Grain size effects on the mechanical properties of some squeeze cast light alloys

T. M. YUE Dept of Manufacturing Engineering, Hong Kong Polytechnic, Hung Hom, Hong Kong H. U. HA Southampton Institute of Higher Education, East Part Terrace, Southampton, UK N. J. MUSSON

Pirelli Cables Ltd., Bishopstoke, Hampshire, UK

Mechanical property-grain size relationships have been examined for squeeze cast Al–4.5% Cu alloy, for an aluminium alloy with a composition corresponding to wrought 7010, and for a magnesium alloy AZ91. The general trend of the results obtained showed that the tensile properties and the fatigue strength improved as grain size decreased and the reverse was found to be the case for the fatigue crack propagation resistance and fracture energy of these castings. However, the results also showed that no simple common relationship existed between grain size and the tensile properties of the different alloys. The results are discussed in respect of their microstructures.

1. Introduction

The grain size effect on the mechanical properties of castings has never been unequivocally assessed due to inherent defects which make their own contribution and which tend to mask grain size-mechanical property relationships. In this respect porosity presents a major problem in assessing these relationships. Recent advances in the production of pore-free material has overcome this obstacle, and thus enabled the true mechanical properties of the cast microstructure, free of defects, to be determined.

The influence of grain size on the tensile properties of metals and alloys has been widely reported in the literature [1-5]. It is, perhaps, not surprising that it is those microstructures where grain boundaries represent the prime obstacles to dislocation movement that this influence can be most clearly demonstrated, and where the Hall-Petch relationship can be established. The presence in a microstructure of other dispersed features that also impede or modify dislocation movement must tend to reduce the grain size contribution. Furthermore, the presence of discontinuities such as pores, by virtue of their strength reducing effects, may swamp any grain size effects. The picture has been further complicated by the various shapes that porosity can take in different castings. Ruddle [2] has shown that the tensile properties of some Al-Cu alloys increased markedly with a decrease in grain size, but this might have also been due to changes in the form of the intergranular shrinkage porosity that related to these different grain sizes. The effect of porosity on the tensile properties of an aluminium alloy has been studied by Surappa et al. [6] and the results show that the properties depend mainly on the size of the dominant cavity, rather than the volume percent porosity obtained by density measurements. On the other hand, Khan and Murthy [7] have found a relationship between volume-percent porosity and tensile strength for an aluminium-10% magnesium alloy, the results revealing that even a minor amount of porosity decreases the tensile strength significantly. Apart from tensile property effects Chien [8] has found that the amount and shape of microporosity also affected the fatigue strength of a high strength Al-alloy. With respect to grain size effects themselves, Campbell [9] has suggested that grain refinement can produce only limited increases in the mechanical properties of aluminium alloy castings. However, for the alloys that he was considering, this could be due to the fact that silicon, which was the predominant phase present, was distributed mainly at dendrite boundaries and this phase was mainly responsible for the tensile properties developed. It should be noted in this context, that with most Al-Si casting alloys, the as-cast dendrite structure remained, even after heat treatment. It has been suggested that in Al-Si casting alloys, other microstructural features, such as the secondary dendrite arm spacing (SDAS) as well as the morphology of the silicon phase, are the prime governing features in controlling their mechanical properties [10].

The casting materials chosen for the present work do not retain a strongly developed dendrite pattern after heat treatment, and consequently have a well defined polygonal grain structure. With such characteristics it was anticipated that the effect of grain size on properties would be more clearly demonstrated. Nevertheless, as has been mentioned, with porosity present in castings, their strength and ductility dependence on grain size cannot be properly assessed, and thus this porosity must be eliminated if a true grain size effect is to be studied. In the past fifteen years it has been demonstrated that the squeeze casting process can produce 100% dense castings both with conventional casting alloys and with compositions of wrought forms [11, 12]. This paper presents some preliminary results obtained on the grain size/mechanical properties relationships of some squeeze cast aluminium and magnesium based alloys.

2. Materials

Three different alloys were used in this investigation, their nominal compositions are given as follows:

(i) A binary Al-4.5% Cu alloy;

(ii) A high strength Al-alloy with the 7010 composition (6.5 Zn-2.5Mg-1.6Cu), which in the wrought form is widely used in the aerospace industry; and (iii) A magnesium alloy AZ91 (9Al-1ZN).

These materials were squeeze cast in a 100 mm diameter cylindrical die under pressures between 50 MPa and 300 MPa. By varying the casting parameters a range of different grain size castings was produced. However, the SDAS of the cast microstructures were not greatly affected by the casting parameters being in the range of 20 μ m to 45 μ m, and, as has been mentioned earlier, these dendrite structures were largely eliminated by heat treatment.

3. Microstructure and heat treatment

Typical examples of the fully heat-treated grain structures of the alloys are shown in Figs 1 and 2. No porosity was observed in any of the castings produced. In both of the aluminium alloys used, fine cellular as-cast grains were obtained (Fig. 3) by selecting the correct casting parameters. Some of the castings were tested in their fully heat treated (FHT) conditions.

Heat treatment conditions: Al-4.5%Cu8 h at 535 °C (WQ)^a then For peak aged: 16 h at 140 °C (AC)^b ^aWQ cold water quench ^bAC air cool 7010 24 h at 475 °C (WQ) then For peak aged: 24 h at 120 °C (AC) For overaged: 8 h at 110 °C followed by 15 h at 175 °C (AC) AZ91 16 h at 420 °C (WQ) then For peak aged: 4 h at 205 °C (AC)

4. Results

4.1. Tensile properties

Tensile properties as a function of the inverse square root of the grain size of the squeeze cast materials are shown in Figs 4–6. The results show that no simple common relationship existed between the data from



Figure 1 Fully heat-treated A1-7010. (a) Coarse grain; (b) fine grain.



Figure 2 Fully heat-treated Mg-AZ91. (a) Coarse grain; (b) fine grain.



Figure 3 Fine grained as-cast Al-4.5 Cu structure.

the different alloys. The % elongation and ultimate tensile strength of the 7010 material, tested in the overaged condition, increased with decreasing grain size up to a critical grain diameter, beyond which no appreciable increase was obtained (Figs 4 and 6). The 0.2% yield strengths of the 7010 overaged alloy (Fig. 5) did not appear to change significantly with grain size nor did the as-cast Al–4.5% Cu material. The Al–4.5% Cu fully heat treated did show a change, but a well defined Hall–Petch relationship was only obtained for the AZ91 alloy.

4.2. Fracture toughness

A three-point bend technique using slotted rectangular bars [13, 14] was employed to determine the energy of fracture both of the AZ91 and 7010 materials. Although, the test itself may not provide a valid $K_{\rm IC}$ measurement, work by Ahmady and Kaufman [13, 15] have shown that there is an approximate correlation between unit energy of fracture and valid $K_{\rm IC}$ data.

The variation in energy of fracture per unit area with grain size for squeeze cast AZ91 and 7010 materials is shown in Fig. 7. The related fracture paths of the 7010 material are illustrated in Fig. 8. These figures reveal that, disregarding differences in grain size, the crack follows either an intergranular or interdendritic path. For the two alloys studied, the crack path of the coarse grained specimen was found always to have larger amplitude of deviation away from the plane of maximum tensile stress than that of the finer grain specimen.

4.3. Fatigue properties of the 7010 and AZ91 materials

Total fatigue life, in which the crack initiation period is of considerable significance, has been determined on plain fatigue specimens using a single rotating cantilever Rolls-Royce fatigue machine of reversed loading cycle, i.e. the stress ratio R = -1. The fatigue properties of the squeeze cast 7010 and AZ91 materials, both



Figure 4 Relationship between the elongation and the inverse square root of the average grain diameter of the squeeze cast materials. \bigcirc Al-4.5 Cu (as cast); \spadesuit Al-4.5 Cu (FHT); \square Al-7010 (overaged); \blacktriangle Mg-AZ91 (FHT).



Figure 5 Relationship between the 0.2% proof stress and the inverse square root of the average grain diameter of the squeeze cast materials. See Fig. 4 for key.



Figure 6 Relationship between the UTS and the inverse square root of the average grain diameter of the squeeze cast materials. See Fig. 4 for key.

in their peak aged condition, are presented as S/N curves in Fig. 9(a) and (b) respectively. There was a significant improvement in the fatigue strength of the two squeeze cast materials when the grain size was reduced. For both squeeze cast materials, transgranular cracks frequently changed direction at grain boundaries (Fig. 10). A change of fatigue cracking



Figure 7 Fracture energy versus the average grain diameter of Al-7010 and Mg-AZ91. \Box 7010 averaged; \bullet AZ91 peak aged.

mode was also observed, from being largely Stage I cracking in the coarse-grained structure (Fig. 11(a)), to predominantly Stage II cracking in the fine-grained structure (Fig. 11(b).

The fatigue crack propagation resistance of the squeeze cast material was assessed using the Pearson bend fatigue machine [16] with specimens containing an edge crack. In this study the stressing form of $1.22\sigma_a \pm \sigma_a$ was used. Since results of similar trends

were observed for both the 7010 and the AZ91 materials only the relationship between crack growth rate and stress intensity factor range for the 7010 material is presented (Fig. 12). The crack path in these two squeeze cast materials was primarily transgranular; however, the amplitude of crack deviation was much greater in specimens with a coarse grained structure.

5. Discussion

The experimental results show that the squeeze cast alloy of the 7010 composition, when tested in the overaged condition, had no yield strength sensitivity to grain size, and only slight tensile strength sensitivity.

The materials and conditions that did show a response to changing grain size were Mg-AZ91 (FHT) and Al-4.5% Cu (FHT). This behaviour implies that microstructures containing large amount of dispersed particles show little strength sensitivity to grain size. This is in agreement with the earlier premise that grain size effects are only likely to be found where the grain boundaries themselves represent the prime obstacle to slip deformation. In this case, the particles themselves replace the boundaries as the main obstacles of free dislocation movement.

Looking now at the Mg–AZ91 (FHT) which has a c.p.h. lattice with considerable slip anisotropy which implies inherent difficulty of slip transfer across grain



Figure 8 The fracture path of a 3-point bend specimen of Al-7010 material. (a) Coarse grain, (b) fine grain.



Figure 9 S/N curves for the FHT cast materials. (a) Al-7010; grain size \Box 70 µm, \bullet 800 µm; (b) Mg-AZ91, grain size \Box 346 µm, \bullet 543 µm.



Figure 11 Fracture surfaces of Al-7010 material tested in rotating bend fatigue, (a) Coarse grain; (b) fine grain.



Figure 10 Transgranular cracks were frequently interrupted by grain boundaries in Al-7010 rotating bend specimens (electropolished).

boundaries. The smaller the grain size the smaller is the free path for dislocation movement, and the more difficult transmission of plastic deformation to neighbouring grains will be. Thus this AZ91 material shows a close correlation between strength and grain size. Considering this in more detail, the linear increase in yield strength of AZ91 when plotted against $d^{-1/2}$ shows that for this c.p.h. alloy, a Hall–Petch model of strengthening applies; that is, dislocation pile-ups



Figure 12 Fatigue crack growth rate, da/dN, versus stress intensity factor, ΔK , in 7010–T6 Al-alloy. Grain size \blacktriangle 380 µm, \blacklozenge 70 µm.

occur at the principal obstacles in the microstructure – the grain boundaries – with the consequent production of long range internal stresses. A c.p.h. metal, with its restricted slip systems, does not have sufficient independent shear mechanisms to allow unrestricted change in shape of a polycrystalline aggregate. Thus, there is less likelihood of a contribution from secondary strengthening mechanisms which would obscure the simple grain size relationship.

The f.c.c. Al-4.5% Cu (FHT) also shows some sensitivity of strength to grain size in spite of the expected isotropic behaviour of the lattice. However, this material in the fully heat-treated condition will have a boundary precipitate network and an associated precipitate-free zone which results in characteristic boundary sliding coupled with difficulty of boundary penetration by dislocations. For the same reasons as for the magnesium base alloy, reducing the grain size will make boundary slip penetration more difficult and will thus enhance strength.

The strengthening in the f.c.c. Al-4.5% Cu alloy (FHT) does not follow a well defined Hall–Petch relationship, although yield strength does generally increase with decreasing grain size. In other work [17] observations on pure aluminium, with its less inhibited slip systems, revealed that considerable cross slip occurred particularly near grain boundaries, when subjected to tensile stress, and this was found to be more evident in the small grain size material than in the large. It is likely that this complex deformation strengthening obscured any grain size relationship, and the introduction of other microstructural obstacles, particles etc, will suppress it even further, as can be seen with the overaged 7010 and the as-cast Al–4.5% Cu alloy.

The effect of grain size on ductility has also been investigated and Fig. 4 shows that all of the materials tested showed increased ductility with reduced grain size. However, with what might be described as dispersed particle mictrostructures, for example overaged 7010 and as-cast Al-4.5% Cu, further improvement was negligible when the grain size was reduced below 0.25 mm. As mentioned earlier, cross slip was very much in evidence in the grain boundary region of pure aluminium and has similarly been observed in Al-4.5% Cu and 7010 alloy. AZ91 with its restricted slip systems would also be expected to exhibit more intense deformation in the grain boundary regions. Thus the smaller the grain size the greater the volume fraction of material undergoing cross slip for a given total strain, and the more homogeneous the deformation becomes. Fracture will be expected to occur after exhaustion of all possible modes of deformation, and the utilization of the total volume of material with potential for slip. The smaller grain size material will be more efficient in this respect and provide a greater total strain capability. By the same token, ineffectual use of the total potential slip volume in the material has limited the strength that can be developed. The c.p.h. metal with its restricted systems will, at all grain sizes, be disadvantaged with respect to ductility when compared with the f.c.c. metal.

Referring now to the fracture energy and the fatigue results, it might be expected that a fine grain size, which confers increased ductility, should also enhance fracture toughness in terms of fracture energy, but some researchers have shown this to be untrue [18]. The present work also shows that ductility and toughness are not directly related. The inferior fracture toughness of the finer grained material is believed to be due to the fact that a small grain size allows an intergranular crack to follow a less devious path when compared with the coarse-grained material. Any crack deviation will cause a decrease of the effective stress intensity at the crack front and will, therefore, increase the energy needed to propagate the crack.

The relatively poor fatigue strength observed in the coarse-grained squeeze cast material is considered to be the consequence of easy fatigue crack initiation. In the corase-grained squeeze cast material, because of the longer mean free path for slip, State I fatigue cracks can grow deeper into the test piece before any Stage II cracking develops (Fig. 11). Conversely, the relatively coarse-grained material has a better fatigue crack propagation resistance (lower da/dN for a given ΔK value) than the fine grain size material. This is thought to be due, as for the fracture energy case, to the greater deviations that the crack tip makes from the generalized fracture plane when compared with the finer grain size material. Again this results in a reduction in the energy per unit crack front length in the coarse-grained material. Work by Pitcher et al. [19] also found that the fatigue crack growth rates of some cast Al-alloys were significantly influenced by the nature of the crack path. Other workers [20, 21]have also shown that a tortuous fatigue fracture path will cause a reduction in the effective stress intensity factor range at the crack front.

6. Conclusions

1. A well defined tensile property/grain size dependence of the Hall–Petch type was found for squeeze cast magnesium alloy AZ91.

2. The tensile properties of squeeze cast Al-4.5% Cu alloy in the fully heat-treated state were grain size dependent, but not with a clearly defined Hall–Petch relationship.

3. The tensile properties of the squeeze cast 7010 composition aluminium alloy, with a more complex microstructure containing out-of-solution intermetallic particles showed virtually no tensile property/grain size dependence.

4. For squeeze cast aluminium alloy to show a well defined relationship between grain size and tensile properties there appear to be two main requirements:

- (a) That the grain boundaries should be the prime obstacles to slip in the microstructure and
- (b) There should be only limited secondary slip hardening, either by dislocation/dislocation or dislocation/particle interactions.

5. The fracture energy and fatigue crack propagation results confirm well-established principles that coarse microstructures, by imposing tortuous crack paths, exhibit higher crack resistance than those microstuctures where the crack path is more closely confined to the general crack plane. On the other hand, fatigue strength, on the basis of the S/N data, shows that fine-grained material has a higher fatigue strength than coarse-grained material due to the greater difficulty of fatigue crack initiation and early growth in small grains.

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