Influence of Strain Magnitude on Microstructure, Texture and Mechanical Properties of Alloy 825 during hot-forging

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Abstract

Alloy 825 is a nickel-base alloy used in applications with high stresses and corrosive environments. It is commonly hot forged, but there are few data about how this affects the microstructure, which is critical for both mechanical and corrosion performance. Here, Alloy 825 was hot forged in a commercial thermomechanical process to three industrially-relevant strains and the microstucture was examined using scanning electron microscopy and EBSD. Dynamic recrystallization was prevalent, so increasing the forging strain leads to smaller grains. Data were combined to allow each of dislocaiton density, recrystallized grain size and 0.2% proof stress to be calculated as a function of forging strain alone. The grain size or dislocaiton density are related by a powder law finciton with an exponent of ~ -1.5 and the proof stress can be related to either via a Hall-Petch relation. All forging strains were sufficient to meet the criteria of the relevant industrial standard for this material. The maximum yield strength and ultimate tensile strength were obtained after forging to a true strain of 0.9 were $413\,\mathrm{MPa}$ and $622\,\mathrm{MPa}$, respecitvely, with a ductlity of 40%. This may be used to tailor thermomechanical treatments to achieve precise mechanical properties.

Keywords: Alloy 825, Hot forging, Grain structure, Yield strength, Strengthening mechanisms

1. **Introduction**

Alloy 825 is a nickel-based alloy typically supplied in the wrought hot finished annealed bars, or cast into a final shape [1–3]. It is used in pickling tanks and vessels [4], oil and gas industries [5], agitators [6] and heat-exchanger systems [7]. The components in these applications are subjected to a complex combination of elevated temperatures, high stress, and hostile environmental conditions [8]. The high contents of nickel, chromium and molybdenum give good corrosion resistance and high strength. The casting structure is broken down by thermomechanical processing to obtain a uniform chemistry and microstructure. Thereafter, the material is typically subjected to an appropriate annealing process to develop the optimum combination of a good corrosion resistance and mechanical properties [9]. To ensure the mechanical properties and corrosion resistance are suitable for the application, particular attention must be paid to grain size and precipitate populations, as both grain boundaries and precipitates contribute to strengthening, but both grain boundaries the regions around precipitates may be sensitive to chemical attack. Previous work showed that a suitable heat treatment, called a *stabilization treatment* or *soft annealing*, will precipitate the maximum possible volume fraction of Ti(C, N) inside grains. This will provide strengthening while also avoiding the precipitation of $Cr_{23}C_6$-type carbides at grain boundaries, which would deplete regions near grain boundaries of chromium and lead to grain boundary sensitization [9]. In forged products of alloy 825, work hardening, recovery, and recrystallization are possible during hot-forging and stabilization [8,10–15]. It is well known that recrystallization generates fine grains, which is beneficial for both strength and toughness [16–18]. Differences in the grain size within the material are often observed due to inhomogeneous local strains [19]. This can lead to differences in mechanical properties due to variations in both grain size and dislocation density. Therefore, it is important to understand the evolution of strain within the material during the hot forging process. However, there is a lack of research around the behavior of alloy 825 during hot deformation. While some studies do exist, they only focus on dynamic recrystallization at very high reduction ratios (true strain, ε_t: $0.7 \leq \varepsilon_t \leq 2.5$) [15]. Industrial processes, in particular hot forging processes, often operate at lower strains, so the results of those studies may not be applicable. The current study addresses this deficiency by examining the effects of lower

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52 (industrially relevant) reduction ratios on both microstructure and mechanical properties. The findings

53 should be applicable to all thermomechanical processes at similar tempeatrues and strains.

54

55 This article will discuss the application of multiple characterization techniques to conduct a thorough

56 investigation of recrystallization in Alloy 825, which should be applicable to other Ni-Fe-Cr-Mo-Cu

57 alloys. Additionally, the evolution of crystallographic texture has been analyzed. An understanding of

58 the relationships between deformation conditions, thermomechanical history, and crystallographic

59 texture is essential for understanding the resulting properties of forged Ni-based superalloy bar.

60

61 The structural strengthening is commonly discussed in terms of Hall-Petch relationship [20]. However,

62 the strength of Alloy 825 and alloys subjected to large strain deformation is rather difficult to express

63 by a simple Hall-Petch equation due to the development of complicated hierarchical microstructure

64 including well developed dislocation substructures with large internal stresses. There are several

65 approaches to evaluate the strength after large strain deformation. Some of them consider the

66 subgrain size as the main strengthening contributor [16,17,19] . Others include the grain boundary and

67 dislocation strengthenings as independent and linearly additive contributors [15,20,21].

68

69 The primary objectives of the present work are to understand the microstructral evolution and the

70 dependency of microstructure changes on the deformation level during hot forging of Alloy 825.

71

72 2. **Materials and methods**

73 *2.1   Materials used and thermomechanical treatment*

74 All material in this study came from three billets of Alloy 825, which originated from the same cast

75 ingot (composition in Table 1). The ingots were cast after air melting in an electric arc furnace and

76 refinement using an argon oxygen decarburization process.

77

3

78 Table 1: Nominal composition for the tested material. All values are expressed in wt%. Combustion

79 analysis in accordance with ASTM E1018-11 was used for carbon and nitrogen and X-Ray

80 Fluorescense spectrometry was used for all other elements in accordance with ASTM E572-13.

	C	Si	Mn	Cr	Fe	Mo	Ti	Cu	N	Ni
Alloy 825	0.02	0.20	0.800	22.00	balance	3.000	0.700	1.800	0.018	41.5
Uncertainty	0.01	0.01	0.001	0.03		0.003	0.002	0.005	0.001	0.03

81

82 ### 2.1.1 *Initial microstructure*

83 The ingot was homogenized at 1200 °C for 6h followed by hot rolling at the same temperature with

84 80% thickness reduction, after which the material was allowed to air cool. The starting billets (after

85 hot rolling at 1200 °C) had an initial mean recrystallized grain size of 67 ± 3 μm, mesaured using

86 electron backscatter diffraction (EBSD) and the mean linear intercept method. One sample machined

87 from hot rolled billet was separately solution-annealed at 1200 °C for 60 minute in a resistance

88 furnace and then quickly water quenched to simulate the starting microstructure before hot forging.

89 ### 2.1.2 *Strain magnitude during hot forging process*

90 Following established practice, the billets were soaked at 1200 °C for 3 min mm^{-1} and then hot forged.

91 The forging process was performed on a hydraulic press with flat dies and at a strain rate of ~0.5 s^{-1}.

92 The hot-forging of all three billets was performed at temperatures maintained between $950\,℃$ and

93 $1180\,℃$ (Fig. 1). Samples were reheated during each forging process and the final forged bars were

94 quenched in water from between $950\,℃$ and $980\,℃$. Different samples were subjected to total

95 accumulative strains of 0.45, 0.65 or 0.9 with a pass strain of ~0.1 (i.e. 10% reduction per pass) to

96 study the structural changes during deformation (Table 2). The samples were rotated by 90° from

97 one pass to the next. The true strain was estimated by the formula $\varepsilon = \ln R_{\mathrm{R}}$, where R_{R} is the

98 reduction ratio (ratio of the starting cross-sectional area to the final cross-sectional area). Alloy

4

99   production and processing took place at Sandvik Materials Technology facilities in Sanvdiken,

100   Sweden. Material was sectioned for microscopy parallel to the forging (axial) direction from the centre

101   of the solid bar.

102

103   Table 2: Sample designations used in the current work.

Sample designation	Solution annealed	A	B	C
True strain, ε	0.00	0.45	0.65	0.90

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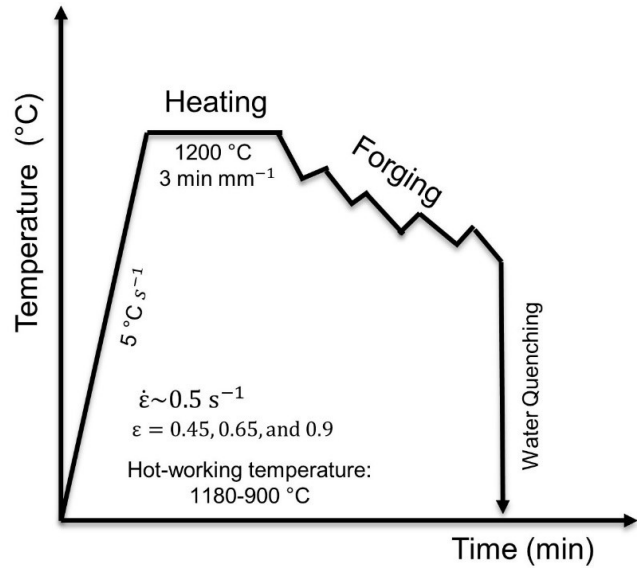
105
106   Fig. 1—Schematic diagram showing thermo-mechanical processing cycle. "min mm^{-1}" refers to the

107   heat treatment time per millimetre of rod radius.

5

2.2 Microstructure evolution

2.2.1 Electron backscatter diffraction (EBSD)

Electron backscatter diffraction (EBSD) was conducted using a Zeiss Sigma field emission gun scanning electron microscope (Carl Zeiss Microscopy GmbH, Oberkochen, Germany). The data were acquired and processed using the software TSL OIM Analysis 7 (AMETEK, Inc., Berwyn, PA, USA). The operating voltage was 20 kV. Energy dispersive X-ray spectroscopy (EDS) was also performed to analyse compositions. An orientation imaging microscopy (OIM) map and the misorientation angle of grains were calculated from the EBSD results. The OIM software was used for evaluation of the mean grain size (d) and kernel average misorientation (KAM). Samples for microstructural investigations were mounted in phenolic resin and prepared using standard grinding and polishing procedures. Specifically, the samples were jet polished at temperatures between 8 °C and 18 °C in 3 M sulfuric acid dissolved in ethanol (630 ml ethanol, 123 ml sulfuric acid). The electrolytic polishing voltage, current and time were 30-40 V, 1-2 A, and approximately 30 s, respectively. The areas of observation in this study were in the centre of each sample.

EBSD maps of a solution-annealed sample were obtained for areas 2.313 mm × 1.737 mm with a step size of 3 µm. The EBSD patterns with confidence index below 0.1 were omitted from analysis (such pixels are colored black in images). A total of four scans was used to evaluate the solution-annealed grain size, texture and twin boundaries fraction. The grain size was evaluated by a linear intercept along the forging direction, counting all boundaries with misorientation of $\theta \geq 10°$. To ensure statistically representative results, a minimum of 1500 grains were measured in annealed sample.

A step size of 0.5 µm for higher-resolution local scans was used to characterize the overall deformed microstructure and also subjected to a cleanup procedure, in which only pixels where a confidence index ≥ 0.1 were accepted. The grain size was evaluated by a linear intercept method on orientation imaging maps, counting all high-angle boundaries with misorientation of $\theta \geq 10°$, along the forging direction. The twin boundaries were omitted from the grain size calculations for the recrystallization

6

135   analysis, whereas the strengthening was analyzed using the grain size including the twin boundaries.

136   To ensure statistically representative results, a minimum of 3000 grains was measured in each

137   deformed sample.

138

139   ### 2.2.2   Identification of dynamically recrystallized grains and dislcoation density

140   There are several ways in which EBSD data may be processed. Previous literature has shown that

141   the most reliable technique to identify if a grain has undergone dynamic recrystallization without

142   further deformation is grain orientation spread (GOS) [22], which is the mean difference between the

143   crystal orientation at each pixel within a grain and the mean grain orientation. One mechanism by

144   which a point within a grain may not align with the mean orientation is the distortion caused by the

145   presence of dislocations. Grains that are recrystallized contain few dislocations and so the average

146   distortion will be lower than in a deformed grain that contains many dislocations. In literature, some

147   threshold is applied to classify a grain as either recrystallized or deformed, typically (GOS$\leq$ 1° [22],

148   GOS$\leq$ 2° [23,24], GOS$\leq$ 2.6° [25], GOS$\leq$ 3.0° [26,27], GOS$\leq$ 5° [28]). Grains were defined as each region

149   within which the local misorientation did not exceed 5˚, this is the so-called *grain tolerance angle* [21,29].

150   A minimum size of ten pixels was also set to define a grain. For each sample, at least three EBSD

151   scans with size step of 0.75 µm was acquired, covering an area of 2319 µm × 1737 µm (~4.03 mm^2),

152   578.5 µm × 434 µm (~0.25 mm^2), and 387 µm × 295.25 µm (~0.114 mm^2).

153

154   Dislocation density, ρ, itself is typically measured using a different statistic called the kernel average

155   misorientation (KAM), which is the average difference in orientation between a single point and a set

156   of points that form the boundary of a region used for analysis (the *kernel*). There is a well established

157   equation to relate dislocaiton density to the KAM statistic, known as Frank's rule, which depends on

158   the the kernel average misorientation angle, θ_{KAM}, the Burgers vector of the dislocation density, b,

159   the step size of the EBDS scan, s and a constant that depends on the scanning geometry, κ

160   (Equation 1) [30–32]. There is no such established relationship between GOS and dislocation density,

161   so that technique may not be used here [23,33] .

7

$$\rho = \kappa\theta_{\text{KAM}}(bs)^{-1} \qquad\qquad \text{Equation 1}$$

162

163   The KAM step size is used 0.75 µm, which satisfies the requirement that the KAM step size must be

164   smaller than subgrain size (in this case, approximately ~1 µm) in order to provide reliable results for

165   the dislocation density. In this work, the first neighbor was considered for calculating the KAM values.

166

167    $\kappa = 2$ represents pure tilt boundaries and $\kappa = 4$ represents pure twist boundaries [32]. Some studies

168   use $\kappa = 2/\sqrt{3}$, as this relates the EBSD step size to the (hexagonal) surface area that is closest to

169   each step location [30,31]. In this study, $\kappa = 2$ is used, as the pixels are in square shape not hexagonal,

170   and as the forging deformation under consideration leads overwhelmingly to the formation of tilt

171   boundaries [34–36]. The dislocation density may, therefore, be calculated from values that are either

172   known (κ, b, s) or may be measured (θ_{KAM}). The kernel average misorientation gives an overestimate

173   of dislocation density because of the presence of low-angle dislocation sub-boundaries that are grain

174   boundaries in practice, but are included in the dislocation density calculation [30,31].

175

176   ### 2.2.3   *Recrystallized grain size and twin boundaries*

177   Grain boundaries were identified from EBSD data as high-angle boundaries with misorientations, $\theta \geq$

178    $10°$ when observed on the plane at 90° to the forging axis. The mean grain size was measured by

179   applying the linear intercept method measured on an EBSD map. Boundaries identified as low-angle

180   ($\theta < 10°$) were attributed to sub-grain boundaries formed from regions of high dislocation density and

181   not considered grain boundaries. For each sample, at least three EBSD scans with size step of 0.75

182   µm was acquired, with each map covering an area 2319 µm x 1737 µm, and used to evaluate the

183   deformation texture and the number of twin boundaries. To ensure statistically representative results,

184   a minimum of 3500 grains was measured in each deformed sample. The microstructure and data

185   reported in this study is a representative microstructure or average of the values obtained from these

186   scans/maps.

187

188   The TSL OIM Analyzer software was also used to identify twin boundaries in order to be ignored

189   (excluded) from grain size calculations. Twin boundaries were defined when the misorientation angle,

190    $\theta_m = 60°$ and the local orientation lies within 5° of a $\langle 111 \rangle$ axis. For the grain boundary analysis,

191   boundaries with a misorientation between $2°$ and $10°$ were considered to be low-angle grain

192   boundaries. High-angle grain boundaries were further classified into Σ3 (twin boundaries) and other

193   high angle boundaries. Boundaries with a misorientation angle, θ_m: $10° < \theta_m < 60°$ are characterized

194   by near random distribution. The maximum deviation from the ideal orientation for Σ3 boundaries was

195   8.66° according to the Brandon criterion [37]. A fraction of Σ3 boundaries was calculated as a ratio of

196   the length of Σ3 boundary segments to the total length of all high-angle grain boundary segments. A

197   ratio of the length of Σ3 boundaries to the scan area was used to obtain a density of this boundary

198   type.

199   *2.2.4   Crystalographic texture*

200   The texture and misorientation analysis was performed on regions containing fully recrystallized

201   grains and separately on the overall microstructure, including grains that were not recrystallized. The

202   classification of recrystallized and non-recrystallized regions in current analysis was based on the

203   grain orientation spread (GOS) of individual grains.

204   *2.2.5   Estimation of stacking fault energy, γ_{SFE}*

205   In the current material, the stacking fault energy, γ_{SFE} is calculated as a function of composition

206   (Equation 2, where the symbol for each element represents the content of that element in wt%) [38,39].

$$\gamma_{SFE} = 1.59Ni - 1.34Mn + 0.06Mn^2 - 1.75Cr + 0.01Cr^2 + 15.21Mo - 5.59Si - 60.69(C + 1.2N)^{0.5} + 26.27(C + 1.2N)(Cr + Mn + Mo) + 0.61[Ni(Cr + Mn)]$$

        Equation 2

207   *2.3   Tensile specimens and testing*

208   Three tensile specimens were used for each hot forging condition. Longitudinal samples for

209   microstructural examination and tensile testing were extracted from a location at center and in

210   distance approximately 3 times the outer diameter of a bar (~250 mm) from a hot-forged end surface.

211 The tensile specimens were machined from the bars processed at different conditions and parallel to

212 the forging direction. Round bar specimens with 10 mm diameter and 50 mm gauge length were

213 used. The room temperature tensile tests were carried out at a strain rate of 0.001 s^{-1} on screw-driven

214 Instron 4488 electromechanical tensile test machine. The yield strength, σ_Y, ultimate tensile strength,

215 σ_{UTS}, and total elongation at failure, e_f, were determined from the output of the testing machine form

216 software provided by Inersjö Systems AB. To compare the results of orientation measurements

217 before and after tensile testing, all parameters used for the EBSD measurements were kept the same.

218 **2.4   *Hardness testing***

219 The average hardness was determined after testing a minimum of ten readings from each processing

220 condition. Hardness testing was performed with Vickers method with a 500 g load in accordance with

221 ASTM E384. The hardness measurements were carried out using an automated universal hardness

222 testing machine (QATM, Qness 30 A+, ATM Qness GmbH, Mammelzen, Germany).

223 **2.5   *Transmission electron microscopy***

224 Transmission electron microscopy was used to characterize the microstructure of the as-wrought

225 material (i.e. before initial heating). Imaging was performed using a Tecnai F20 scanning

226 transmission electron microscope (STEM) from Thermo-Fisher Scientific using a 200 kV accelerating

227 voltage with a high angle annular dark field detector. Selected area electron diffraction in the same

228 orientation was used for careful dark-field imaging to identify precipitates for compositional analysis

229 by energy-dispersive X-ray spectroscopy (STEM-EDS).

230 3. **Results**

231 ***3.1. Initial microstructure before hot forging***

232 Fig. 2 shows the initial solution-annealed microstructure with the mean initial grain size (d_0) 122 ± 11

233 μm, if twins are ignored for the purpose of measuring grain size. The material contians a small number

234 of large grains, between 180 μm and 500 μm in size, together with a large number of much smaller

235 grains, and exhibits annealing twins (Fig. 2a). Twin boundaries (Σ3, red) are common in the

236 microstructure and represent $52.7 \pm 2.2\%$ of all boundaries. Lower-coincidence boundaries (Σ9,

237 yellow) make up approximately $1.1 \pm 0.3\%$ of boundaries and the remainder of boundaries are high

10

238   angle grain boundaries (black). The grains appear to be equiaxed with a strong fibre texture of ⟨112⟩

239   along the forging direction (FD). Cubic precipitates could be observed in the body of several grains

240   (Fig. 3). Energy dispersive X-ray spectroscopy analysis suggested a composition of 79.3 ± 0.3

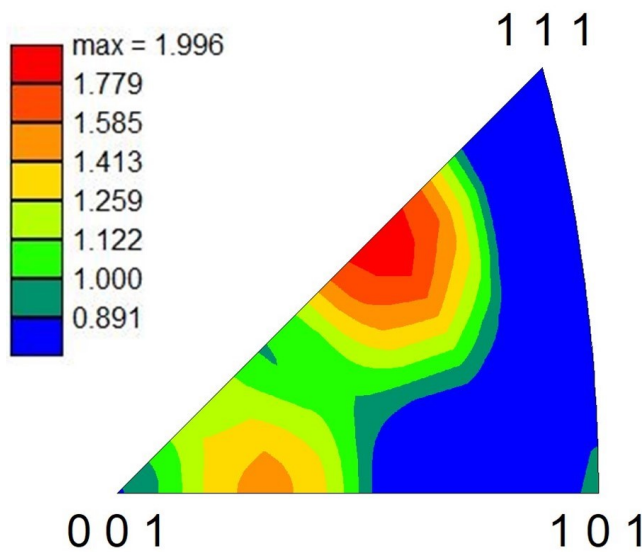241   wt% Ti, 20.0 ± 0.3 wt% N, and 0.7 ± 0.2 wt% Cr; carbon was not detected.

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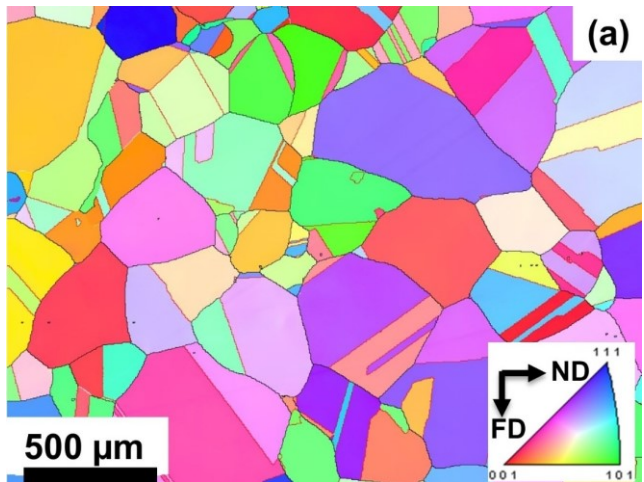
244   Fig. 2—(a) Inverse pole figure map parallel to forging direction (FD). Twin-type boundaries are

245   highlighted in red, lower-coincidence low-angle boundaries are presented as yellow and high angle

246   grain boundaries are black. (b) Pole figure showing the distribution of crystallographic poles oriented

247   parallel to the forging direction (FD) for Alloy 825 solution-annealed at 1200 °C for 3 min mm^{-1}, as
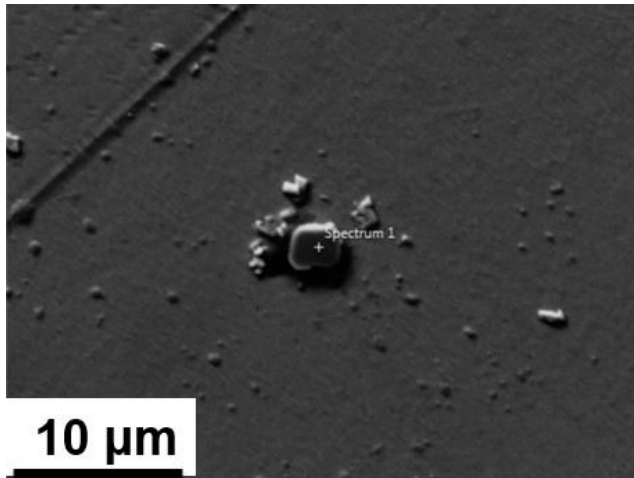
248   used in this study prior to hot-forging.


249

250   Fig. 3—Secondary electron SEM image of the morphology of a titanium nitride precipitate in the grain

251   interior.

3.2. Evolution of microstructure under strain

253   Inverse pole figures taken perpendicular to the forging direction (FD) from the highly deformed zones

254   of the as hot-forged samples show the microstructures after the three different levels of deformation

255   (Fig. 4). The iamges show a range of microstructural features, including recrystallized grains, grain

256   boundaries, twins, subgrain boundaries. It is apparent that the as-forged microstructure consists of

257   equiaxed recrystallized grains in a narrow range of sizes a high proportion of twin boundaries and

258   high angle grain boundaries, but is almost free of subgrain boundaries (Table 3, Fig. 4a). Samples B

259   and C (true strain levels of 0.65 and 0.9, respectively) also contain intergranular equiaxed grains.

260   After strain to a level of 0.65, the original equiaxed grains are almost identical to those in the starting

261   microstructure (Fig. 4b cf. Fig. 4a), with the exception that there are more subgrain boundaries

12

262   (subgrain size ~10 µm) (arrows in Fig. 4b). Following strain to a total level of 0.9, subgrain boundaries

263   (subgrain size ~2.5 µm), large non-recrystallized grains (arrows in Fig. 4c) and very fine recrystallized

264   grains can be observed at a strain level of 0.9. Overall, increasing the total deformation strain leads

265   to an decrease in prevalence of high-angle grain boundaries (Table 3). Increasing the strain level

266   from 0.45 to 0.90 has resulted in approximately half as many Σ3 twin boundaries. The number of

267   high-angle boundaries also decreases sharply, which implies that low-angle boundaries become

268   more prevalent.

269

270

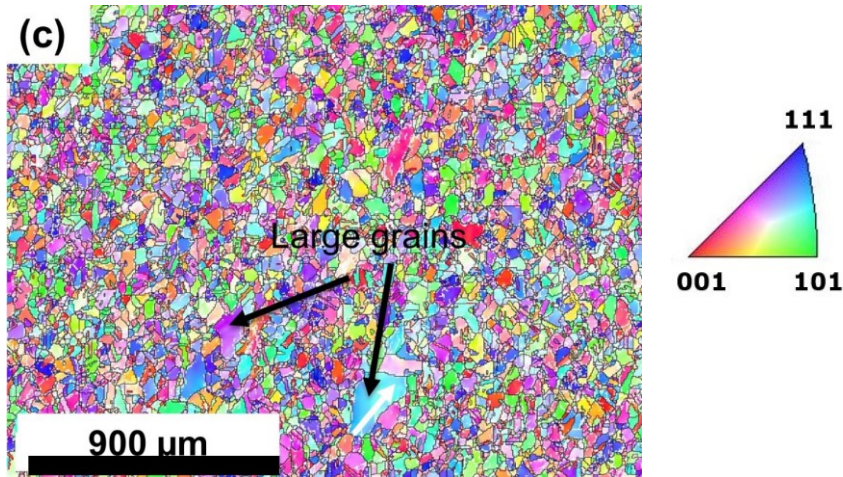271

13

(c)

111

001   101

Large grains

900 μm
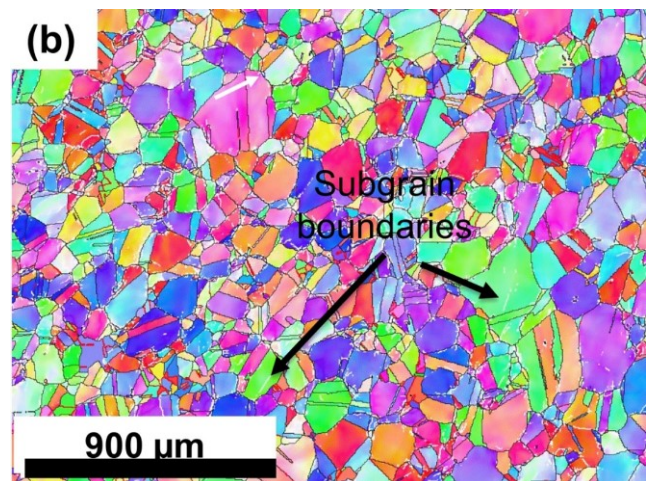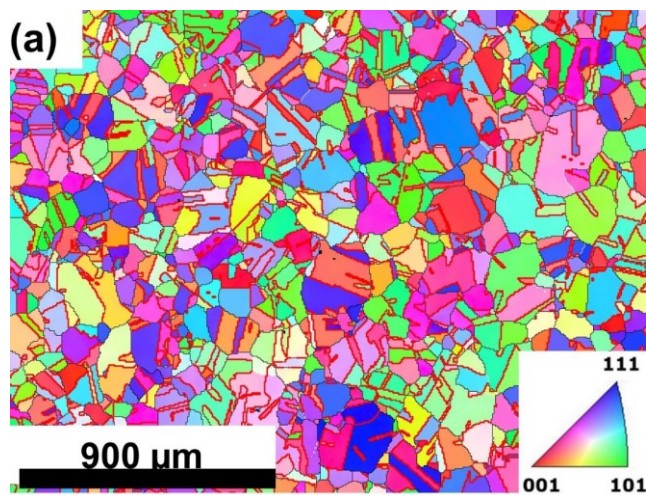
Fig. 4—Inverse pole figure maps measured using EBSD parallel to the forging axis of the as-forged microstructures deformed to deformation level of (a) $\varepsilon = 0.45$, (b) $\varepsilon = 0.65$, and (c) $\varepsilon = 0.9$. Each image is overlaid with high angle grain boundaries (black), low angle grain boundaries (white), and twins (red lines). The white arrows indictate directions for measurment of local misorientations presented in Fig. 6.

Table 3: The mean of recrystallized grain size (diameter) of sample A, B, and C.

	Sample A	Sample B	Sample C
Percentage of grain boundaries that are high angle grain boundaries (includes Σ3 twin boundaries)	99.0 ± 0.1	66 ± 16	61 ± 9
Percentage of grain boundaries that are Σ3 twin boundaries	57 ± 5	27 ± 4	26 ± 1
The grain size ratio (d/d_0)	0.33 ± 0.06	0.29 ± 0.06	0.18 ± 0.01

3.2.1   Grain refinement

The mean size of the grains of the equiaxed grains, d, are 40 ± 7 μm, 35 ± 7 μm, 22 ± 3 μm for samples deformed to a total strain of 0.45, 0.65, and 0.9, respectively. The large grains in samples are larger than 125 μm (Fig. 4c). All samples contained a plurality of grains that are ~25 μm and the

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283   grain size distribution of each sample shows a progressive decrease in frequency as grain size

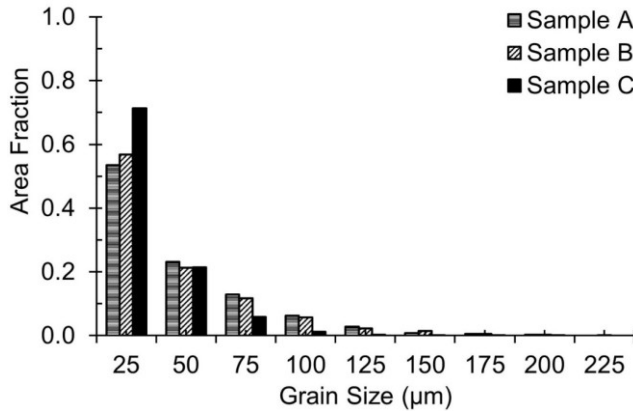284   increases (Fig. 5).

285

286   Fig. 5—Distributions of grain size in the as-forged Alloy 825 bar after various strain levels.

287   3.2.2   Misorientation within grains

288   Analysis of the crystallographic misorientation in the deformed material (using the data from Fig. 4),

289   shows that the large grains in sample B has an abrupt change in orientation of 60°, which is indicative

290   of a twin. The twin in question is straight-sided, implying that it is an annealing twin that has survived

291   the thermomechanical treatment. The same sample also exhibits a gradual increase in misorientation,

292   relative to the starting point, which implies the present of dislocations. However, in sample C, the

293   misorientation increases gradually to a similar level, but does not show any abrupt change and

294   accumulates across the grain (Fig. 6b). This implies a similar accumulation of dislocations but not

295   twins. Examination of the misorientation between adjacent measurements point (essentially the

296   magnitude of the differential of the total misorientation to the starting point), shows a near-constant
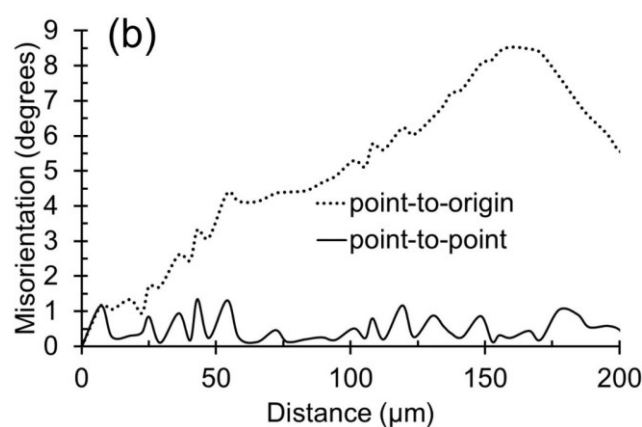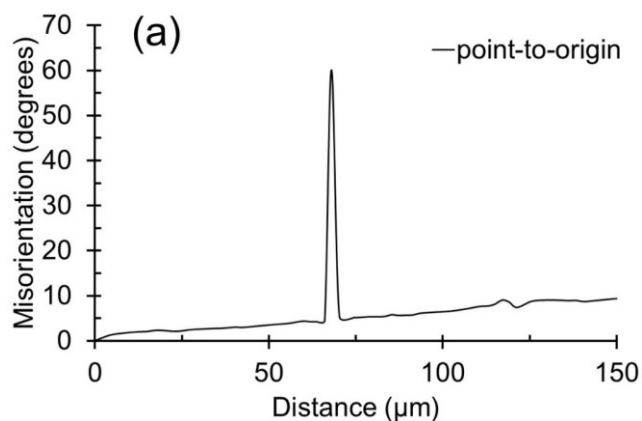
297   value.

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298

299

Fig. 6—Misorientations within the grains evolved during hot-forging for (a) sample B (b) sample C along the white arrows indicated in Fig. 4 b and c, respectively.

302

3.2.3   Dislocation density and recrystallization hot-deformed samples

Total area and grain orientation spread (GOS) was used to distinguish grains that had undergone recrystallization and no subsequent deformation from those that had either recrystallized and had undergone subsequent deformation, or not recrystallized at all (Fig. 7). For $GOS \leq 1°$, the solution-annealed sample and sample A (deformed to a total strain of 0.45) show only one prominent peak that begins at $0°$ and persists up to $0.6°$. However, the more severely deformed specimens B and C (deformed to a total strain of 0.65 and 0.9, respectively) exhibit grain area distribution with $GOS > 1°$.

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310   This implies that no grains in samples B and C show high densities of dislocaitons. It also shows that

311   while some grains have high dislocation densities in samples subjected to higher levels of strain,

312   others do not. It is unlikely that strain would be concentrated in a few grains due to deformation, but

313   the findings are explained by recrystallization. Based on the findings for the undeformed sample, it is

314   assumed that all GOS values below 1° imply that grains are non-deformed or recrystallized. This

315   value is consistent with published literature [22,40,41]. However, a threshold value of up to 3° has been

316   reported [27,40] Grains with a GOS > 1° were considered to be deformed – either they did not undergo

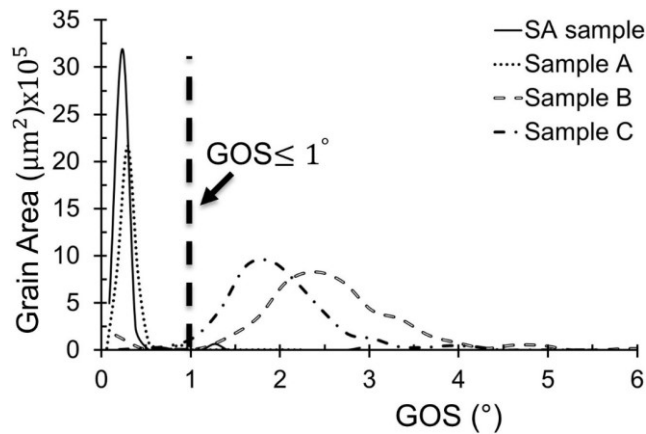317   recrystallization or they recrystallized and then underwent subsequent deformation.

318

319

320   Fig. 7—Grain orientation spread (GOS) plotted agianst the total area of the analysed microstructure

321   that had that GOS at various strain levels from 0.0 ("SA sample") to 0.9 (Sample C).

322   3.2.4   Influence of strain magnitude on grain boundary type

323   However, an increase in deformation level in forged samples B and C led to decreases Σ3 twin

324   boundaries (Table 3), containing a large number of sub-grains (low angle grain boundaries, LAGBs).

325   Results indicate that annealing twinning can occur in the present alloy during hot forging even at such

326   a high deformation level (Fig. 4, red lines) [42]. Twinned grains in these samples also contain large

327   internal distortions after hot forging.

17

328   Sample A shows a large prevalence (57%) of grain boundaries with a misorientation of $60°$ (Fig. 8a),

329   corresponding to annealing twins, as well as other high angle boundaries. Very few boundaries are

330   low-angle grain boundaries. An increase in the strain level leads to an increase in the fraction of low

331   angle boundaries: the grain boundary misorientation distribution for both samples exhibits two sharp

332   peaks corresponding to low-angle boundaries and twins (Fig. 8b and 8c). The misorientation

333   distribution outside these two peaks resembles a random distribution [43], albeit not as high, since

334   much of the distribution lies in the two peaks.
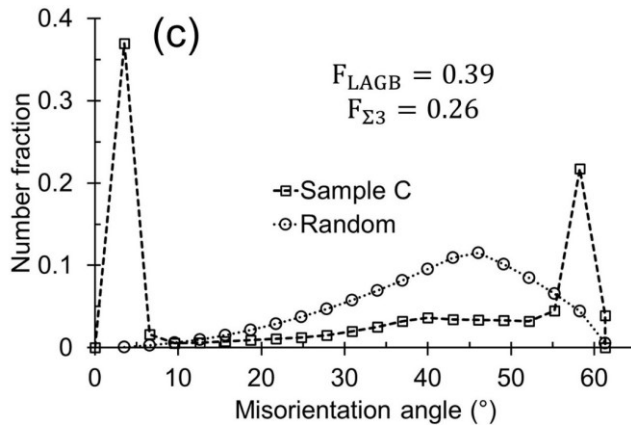
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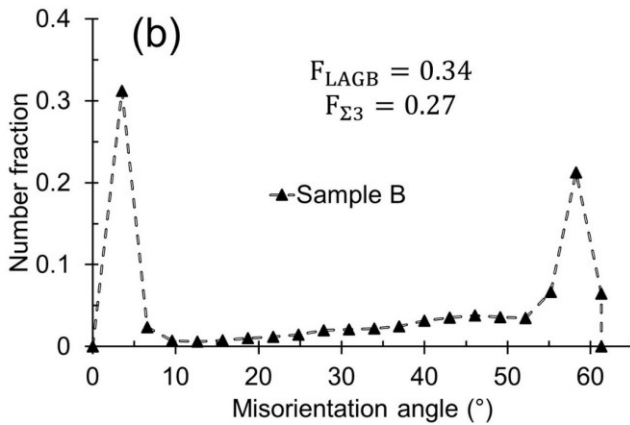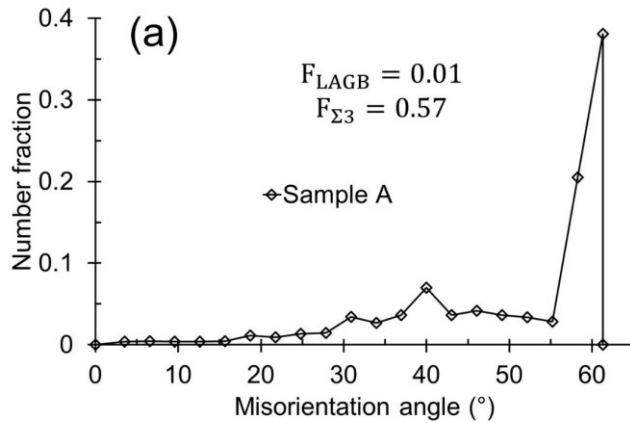
Fig. 8—Misorientation distributions for grain boundaries evolved in alloy 825 subjected to hot forging in (a) sample A, (b), sample B and (c) sample C. The distribution for a random misorientation has been calculated [43] for comparison to sample C.

3.2.5   Deformation Textures

Once grains were identified as recrystallized or deformed, they were analyzed for texture evolution for different strain levels during hot forging. The evolution of different texture components was carried out separately for both deformed and recrystallized regions, and overall microstructure. Whereas the undeformed sample showed no strong texture (Fig. 2), crystal orientation maps of hot-forged samples perpendicular to the forging direction (FD) show that at low strain, where recrystallization was not as prevalent, showed an extremely strong intensity for both $\langle 111 \rangle$ and $\langle 102 \rangle$ orientations, with both being about five times as strong as would be the case for a random texture (Fig. 9). The highest intensity parallel to $\langle 111 \rangle$ is consistent with stable deformation in a face-centered cubic metals. Within recrystallized grains, there is a seemingly random texture, in which the maximum intensity of any one orientation is not more than double that of a purely random texture. This is consistent with a lack of deformation in those grains.
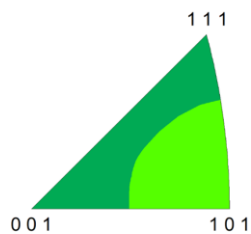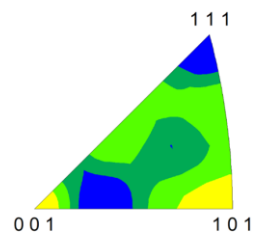
19

Sample A   Sample B   Sample C

[100]   [100]   [100]
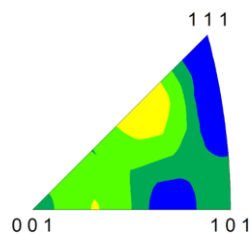
Max.=2.056   Max.= 2.096   Max.=1.096

Texture of the overall microstructure

[100]   [100]   [100]
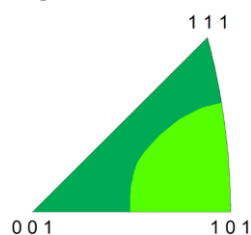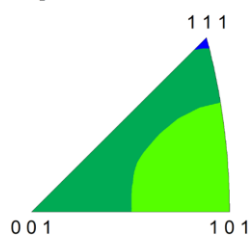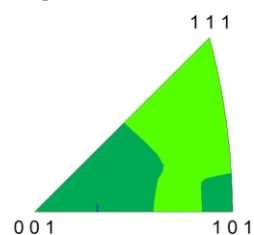
Max.=1.263   Max.=1.264   Max.=1.323

Texture of recrystallized grains

[100]   [100]   [100]
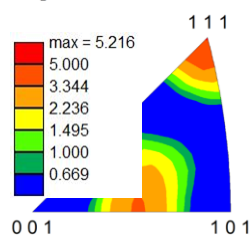
Max.=5.216   Max.=1.316   Max.=1.091

Texture of non-recrystallized grains

355 Fig. 9—Pole figures of the hot-deformed samples A, B and C. The color map used to show the pole

356 intensities is shown in the inverse pole figures and is used for all subfigures. The maximum intensity

20

357   in the pole figure is given below each figure. The direction indicated is perpendicular to the plane of

358   the pole figure.

359

360   ***3.3. Room-Temperature tensile Properties***

361   An increase in strain level leads to significant strengthening of Alloy 825 (Fig. 10). The effect of strain
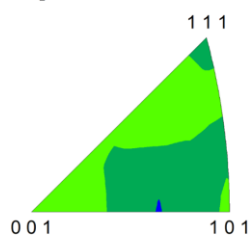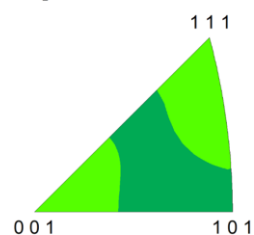
362   level on the 0.2% proof strength, $\sigma_{0.2}$, is much more pronounced than that on the ultimate tensile

363   strength, σ_{UTS}. The former increases by approximately one third from 305 MPa to 413 MPa, while the

364   latter increases by only 5% from 593 MPa to 622 MPa. This increase correlates with a twofold

365   decrease in the grain size ratio (d/d_0) during section forging (Table 3). For comparison, the available

366   data are also presented [44–48]. The deformation level at which the ultimate tensile strength is recorded

367   decreases with an increase in the deformation level (Fig. 10a), as does the ductility of the samples

368   (Fig. 10a, Table 4). However, all samples show uniform elongation to large plastic strains, up to 0.3,

369   (Fig. 10b) and meet the requirements for the *Standard specification for Ni-Fe-Cr-Mo-Cu alloy UNS*

370   *N08825 forgings, annealed* [2].

371

372
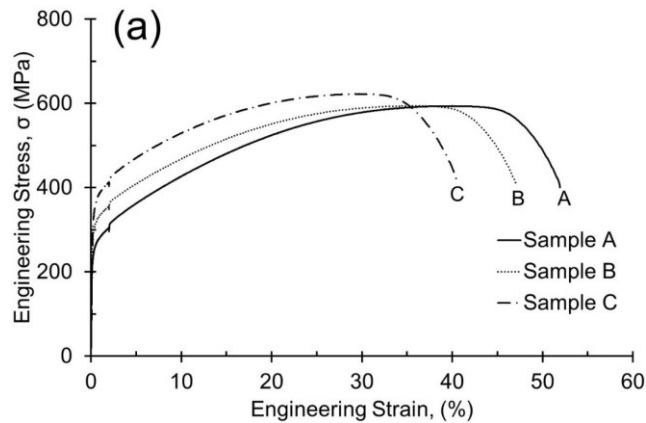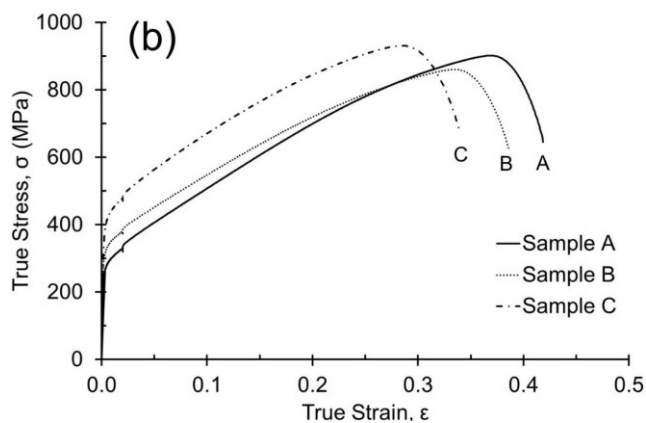
Fig. 10—(a) Engineering tensile stress–strain (b) and true stress–strain curves for Alloy 825 processed by hot forging at indicated samples. All the samples statisfy the minimum yield strength of 241 MPa [2].

Table 4: Room temperature mechanical properties of as-hot forged samples.

Sample	0.2% Proof stress / MPa	Ultimate Tensile Strength / MPa	Failure strain, ε_f (%)
A	305 ± 8	593 ± 3	52 ± 3
B	355 ± 5	594 ± 2	47 ± 2
C	413 ± 5	622 ± 2	40 ± 2

4. Discussion

4.1. *Microstructural evolution*

The reduction of grain size with increasing forging strain is consistent with similar thermomechanical treatments in the temperature range at which recrystallization is possible [49]. The microstructure that develops after hot forging (Fig. 4) is typical of the development of discontinuous dynamic recrystallized grains [50,51]. All three forged samples contain both fine and coarse grains. One potential explanation for this in general materials science is abnormal grain growth [52–55]. However, in the current study, the material is deformed and allowed to recrystallize without large amounts of any second phase to pin grain boundaries (Fig. 2) or any other external factor that would favor one grain orientation over others, such as a magnetic field. Therefore, abnormal grain growth can be rejected as the cause of the grain size distribution in the current study. It is more likely that incomplete dynamic recrystallization is responsible for the grain size distribution: grains that did not undergo recrystallization simply grew during the thermomechanical treatment and correspond to the coarse grains observed after treatment. Those that did recrystallize are significantly finer. This is supported by the reduction in grain size with increasing forging strain, similar to the findings of Niikura et al., who considered the case of a severely-rolled 42 wt% nickel-based alloy ($0.7 < \varepsilon < 2.5$) during hot-working between $1150\,°C$ and $950\,°C$ [15]. Similar relations also apply to steels containing manganese and copper-nickel alloys, both of which also have a matrix with a face-centered cubic crystal structure [56]. This implies that the same approach may be extended to the current alloy. The presence of the observed subgrain boundaries inside grains is evidence of the progress of continuous dynamic recrystallization (CDRX), strain-induced grain boundaries, (dynamic recrystallization by progressive lattice rotation), where recrystallized grains also can nucleate in the body of prior grains [57]. Also, a varity of small and large dynamic recrystallized grains as well as large deformed grains overall forged microstructure is also evidence of the discontinuous dynamic recrystallization (DDRX) [51]. Since most of the decrease in twin prevalence occurred from sample A to sample B it seems that most twins are destroyed between a strain of 0.45 and 0.65 early in the deformation process. The change in the orientation along the white arrow indicated in Fig. 6b is represented in Fig. 4c. The lattice curvature

23

407   over the grain (point-to-origin) achieves 9 degree, although the misorientation between any

408   neighboring points (point-to-point) does not exceed 1.5 deg. The selected grain in Fig. 4c contains

409   annealing twins that suggests its discontinuous recrystallization origin, i.e., nucleation followed by

410   growth in course of dynamic or post-dynamic recrystallization. The large internal distortions as shown

411   in Fig. 4c suggest dynamic or post-dynamic recrystallization [19,58–60]. The large internal distortions as

412   shown in Fig. 4c testify to rather high dislocation densities evolved in the alloy samples subjected to

413   hot forging irrespective of discontinuous recrystallization taking place during and/or after deformation.

414

415   4.2. *Texture evolution*

416   The lack of ⟨111⟩ orientations in the recrystallization texture at all strain levels may have been caused

417   by dynamically recrystallized grains that, after nucleating, rotated toward the hot-forged texture under

418   subsequent deformation [61]. Some studies have proposed that the randomness in recrystallized

419   textures of low stacking fault energy materials is caused by annealing twins, which may hinder

420   recrystallized texture development [62]. This is consistent with both the orientation maps (Fig. 9) and

421   the pole figures of the samples (Fig. 4). It can be seen that the measured textures are weak, with a

422   maximum intensity of not more than double that of a random texture (Fig. 9). Coryell et al. [61] have

423   reported somewhat similar results from nickel-superalloy 945 after the uniaxial compression testing

424   and have shown by EBSD that after deformation to a strain of 1.0 at temperatures 950°C-1150 °C

425   and strain rates of 0.001-1.0 s^{-1}, the microstructure consisted of recrystallized grains that were

426   randomly oriented and contain twins as well as the ⟨111⟩ components were not present in most

427   deformation conditions. Furthermore, the peak in the misorientation angle distribution plots (Fig. 8,

428   sample A) correspond to a 60° misorientation, as has clearly been shown in the misorientation axis

429   distribution in Fig. 9 (Sample A), that is a characteristic of coherent twin boundaries [63]. The decrease

430   in the fraction of Σ3 twin boundaries with increased strain level is due to the formation of subgrain

431   boundaries with a misorientation angle between 2° and 10° (low angle grain boundaries) during

432   straining. This is in stark contrast to materials thermomechanically processed by severe plastic

433   deformation ($\varepsilon \sim 3$) [64].

24

434 In the current material, the stacking fault energy (SFE) was calculated to be 88 ± 5 mJ m^{-2} [38,39,65],

435 which is close to other values in similar face-centred cubic crystal materials, such as copper

436 (78-80 mJ m^{-2} [52]). As a result, the primary deformation mechanism is slip, but twinning may also

437 occur at low temperatures and high strain rates [52]. Twinning is also the preferred deformation mode

438 during rolling in regions oriented at {112}⟨111⟩ and {100}⟨001⟩ [52]. For face-centred cubic metals, a

439 ⟨110⟩ texture is most frequently reported but in some low stacking fault energy materials ⟨111⟩

440 components also form [52].

441

442 4.3. *Effect of strain level on the recrystallized grain fraction, F_G*

443 The fraction of recrystallized grains (F_G) can be described using a simplified version of the Johnson–

444 Mehl–Avrami–Kolmogorov equation (Equation 3) [66,67]:

$$F_G = 1 - \exp(-K\varepsilon^n) \qquad\qquad \text{Equation 3}$$

445 where F_G is the fraction of grains, K and n are material constants and depend on the grain size [66].

446 In the current study, F_G was taken as the area fraction of grains with a size below 25 μm (from Fig.

447 5). The grain refinement kinetics in the hot-forged samples after different strain levels are represented

448 in Fig. 11, which shows F_G as a function of total hot-forging strain. Regression analysis reveals that

449 $K = 1.265 \pm 0.028$ and $n = 0.69 \pm 0.054$. This suggests that a hot forging strain, $\varepsilon > 4$ is sufficient to

450 achieve almost complete recrystallization. However, this is unlikely to be accurate, since the rate of

451 nucleation (number of nuclei per unit time per unit of volume) and rate of growth (length of growth per

452 unit time) are not constant throughout hot forging process, but they are assumed to be constant when

453 deriving Equation 3. In addition, the influence of the increasing grain size (d) will change the shape

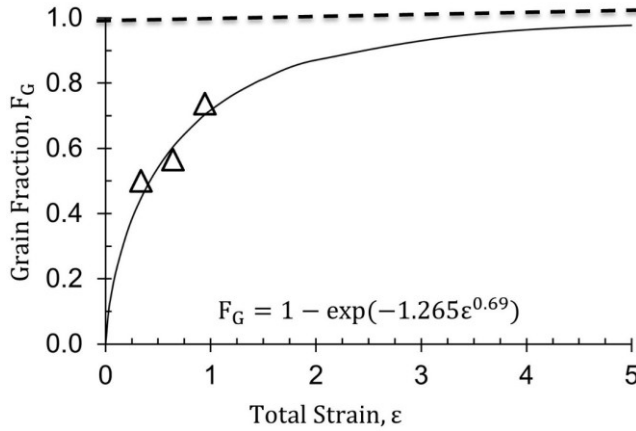454 of the curve toward that the curve in Fig. 11 [66].

455

456

457　Fig. 11—The effect of strain magnitude on the fraction of refined grains (F_G) in the deformed samples

458

459　4.4. *Effect of strain magnitude on the recrystallized grain size*

460　As the reduction of the sample cross section proceeds during deformation, it will lead to finer grain

461　size, if the number of grains through the cross section of sample stays constant. Assuming that the

462　transverse grain size follows the change in cross section of forged sample, the grain size can be

463　represented by a simple function (indicated by dashed line in Fig. 12 and Equation 4, where d is the

464　recrystallized grain size, d_0 in the solution-annealed grain size approximately $122\,\mu\text{m}$, n is a

465　materials-dependent constant and ε is the total forging strain). It is clearly seen in Fig. 12 that the

466　transverse size of the grains decreases much faster than that of the whole sample in the range of

467　relatively small strains below 0.45. The change in the grain size in largest strain follows a common

468　tendency, which is characterized by a quasi-steady-state behavior, where the grain size becomes

469　strain-invariant as reported for various metallic materials subjected to large strain deformation [68].

$$d = d_0\exp(-n\varepsilon) \qquad\qquad \text{Equation 4}$$

470　In this study, regression analysis showed that $n = 2$. This value of n is remarkably higher than those

471　of 1.2-1.4 in stainless steel with dynamically recrystallized microstructures [69,70]. For comparison,

472　$n = 1$ in nickel [70].

26

The plot shows Grain Size, d (μm) on the vertical axis (0 to 150) versus True Strain, ε on the horizontal axis (0.0 to 1.0), with the dashed line labeled:
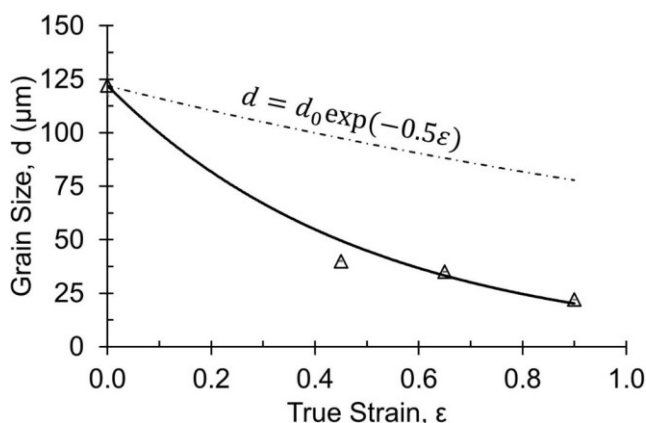
$$d = d_0 \exp(-0.5\varepsilon)$$

473

474 Fig. 12—Effect of the hot forging strain on the recrystallized austenite grain size (open triangles) and

475 calculated (dashed line) values in Alloy 825 samples.

476 4.5. *Evolution of dynamically recrystallized grains*

477 Grains with GOS $\leq 1.0°$ can be considered to be effectively free of dislocations [21] and are

478 considered to be "recrystallized" [21] with no further deformation occurring within the grain after

479 recrystallization. Following forging, there are significant populations of both recrystallized grains and

480 non-recrystallized, deformed grains. Increasing the forging strain increases the fraction of grains that

481 are classified as "deformed" but not recrystallized (Fig. 7), as well as increasing the fraction of grains

482 that underwent recrystallization. It is probable that many of the grains that are identified as deformed

483 did form by recrystallization but then underwent subsequent deformation. This deformation could

484 cause the dislocation density to increase to the point that GOS $> 1.0°$. Other grains that do not

485 undergo recrystallization will accumulate deformation during forging, so an increase in the frequency

486 of "deformed" grains is not inconsistent with increased deformation and dynamic recrystallization. In

487 a recent paper on the effect of strain on the evolution of microstructure during hot-forging of a nickel-

488 based superalloy, the fraction of recrystallized grains was shown to increase with deformation [64]. In

489 that case, the material was air-cooled at $1\,°\text{C}\,\text{s}^{-1}$ to room temperature. Static recovery and

490 recrystallization would almost certainly occur during cooling. In the current study, all forged materials

491 were quenched in water immediately after forging and so such mechanisms are suppressed. It would

492 be expected that the continuous deformation keeps causing grains to recrystallize, after which they

27

493   deform again, resulting in a large number of fine, "deformed" grains, as was observed (Fig. 7, Table

494   3). In the current material, the stacking fault energy, γ_{SFE} is calculated to 88 ± 5 mJ m^{-2} (Equation

495   2), which is significantly higher than those in which annealing twins have been found to block

496   dislocations and so it is unlikely that the twins play a significant strengthening role. Therefore, twins

497   may be ignored when evaluating strengthening mechanisms in the current alloy.

498

499   4.6. *Hardness of deformed samples*

500   The hardness values averaged over 10 measurements on the solution-annealed sample was $1375 \pm$

501   64 MPa. Hardness is observed to increase with deformation strain (Fig. 13). Despite experimental

502   scatter, represented by the error bars (one standard deviation about the mean value for each

503   condition), the rise in hardness is significant.

504

505
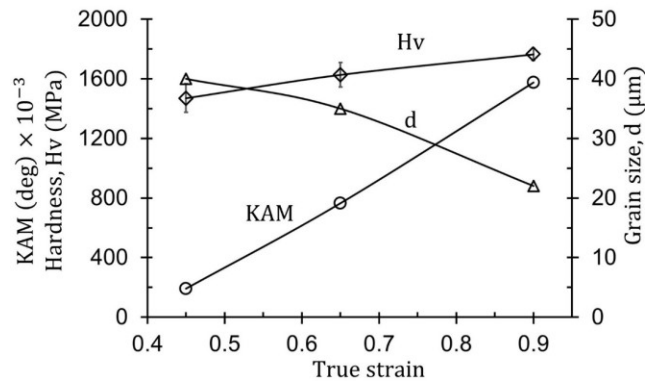
506   Fig. 13—Influence of strain magnitude on the average grain size, d, hardness, H_V, and the kernel

507   average misorientation angle, θ_{KAM} in Alloy 825. Twin boundaries were excluded from the grain size

508   calculation.

509

510   4.7. *Strengthening mechanisms*

511   The relationship between the 0.2% proof strength ($\sigma_{0.2\%}$) and the recrystallized grain size is

512   represented in Fig. 14. The current Alloy 825 samples processed by hot forging and subsequent

28

513   water-quenching obey the following Hall-Petch-type relationship (Equation 5, where the 0.2% proof

514   strength is expressed in MPa and the grain size, d, is measured in micrometres)

$$\sigma_{0.2} = (38 \pm 22) + (1.8 \pm 0.5)d^{-1/2} \qquad \text{Equation 5}$$

515   The data in Fig. 14 and Equation 5 suggest that there may be an additional strength contribution for

516   the present samples, since the Hall-Petch coefficient (the grain size strengthening factor) has a large

517   value of K_G = 1.8 MPa m$^{0.5}$, which is significantly large than those in other studies on Nickel-based

518   superalloys (0.71–0.75 MPa m$^{0.5}$) [71–74] or austenitic stainless steels with statically recrystallized

519   microstructures (0.27–0.64 MPa m$^{0.5}$) [75]. The correlation coefficient of linear regression for yield

520   strength is 0.92, suggesting that the linear fit to the data is certainly reasonable.

521
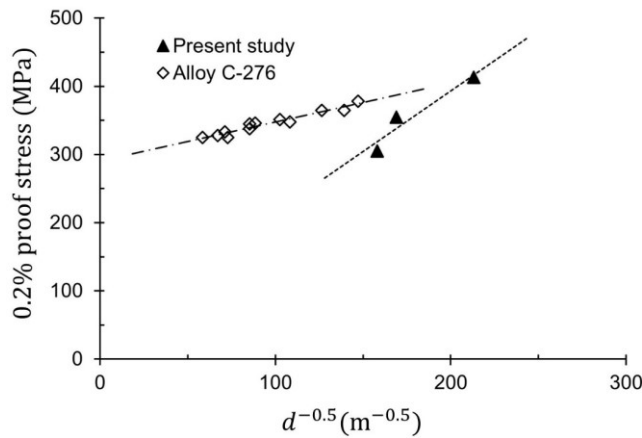
522   Fig. 14—The 0.2% proof stress ($\sigma_{0.2\%}$) of hot-forged as a function of the inverse of the square root of

523   the average static grain size.

524   The additional strength contribution in the present study is very likely to be attributed to the high

525   dislocation density (work hardening). This has been identified as the reason for the deviation from a

526   Hall-Petch-type relationship of conventionally recrystallized austenitic stainless steels [75] or nickel-

527   based superalloys [71–74] with relatively coarse grains. Assuming the strength contributions from grain

528   boundaries and dislocations being independent and linearly additive, as has been reported elsewhere

529   [76–80], the modified relationship for the offset yield strength should include an additional term for the

530   dislocation strengthening, which is much similar to Taylor-type equation (Equation 6, where σ_0 is the

29

531   inherent resistance of the material to dislocation glide excluding grain refinement and work hardening,

532   α is a proportionality constant and depends on the strain rate and the temperature [81]; M is the Taylor

533   factor, equal to 3.1 [82]; $G = 7.6 \times 10^{10}$ Pa is the shear modulus of the material [45], $b = 2.54 \times 10^{-10}$ m

534   is the Burgers vector in the material, K_g is the Petch-coefficient and d is the grain size [83–88].

$$\sigma_y = \sigma_0 + \alpha_\varepsilon MGb\rho^{1/2} + K_G d^{-1/2}$$ Equation 6

535   Using the relationship between KAM and dislocation density (Equation 1) and noting that those grains

536   with low angle grain boundaries are likely to have a dislocation density that is orders of magnitude

537   higher than other grains, a new expression for the yield strength can be derived to reformulate

538   Equation 6 in terms of known or measureable quantities only (Equation 7). The second term of

539   Equation 7 quantifies the contribution due to the low angle grain boundaries. Such boundaries form

540   from dislocation substructures and so the effective size depends on dislocation density, which is

541   related to the total strain. The dislocation density is measured from the kernel average misorientation

542   data (Table 5). The final term gives the strengthening contribution from high angle grain boundaries.

$$\sigma_y = \sigma_0 + \alpha_\varepsilon MGb((\kappa\theta_{KAM}(bs)^{-1})_{LAGB})^{1/2} + K_G d_{HAGB}^{-1/2}$$ Equation 7

543

544   Table 5. Dislocation density, ρ, calculated using Table 1. Values of θ were measured during EBSD

545   of the as-forged material and s is the step size of the EBSD scan.

Sample	$10^3\theta s^{-1}$ / m^{-1}	$10^{13}\rho$ / m^{-2}	$d^{-0.5}$ / m$^{0.5}$	$MGb\rho^{0.5}$ / MPa
A	1.11	0.875 ± 0.2	158.1	174.8
B	4.45	3.5 ± 0.3	169.0	349.5
C	9.16	7.2 ± 0.8	213.2	501.6

546

547   The relationship between the yield strength, grain size and dislocation density (Equation 6) can be

548   used to derive the unknown parameters K_G, α_ε and σ_0. Combining the measured proof stresses,

30

549   dislocation densities derived from KAM measurements and grain sizes from EBSD measurements

550   (summarized in Table 4) and using Gaussian elimination gives the values of each quantity as:

551   0.42 MPa m$^{0.5}$, 0.26, and 193 MPa, respectively. The value of K_G is of the same magnitude of similar

552   materials reported in literature [78,81,89–91] and so it is a reasonable result. The value of α_ε is slightly

553   lower than published results for work-hardened austenitic stainless steels (~0.3) [76,91,92]. This is

554   consistent with materials subjected to a stabilization treatment in which dislocations interact more

555   weakly than work-hardened materials with internal stress fields caused by the accumulated

556   dislocations [36]. The value of σ_0 is consistent with the strengthening mechanisms that contribute to it.

557   The value of 193 MPa in the Hall-Petch equation is also reasonable. Almost the same values of

558   around 200 MPa have frequently reported for austenitic steels by various authors [76,93,94].

559

560   An increase in the total strain leads to an increase in the grain boundary and dislocation

561   strengthening, although the dislocation strengthening prevails over the grain size strengthening. Each

562   contribution can be quantified ($\sigma_0 = 193\ \mathrm{MPa}$ from tensile test data, grain boundary strengthening and

563   work hardening from Table 5 and Equation 7) and compared (Fig. 15).
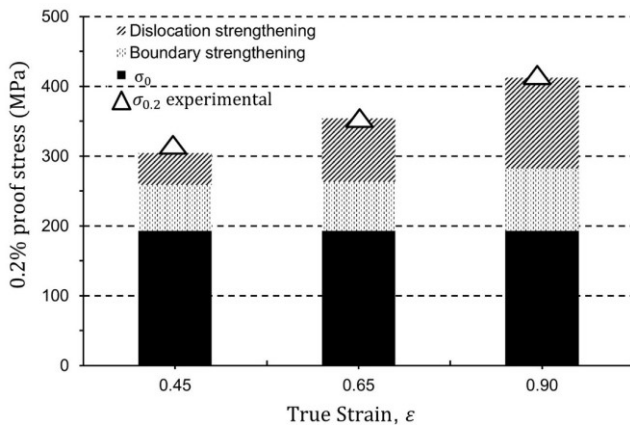

564

565   Fig. 15—Contribution of different strengthening mechanisms to general yield strength of hot forged

566   Alloy 825 subjected to different strain levels.

567 Dislocation density will increase with the extent of deformation, as more dislocations are generated

568 as strain proceeds up to some equilibrium level when recrystallization annihilates dislocations as

569 quickly as they are produced. The dynamically recrystallized grain size decreases with an increase

570 in deformation strain, as more grains contain sufficient dislocation density to drive the nucleation of

571 new grains. It should, therefore, be possible to relate the dislocation density directly to recrystallized

572 grain size. Indeed, analysis of the current data shows that the dislocation density, $\rho^{0.5}$, obeys a power

573 law relationship with the dynamically recrystallized grain size, d_{DRX} (Equation 8).

$$\rho^{0.5} = 0.862\left(d_{\text{DRX}}^{-0.5}\right)^{3.02} \qquad \text{Equation 8}$$

574 Substituting Equation 8 into Equation 7 and replacing the variables with the values derived in this

575 section allows the calculation of 0.2% proof stress, $\sigma_{0.2,\text{calc}}$, as a function of recrystallized grain size,

576 d_{DRX} (Equation 9).

$$\sigma_{0.2,\text{calc}} = 193 + 0.42 d_{\text{DRX}}^{-0.5} + 1.3 \times 10^{-6} d_{\text{DRX}}^{-1.5} \qquad \text{Equation 9}$$

577

578 Substituting Equation 4 into Equation 7 and replacing the variables with the values derived in this

579 section and subsection 4.4 allows the prediction of 0.2% proof stress as a function of strain hardening,

580 ε (Equation 10).

$$\sigma_{0.2} = 193 + [d_0\exp(-n\varepsilon)]^{-0.5}\{0.42 + 1.3 \times 10^{-6}[d_0\exp(-n\varepsilon)]^{-1}\} \qquad \text{Equation 10}$$

581 Where d_0 is initial grain size ($\sim$0.122 m), $n = 2$ for Alloy 825, and ε is a true strain, equal to $\ln R_{\text{R}}$

582 (reduction ratio). The experimental yield strengths are approximately one third higher than those

583 calculated by Equation 10 (Fig. 16). Using regression, a constant factor of 1.34 leads to good

584 agreement between the calculated and measured values, with a correlation coefficient, $R^2 = 0.99$.

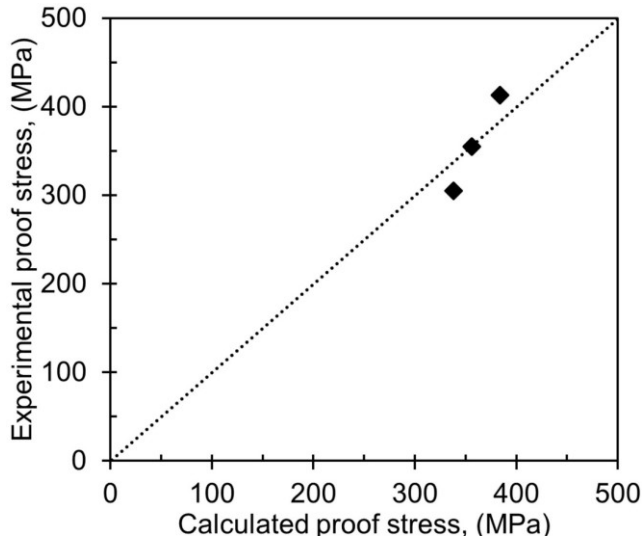$$\sigma_{0.2,\text{exp}} = 1.34\sigma_{0.2,\text{calc}} \qquad \text{Equation 11}$$

585

32

586

587 Fig. 16- Relationship between the experimental and calculated (Equation 11) proof strength of Alloy

588 825 samples subjected to different hot forging strain levels.

589 It is not apparent from the current data why the calculated proof stress is different to the measured

590 value. Further tests are needed to improve the coefficients derived and to reduce statistical scatter.

591 Both of these should improve the reliability of the derived model. However, it seems feasible to derive

592 a relationship between the proof stress and reduction ratio during hot forging. This has the potential

593 to allow customization of the process to achieve a desired proof stress.

594

595 4.8. *Effect of strain magnitude on the dislocation density*

596 The dislocation density calculated by using KAM depends significantly on the OIM step size, the

597 correct value of which depends, in turn, on the value of dislocation density [35]. For each sample, at

598 least two EBSD scans with size step of 5 µm, 2.5 µm, 1.5 µm, 0.75 µm, 0.5 µm, 0.25 µm, and 0.1 µm

599 was used to evaluate the θ_{KAM} value. For a constant dislocation density, the amount by which the

600 KAM method underestimates the dislocation density increases as step size increases. Similarly, the

601 underestimate increases at constant step size as the real dislocation density increases [35,95]. In this

602 study, the KAM values increase as the hot-forged strain increases (Fig. 18), consistent with an

33

603   increase in dislocation density as discussed previously (Fig. 17). The dislocation density can be

604   considered as a unique source of internal stresses. It has been suggested that the measured

605   dislocation densities (solid triangles in Fig. 17) during hot forging can be approximated by a maximum

606   exponential growth function of true strain (solid lines in Fig. 17) (Equation 12, where ρ_0 is the

607   dislocation density in solution annealed sample, ε is the true strain and β and n are materials

608   constants) [80,96].

$$\rho = \rho_0 + \beta(1 - \exp(-n\varepsilon)) \qquad\qquad \text{Equation 12}$$

609   In the current study, $\rho_0 \approx 3.6 \times 10^{12}$ m^{12}. Regression reveals that the best estimate for $\beta = 8.8 \times$

610   10^{13} m^{-2} (cf. previously reported values of 20×10^{15}m^{-2} [80] and 5.75×10^{15}m^{-2} [96], both in

611   austenitic stainless steels) and $n = 0.7$ (cf. previously reported values of 0.25 [80] and 1.03 [96]). The

612   low value of β in the current alloy (Ni-based alloy) differs from the values reported for S304H

613   austenitic stainless steel due to Alloy 825 has a stacking fault energy of approximately 88 mJ m^{-2} in

614   contrast to the austenitic stainless steel, which has a low stacking fault energy of approximately

615   20 mJ m^{-2}. Therefore, recovery should develop somewhat faster in nickel, compared to austenitic

616   stainless steel, which then reduces dislocation density. In the steel, the deformation was performed

617   at higher strain up to 4.0 and below 600 °C, but in the current study, there is a lower strain, which is

618   induced at temperatures above 950 °C. This will allow dislocations to accumulate in the austenitic

619   stainless steel, which can reduce the stacking fault energy in the different deformed samples [97,98].
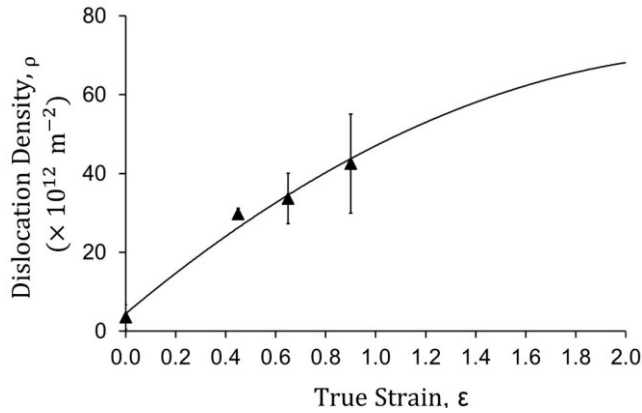
620

621　Fig. 17—The effect of forging strain level on the dislocation densities of experimental (solid triangle)

622　and calculated (line, Equation 12) values in the present Alloy 825.

623

624　4.9. *Chromium- and molybdenum-rich precipitates*

625　Fine precipitates could also be observed sparsely throughout the samples at grain boundaries after

626　hot forging (Fig. 18a). These were analyzed using STEM-EDS and were found to be rich in chromium

627　and molybdenum (Fig. 18b, Table 6). During analysis, these precipitates were found to be elongated

628　along the boundaries with a length of between 150 nm and 500 nm. These findings are also

629　consistent with other published studies in similar materials [9,99–101]. Furthermore, many annealing

630　twins can also be seen in the hot-forged microstructure, which is also consistent with published

631　studies of similar alloys [102,103]. While the presence of grain boundary precipitates could conceivably

632　affect the subsequent behavior of the material, the volume fraction of precipitates is low and the grain

633　boundary precipitates only occur sporadically in the material and so are unlikely to affect the bulk

634　behavior and properties of the material to a significant extent.

35

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636
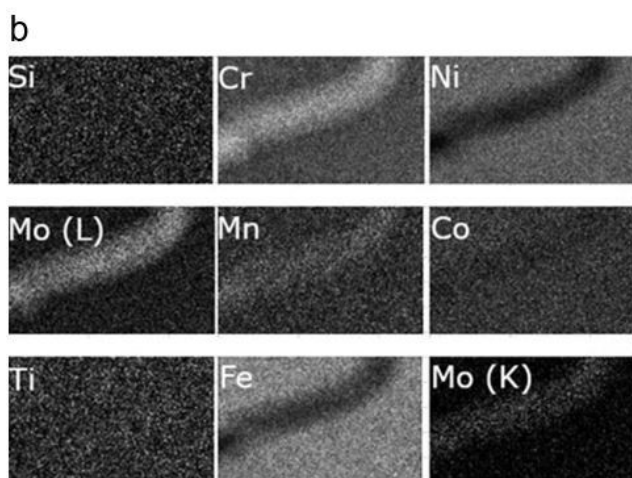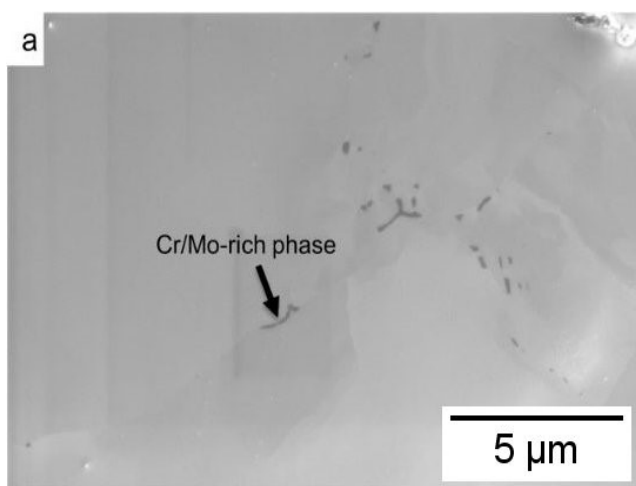
637 Fig. 18—SEM micrograph of initial billet before homogenization and forging: (a) precipitates

638 decorating grain boundaries; (b) STEM-EDS mapping of elements in the precipitate indicated in (a).

639

640 Table 6: Chemical compositions (wt%) of the matrix and precipitate depicted in Fig. 19, measured

641 using scanning TEM-EDS.

Element	Cr	Fe	Ni	Mo
GB phase	46.78	16.24	19.11	17.86
Matrix	23.98	32.73	40.13	3.16

36

642

5. **Conclusions**

The influence of strain magnitude on the mecrostructural evolution, texture and mechanical properties of Alloy 825 was studied. The main conclusions of this study are summarized below:

1) The average grain size decreases with increasing strain during forging, due to increased recrystallization. Both continuous and discontinuous dynamic recrystallization mechanisms operated during the hot forging process.

2) The area fraction of recrystallized grains (F_G) with sizes below 25 µm increased with increasing strain, ε. The fraction of grains that are recrystallized can be described using a simplified modification of the Johnson–Mehl–Avrami–Kolmogorov equation: $F_G = 1 - \exp(-1.265\varepsilon^{0.69})$. This suggests that a near-fully recrystallized microstructure can be developed in the Alloy 825 tested at strains of ~ 4.

3) Hot forging results in nonrecrystallized grains oriented toward a $\langle 110 \rangle$ fiber forging texture, which is consistent with other face-centred cubic materials. An exception occurs at the highest strain level tested in this study (0.90), where the microstructure is only one-third recrystallized. In this deformation level, there is a $\langle 111 \rangle$ fibre texture.

658

37

659   4)   The dislocation density of hot-forged samples increases with increased forging strain. In addition,

660   the microstructures were characterized by high dislocation densities in deformed grains. The

661   change in the dislocation density during hot forging may be expressed as

662 $$\rho = 5.39 \times 10^{12} + \beta(1 - \exp(-0.7\varepsilon)$$

663 where $\beta = 8.75 \times 10^{13} \ \mathrm{m}^{-2}$ for deformed samples.

664   5)   A power law function was obtained between the grain size, d and the dislocation density, ρ:

665        $\rho^{0.5} = 0.862\left(d_{\mathrm{DRX}}^{-0.5}\right)^{3.02}$ for Alloy 825 processed by hot forging with different strain levels and

666   subsequent water quenching. Both the grain size and substructural strengthening contributed to

667   the mechanical properties. Thus, the yield strength could be expressed as a function of grain

668   size by a modified Hall-Petch relationship:

669 $$\sigma_{0.2} = 193 + 0.42 d_{\mathrm{DRX}}^{-0.5} + 1.3 \times 10^{-6} d_{\mathrm{DRX}}^{-1.5}.$$

670   6)   The experimental 0.2% proof strength, $\sigma_{0.2}$, may be obtained by multiplying the calculated yield

671   strength by a factor of 1.34 and can also be expressed through initial grain size, d_0, and total

672   forging strain, ε, by modified Hall-Petch relationship:

673 $$\sigma_{0.2,\mathrm{calc}} = 193 + [d_0 \exp(-2\varepsilon)]^{-0.5}\{0.42 + 1.3 \times 10^{-6}[d_0 \exp(-2\varepsilon)]^{-1}\}$$

674 $$\sigma_{0.2,\mathrm{exp}} = 1.34\sigma_{0.2,\mathrm{calc}}$$

675   7)   The maximum yield strength and ultimate tensile strength were obtained after forging to a true

676   strain of 0.9 and were 413 MPa and 622 MPa, respecitvely, with a ductlity of 40%.

677 **Acknowledgement**

680

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