1	Effects of Iron-rich Intermetallics and Grain Structure on Semisolid Tensile Properties of
2	Al-Cu 206 Cast Alloys near Solidus Temperature
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4	Amir Bolouri, Kun Liu, XGrant Chen*
5	Department of Applied Science, University of Quebec at Chicoutimi,
6	Saguenay, QC, Canada, G7H 2B1
7	Abstract
8	The effects of iron-rich intermetallics and grain size on the semisolid tensile properties of
9	Al-Cu 206 cast alloys near the solidus were evaluated in relation to the mush microstructure.
10	Analyses of the stress-displacement curves showed that the damage expanded faster in the mush
11	structure dominated by plate-like $\beta$ -Fe compared to the mush structure dominated by Chinese
12	script-like $\alpha$ -Fe. While there was no evidence of void formation on the $\beta$ -Fe intermetallics, they
13	blocked the interdendritic liquid channels and thus hindered liquid flow and feeding during
14	semisolid deformation. In contrast, the interdendritic liquid flows more freely within the mush
15	structure containing $\alpha$ -Fe. The tensile properties of the alloy containing $\alpha$ -Fe are generally higher
16	than those containing $\beta$ -Fe over the crucial liquid fraction range of ~0.6 to 2.8%, indicating that
17	the latter alloy may be more susceptible to stress-related casting defects such as hot tearing. A
18	comparison of the semisolid tensile properties of the alloy containing $\alpha$ -Fe with different grain
19	sizes showed that the maximum stress and elongation of the alloy with finer grains were
20	moderately higher for the liquid fractions of ~2.2-3.6%. The application of semisolid tensile
21	properties for the evaluation of the hot tearing susceptibility of experimental alloys is discussed.
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Keywords: Al-Cu 206 cast alloy; Semisolid, Tensile properties; Fe-rich intermetallics; Grain
size.

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- 27 \* Corresponding author:
- 28 X.-Grant Chen, Department of Applied Science, University of Québec at Chicoutimi,
- 29 Saguenay (QC), Canada G7H 2B1, Tel.: 1-418-545 5011 ext. 2603; Fax: 1-418-545 5012
- 30 E-mail: <u>xgrant\_chen@uqac.ca</u>

### 32 **1. Introduction**

The semisolid tensile behavior of solidified aluminum alloys has recently received 33 considerable attention [1-5]. Due to the thermal gradients and solidification shrinkage during the 34 casting process, the semisolid microstructure is frequently subjected to tensile stresses, which 35 can lead to casting defects such as hot tearing and porosity [6-8]. The response of the solidified 36 37 microstructure to the applied stress depends on the deformation behavior, tensile properties and liquid flow within the semisolid structure (mush structure) [9-11]. To investigate the mechanical 38 properties of aluminum alloys in the semisolid state, three major mechanical tests in shear [7,12-39 40 13], compression [7,14-15] and tension [4,7,11,16-19] have been developed. It is widely accepted that the semisolid tensile test induces a similar stress-strain condition to that during 41 solidification of the aluminum alloy [7,17]. Therefore, the semisolid tensile test can provide 42 accurate quantitative results for the tensile properties of the semisolid alloy [2,3,17,20-26]. 43 Moreover, the microstructural observations of the mechanically tested samples may show that 44 the microstructural evolution is similar to those during solidification [1,27-30]. These 45 capabilities provide a deeper understanding of the deformation mechanisms of the mush structure 46 under the stresses from the solidification process [8]. 47

The tensile behavior and deformation mechanisms of the mush structure as a function of the solid fraction have been the subject of a number of studies [20-27]. Studies to determine the semisolid tensile properties of different aluminum alloys, including AA5182 [2,4], AA3014 and AA6111 [3,4], 7xxx [5] and AA6061 [17], have been conducted. The results from those prior studies indicate that all semisolid materials lost their ductility at a solid fraction, fs, of ~0.95– 0.98, and their strength at a fs of ~0.90–0.95 [3]. However, there is limited information on the effect of constitutive phases such as Fe-rich intermetallics on the semisolid tensile properties of aluminum alloys during the last stage of solidification. The morphology, size and distribution of
these intermetallics are important for the formation of casting defects such as hot tearing [28,31].
Furthermore, a careful examination of the effect of grain structure on the tensile properties of the
mush is rarely found in the literature.

Al-Cu cast 206 alloys possess great potential to achieve excellent mechanical properties 59 comparable to those of forged and wrought aluminum alloys [32,33]. In addition, they have a 60 promising high temperature tensile strength [34]. However, 206 cast alloys are susceptible to hot 61 tearing during the casting process [35,36]. Iron is one of the most common impurities in 62 63 aluminum alloys. Due to its extremely low solid solubility in aluminum, iron often precipitates in the form of different iron-rich intermetallic phases during solidification. The most common iron-64 rich intermetallics in 206 alloys are plate-like β-Fe (Al<sub>7</sub>CuFe) and Chinese script-like α-Fe 65  $(Al_{15}(FeMn)_3(SiCu)_2)$  intermetallics, depending on the chemistry of the alloy [37,38]. The plate-66 like  $\beta$ -Fe intermetallics are considered detrimental to the mechanical properties of the alloys 67 because they act as stress concentrators and crack initiators and promote shrinkage porosity by 68 blocking interdendritic feeding [28,39,40]. To counteract the detrimental effect of the plate-like 69 β-Fe intermetallics in 206 alloys, Mn and Si are added to transform the iron-rich intermetallics 70 71 from platelet  $\beta$ -Fe to Chinese script-like  $\alpha$ -Fe. [39,40]. In addition to affecting the mechanical properties of Al-Cu 206 alloys at ambient temperature, these intermetallics may also 72 significantly influence the semisolid tensile properties of the mush [31]. 73

In the present study, semisolid tensile tests were conducted on 206 alloys with liquid fractions less than 0.1 (near the solidus), which is critical for stress-related defect formation during casting and solidification [1]. The effects of different iron-rich intermetallics and grain sizes on the tensile properties of the mush were studied. The microstructural evolution at

different liquid fractions and the fracture surfaces of tensile samples were examined using 78 scanning electron microscopy. The mush deformation mechanisms for different liquid fractions 79 were discussed. 80

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#### 2. Experimental procedures 82

2.1. Preparation of alloys and cast samples 83

Commercially available pure aluminum (99.7%) and pure Mg (99.9%), Al-50Cu, Al-84

25Fe, Al-50Si, and Al-25Mn master alloys were used to prepare three experimental Al-4.5Cu 85

206 alloys. The chemical compositions of the alloys were analyzed by optical emission 86

0.3%. They were designated based on their Fe, Si and Mn contents as following: Alloy 311

spectroscopy and are shown in Table 1. Three experimental alloys contained a fixed Fe level of

containing 0.1% Si and 0.1% Mn for forming plate-like β-Fe (Al<sub>7</sub>CuFe) intermetallics and Alloy 89

333 containing 0.3% Si and 0.3% Mn for forming Chinese script-like  $\alpha$ -Fe (Al<sub>15</sub>(FeMn)<sub>3</sub>(SiCu)<sub>2</sub>) 90

intermetallics [37-39]. Alloy 333-GR has the same chemical composition as Alloy 333, but 91

0.02% Ti was added to form an Al-5Ti-1B master alloy for grain refining. In this study, all 92

compositions are given in wt.%, unless otherwise specified. 93

94 The alloy batch and melting was conducted in an electric resistance furnace. The temperature of melt was held at 1033 K (750 °C) for 30 minutes and the melt was gently stirred. 95 Pure argon gas was used for 20 minutes degassing at a flow rate of 2 L/min through a rotating 96 97 graphite impeller at a speed of 150 rpm. A standard ASTM B108 permanent mold, preheated at 633 K (350 °C), was used to cast the as-cast samples. For semisolid tensile testing, cylindrical 98 specimens with a total length of 120 mm and a diameter of 10 mm were machined from the 99 100 standard ASTM B108 cast samples. Even screw threads were precisely machined at the both

ends of the cylindrical specimens providing that the final distance between two installed nuts was
95±0.1 mm (Fig. 1a).

103 2.2. Semisolid tensile testing

A Gleeble 3800 thermomechanical testing unit was used for the semisolid tensile testing. 104 Each specimen was fixed in a horizontal orientation between the two grips and covered by a free 105 movable ceramic tube to prevent aluminum liquid leakage during the last stage of tensile 106 deformation (Fig. 1a). The specimen was heated rapidly via electro-resistance heating. Due to the 107 heat losses from the water-chilled grips at the ends of the specimen, there is a parabolic 108 109 temperature profile along the specimen length with the hottest point located in the middle of the specimen [2,19]. Temperature control is critical during tensile testing because the microstructural 110 changes that occur near the solidus strongly influence the mechanical properties. The 111 temperature was monitored by three K-type thermocouples spot-welded at the middle and  $\pm 8$ 112 mm on either side of the center of the sample (Fig. 1a). A two-step heating profile was 113 programmed. The temperature was controlled by the middle thermocouple and the specimen was 114 heated to 733 K (450 °C) at a rate of 2 °C/s and held for 45 s. Subsequently, the specimen was 115 heated to 5-7 K below the target temperature at a rate of 1 °C/s. Further temperature increases to 116 117 the target temperature were conducted manually to avoid overheating. The specimen was held at the target temperature for 30 s. 118

Fig. 1b shows the results of a sensitivity analysis for 15 test samples for a target temperature of 797 K (524 °C). All of the heating data indicated that the temperature profiles along the length of the specimen were parabolic. Great effort was made to ensure that the temperature profiles were symmetric and that the maximum temperature drop in the vicinity of the target temperature was approximately one degree in the middle zone (5-6 mm) of the specimen.

The tensile tests were first conducted for fully solidified alloys at a few degrees below the solidus. Subsequently, the semisolid tensile tests were conducted at 2-degree increments until a maximum temperature was reached. This maximum temperature was determined such that a negligible fracture stress was present and corresponds to a solid fraction of ~0.9. A minimum of three tests were conducted at each temperature. The specimens were loaded at a strain rate of ~10<sup>-3</sup> s<sup>-1</sup>. The data acquisition rate was 200 data per second.

131 2.3. Material characterization

Differential scanning calorimeter (DSC) analysis was performed to determine the solidus 132 and liquidus temperatures of experimental alloys. DSC analysis was conducted on heating 133 (melting) and cooling (solidification) paths. In the present work, DSC data on heating was used 134 to calculate the liquid fraction of the alloys due to the fact that Gleeble semisolid tests were 135 136 conducted on heating process. The liquid fraction of the alloys as a function of temperature was calculated with a method proposed in Ref [41]. The liquid fraction curves vs temperature for 137 Alloys 311 and 333 are shown in Fig. 2. After tensile tests, the samples were sectioned parallel to 138 139 the loading direction, and prepared for metallographic observations. An optical microscope and a scanning electron microscopy (SEM) equipped with energy-dispersive X-ray spectroscopy 140 141 (EDS) are used to examine the microstructure and fracture surface of specimens.

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### 143 **3. Results and discussion**

#### 144 **3.1.** As-cast microstructure

The typical as-cast microstructures of the prepared alloys are shown in Fig. 3. In general,
the microstructure consists of α-Al dendrites, iron-rich intermetallics and eutectic Al<sub>2</sub>Cu or

 $Mg_2Si+Al_2Cu$ , which are labeled in the micrographs. For Alloy 311, the iron-rich intermetallics 147 are plate-like  $\beta$ -Fe(Al<sub>7</sub>CuFe), which are interlocked with the low melting Al<sub>2</sub>Cu and are 148 distributed in the interdendritic regions (Fig. 3a). For Alloys 333 and 333-GR, the Chinese 149 script-like  $\alpha$ -Fe (Al<sub>15</sub>(FeMn)<sub>3</sub>(SiCu)<sub>2</sub>) is the dominant iron-rich intermetallic (Figs. 3b and c). In 150 addition, a binary eutectic Al<sub>2</sub>Cu and a ternary eutectic Mg<sub>2</sub>Si+Al<sub>2</sub>Cu are distributed in the 151 152 interdendritic regions. The iron-rich intermetallic transformation from the plate-like  $\beta$ -Fe to the Chinese script-like  $\alpha$ -Fe is due to the increased Mn and Si contents in the 333 and 333-GR alloys 153 compared to Alloy 311 [37]. 154

The grain structure and size were determined using Electron Backscatter Diffraction (EBSD) technique. Fig. 4 shows the EBSD grain maps for Alloys 311, 333 and 333-GR. The different colors represent aluminum grains with different orientations. All three alloys have uniform, equiaxed grains. For Alloys 311 and 333, the average grain sizes are  $280 \pm 28 \ \mu m$  and  $323 \pm 30 \ \mu m$ , respectively, which are comparable because both alloys were cast using the same casting conditions and not grain-refined. The addition of 0.02% Ti from the Al-5Ti-1B master alloy significantly reduced the average grain size of Alloy 333-GR to 94±15 \mum m (Fig. 4c).

### 162 **3.2.** Evolution of stress-displacement curves

Tensile tests near the solidus were performed on samples with different liquid fractions. The fracture behavior greatly depends on the liquid fraction, and similar behaviors were observed for all three experimental alloys (Alloys 311, 333 and 333-GR). For example, typical curves for samples in the solid state (a few degrees below the solidus temperature) and the semisolid state (above the solidus temperature) are shown in Fig. 5 to illustrate the effect of the liquid fraction on the stress-displacement curves in Alloy 333. The stress values are engineering stresses. The engineering stress values ( $\sigma$ ) are calculated as  $\sigma = \frac{F}{A_0}$ , in which F is the force and

 $A_0$  is the cross-section area of specimen before deformation. For the samples tested in the solid 170 state (Fig. 5a), there is a gradual decrease in the stress until the final fracture after the maximum 171 stress is reached. The alloy necks and is remarkably ductile until fracture (a total displacement of 172 3.68 mm), which is consistent with the hot tensile behavior of solid metals. For the specimens 173 tested in the semisolid state with a 0.02% liquid fraction (Fig. 5b), there is a limited amount of 174 plastic deformation before the final fracture. This fracture behavior occurs in the mush at 175 temperatures just above the solidus as the amount of the coherent solid skeleton is sufficient to 176 sustain the tensile stress allowing it to plastically deform [42]. However, the small amount of 177 liquid (0.02%) in the microstructure introduces isolated pockets of liquid that considerably 178 reduce the elongation value compared to the fully solid alloy (Fig. 5a). At a liquid fraction of 179 180  $\sim 0.1\%$ , the alloy undergoes brittle fracture that occurs at the peak stress without any plastic deformation (Fig. 5c). This behavior is identical for the samples with liquid fractions of ~0.1-181 2.2%. The maximum stress decreases from 13 MPa at a liquid fraction of ~0.1% to 6.5 MPa at a 182 183 liquid fraction of ~2.2%. Further increases in the liquid fraction (>~2.6%) result in the development of end-pieces in the stress-displacement curves after the maximum stress is reached 184 185 (Fig. 5d). Instead of the aforementioned brittle behavior (Fig. 5c), the stress increases sharply 186 until the peak stress is reached and then continuously decreases (end-pieces) before a final rupture slightly below the maximum stress (Fig. 5d). This may indicate the presence of plastic 187 deformation and necking in the mush structure after the maximum stress. The size of the end-188 pieces decreases as the liquid fraction increases, as shown in Fig. 5d. 189

Similar types of stress-displacement curves and therefore a similar fracture behavior were
obtained for all three alloys. However, the specific fracture behavior (as previously explained)
occurred over a different range of liquid fractions for each alloy. For example, Alloy 311

exhibits brittle fracture for liquid fractions of 0.5-0.8%, but Alloy 333-GR exhibits brittle
fracture for liquid fractions of 0.1-3.7%.

## 195 **3.3.** Effect of iron-rich intermetallics on semisolid tensile behavior

As mentioned in Section 3.1, Alloy 311 contains plate-like  $\beta$ -Fe intermetallics, while Alloy 333 has predominant Chinese script-like  $\alpha$ -Fe intermetallics (see Fig. 3). The semisolid tensile deformation and properties of Alloys 311 and 333 were compared to examine the effect of different iron-rich intermetallics on the semisolid tensile behavior for various liquid fractions.

#### 200 3.3.1. Stress-displacement curves

201 The stress-displacement curves for Alloys 311 and 333 at different liquid fractions are shown in Fig. 6. For a given liquid content, the mush structure of Alloy 333 tolerates larger 202 displacements before reaching the maximum failure stress compared to the mush structure of 203 Alloy 311. Because tension was applied to the samples at a constant strain rate, for a given liquid 204 fraction, the stress reaches its maximum value more quickly in the mush structure of Alloy 311 205 compared to the mush structure of Alloy 333. Assuming that crack initiation and its full (Fig. 6a) 206 or partial propagation (Fig. 6c) occur before the maximum stress is reached [2], it is reasonable 207 to suggest that the expansion of damage in the mush structure of Alloy 311 occurs more quickly 208 209 than in the mush structure of Alloy 333. On the other hand, the semisolid structure of Alloy 311 is weaker than the semisolid structure of Alloy 333 because Alloy 311 reaches lower maximum 210 stresses (Figs. 6a and b). At larger liquid fractions of ~2.8%, both alloys exhibit a similar range 211 212 of strengths (Fig. 6c), which will be discussed in detail in the following sections.

213 3.3.2. Deformation behavior

To further understand the fracture mechanisms in the semisolid zone, a set of interrupted semisolid tensile tests was conducted. At a fixed liquid fraction, the specimen was loaded to a 216 limited deformation (corresponding to a predetermined displacement), and the test was terminated. For one liquid fraction (corresponding to a single test temperature), different 217 displacements were applied. The minimum displacement was 0.03 mm and was increased in 218 increments of 0.01-0.02 mm. The unfractured specimens were carefully removed from the 219 testing machine and then sectioned and prepared for metallographic observation. These results 220 provide insight into the evolution of the semisolid microstructure during tensile deformation. The 221 tensile tests were conducted at liquid fractions in which both Alloys 311 and 333 exhibited 222 identical characteristics in the stress-displacement curves with similar maximum stress values. 223

224 The stress-displacement curve for Alloy 311 with  $\sim 2\%$  liquid fraction is shown in Fig. 7a. SEM images of the damage distribution within the semisolid microstructure at different 225 deformation levels (displacement) are shown in Figs. 7b-d. For a displacement of 0.03 mm (a 226 very small deformation level), the formation of primary voids at triple points and crack 227 propagation through the aluminum cell/grain boundaries can be clearly observed in Fig. 7b. The 228 triple points and cell/grain boundaries are in the low melting eutectic regions in the as-cast 229 microstructure that is the first to remelt during heating of the semisolid zone. When the semisolid 230 is deformed, the existing low melting liquid at the triple points and cell/grain boundaries is 231 232 sucked to the regions being deformed to accommodate the deformation [21,29]. No evidence of void nucleation on the  $\beta$ -Fe intermetallics was observed via SEM in the deformed mush, which 233 is consistent with previous research [28]. In other words, as the displacement increases to 0.05 234 235 and 0.07 mm (Figs. 7c and d), the voids open considerably and the cracks grow rapidly. For a displacement of 0.07 mm, the sample completely fractures and plate-like  $\beta$ -Fe intermetallics 236 were observed on the crack surfaces (Fig. 7d), suggesting that the  $\beta$ -Fe intermetallics facilitate 237 238 crack propagation, although there was no direct initiation of voids through the  $\beta$ -Fe plates.

239 The stress-displacement curve and corresponding semisolid microstructures of Alloy 333 with a ~2.7% liquid fraction are shown in Fig. 8. For displacements of 0.03 and 0.05 mm, there 240 is little evidence of voids and cracks. When the displacement was increased to 0.06 mm, several 241 interconnected voids formed (Fig. 8b). Furthermore, a number of micro-necks are present 242 (indicated by arrows). These micro-necks are the bridged dendrite arms through the  $\alpha$ -Fe 243 intermetallics. By further increasing the displacement to 0.07 and 0.08 mm (Figs. 8c and d), the 244 micro-necks stretch and plastically deform [43]. Some micro-necks begin to break at a 245 displacement of 0.08 mm (Fig. 8d). From Figs. 8b-d, it is evident that after reaching the 246 247 maximum stress (at the displacement of  $\sim 0.07$  mm), further deformation is required for the micro-necks to completely fracture, which occurs at the lower stresses indicated by the end-248 pieces. A similar deformation behavior was also observed in Alloy 333-GR because the 249 microstructures of Alloys 333 and 333-GR have similar characteristics such as the same Chinese 250 script  $\alpha$ -Fe intermetallics and eutectic phases. It should be noted that this micro-necking effect is 251 not typical and was rarely observed in the mush microstructure of Alloy 311. These 252 microstructural differences result in the remarkable difference in the end-pieces of the stress-253 displacement curves for Alloys 311 and 333, as shown in Fig. 6c. 254

255 3.3.3. Tensile properties

Fig. 9 shows the evolution of the maximum stress and displacement at fracture as a function of the liquid fraction for Alloys 311 and 333. The displacement at fracture is used here instead of the traditional elongation in the tensile testing because of the non-uniform temperature profile within the Gleeble tensile specimen. For each condition, the reported displacement at fracture is the average value of a minimum of three tests. In general, as the liquid fraction within

the mush microstructure increases, the maximum stress and the displacement at fracture decrease
for both alloys.

At very low liquid fractions of  $<\sim 0.5\%$ , the maximum stress values are similar to those 263 obtained for the solid state and are similar for both alloys (311 and 333). This indicates that the 264 strength of the alloys is mainly determined by the solid phase deformation. However, the 265 displacement values at fracture are sensitive to increases in the liquid content of the mush (Fig. 266 9b). When the liquid fraction increases from a minimum value to  $\sim 0.5\%$ , the displacement value 267 at fracture decreases from  $\sim 0.5$  to 0.15%. At high liquid fractions above 3%, plateaus in the 268 maximum stress and displacement values are observed. Moreover, the plateaus occur at similar 269 values for both alloys. It is likely that at this stage, the liquid completely wets the solid grains, 270 separating them with liquid films such that the semisolid tensile properties of the alloys are 271 mostly determined by the liquid phase [11]. 272

The crucial zones in Fig. 9 are located in samples with liquid fractions of  $\sim 0.6-2.8\%$ . In 273 this case, Alloy 333 exhibits superior semisolid tensile properties (higher maximum stress and 274 displacement at fracture) compared to Alloy 311. These differences may be explained by 275 considering the influence of the  $\beta$ -Fe and  $\alpha$ -Fe intermetallics on the deformation of the mush. 276 277 Several studies [1,11,27,42] have indicated that during semisolid tensile deformation, the flow behavior of liquid metal within the mush structure is the key parameter in determining the 278 semisolid deformation characteristics of alloys. Indeed, liquid flow is promoted in the direction 279 280 of the deformed regions [23]. If sufficient liquid feeding is provided, the applied deformation can be accommodated by the mush structure, which results in a higher deformation prior to fracture 281 [27]. As discussed in Section 3.3.2, no evidence of void formation on iron-rich intermetallics was 282

observed. Therefore, the role of iron-rich intermetallics in the prevention of liquid feeding isclosely examined.

As shown in Fig. 10a, the fracture surface of the Alloy 311 specimen with a liquid fraction of ~0.1% clearly shows that the low melting eutectic Al<sub>2</sub>Cu phase is surrounded and separated by large  $\beta$ -Fe intermetallic plates. A similar structure is observed in the as-cast microstructure in Fig. 2a. During semisolid tensile deformation, the flow of the Cu-rich eutectic liquid (Al<sub>2</sub>Cu firstly remelting above the solidus) is blocked by the plate-like  $\beta$ -Fe intermetallics, which prevents sufficient liquid feeding to the regions being deformed. Fig. 10b shows that at a high liquid content of ~3%, the liquid Al<sub>2</sub>Cu is immediately blocked by the  $\beta$ -Fe.

Another example of the blocking effect of plate-like  $\beta$ -Fe in the 311 sample with ~2.1% 292 liquid content undergoing a displacement of 0.05 mm (the sample shown in Fig. 7c) is shown in 293 the longitudinal cross-section shown in Fig. 11. An interdendritic channel was completely 294 blocked by two β-Fe plates, and the eutectic liquid was unable to flow to and feed the required 295 regions during tensile deformation (Fig. 11a). Fig. 11b also shows that the  $\beta$ -Fe intermetallic 296 plates blocked the interdendritic liquid in region 'A', and thus, no feeding occurred between 297 regions 'A' and 'B'. The blocking effect of the  $\beta$ -Fe significantly reduces the permeability of the 298 299 mush structure, resulting in void/crack formation and propagation [22].

Figures 10c and d show that in the mush structure of Alloy 333, the low melting eutectic Al<sub>2</sub>Cu phases are not necessarily located close to the  $\alpha$ -Fe. Thus, there are more free paths in the interdendritic channels for the liquid to flow in Alloy 333 than in Alloy 311. It is apparent that the anticipated differences in liquid flow behavior within the mush structure (feeding capabilities) between Alloys 311 and 333 are not only related to the morphology of the iron-rich intermetallics but also to their distribution. In Alloy 311, the precipitation temperature for the  $\beta$ -

306 Fe intermetallics is similar to the precipitation temperature for the Al<sub>2</sub>Cu eutectic phase [37]. Therefore, most of the β-Fe intermetallics are closely distributed and interlocked with the Al<sub>2</sub>Cu 307 eutectic phase (Figs. 2a and 10a). With their large and long plate-like morphology,  $\beta$ -Fe 308 intermetallics can easily block the interdendritic channels, thereby hindering the eutectic liquid 309 flow and feeding during semisolid deformation (Fig. 10b). In contrast, the  $\alpha$ -Fe intermetallics 310 311 precipitate much earlier than the main eutectic phases because the precipitation temperature of the α-Fe intermetallics is much higher than that of the Al<sub>2</sub>Cu or Al<sub>2</sub>Cu+Mg<sub>2</sub>Si eutectic phases 312 [37]. As a result, some of the  $\alpha$ -Fe intermetallics may not be located in the interdendritic regions. 313 314 In addition, for the intermetallics located in the interdendritic regions, the  $\alpha$ -Fe is less effective at blocking flow than the β-Fe due its branched morphology. Therefore, the liquid can flow and 315 feed within the mush structure before it is blocked, allowing it to accommodate a larger amount 316 of deformation before fracture (Fig. 9b). It is generally believed that  $\beta$ -Fe intermetallics operate 317 as stress concentrators within the mush structure due to their morphology and distribution, which 318 weakens the mush [44]. The deleterious effect of  $\beta$ -Fe is clearly reflected in Fig. 9a in which 319 Alloy 311 exhibits considerably lower strengths compared to Alloy 333 for liquid fractions of 320 ~0.6-2.8%. 321

322 3.3.4. Spike formation

During semisolid failure, a number of small spikes are often observed on the fracture surface after the semisolid tensile tests [27]. SEM images of the spikes along with their approximate chemical compositions (as a reference) are shown in Figs. 12a and b for Alloys 311 and 333, respectively. In Alloy 333, spikes that, based on their chemical compositions, resulted from either the necking of interdendritic  $\alpha$ -Fe bridges (A in Fig. 12a) or the rupturing of accumulated eutectic liquid (B in Fig. 12a) were frequently observed. However, in Alloy 311,only a few spikes that formed via eutectic liquid rupturing were observed (Fig. 12b).

Recently, by utilizing synchrotron X-ray radiography, Phillion et al. [27] conducted in 330 situ observations of the semisolid deformation behavior of the Al-Cu system and proposed that 331 rupturing of the accumulated liquid was the principle source for the spikes. Farup et al. [6] 332 333 identified two spike formation mechanisms: (1) the necking of solid bridges at the grain boundaries and (2) the break-up of the liquid phase. This study shows that depending on the 334 microstructure of the alloy, either of these mechanisms or a combination of both may contribute 335 336 to the semisolid tensile properties as well. For Alloy 311, although there are  $\beta$ -Fe bridges between the aluminum dendrite cells/grains, the mechanism for necking through the solid 337 bridges is not active, due to the discontinuous nature of the individual large plates. On the other 338 hand, their plate-like morphology limits the liquid flow, and consequently, liquid accumulation 339 and rupturing rarely occur, suggesting that little plastic deformation occurs before failure. The 340 scenario is different for Alloy 333 with the Chinese script-like  $\alpha$ -Fe. The eutectic liquid that 341 contains the Chinese script-like  $\alpha$ -Fe may act as a continuous structure at the cell/grain 342 boundaries due to the branched morphology that sustains the deformation by necking. This 343 induces a further plastic deformation prior to final fracture. In a partially fractured sample 344 (loaded to 0.08 mm with a liquid fraction of 2.7%, Fig. 8), an  $\alpha$ -Fe bridge mixed with eutectic 345 liquid at the interdendritic boundaries is shown in Fig. 12c. The collapse of these micro-necks 346 347 results in the formation of spikes on the fracture surfaces, indicating that plastic deformation occurs during semisolid failure. 348

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#### **350 3.4. Influence of grain refinement on semisolid tensile properties**

To examine the effect of the grain structure on the semisolid tensile behavior, the semisolid tensile properties of Alloys 333 and 333-GR were compared. These alloys have the same chemical compositions but different grain sizes (Fig. 4). The average grain size of Alloy 333-GR (94  $\mu$ m) is significantly smaller than that of Alloy 333 (323  $\mu$ m).

The semisolid tensile properties of Alloys 333 and 333-GR as a function of the liquid fraction are shown in Fig. 13. At liquid fractions above ~4%, the tensile properties are nearly identical for both alloys, indicating that the tensile properties are controlled by the liquid phase. For both alloys (333-GR and 333) with liquid fractions of ~3.6-2.2%, a decrease in the liquid fraction results in an increase in both the maximum stress and displacement values, followed by a quasi-plateau. Ultimately, a rapid increase in the properties occurs at liquid contents of 0.6% and 0.1% for Alloys 333 and 333-GR, respectively.

Although the general trend for the semisolid tensile properties of both alloys is similar, 362 the maximum stress and displacement at fracture of the alloy with fine grains (Alloy 333-GR) 363 are somewhat higher than those of the coarse-grained alloy (Alloy 333) for liquid fractions of 364  $\sim 2.2-3.6\%$  (Fig. 13). In this liquid fraction range, a continuous liquid layer/film is assumed to 365 exist in the interdendritic regions. In the alloy with fine grains, the liquid is distributed more 366 367 uniformly around the dendrites and there are more  $\alpha$ -Al grain bridges, which lead to a higher strength and increased displacement at fracture. As the liquid fraction decreases, the liquid 368 layer/film becomes discontinuous and the effect of the fine grains is weaker. Therefore, both 369 370 alloys exhibit a quasi-plateau with similar semisolid properties.

371 As discussed earlier, Alloy 333 containing the Chinese script-like  $\alpha$ -Fe has remarkably 372 higher semisolid tensile properties during the last stage of solidification compared to Alloy 311 373 containing the plate-like  $\beta$ -Fe (Fig. 9). This indicates that the  $\alpha$ -Fe intermetallics in Al-Cu 206

alloys play a major role affecting the semisolid tensile behavior near the solidus. On the other hand, for the grain-refined alloy (Alloy 333-GR containing the same  $\alpha$ -Fe as Alloy 333), a moderate improvement in the semisolid properties is observed. It is suggested that with the same iron-rich intermetallic phase in the microstructure, the grain refinement can give a supplementary benefit on semisolid tensile properties.

#### 379 **3.5.** Application to hot tearing

Classical hot tearing is considered as the inability of a material to accommodate stress 380 and strain during the last stage of solidification and is linked to the intrinsic mechanical 381 382 properties of the mush state and the interdendritic liquid flow. Generally, the mush structure dominated by  $\alpha$ -Fe (Alloy 333) has a higher load-bearing capacity compared to the mush 383 structure dominated by  $\beta$ -Fe (Alloy 311) for liquid fractions of  $\sim 1-3\%$  (Fig. 9a). Thus, under the 384 same solidification conditions, the stress arising from solidification shrinkage and thermal 385 contraction that provokes catastrophic failure (hot tearing) is higher for the mush structure 386 dominated by  $\alpha$ -Fe (Alloy 333). On the other hand, Alloy 333 can sustain more deformation 387 before failure and is more ductile (Fig. 9b). The higher stress and ductility result in a lower 388 susceptibility to hot tearing in Alloy 333. 389

A critical transition for hot tearing can be defined as the *critical liquid content for* stress/ductility below which the load-bearing capacity of the mush structure sharply increases and the ductility is rapidly enhanced, which allow it to sustain a noticeable amount of deformation prior to failure [2, 3]. As shown in Fig. 9, as the liquid content during the last stage of solidification decreases, the stress/ductility dramatically increases at deformations of ~1% and ~3% for Alloys 311 and 333, respectively. This indicates that Alloy 333 can sustain the stress and deformation earlier and at higher liquid contents than Alloy 311. At the critical liquid

content, the interdendritic liquid film separation is the main failure mechanism for the mush 397 structure due to the very low permeability of the structure [42]. This means that if an appropriate 398 399 stress is applied to the mush structure to create a void/crack, the crack will propagate easily with only limited resistance to mush deformation. Therefore, it is reasonable to believe that a lower 400 critical liquid content for stress/ductility indicates a higher susceptibility of a material to hot 401 402 tearing. The critical liquid content for Alloy 311, which is dominated by  $\beta$ -Fe, is much lower than that of Alloy 333 containing  $\alpha$ -Fe (~1% vs. ~3%). In addition to the reduced interdendritic 403 feeding for potential crack healing in Alloy 311 (see Section 3.3.3), it can be concluded that the 404 405 mush structure of Alloy 311 is more prone to hot tearing than the mush structure of Alloy 333.

As shown in Fig. 13, the semisolid tensile properties of Alloys 333 and 333-GR are only moderately different. Hence, a significant improvement in the hot tear resistance as a result of grain refinement of the same  $\alpha$ -Fe-containing microstructure cannot be expected. However, in Alloy 333-GR, the critical liquid content for stress/ductility occurs at ~4% compared to ~3% in Alloy 333 (Fig. 13). Therefore, Alloy 333-GR is less susceptible to hot tearing than Alloy 333.

Additionally, it should be noted that during the semisolid tensile tests, the applied tension 411 (stress-displacement) represents the intentionally induced voids/cracks and deformation in the 412 413 mush structure. Moreover, it has been shown that the hot tearing failure mechanism of the mush structure follows the formation of voids/cracks and their growth within the structure [27]. Once 414 415 the voids/cracks form, an increased stress is required for the voids/cracks to propagate, which 416 leads to final failure (hot tearing). Therefore, the rate at which the stress increases with the displacement (strain) can be used to evaluate the hot tearing susceptibility. For similar liquid 417 contents, hot tearing is more likely to occur at higher rates. In the present work, the rate  $\left(\frac{d\sigma}{du}\right)$  of 418 increase in the stress ( $\sigma$ ) as a function of the displacement ( $\mu$ ) has been calculated for Alloys 311, 419

420 333 and 333-GR (Figs. 6 and 14). It is found that  $\frac{d\sigma}{d\mu}$  is always higher in Alloy 311 than in Alloy 421 333 (Fig. 6), indicating that Alloy 311 has a higher susceptibility to hot tearing, which is 422 consistent with the fact that Al-Cu 206 alloys containing β-Fe intermetallics are very prone to hot 423 tearing [28]. The stress-displacement curves for Alloys 333 and 333-GR are shown in Fig. 14 for 424 two liquid fractions. Alloy 333-GR has a lower  $\frac{d\sigma}{d\mu}$  compared to Alloy 333, demonstrating that 425 grain refinement increases the resistance to hot tearing, although it may be limited in the current 426 study.

427

#### 428 4. Conclusions

(1) The effects of iron-rich intermetallics on the semisolid tensile behavior of Al-Cu 206 alloys
are significant. The tensile properties (the maximum stress and displacement at fracture) of
the alloy dominated by α-Fe are generally higher than those of the alloy dominated by β-Fe
for liquid fractions of ~0.6-2.8%. The mush structure dominated by α-Fe has a higher loadbearing capacity and can sustain more deformation before failure compared to the mush
structure dominated by β-Fe, resulting in a lower susceptibility to hot tearing.

435 (2) Although no evidence of void formation on the β-Fe intermetallics was observed, the 436 interdendritic liquid channels are clearly blocked by the plate-like β-Fe, which hinders liquid 437 flow and feeding during semisolid deformation. In contrast, interdendritic liquid flow occurs 438 more freely within the mush structure containing Chinese script-like  $\alpha$ -Fe due to their 439 branched morphology and distribution.

(3) Comparing the semisolid tensile properties of the alloys containing α-Fe with different grain
 sizes, the maximum stress and displacement at fracture of the alloy with finer grains were

- 442 moderately higher for liquid fractions of ~ 2.2 to 3.6%, indicating a supplementary benefit
  443 of the grain refinement on semisolid mechanical properties.
- (4) The critical liquid content for stress/ductility and the rate of the stress increase with respect to
  the displacement in the semisolid tensile tests are proposed as indicators of the hot tearing
  susceptibility of aluminum alloys.
- 447

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525

- 527 Table
- 528

Table 1 Chemical composition of Al-Cu 206 alloys

	Elements (wt.%)						
Alloys	Cu	Mg	Fe	Si	Mn	Al	
311	4.69	0.31	0.32	0.11	0.12	Bal.	
333	4.64	0.33	0.34	0.32	0.33	Bal.	

529

# 531 Figure captions

- Fig. 1. (a) Image for specimen configuration and the position of thermocouples and (b)
- temperature distribution profiles along the length of specimens.
- Fig. 2. Liquid fraction as a function of temperature (a) Alloy 311 and (b) Alloy 333 with insertsof the enlargement near solidus temperature.
- Fig. 3. As-cast microstructures of samples (a) Alloy 311, (b) Alloy 333 and (c) Alloy 333-GR.
- Fig. 4. EBSD maps for grain size analyses (a) Alloy 311, (b) Alloy 333 and (c) Alloy 333-GR.
- Fig. 5. Stress-displacement curves for Alloy 333 at different liquid fractions (a) fl=0, (b) fl=0.02%, (c) fl=0.1-2.2% and (d) fl=2.6-5.7%.
- Fig. 6. Stress-displacement curves for Alloys 311 and 333 at different liquid fractions (a) fl=0.6-541 0.7%, (b) fl=1.6% and (c) fl=2.8%.
- 542 Fig. 7. (a) Stress-displacement curves for Alloy 311 at the liquid content of ~2%, and (b-d)
- fracture profiles after different displacements for specimens tested at the liquid fraction of  $\sim 2\%$ .
- Fig. 8. (a) Stress-displacement curves for Alloy 333 at the liquid content of ~2.7%, and (b-d)
  fracture profiles after different displacements for specimens tested at the liquid fraction of
  ~2.7%.
- Fig. 9. The semisolid tensile properties of Alloys 311 and 333 as a function of liquid fraction (a)
  average maximum stress and (b) average displacement at fracture.
- Fig. 10. SEM pictures from fracture surfaces of Alloys 311 and 333 at different liquid contents
  (a) Alloy 311, fl=0.1%, (b) Alloy 311, fl=3%, (c) Alloy 333, fl=0.1% and (d) Alloy 333, fl=3%.
- 551 Fig. 11. Fracture profiles of Alloy 311 after 0.05 mm displacements for a specimen tested at
- 552 liquid fraction of  $\sim 2\%$  (a) and (b)  $\beta$ -Fe plates blocked the interdendritic channels at two different 553 locations.
- Fig. 12. SEM images from the fracture surfaces showing the spikes in (a) Alloy 311 and (b) Alloy 333 as well (c) the micro-necking of an  $\alpha$ -Fe bridge mixed with eutectic liquid in a not-
- 556 fully-fractured sample (Alloy 333 with displacement of 0.08 mm).
- 557 Fig. 13. The semisolid tensile properties of Alloys 333 and 333-GR as a function of liquid
- 558 fraction (a) average maximum stress and (b) average displacement at fracture.
- Fig. 14. Stress-displacement curves for Alloys 333 and 333-GR (a) fl=1.7-1.8% and (b) fl=2.72.8%.

# Figures



Fig. 1. (a) Image for specimen configuration and the position of thermocouples and (b) temperature distribution profiles along the length of specimens.



Fig. 2. Liquid fraction as a function of temperature (a) Alloy 311 and (b) Alloy 333 with inserts of the enlargement near solidus temperature.



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Fig. 4. EBSD maps for grain size analyses (a) Alloy 311, (b) Alloy 333 and (c) Alloy 333-GR.



Fig. 5. Stress-displacement curves for Alloy 333 at different liquid fractions (a) fl=0, (b) fl=0.02%, (c) fl=0.1-2.2% and (d) fl=2.6-5.7%.



Fig. 6. Stress-displacement curves for Alloys 311 and 333 at different liquid fractions (a) fl=0.6-0.7%, (b) fl=1.6% and (c) fl=2.8%.





Displacement of 0.05 mm



Displacement of 0.07 mm

Fig. 7. (a) Stress-displacement curves for Alloy 311 at the liquid content of  $\sim 2\%$ , and (b-d) fracture profiles after different displacements for specimens tested at the liquid fraction of  $\sim 2\%$ .



Displacement of 0.07 mm



Fig. 8. (a) Stress-displacement curves for Alloy 333 at the liquid content of  $\sim 2.7\%$ , and (b-d) fracture profiles after different displacements for specimens tested at the liquid fraction of  $\sim 2.7\%$ .



Fig. 9. The semisolid tensile properties of Alloys 311 and 333 as a function of liquid fraction (a) average maximum stress and (b) average displacement at fracture.



Low liquid content of ~0.1%



Low liquid content of ~0.1%



High liquid content of ~3 %



High liquid content of ~3 %

Fig. 10. SEM pictures from fracture surfaces of Alloys 311 and 333 at different liquid contents (a) Alloy 311, fl=0.1%, (b) Alloy 311, fl=3%, (c) Alloy 333, fl=0.1% and (d) Alloy 333, fl=3%.



Fig. 11. Fracture profiles of Alloy 311 after 0.05 mm displacements for a specimen tested at liquid fraction of ~2% (a) and (b)  $\beta$ -Fe plates blocked the interdendritic channels at two different locations.







Fig. 12. SEM images from the fracture surfaces showing the spikes in (a) Alloy 311 and (b) Alloy 333 as well (c) the micro-necking of an  $\alpha$ -Fe bridge mixed with eutectic liquid in a not-fully-fractured sample (Alloy 333 with displacement of 0.08 mm).



Fig. 13. The semisolid tensile properties of Alloys 333 and 333-GR as a function of liquid fraction (a) average maximum stress and (b) average displacement at fracture.


Fig. 14. Stress-displacement curves for Alloys 333 and 333-GR (a) fl=1.7-1.8% and (b) fl=2.7-2.8%.



(a)





### 960 980 1000



#### 960 980 1000































# Displacement of 0.03 mm



Displacement of 0.05 mm



# Displacement of 0.07 mm









Displacement of 0.07 mm

20kU X1,700 10µm 16 60 SEI

**d**)

Displacement of 0.08 mm



Average Displacement at Fracture



# (a) Alloy 311

β-Fe

#### Al<sub>2</sub>Cu

20kU X1,200 10Mm 24 50 BES

# Low liquid content of ~0.1%

# (b) Alloy 311

 $-\beta$ -Fe



20kU X1,800 10µm



High liquid content of ~3%



#### Low liquid content of ~0.1%

# (d) Alloy 333

### Al<sub>2</sub>Cu in interdendritic channels

20kU

### 19 50 BES

α-Fe

# High liquid content of ~3 %

00 50 ym





		Contraction and March March 19	
(a) Alloy 333	Element	Wt.%	
	MgK	3.31	
	AIK	48.35	
	Si K	1.70	
	<u>Mn</u> K	0.76	
	Fe K	3.28	
	CuK	42.60	
1 Starten I			
- ( A ) KEY			
Element Wt.%			
Mg K 0.75	3	Sec. S	
- AIK 50.22	AV. Carrie	1 Miles	
Si K 0.71 B			
Cu K48.32 //			
		2-1	
		or t	
ZUKU	7 00	SEI	

#### (b) Alloy 311 Wt.% Element Mg K 1.98 AIK 63.92 Cu K 34.10 20kU X900 20µm 45 SEI 14
## (c) Alloy 333

20kU X1,700 10µm

16 60 SEI







