268 summarised in Table 3 for locations A, B and C from quarters of double cone test pieces. It is evident
 269 that the ideal mean grain size of 20 – 40 μm grain size was achieved for forging temperatures of
 270 1050, 1075 and 1100 $^{\circ}\text{C}$ but not at 1125 $^{\circ}\text{C}$. Since the As Large As (ALA) grains were smaller in
 271 material forged at 1050 and 1075 $^{\circ}\text{C}$ than at 1100 $^{\circ}\text{C}$, the next series on experiments on
 272 Microstructure 2 considered forging temperatures below 1100 $^{\circ}\text{C}$. Visible grains occurred in
 273 locations between B and C (see Fig. 4, top left), although the largest ones were found in areas of
 274 effective strain between 0.1 and 0.17.
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279 Fig.8 Backscattered electron (BSE) images (X100) for forged Microstructure 2 material after
 280 solution heat treatment (SHT) at 1185 $^{\circ}\text{C}$ for 1 hour. The images show grain structures that
 281 were found at locations A, B and C (Fig. 4) in double cone test pieces forged at 1030, 1045,
 282 1060 and 1075 $^{\circ}\text{C}$ using a constant strain rate of 0.2 strain per min.
 283

284 BSE images for forged Microstructure 2 material after SHT at 1185 $^{\circ}\text{C}$ for 1 hour are presented in Fig.
 285 8. The grain structure is similar across the 4 forging temperatures and 3 locations in the DC test
 286 pieces. One difference is the slightly larger average grain size in the low strain location C for the DC
 287 forged at 1075 $^{\circ}\text{C}$ (Table 4). However, the grain structure does appear almost “duplex” at the other
 288 forging temperatures, with visible grains apparent, along side the smaller, average grains. These
 289 observations are consistent with the flow stress behaviour in Fig. 6, i.e. for the constant strain rate of

290 0.2 per min, a forging temperature of 1075°C shows little reduction in true stress with increasing
291 true strain whereas the lower temperatures do.

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293 Table 4 A summary of grain size in forged Microstructure 2 material after solution heat treatment
294 (SHT) at 1185°C for 1 hour. See Fig. 4 for locations A, B and C.
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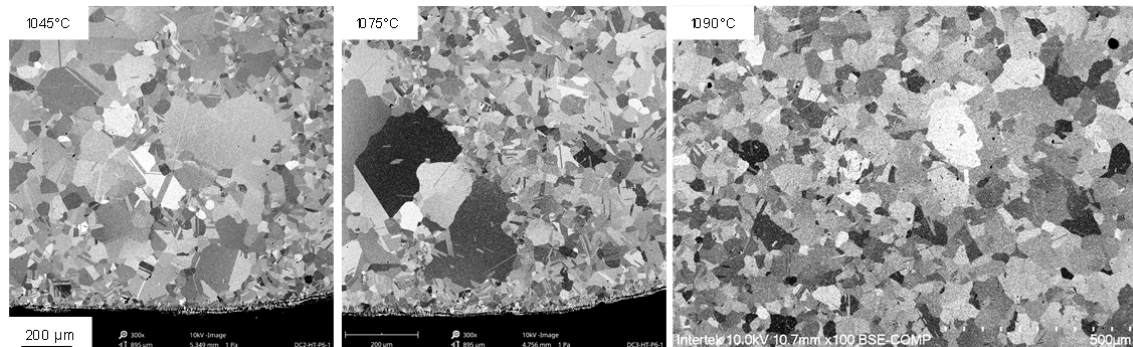
Forge T (°C)	Mean grain size (µm)		
	A	B	C
1030	29	30	18
1045	31	33	19
1060	28	30	20
1075	18	20	36

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297 At location D in DC test pieces, a small area is subject to a spike in maximum instantaneous strain
298 rate at the end of the forging process. For forging at a constant strain rate of 0.2 strain per min., the
299 predicted strain rate can be as high as 0.0155 (Fig. 4, top right). The higher strain rate produces a
300 localised area of visible grains for forging temperatures of 1030, 1045, 1060 and 1075°C. Examples
301 are provided in Fig. 9 for forging temperatures of 1045 and 1075°C. The location of visible grains
302 coincides with the small area of high strain rates. However, at a higher forging temperature of
303 1090°C, visible grains were not found at location D although an increase in average grain was evident
304 (Fig. 9, right).

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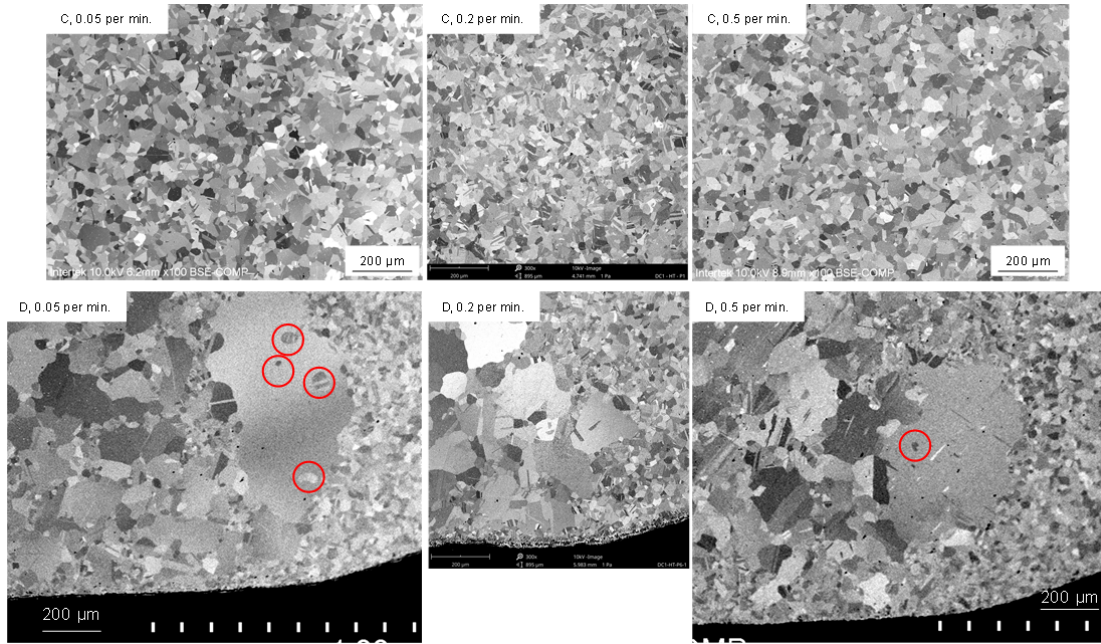
309 Fig. 9 Backscattered electron (BSE) images for forged Microstructure 2 material after solution
310 heat treatment (SHT) at 1185°C for 1 hour. The images show grain structures that were
311 found at location D (Fig. 4) in double cone (DC) test pieces forged at 1045°C (left), 1075°C
312 (centre) and 1090°C (right) using a constant strain rate of 0.2 strain per min.

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314 As stated earlier, DC test pieces were also forged at 0.05 and 0.5 strain per min. at 1060°C. Post SHT
315 grain structures for Microstructure 2 material, forged at these constant strain rates, are compared
316 with those from 0.2 strain per min in Fig. 10. Neither the average grain size in low strain, location C
317 (top row, Fig. 10) nor the propensity for visible grains in the high strain rate area, location D (lower
318 row, Fig. 10) appear to have changed significantly from adjusting the forging strain rate.

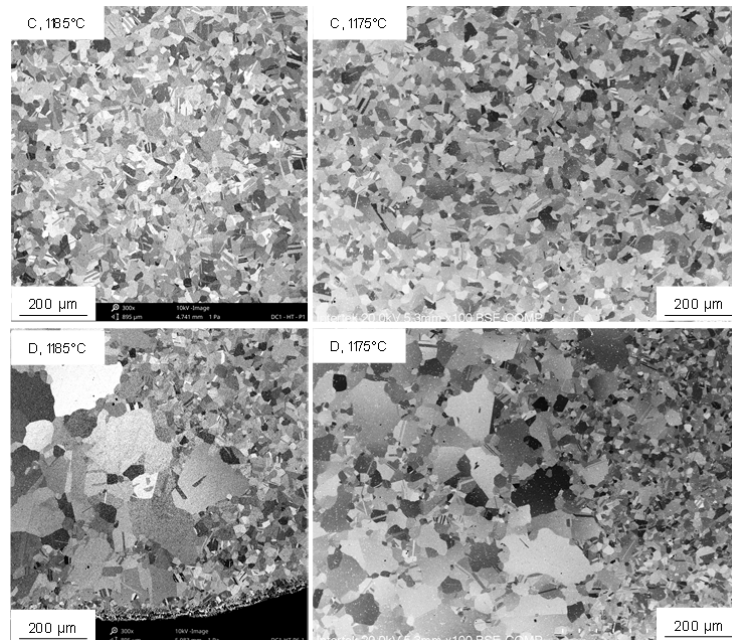
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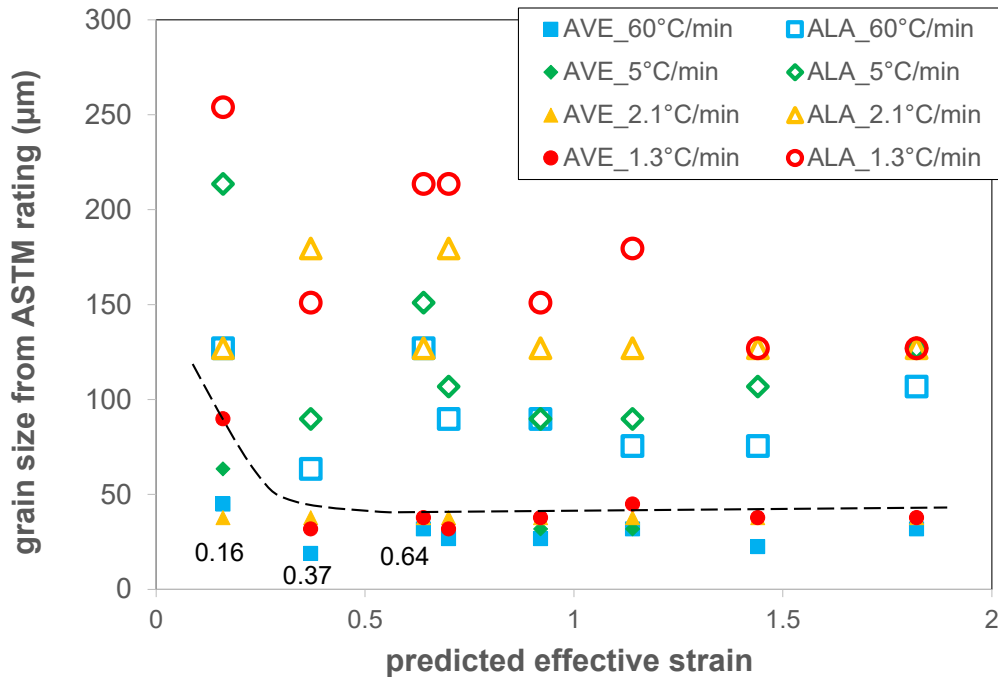
Fig. 10 Backscattered electron (BSE) images for forged Microstructure 2 material after solution heat treatment (SHT) at 1185°C for 1 hour. The images show grain structures that were found at location C (top row) and D (lower row) in double cone test pieces forged at 1060°C using a constant strain rate of 0.05 strain per min. (left), 0.2 strain per min. (centre) and 0.5 strain per min. (right). Red circles highlight isolated “normal” grains.



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Fig. 11 Backscattered electron (BSE) images for forged Microstructure 2 material after solution heat treatment (SHT) at 1185°C (left) and 1175°C (right) for 1 hour. The images show grain structures that were found at location C (top row) and D (lower row) in double cone test pieces forged at 1060°C using a constant strain rate of 0.2 strain per min.

336 Whilst forging conditions has thus far been examined in this study, the effect of SHT temperature
 337 should also be considered. The majority of forged DC test pieces have received a SHT of 1185°C for 1
 338 hour. Figure 11 compares the grain structure of Microstructure 2 material, forged at 1060°C and a
 339 constant strain rate of 0.2 strain per min., following SHT at 1185 and 1175°C. The average grain size
 340 in low strain, location C (top row, Fig. 11) does not appear to have changed significantly from
 341 reducing the SHT temperature by 10°C. However, the number density of visible grains in location D
 342 has increased marginally although the size of the largest visible grain has not changed significantly.
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 347 Fig. 12 Effect of forging strain and heating rate during SHT on grain size: AVE is average grain size
 348 from ASTM E112, ALA is As Large As grain size from ASTM E930. See Fig. 3 for location of
 349 samples, extracted from the centre section of a large forging (Microstructure 2). SHT was 1
 350 hour at 1185°C.
 351

352 Grain size data from samples, extracted from the centre section of a large forging (Fig. 3), after SHT
 353 are presented in Fig. 12. The SHT was 1 hour at 1185°C. The chart illustrates the effects of forging
 354 strain and heating rate during SHT on grain size. Whilst the data set does not show perfect
 355 correlations, with likely experimental variation in grain size masking trends, the general trend is for
 356 average and ALA grain size to increase with reducing forging strain and heating rates.
 357

358 Discussion

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 360 The error in not soaking Microstructure 2 RCC test pieces for compression testing has offered an
 361 opportunity to examine the effects of soak time at forging temperatures on flow stress behaviour. It
 362 is evident from Fig. 7 that the flow stresses for “unsoaked” Microstructure 2 material are higher than
 363 those from the initial “soaked” Microstructure 1, despite showing a greater fraction of smaller
 364 grains. Flow stresses at these forging temperatures tend to increase with increasing grain size.
 365 Whilst Fig. 7 also showed that extending the soak time from 4 to 8 hours in Microstructure 1
 366 material had little effect on the stress-strain behaviour, albeit with some test-to-test scatter, it is
 367 likely that secondary γ' precipitates in the starting material will coarsen and potentially dissolve

368 during extended times at temperatures of 1030 to 1090°C. Precipitation simulations, using the
369 mean-field approach described in [16], indicate that from an assumed heating rate of about 25-
370 30°C/min., secondary γ' precipitates will dissolve completely before 1090°C but will still be present
371 after 15 minutes at 1030 and 1060°C. Work has not yet been undertaken to detect the dissolution of
372 secondary γ' precipitates during heating and thermal exposure. However, researchers from NASA
373 [17] have reported the use of differential thermal analysis (DTA) to understand phase formation in
374 the Low Solvus High Refractory (LSHR) alloy. Using a heating rate of 5°C/min., they showed a distinct
375 spike in DTA heat flow data, with the peak around 1090°C, for the first heating cycle and attributed
376 this to dissolution of secondary γ' precipitates. The onset of the spike occurred about 1075°C. As
377 LSHR and the new alloy have similar compositions and volume fractions of γ' precipitates (51-53%),
378 the LSHR DTA data can be extended to the new alloy, at least for the purposes of the discussion.
379 Given that secondary γ' precipitates have a significant effect on flow stress behaviour, it is curious
380 that there is a change in the shape of flow stress curves about 1075°C, with lower forging
381 temperatures producing a reduction in true stress with increasing true strain and higher forging
382 temperatures producing no or little change in true stress with increasing true strain (Fig. 6). If these
383 precipitates do influence flow stress behaviour, along with starting grain size, do they also influence
384 DRX? This question will not be addressed further here but is extended to the academic community
385 for further investigation.

386

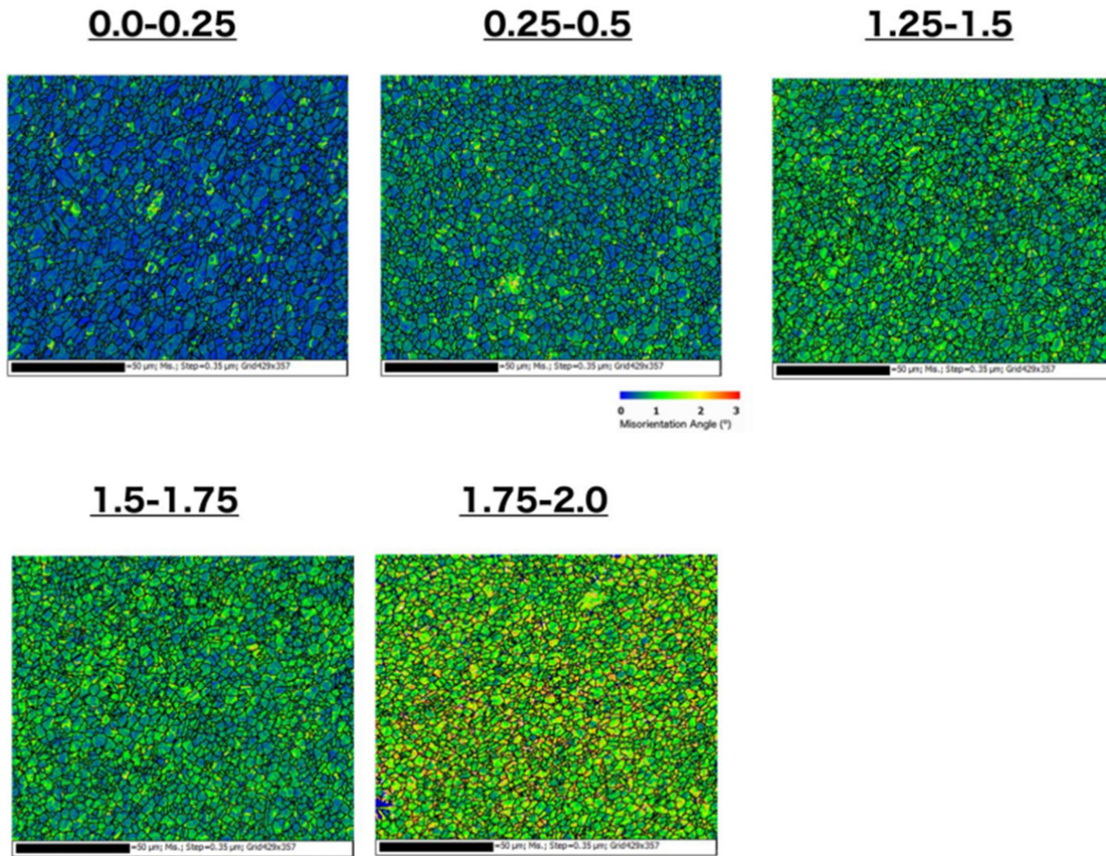
387 The compression tests on DC test pieces have identified potential forging conditions that could be
388 used to isothermally forge large complex shapes for disc rotors. The evidence in Figs. 8-12 suggests
389 that the ideal 20-40 μm grain size could be produced in most areas of forgings. However, particular
390 attention should be given to areas of low forging strain and areas that are subjected to high forging
391 strain rates as these have the potential to develop large visible grains. It should be noted that the
392 heating rates, SHT temperature and duration that have been used in this study are possible worst
393 cases that could exaggerate the occurrence and size of visible grains. As stated earlier, T_{solvus} for the
394 new alloy is no higher than 1160-1165°C but grain growth could begin at slightly lower
395 temperatures. As such, the SHT temperature could be reduced from 1185°C. This is prudent to
396 minimise both the extent of visible grains and the propensity for quench cracks [18].

397

398 The incidence of visible grains reported in this study from low forging strain areas can be rationalised
399 by the mechanism proposed by Bozzolo et al. [11, 12], *i.e.* that selective grain growth is due to the
400 activation of a few nuclei that have sufficient retained strain or stored energy to exceed a critical
401 value. Furthermore, this critical value decreases with increasing heating time for super-solvus
402 solution heat treatment. As discussed earlier, KAM data from EBSD is a useful means of
403 understanding levels of retained strain. Figure 13 shows KAM images from EBSD that was
404 undertaken on 5 samples from the forging centre section (Fig. 3). The sampled area was 429 x 357
405 μm and the step size was 0.35 μm . It is evident from Fig. 13 that increased forging strains produce
406 more homogeneous misorientations. The observed rotations of 1-3° in the 0 – 0.25 forging strain
407 sample are very localised and heterogeneous, suggesting that not all grains show the same level of
408 retained strain.

409

410 The proposed mechanism for selective grain growth can also explain the presence of visible grains at
411 very localised areas that are subject to high strain rates. In terms of the DC test pieces, the forging
412 strains in these small areas (Fig. 4 top left) may not be sufficiently high (0.25 – 0.5) to exceed a
413 critical stored energy level but will create more nuclei for recrystallisation than forging strains below
414 0.25 (as illustrated in Fig. 13). However, the small areas in location D that receive high strain rates
415 will show a higher number density of nuclei and a higher rate of recrystallisation than the
416 surrounding regions that receive lower strain rates, which may trigger the development of visible
417 grains.



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420 Fig. 13 Kernel Average Misorientation (KAM) images from EBSD of 5 samples in Fig. 3 in the as-
421 forged condition. The legend indicates rotations from local misorientation of up to 3°.
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Two further observations can be made from the grain structure images. The lower BSE images in Figure 10 show isolated “normal” grains within large visible grains. Examples of such occurrences are indicated by red circles. Secondly, the “normal” grains show annealing twins as expected, whereas the visible grains appear to be free of annealing twins [19]. Understanding these observations is the subject of future research.

430 Summary

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The results of experimental work and process simulations have been presented to understand forging and heat treatment conditions that produce visible grains in a new powder Ni superalloy that has been developed for disc rotor applications. These have identified potential forging conditions that could be used to isothermally forge large complex shapes for disc rotors. The evidence presented suggests that the ideal 20-40 μm grain size could be produced in most areas of forgings. However, particular attention should be given to areas of low forging strain and areas that are subjected to high forging strain rates as these have the potential to develop large visible grains. It should be noted that the heating rates, solution heat treatment temperature and duration that have been used in this study are possible worst cases that could exaggerate the occurrence and size of visible grains. The incidence of visible grains reported in this study from low forging strain areas can be rationalised by the mechanism proposed by Bozzolo et al., *i.e.* that selective grain growth is due to the activation of a few nuclei that have sufficient retained strain or stored energy to exceed a

444 critical value. Furthermore, this critical value decreases with increasing heating time for super-
445 solvus solution heat treatment.

446

447 It is recommended that caution should be exercised in using laboratory compression test pieces and
448 heat treatment trials to simulate conditions in full-scale forgings. Wherever possible, relevant
449 starting materials/microstructures, soak times and heating rates for industrial forgings should be
450 used for laboratory test pieces to ensure that the data and information that are generated are
451 representative.

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461

462 **Conflict of Interest Statement**

463 On behalf of all authors, the corresponding author states that there is no conflict of interest.

464

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