



## Large UFG Al alloy plates from cryomilling

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**Abstract:** To assess potential routes for the production of high-strength Al 5083, gas-atomized powder was cryogenically ball-milled, to obtain a nanocrystalline structure, then hot vacuum degassed and consolidated by two methods: (1) hot isostatic pressing (HIPping) and extrusion, and (2) two-step quasi-isostatic (QI) forging. The consolidated billet in both cases was hot-rolled to produce 19 mm plate. Despite grain growth and differences in the consolidation and deformation processing, a similar, ultra-fine grained microstructure was obtained in both plates, with elongated grains predominantly in the range 100–500 nm. The cryomilled plates had similar tensile strengths in the plane of the plate, which were up to 60% greater than conventionally processed and work-hardened Al 5083. However, the QI-forged plate had significantly higher fracture toughness than the HIP/extruded plate, which was brittle for crack surfaces in the plane of the plate. The disparity in toughness values was attributed to differences in the prior powder particle boundary structure.

Key words: Nanocrystalline powder; Hot isostatic pressing; Extrusion; Quasi-isostatic forging; Ultra-fine grained microstructure; Mechanical properties

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## 1. Introduction

The nanocrystalline structure of metal powder produced by cryogenic ball-milling can result in extremely high strength of the consolidated material (Witkin and Lavernia, 2006). For Al-containing alloys, the incorporation of non-metallic elements during cryomilling, particularly nitrogen from the surrounding liquid, imparts excellent thermal stability and results in an ultra-fine grained (UFG) structure after the powder has been consolidated at high temperature (Hayes et al., 2004 and Newbery et al., 2007a). In order to retain acceptable ductility in the consolidated alloys, the cryomilled powder must undergo shear deformation during the consolidation process to break up and disperse prior particle boundaries (PPBs) (Newbery et al., 2007a).

A combination of hot isostatic pressing (HIPping) and extrusion has often been used to produce cryomilled rod or bar of high strength with greater ductility than as-HIPped (Witkin et al., 2006). Using an extrusion die of the appropriate rectangular shape, HIP/extrusion reportedly can be used to produce plate stock from a cryomilled Al alloy if the extrusion is subsequently rolled (Bampton and Perez, 2006). However, to produce a more convenient preform for rolling, forging is more suitable. Recently, quasi-isostatic (QI) forging, formerly known as Ceracon forging, has been used to produce cryomilled Al 5083 plate with enhanced mechanical properties (Newbery et al., 2007b). During QI forging, the material is surrounded by a particulate pressure transfer medium (PTM) within a closed die. The forging process can also be used to consolidate the canned powder, replacing the need for HIPping as the primary step (Newbery et al., 2007c).

The Al–Mg alloy Al 5083 possesses an excellent combination of high specific strength, weldability, ballistic properties and corrosion resistance. These properties are desirable for the construction of military ground vehicles, particularly those with amphibious capabilities (Anon, 2006). The design



of such vehicles is typically based on a box frame constructed by welding plate. Al 5083 is commonly used for military applications in the ‘armor grade’ (H131) condition, which is cold-worked to optimize ballistic resistance. Increases in strength and ballistic performance above that possible by cold working are achievable by the use of cryomilling, which would substantially reduce vehicle weight, increasing range and effectiveness (Newbery et al., 2006). To obtain such performance improvements, the production capabilities of cryomilling technology must be scaled up to produce the required plate sizes, firstly for comprehensive assessment of the material, and then for fabrication of the vehicles.

The objective of this study is to compare and evaluate two powder consolidation processes – (1) HIP and extrusion and (2) two-step QI forging – for producing large plates (~19 mm × 450 mm × 250 mm) from cryomilled Al 5083, with respect to microstructure and mechanical properties, especially fracture toughness.

## 2. Experimental procedure

### 2.1 Cryomilling and degassing

Two 20 kg batches of gas-atomized –325 mesh (<44 μm) Al 5083 (Al–4.4Mg–0.7Mn–0.15Cr wt.%) powder produced by Valimet, Inc. (Stockton, CA) were cryomilled for 8 h in liquid N<sub>2</sub> by DWA Aluminum Composites (Chatsworth, CA) using a modified Szegvari attritor. Stainless steel (440C) milling balls were used with a ball-to-powder weight ratio of 32:1, and 40 g (0.2 wt.%) ‘stearic acid’ (~50/50 palmitic and stearic acids) was added to improve yield. After milling, the powder was transferred to a glove box under liquid N<sub>2</sub>, ensuring that atmospheric contamination of the cryomilled powder was minimized. Samples of powder from each of the cryomilled batches were

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used to fill two welded Al 6061 cans. The powder was degassed by heating each can to 450 °C, while simultaneously applying a vacuum through a stem in the can's lid. When a pressure of  $<10^{-6}$  Torr was obtained, a valve on the stem was closed and the can was cooled to ambient temperature. The stem was then sealed permanently by crimping and welding.

## 2.2 HIP and extrusion

A 295 mm diameter, 165 mm high can containing 14.0 kg cryomilled powder was HIPped by Kittyhawk, Inc. (Garden Grove, CA) at a maximum pressure and temperature of  $103 \pm 1$  MPa and  $396 \pm 4$  °C for 4 h. The can was removed by machining to leave a cylindrical billet of consolidated material, 223 mm diameter and 105 mm height. To prepare the billet for extrusion, two cylindrical Al 6061 sections (223 mm diameter) were prepared (the header and the follower) and fitted along with the cryomilled billet into a thin-walled Cu jacket, 231 mm O.D., to give a 45 kg preform of total length  $\sim 360$  mm. The assembled preform was extruded through a 127 mm  $\times$  51 mm rectangular cross-section die by HC Starck (Coldwater, MI), using a billet pre-heat temperature of 204 °C, a starting force of 20.4 MN and a ram speed of 0.5 mm/s. The Cu sheath and the header and follower were removed, leaving a 430 mm length of extruded Al 5083, weighing 6.8 kg, 48% of the cryomilled powder.

## 2.3 Quasi-isostatic forging

A 150 mm diameter, 255 mm high can containing 5.6 kg of cryomilled powder was QI-forged by Advance Materials & Manufacturing Technologies, LLC (Roseville, CA) employing a 330 mm diameter die and a load of 22 MN, equating to a die pressure of 260 MPa. The can, pre-heated to 454 °C, was placed in the die within a bed of graphitic particulate, the PTM, pre-heated to 500 °C.

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After the first forging, the can was removed by machining, leaving a cylindrical billet of consolidated material, 127 mm diameter and 133 mm height. This billet was then reheated to 407 °C and forged a second time using the same die and load as before. After the second forging, the top and bottom surfaces were milled to form a disc, ~223 mm diameter and thickness of 46 mm, in preparation for rolling. The weight of the billet was now 4.3 kg, corresponding to 78% of the cryomilled powder.

## 2.4 Rolling

HIP/extruded and QI-forged billets were pre-heated to 450 °C, then uni-axially rolled by Niagara Specialty Metals (Akron, NY). The rolling schedule entailed a 5 mm reduction per pass until a thickness of 25 mm was reached, followed by two more passes of ~3 mm reduction. The plates were re-heated between passes, typically for a period of about 10 min. Finally, the plates were passed through flattening rollers, before and during which they were allowed to cool, achieving a final transverse (T) thickness of  $19.3 \pm 0.5$  mm. The HIP/extruded plate, rolled normal to the extrusion axis, was approximately rectangular, with dimensions of ~450 mm  $\times$  295 mm, in the extrusion (E) and rolling (R) directions, respectively. The QI-forged plate was oval, with maximum dimensions of ~454 mm  $\times$  229 mm, the shorter dimension being normal (N) to the rolling direction (R).

## 2.5 Standard plate

A rectangular section of conventionally processed ‘armor grade’ Al 5083 H131 plate, 19.0 mm transverse (T) thickness, was used for comparison with the cryomilled plates. The precise processing history of this plate was unknown, although it is likely to have been made by a standard ingot metallurgy (IM) route, followed by hot rolling, and then cold worked to achieve the H131 condition.



We assumed that this plate had primary (R1) and secondary/normal (R2) rolling directions parallel to the length ( $L$ ) and width ( $W$ ), respectively.

## 2.6 Microstructural characterization

The concentration of metallic alloying elements was measured using DC plasma emission spectroscopy according to ASTM E 1097-03 by Luvak, Inc. (Boylston, MA). The concentration of non-metallic elements were measured by LECO, Inc. (St. Joseph, MI) using inert gas fusion analyzers for H (RH404), and for O and N (TCH600), and combustion combined with IR detection for C (CS600). Density measurements were performed using an Ar gas displacement pycnometer (Micromeritics AccuPyc-1330) with a 3.5 cm<sup>3</sup> chamber, at least 80% of which was filled by the samples. Four sets of 20 measurements were made for each material, achieving an accuracy of close to  $\pm 0.001$  g/cm<sup>3</sup>.

Standard metallographic polishing was performed on the plates, sectioned in different orientations with respect to the extrusion, forging or rolling directions, for examination in an optical microscope (Olympus AHMT3). Etching the cryomilled material with detergent emphasized the coarse-grained regions, which appeared lighter than the UFG matrix. Slices of material were thinned for TEM examination (Philips EM420T) by jet polishing in ethanol containing 8% perchloric acid and 10% 2-butoxyethanol. The maximum dimensions of 400 individual grains were measured from TEM images, generating a grain size histogram, from which the number-based, mean grain size and aspect ratio were obtained. The mean grain size and aspect ratio of the standard plate, etched with Kellers Reagent (water containing 2.5% nitric acid, 1.5% hydrochloric acid and 1% hydrofluoric acid), was obtained from optical micrographs using a 10-line intercept method.



## 2.7 Mechanical properties

Tensile behavior in two normal directions within the plane of the plates was assessed using flat dog-bone specimens approximating to sub-size ASTM E 8M (with gauge section  $40 \text{ mm} \times 6 \text{ mm} \times 3 \text{ mm}$ ). The specimens were deformed at a strain rate of  $10^{-3} \text{ s}^{-1}$  using a universal testing machine (Instron 8801), monitored by a dual-camera video extensometer. The presented tensile property data, derived from the resultant stress–strain curves, represents a mean from three measurements, unless stated.

Fracture toughness was measured using a mini-compact tension (CT) specimen of thickness 5.1 mm and width 12.7 mm. Side grooves with depths equal to 5% of the thickness were introduced on both sides of each specimen to enhance crack constraint. CT specimens were made from cryomilled plate with four combinations of crack plane and propagation direction with respect to the extrusion, forging or rolling axes. CT specimens were also prepared to provide two combinations of crack plane and propagation direction for the standard plate. The CT specimens were fatigue pre-cracked in air, and then fractured (with an ATS 904 Universal Test Machine) in accord with ASTM E 399 in ambient air (20 °C and 42% relative humidity) (Anon, 2005). In practice, the plane-strain conditions required for a valid  $K_{Ic}$  measurement were not necessarily met, and so the values obtained were deemed to be  $K_q$ , where  $K_q$  is greater than  $K_{Ic}$ . The  $K_q$  values presented are a mean from three measurements, unless stated. After testing, fracture surfaces were examined (Leo 1550 SEM), and the resultant fractographs presented with the crack growth direction horizontal on the page.



### 3. Results

#### 3.1 Mechanical properties

The concentration of metallic alloying elements for both the plates made from cryomilled powder were within the specification for Al 5083, as shown in Table 1. However, as expected, the levels of the non-metallic elements, H, O, N and C, were all substantially higher than standard plate, as also shown by Table 1. Although the two cryomilled plates were produced from different batches of powder, the compositions were similar. The QI-forged plate was slightly lower in Mn and C.

*Table 1. Chemical analysis of rolled Al 5083 plates. Values in wt.%, except H (ppm)*

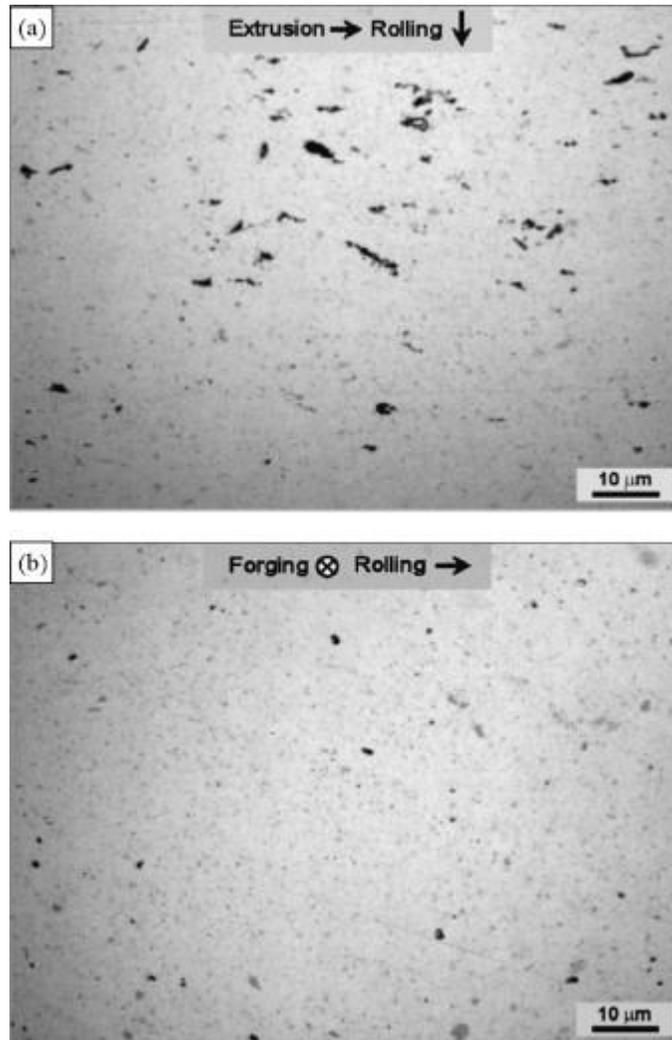
Al5083	Processing	Al	Mg	Mn	Fe	Cr	H	O	N	C
Cryomilled	HIP extrusion	93.9	4.63	0.68	0.26	0.08	11.2	0.47	0.43	0.18
	QI forging	94.2	4.60	0.47	0.21	0.06	10.2	0.48	0.44	0.14
Standard	H131	92.4– 95.6 <sup>a</sup>	4.0– 4.9 <sup>a</sup>	0.4– 1.0 <sup>a</sup>	0.4 max. <sup>a</sup>	0.05– 0.25 <sup>a</sup>	1.3	0.003	<0.005	0.001

*Specification for Al 5083—concentration of alloying elements in standard plate was not measured.*



### 3.2 Porosity

The measured density of the central portion of the HIP/extruded plate was  $2.642 \text{ g/cm}^3$ , 99.3% of the reference value for Al 5083 ( $2.66 \text{ g/cm}^3$ ), indicating that it was less than fully dense. In addition, the density varied with respect to position within the plate, between  $2.639$  and  $2.650 \text{ g/cm}^3$ . This was confirmed by optical microscopy, which revealed the presence of pores as large as  $10 \text{ }\mu\text{m}$ , as shown in Fig. 1(a). Many of these pores were created by the rolling, since the as-extruded billet had a higher density, measured to be  $2.659 \text{ g/cm}^3$ . The density of the QI-forged plate ( $2.670 \text{ g/cm}^3$ ) was greater than the reference value for Al 5083 and 0.5% greater than the standard plate used for comparison here ( $2.656 \text{ g/cm}^3$ ). This high density is attributed to the incorporation of oxides and nitrides into the structure, due to gas atomization and cryomilling. The QI-forged plate exhibited negligible density variation with respect to position—all samples were in the range  $2.670$ – $2.671 \text{ g/cm}^3$ . Microscopic examination revealed a distribution of small pores, mostly  $<1 \text{ }\mu\text{m}$ , as shown in Fig. 1(b).



*Fig. 1. Optical micrographs (unetched) of cryomilled Al 5083 plate showing pores (black) viewed normal to the rolling direction: (a) HIP/extruded, and (b) QI-forged.*

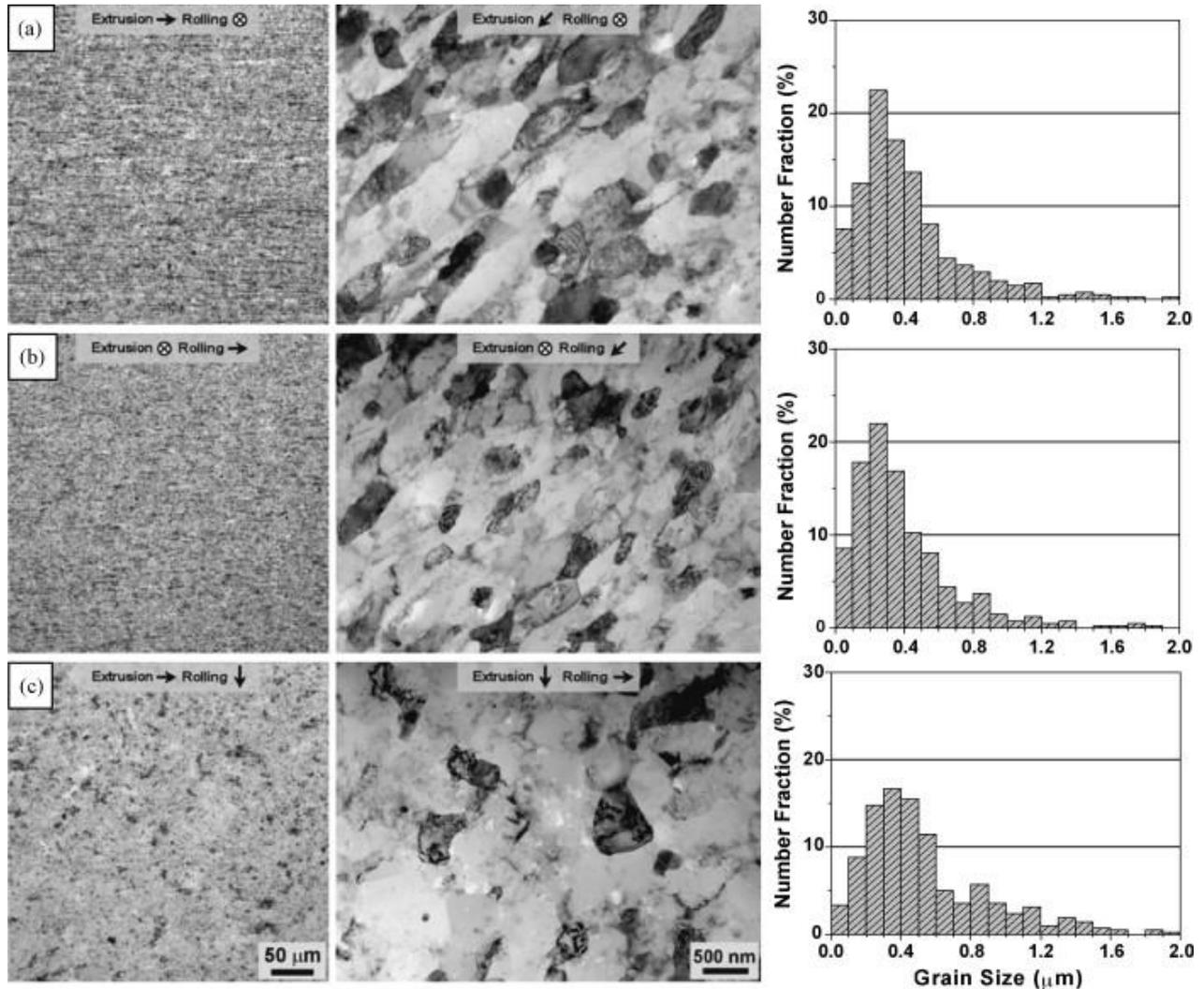
### 3.1 Mechanical properties

The HIP/extruded plate had a uniform optical microstructure predominantly composed of UFGs, interspersed with regions of coarser grains elongated parallel to the extrusion axis, as shown in Fig. 2. TEM confirmed that the grains, also elongated in the extrusion axis, had a wide range of sizes. The mean grain size was approximately  $440 \text{ nm} \times 350 \text{ nm} \times 220 \text{ nm}$ , in the extrusion, rolling and transverse directions, respectively, a ratio of approximately 2:1.6:1, as shown in Table 2. Although

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most grains were less than  $\sim 400$  nm, a significant population of grains larger than  $1 \mu\text{m}$  were present, as also shown in Fig. 2.



**Fig. 2.** Optical (etched) and TEM micrographs, with grain size distributions, of HIP/extruded cryomilled Al 5083 plate viewed in three directions with respect to processing: (a) rolling, (b) extrusion, and (c) transverse axes.

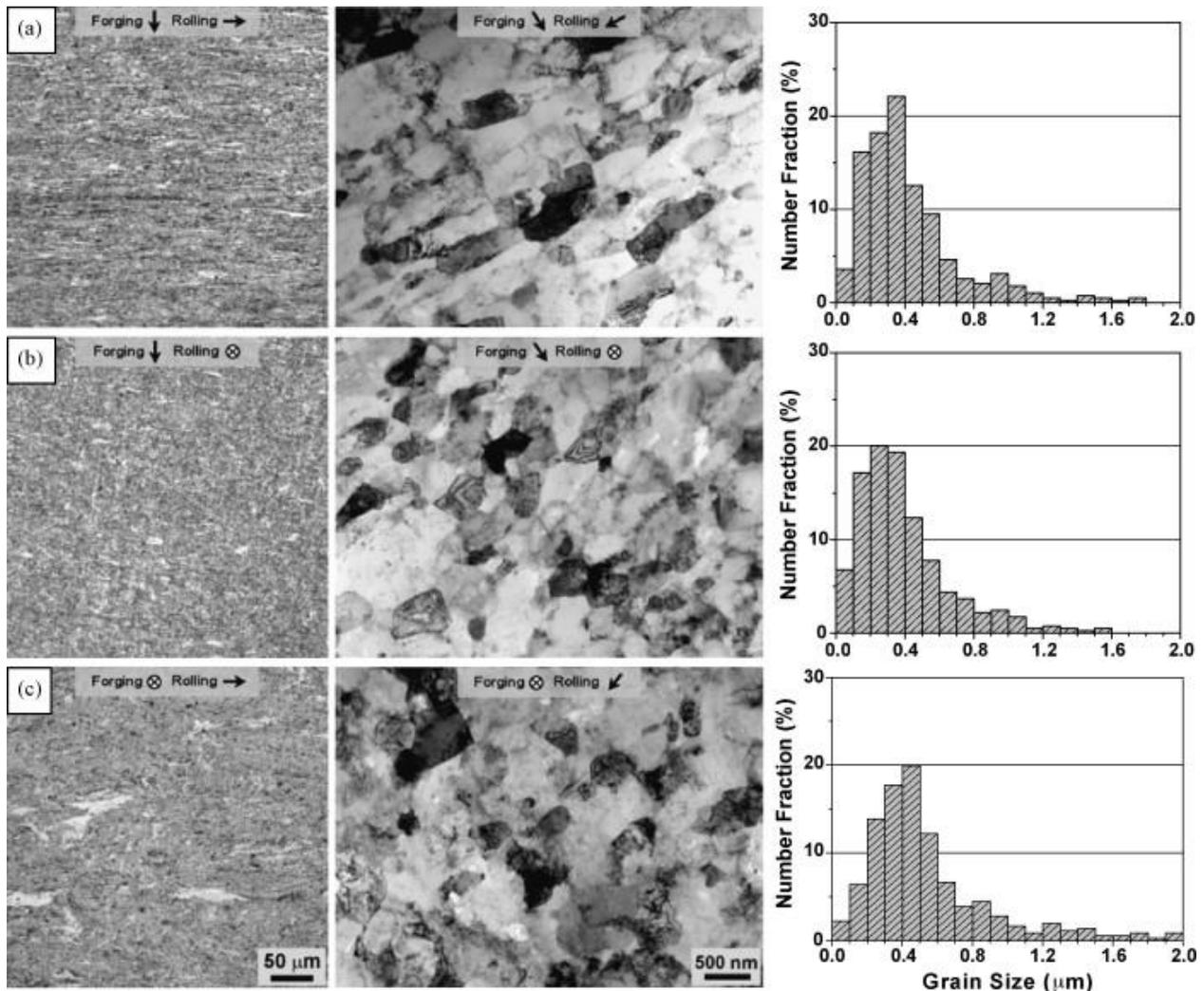
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**Table 2.** Mean grain size and aspect ratio of rolled Al 5083 plates

Al5083	Processing	Viewpoint axis	Measurement method	Length (nm)	Width (nm)	Aspect ratio
Cryomilled	HIP Extrusion	Rolling (R)	Individual grains from TEM images	440 (E)	239 (T)	1.8
		Extrusion (E)		362 (R)	201 (T)	1.8
		Transverse (T)		447 (E)	346 (R)	1.3
	QI Forging	Normal (N)		480 (R)	248 (F)	1.9
		Rolling (R)		438 (N)	240 (F)	1.8
		Forging (F)		496 (R)	428 (N)	1.2
Standard	H131	Width (R2)	Linear intercept from optical images	228 μm (R1)	35 μm (T)	6.5
		Transverse (T)		211 μm (R1)	72 μm (R2)	2.9

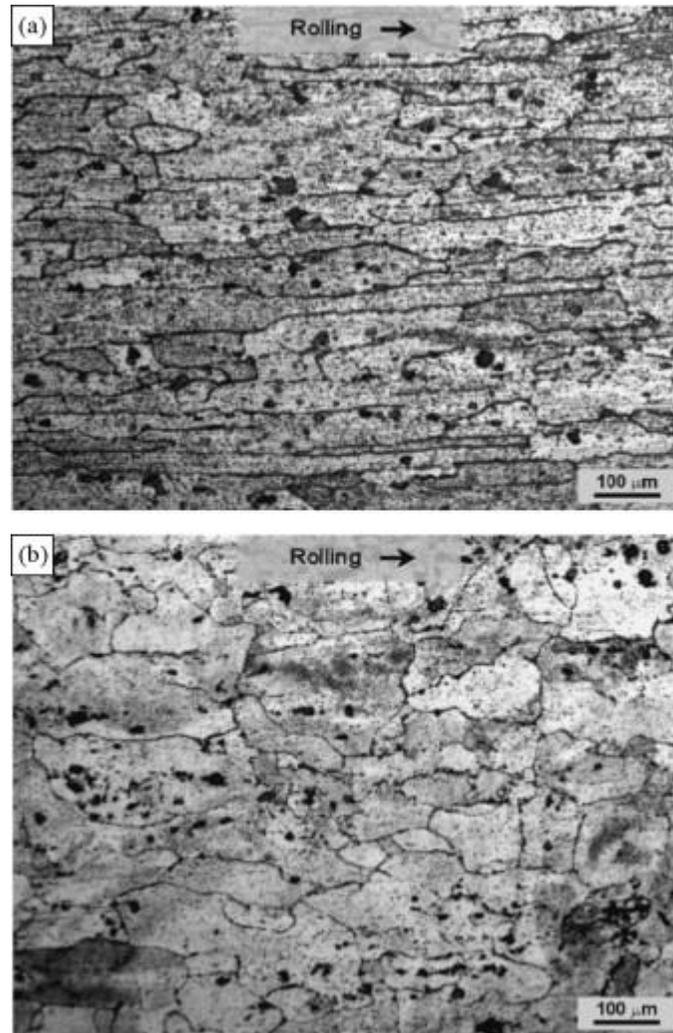
Optically, coarse-grained regions elongated mostly in the rolling axis were more apparent in the QI-forged plate, as shown in Fig. 3. However, TEM images revealed that the proportion of grains larger than 1 μm was slightly less than for the HIP/extruded plate, as also shown by Fig. 3. In general, the grain structure was similar to the HIP/extruded plate, although the mean grain size was larger, as shown in Table 2. The mean grain dimensions were approximately 490 nm × 430 nm × 240 nm in the rolling, normal and forging (transverse) directions, respectively, a ratio of approximately 2:1.8:1.



**Fig. 3.** Optical (etched) and TEM micrographs, with grain size distributions, of QI-forged cryomilled Al 5083 plate viewed in three directions with respect to processing: (a) normal to forging and rolling, (b) rolling, and (c) forging (transverse) axes.

The grains in the standard Al 5083 plate were larger by several orders of magnitude than the cryomilled plates, and they were more elongated, as shown in Fig. 4 and Table 2. The mean grain length parallel to the rolling axis was  $\sim 220 \mu\text{m}$ , almost 500 times that of the cryomilled plates, and the ratio of the mean grain dimension in the primary rolling direction (R1) to that of the transverse direction (T) was 6.5:1, over three times than that of both the cryomilled plates.

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**Fig. 4.** Optical micrographs (etched) of standard Al 5083 H131 plate: viewed normal to the primary rolling direction (R1): (a) normal to the transverse axis (R2), and (b) parallel to the transverse axis.

### 3.4 Tensile properties

Both plates produced from cryomilled powder exhibited high strength, as great as 60% (yield stress) and 35% (UTS) more than the standard Al 5083 H131 plate, as shown in Table 3. The QI-forged plate exhibited a yield stress and UTS close to 420 and 475 MPa, respectively, in both directions with respect to rolling. These strength levels were nearly identical to the HIP/extruded plate in the extrusion direction. However, the strength of the HIP/extruded plate was lower in the rolling

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direction, at 394 and 455 MPa, respectively. The QI-forged plate was more ductile in both directions tested, particularly normal to the rolling direction, elongating over 13%. For the HIP/extruded plate, the ductility was less than the standard H131 plate, and the ductility was higher in the extrusion direction than in the rolling direction.

*Table 3. Tensile properties in the plane of rolled Al 5083 plates*

Al5083	Processing	Direction	0.2% YS (MPa)	UTS (MPa)	Elong. (%)
Cryomilled	HIP Extrusion	Rolling	393 <sup>a</sup>	455 <sup>a</sup>	8.9 <sup>a</sup>
		Extrusion	429	469	10.1
	QI Forging	Normal to rolling	413	475	13.1
		Rolling	427	476	11.5
<hr/>					
Standard	H131	Length (R1)	286 <sup>a</sup>	352	10.6
		Width (R2)	266	354	10.2

<sup>a</sup> Mean value from only two tests.

### 3.4 Tensile properties

The fracture toughness of the QI-forged plate was markedly greater than the fracture toughness of the HIP/extruded plate, and also greater than the standard plate, as shown in Table 4. In particular, the fracture toughness for cracks in the plane of the HIP/extruded plate was low (~7 MPa √m). The toughness in the plane of the plate was low enough that cracks initiated on a plane normal to either the rolling or extrusion directions changed onto this plane during fracture. In contrast, the QI-forged plate was more isotropic—the fracture toughness for cracks on planes parallel to forging was 26–27 MPa √m, only ~4 MPa √m greater than for planes normal to forging. Even for the weaker orientations, the QI-forged plate had greater fracture toughness than the standard plate, which was isotropic, despite its elongated grain structure.

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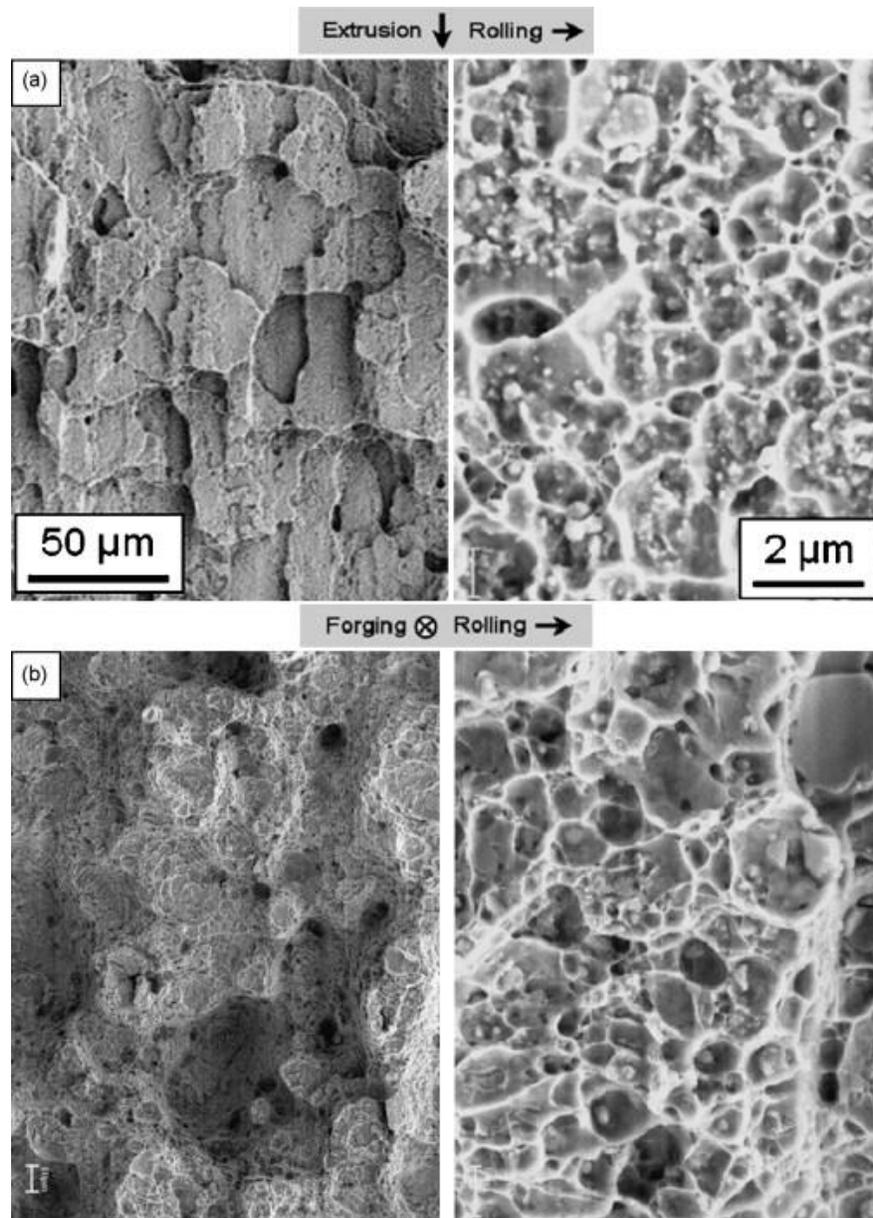


**Table 4.** Fracture toughness of rolled Al 5083 plates

Al5083	Processing	Crack designation	Crack plane normal	Crack propagation	(MPa $\sqrt{m}$ )
Cryomilled	HIP Extrusion	T <sub>R</sub>	Transverse	Rolling	7.2
		T <sub>E</sub>	Transverse	Extrusion	6.8
		E <sub>T</sub>	Extrusion	Transverse	13.8
		R <sub>T</sub>	Rolling	Transverse	16.5 <sup>a</sup>
	QI Forging	F <sub>R</sub>	Forging/transverse	Rolling	22.2
		F <sub>N</sub>	Forging/transverse	90° to rolling	22.7
		N <sub>F</sub>	90° to rolling	Forging/transverse	26.3
		R <sub>F</sub>	Rolling	Forging/transverse	26.5
Standard	H131	F <sub>L</sub>	Transverse	Plate length (R1)	18.6
		W <sub>L</sub>	Plate width (R2)	Plate length (R1)	18.7

*Mean value from only one successful test, since fatigue pre-cracks tended to grow out of the plane—for the one successful pre-crack, the fast-fracture crack propagated 90° out of the intended plane.*

The fracture surfaces for cracks in the plane of the HIP/extruded plate provided clear evidence that inter-particle de-cohesion had occurred during fracture, as shown in Fig. 5(a). Well-defined outlines of prior particles, elongated in the extrusion direction and flattened in the transverse direction, were observed. Closer inspection revealed a dimpled morphology, as also shown in Fig. 5(a), indicating some local ductility to the fracture process. The dimples, however, were shallow.



**Fig. 5.** SEM fractographs for crack surfaces in the plane of rolled cryomilled Al 5083 plates: (a) HIP/extruded ( $T_R$ ), and (b) QI-forged ( $F_R$ ).

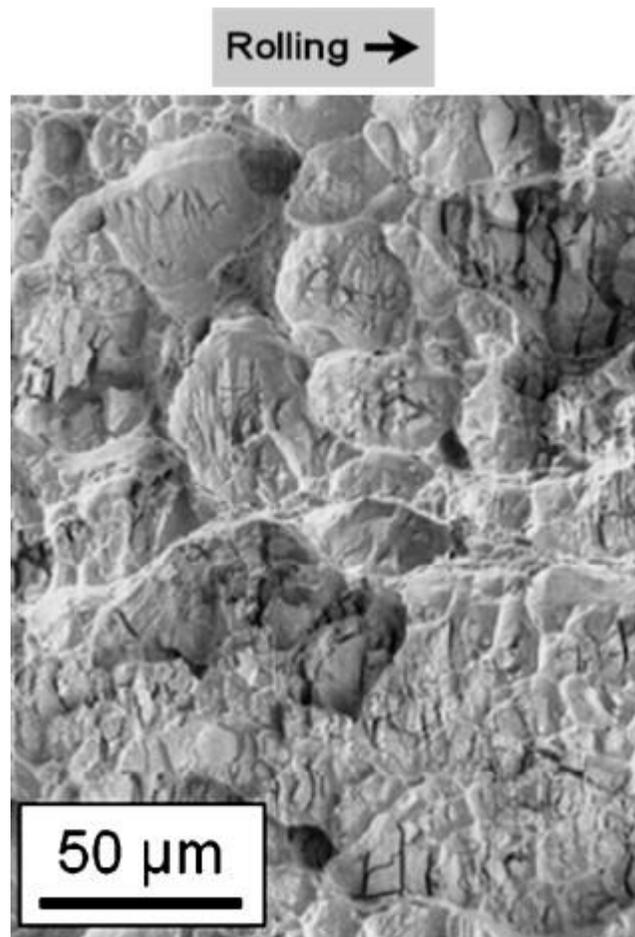
In comparison, the fracture surface of the QI-forged plate with the equivalent crack plane orientation showed a less defined prior particle structure, as shown in Fig. 5(b). Evidence of the prior particle structure was still apparent, the surface undulating over a scale of 10–50 μm, but overall the features were more rounded. The surface showed ductile dimples of 200 nm–2 μm, on a scale similar to the

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grain size observed, and they often contained small particles. These dimples were generally deeper and smoother than those of the HIP/extruded plate.

The fracture surface of standard Al 5083 plate indicated extensive inter-granular failure, as shown in Fig. 6. The fracture morphology consisted of a series of flat plateaus terminating in steep ledges on the scale of 20–200  $\mu\text{m}$ , matching the microstructure shown previously in Fig. 4(b). Surprisingly, despite reasonable fracture toughness, there was little evidence of ductile failure, and most of the surface was smooth and/or cracked.



*Fig. 6. SEM fractograph for a crack in the plane ( $F_L$ ) of standard Al 5083 H131 plate showing the primary rolling direction ( $R1$ ).*

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## 4. Discussion

The two described consolidation methods yielded plates from cryomilled Al 5083 powder that were significantly larger than those made previously. Although the process routes were different, the microstructures of the cryomilled plates were similar. The plates showed a predominantly UFG structure in both cases, in which the grains were elongated in the deformation direction (s). The tensile strengths were significantly greater than standard Al 5083 H131 plate. However, the fracture toughness depended strongly on the consolidation method and the crack orientation with respect to the deformation processing direction—the fracture toughness for the HIP/extruded plate was particularly low for a crack plane in the plane of the plate.

The presence of coarser, micron-sized grains within the UFG structure can be expected to enhance ductility and fracture toughness (Han et al., 2005 and Pao et al., 2006). For the HIPped material, generation of coarse grains arises from filling of the interstices between cryomilled particles by diffusion (Witkin and Lavernia, 2003). The QI-forged plate contained some grains larger than 500 nm, and these probably originated from natural variations in grain size within the as-milled powder, in which grains have been observed to be as large as 200 nm (Newbery et al., 2007c). Although the HIP/extruded plate had a larger population of micron-sized grains, they did not impart greater ductility, which has been observed previously for HIP/forged cryomilled Al 5083 (Newbery et al., 2007c).

The parameters for the HIP/extrusion described here did not result in complete densification of the cryomilled powder. Although the effect of porosity on ductility and fracture toughness are yet to be fully determined, the fracture surfaces gave little indication that the pores had any significance.

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Instead, the fracture surfaces clearly revealed that the primary differences between the plates were the nature of the PPBs. This is a common issue for powder metallurgical Al alloys where the surfaces of the particles are typically covered with a thin layer of oxide and adsorbed water/hydroxides. These surface films weaken the structure and provide pathways for easy crack propagation. PPBs are likely to be especially influential for consolidated cryomilled powder because of the high strength of the matrix.

Because of the repeated fracturing of the particles in liquid N<sub>2</sub> during cryomilling, the surface of the as-milled powder is relatively free from oxide phases. However, during handling of the powder, even in the low O<sub>2</sub>/moisture environment of a glovebox, oxide formation will occur because the partial pressure of O<sub>2</sub> required to oxidize aluminum is extremely low, <10<sup>-10</sup> Torr (Rufino et al., 2007). In addition, the hot vacuum degassing of the powder, performed to remove the stearic acid, may also contribute to oxidation and contamination of the particle surfaces.

Extrusion (and rolling) of the HIP billet deformed the cryomilled particles into flat disks. Extrusion appeared to distort the PPBs, but did little to diminish their deleterious effects on ductility, and the cohesive strength between the prior particles was low. Consequently, the overall fracture toughness for crack surfaces in the plane of the plate was also low. This observation was consistent with low elongations-to-failure in the plate's transverse direction measured by micro-tensile tests (Joshi and Eberl, 2007). Because extrusion decreased the effective PPB surface area in directions within the plane of the plate, the elongations observed for tensile specimens of conventional size were considered acceptable (>8%). However, in terms of ballistics, the low transverse ductility/toughness is likely to have a detrimental effect on resistance to a fragmentary simulating projectile (FSP), causing exit-side spallation and inferior performance.

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One could argue that the extrusion ratio,  $\sim 6$ , was not high enough to sufficiently deform the PPBs. However, even if this was the case, the use of higher ratios would lead to difficulties making plate of reasonable dimensions. Consolidation of the powder by HIPping may have effectively stabilized the PPB structure in place, thus restricting the beneficial shear deformation on the oxide layer during extrusion. Therefore, extrusion of unconsolidated powder is likely to more effectively disperse oxide films and may yield superior performance.

In contrast, QI forging produced greater improvements in fracture toughness for cracks in the plane of the plate, and the toughness was nearly isotropic. This observation was confirmed by good micro-tensile elongations measured through the transverse (forging) thickness of the plate (Joshi and Eberl, 2007). However, crack surfaces from CT specimens indicated that PPBs remained, albeit to a relatively limited extent. Thus, determining the optimum degree of deformation to reduce the presence of PPBs further is a major goal for future process development. An important part of this will be to investigate flow patterns during forging and their effects on PPBs. Due to the small thickness of oxide layers on Al powder, typically  $<5$  nm, precise characterization of the PPBs will be a challenge.

Overall, of the two processing routes considered, QI-forged plate demonstrated superior mechanical performance. However, given that the routes did not employ optimized parameters, there is some uncertainty involved in making an absolute comparison. With regard to other factors, though, the two-step QI forging route is attractive for obvious reasons. It is a simple route, negating the need for HIP, and obtaining fairly high material yield. In preference to extrusion, the forging route is geometrically suitable for making large rolling preforms.

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Although the plates described were larger than those produced previously, they were still smaller than that required for construction of military vehicles, for example. The current size limitations of QI forging will have to be addressed to make cryomilling feasible for commercial application. Comparable property levels must be reproducibly demonstrated on a yet larger scale before this processing route can be implemented in production.

## 5. Conclusions

Two consolidation and deformation processing methods, HIP/extrusion and two-step QI forging, successfully produced, from cryomilled Al 5083 powder, plates of a larger size than has been achieved before. Both of the plates possessed a similar UFG microstructure, with grains mostly in the range 100–500 nm elongated in the deformation directions. The QI-forged plate had grains 40–80 nm greater than the HIP/extruded plate, depending on orientation. The tensile strengths in the plane of the plates were similar and up to 60% greater than conventionally processed Al 5083, with acceptable ductility. However, the fracture toughness of the QI-forged plate was clearly superior. Despite highly deforming the powder particles, extrusion did not sufficiently increase the strength of the PPBs and these were very weak, leading to delamination and low toughness for crack surfaces in the plane of the plate. The QI forging more efficiently broke up the PPBs, leading to relatively isotropic fracture toughness, greater than standard Al 5083 plate.

**Acknowledgements:** Financial support for this work was gratefully received from the Office of Naval Research under contract N00014-03-C-0163. The authors would also like to thank Cliff Bampton and Raul Perez of Pratt & Whitney Rocketdyne for donation of the Cu extrusion jacket

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and discussion of the extrusion route, and Kevin Doherty and Ernie Chin of the Army Research Laboratory for contribution of the standard Al 5083 H131 plate.

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