# THE EFFECT OF MECHANICAL MILLING, CONSOLIDATION ROUTE AND PROLONGED HEAT TREATMENT ON THE MICROSTRUCTURE AND PROPERTIES OF A RAPIDLY SOLIDIFIED Al-Mn-Si ALLOY

**R. HAMBLETON, H. JONES and W.M. RAINFORTH**

Department of Engineering Materials, University of Sheffield, Mappin Street, Sheffield, S1 3JD, UK

ABSTRACT Atomised powder of composition Al-8.3Mn-2.9Si (wt%) was consolidated by one of two routes; extrusion without prior milling or mechanical milling followed by forging. The microstructure and tensile mechanical properties of the specimens were investigated in the as-consolidated condition and after heat treatment for up to 3100h at 425°C. Variations in microstructure and properties, both as-consolidated and after heat treatment, are discussed with respect to the operative processing conditions and strengthening mechanisms.

**Keywords:** *Rapidly solidified Al-Mn-Si alloy, mechanical milling, microstructure, strengthening.*

## 1. INTRODUCTION

The use of conventional Al-based alloys for aerospace applications is limited due to the effect of temperatures above 150°C on their microstructure and mechanical properties. Novel processing methods such as rapid solidification and mechanical alloying have enabled the development of dispersoid-stabilised Al alloys with superior high temperature properties, for example the Al-Fe-V-Si alloys developed by Allied Signal in the 1980's [1]. Other alloy systems such as Al-Mn-Si have also been investigated [2] and our previous paper reported on the effect of silicon additions on melt spun Al-7.5 wt% Mn [3].

The present work reports the effects of consolidation and heat treatment on atomised powder of composition Al-8.3Mn-2.9Si (wt%). This was consolidated by extrusion without prior milling or by mechanical milling followed by forging. Extrusion was carried out at either 350 or 400°C and forging was carried out at either 400 or 495°C. Consolidated material was characterised by means of transmission electron microscopy (TEM) and by tensile mechanical property measurement both as-received and after heat treatment for up to 3100h at 425°C. The measured dispersoid and grain sizes and mechanical properties are discussed with respect to the processing conditions and the operative strengthening mechanisms in order to relate the microstructure and properties of the specimens.

## 2. EXPERIMENTAL PROCEDURES

Atomised Al-8.3Mn-2.9Si powder was produced by Metalloys and supplied in the as-consolidated condition by the DERA, Farnborough. The consolidation conditions used for each sample designation are given in Table 1.

Samples of size approximately 10x10x20mm were cut from forged specimens and from standard tensile test pieces machined from extruded material prior to heat treatment in air for 100, 1000 and 3100h at 425°C. Tensile mechanical properties were measured for all as-consolidated specimens and heat-treated extruded specimens. Vickers hardness measurements were made on all specimens using a 20kg load. TEM was carried out on a Philips EM420 operating at 100kV or a Jeol 200CX operating at 200kV on thin foils prepared by double jet electropolishing. Dispersoid sizes were measured from TEM negatives, using an average from at least 100 particles from a minimum of three regions on a sample.

| Designation | Mech. milling | Degas T (°C) | Extrusion T (°C) | Forging T (°C) |
|---|---|---|---|---|
| E350 | No | 375 | 350 | - |
| E400 | No | 425 | 400 | - |
| F400 | Yes | 400 | - | 400 |
| F495 | Yes | 495 | - | 495 |

Table 1 - Processing conditions for consolidation of atomised Al-8.3Mn-2.9Si powder

## 3. RESULTS

### 3.1 Microstructures

Figs. 1a & b show typical TEM micrographs of Al-8.3Mn-2.9Si as-consolidated at 400°C by extrusion and by forging, respectively. Average dispersoid and grain sizes as-consolidated and after heat treatment are given in Table 2. The microstructure of the extruded material is distinctly bimodal, consisting of fine regions with a high volume fraction of fine second phase particles (size~180nm) and coarse regions with fewer larger particles (size ~1μm) in the α-Al matrix. The fine dispersoids were identified as α-AlMnSi ($Al_{15}Mn_3Si_2$), while the larger particles were either α-AlMnSi or icosahedral phases. The forged material was generally homogeneous and contained a uniform dispersion of α-AlMnSi of size~250nm. E350 and F495 contained the same extruded or forged microstructures, although with different dispersoid sizes, the lower and higher processing temperature leading to a finer and coarser microstructure respectively. Some areas of α-Al that were free of dispersoids were seen in the forged specimens.

After time at 425°C, the average dispersoid size increased in the forged material and fine regions of the extrusions, as illustrated for F495 in Figs. 2a and b. As shown in Table 2, the average grain size was ~1 to 3μm as-consolidated. This grain size increased in the extruded material but remained approximately constant in the forged specimens during heat treatment at 425°C. Dispersoids were observed pinning dislocations in both as-extruded and as-forged conditions and the dislocation density, estimated to be ~$10^{13}$ lines/$m^2$, appeared to remain high after heat treatment for up to 1000h at 425°C (see Fig. 3).

| Specimen | E350 | | E400 | | F400 | | F495 | |
|---|---|---|---|---|---|---|---|---|
| Condition | d (μm) | 2r (nm) | d (μm) | 2r (nm) | d (μm) | 2r (nm) | d (μm) | 2r (nm) |
| As-consolidated | 0.8±0.2 | 111±11 | 1.5±0.3 | 182±11 | 2.1±0.5 | 250±12 | 2.7±0.5 | 551±22 |
| 100h at 425°C | 0.9±0.2 | 262±14 | 1.7±0.4 | 269±16 | 1.1±0.4 | 262±44 | 2.4±0.2 | 676±91 |
| 1000h at 425°C | 1.9±0.2 | 336±13 | 1.5±0.3 | 314±16 | 1.8±0.3 | 271±14 | 3.1±0.2 | 597±25 |
| 3100h at 425°C | 1.6±0.4 | 427±35 | 2.3±0.5 | 419±32 | 1.6±0.1 | 437±57 | 2.6±0.7 | 720±73 |

Table 2 - Average α-Al grain size d (μm) and α-AlMnSi dispersoid diameter 2r (nm) measured in Al-8.3Mn-2.9Si alloys, as-consolidated and after up to 3100h at 425°C.

### 3.2 Mechanical Properties

Tensile mechanical properties and Vickers hardness ($H_v$) measurements for as-consolidated and heat treated specimens are given in Tables 3 and 4. As-consolidated, $H_v$ and strength ($\sigma_{0.2}$ and UTS) of the forged material was higher than that of the equivalent extrusion, e.g. $\sigma_{0.2}$=334 and 214 MPa for F400 and E400 respectively. Higher processing temperatures led to lower $H_v$ and strength and higher ductility whether extruded or forged material, e.g. $\sigma_{0.2}$=334 and 182 MPa for F400 and F495 respectively. The elongation of 14% in as-consolidated E400 was significantly higher than that of 7% measured for F400. After up to 1000h at 425°C, the forged specimens had retained their

hardness (~100 and 80kg/mm$^2$ for F400 and F495 respectively), although these decreased to 75 to 70 kg/mm$^2$ after 3100h at this temperature. The hardness of the extruded material was reduced after only 100h at 425°C (e.g. from 113 to 86kg/mm$^2$ for E350), but it then remained similar after 1000h before reducing further to ~60kg/mm$^2$ after 3100h at 425°C. The ductility of the extruded specimens remained unchanged after 100h at 425°C, but decreased after 1000h at this temperature.

| Specimen | Condition | $\sigma_{0.2}$ (MPa) | UTS (MPa) | E (GPa) | % elongation |
|---|---|---|---|---|---|
| E350 | As-consolidated | 303±3 | 365±2 | 69±2 | 10.2±0.2 |
| | 100h, 425°C | 214±16 | 273±4 | 83±8 | 10.2±0.6 |
| | 1000h, 425°C | 204±7 | 250±2 | 74±1 | 8.5±0.1 |
| E400 | As-consolidated | 214±2 | 280±1 | 87±1 | 13.9±2.3 |
| | 100h, 425°C | 179±5 | 246±2 | 72±10 | 14.0±1.4 |
| | 1000h, 425°C | 171±2 | 239±2 | 78±4 | 9.8±0.8 |
| F400 | As-consolidated | 334±3 | 376±4 | 76±8 | 6.7±0.7 |
| F495 | As-consolidated | 182±2 | 259±3 | 66±7 | 12.5±1.3 |

Table 3 - Tensile mechanical properties of extruded and forged Al-8.3Mn-2.9Si, as-consolidated and after heat treatment for up to 3100h at 425°C.

| Condition | E350 | E400 | F400 | F495 |
|---|---|---|---|---|
| As-consolidated | 113±2 | 89±1 | 105±1 | 82±1 |
| 100h, 425°C | 86±1 | 80±1 | 104±1 | 80±1 |
| 1000h, 425°C | 80±1 | 76±1 | 102±1 | 81±1 |
| 3100h, 425°C | 61±1 | 62±1 | 76±9 | 68±1 |

Table 4 - Vickers hardness (kg/mm$^2$) of extruded and forged Al-8.3Mn-2.9Si, as-consolidated and after heat treatment for up to 3100h at 425°C.

## 4. DISCUSSION

The bimodal microstructure characteristic of the extruded Al-8.3Mn-2.9Si resulted from differences in microstructure of the fine and coarse powder particles that were consolidated to make the alloys. Large powder particles containing α-AlMnSi and icosahedral phase particles of size ~1μm form the coarse regions, while smaller powder particles, which would originally have had a microcellular microstructure, make up the fine regions. The extrusion temperatures allowed the transformation of the microcellular microstructure to the uniform dispersion of α-AlMnSi particles observed in the fine regions. The forged material was more homogeneous with a well-developed substructure due to the prior mechanical milling stage of its processing route. The large regions of α-Al remaining in some forged specimens are attributable to contamination of the mechanical milling apparatus.

The microstructural observations can be used to account for the observed differences in mechanical properties between the specimens and their response to time at elevated temperatures. The contribution $\Delta\sigma_{pt}$ to $\sigma_{0.2}$ from the silicide dispersion can be estimated from the Orowan equation [4]:

$$\Delta\sigma_{pt} = [MY\,Gb\,(1-0.2\upsilon)^{1/2} \ln(3.2\bar{r}/b)] / [2\pi\,(1-\upsilon^{1/2})(L - 1.6\bar{r})] \qquad (1)$$

where M is the Taylor factor (~3), Y ~ 0.9, G is the shear modulus of the matrix (~26GPa for Al), b is the Burgers vector (~0.286nm), υ is Poisson's ratio (0.345 for Al), $L$ is average centre to centre particle spacing and $\bar{r}$ is average particle radius. For randomly distributed spherical particles of

uniform radius r, $L$ is given by $1.23r\,(2\pi/3f)^{1/2}$ [5] where f is the volume fraction of particles. Taking f = 0.23 for αAlMnSi in Al-8.3Mn-2.9Si gives $\Delta\sigma_{pt}$ = 208 and 130MPa for E350 and E400 as-consolidated, from the values of r given in Table 2, accounting for up to two-thirds of measured $\sigma_{0.2}$. The residual contributions to $\sigma_{0.2}$ derive from Hall-Petch (grain size) and matrix contributions derived from solid solution and residual work hardening (dislocation density).

The Hall-Petch constant 2.1MPa $mm^{1/2}$ for Al [6] gives $\Delta\sigma_{gb}$ of 74 and 54 MPa for the grain sizes of E350 and E400 amounting to one quarter of measured $\sigma_{0.2}$, the balance being attributable to solid solution hardening and residual work hardening. Reductions in $\sigma_{0.2}$ on heat treatment for 100 and 1000h at 425°C are attributable mainly to reduction in $\Delta\sigma_{pt}$ resulting from coarsening of dispersoids with some effect also from reduced $\Delta\sigma_{gb}$ for E350, so that residual matrix hardening from dissolved solute and dislocation density then become more significant.

Although $\sigma_{0.2}$ was not measured for the forged samples, contributions to measured $H_v$ can be estimated using the relationship $H_v = \sigma_y/3$ [7]. On this basis the contribution to measured $H_v$ from Orowan hardening (eqn. (1)) is one-third or one quarter for F400 and F495 as-consolidated, while the contributions from Hall-Petch hardening amount to some 15 per cent, leaving more than half the contribution to $H_v$ to be provided by dissolved solute and dislocation density. A dislocation density, $\rho_d$ of 7 and $4\times10^{13}$ $m^{-2}$, similar to that observed, would account for a such a contribution to measured $H_v$ on the basis of $\Delta\sigma_{\perp\rho} = MYGb\rho_d^{1/2}$. Heat treatment for 1000h at 425°C did not reduce hardness of F400 or F495 significantly, which is consistent with retention of the dislocation density and only a small increase, if any, in dispersoid size and grain size.

## 5. CONCLUSIONS

- The Al-8.3Mn-2.9Si extrusions had a bimodal microstructure with regions of 'fine' (size ~180nm) and 'coarse' (size ~1μm) α-AlMnSi dispersoids. The α-Al subgrain size in the coarse regions was 0.8 to 1.5μm as-extruded. Both dispersoid and subgrain sizes were increased by heat treatment for up to 3100h at 425°C.
- The forged alloys had a more homogeneous and refined microstructure with increased $\rho_d$ and substructure compared with the extruded alloys. This was due to the introduction of a mechanical milling stage prior to forging. Homogenisation was not complete, with some isolated pockets of coarser dispersoids and large regions of α-Al from prior contamination of the mechanical milling apparatus. The as-forged α-Al subgrain size of 1 to 2μm in the forged alloys was not changed significantly by heat treatment for 3100h at 425°C.
- The main contribution to strength of the extrusions appears to have derived from dispersion (Orowan) plus Hall-Petch hardening whereas the strength of forged materials seems to be dominated by the dislocation density in the matrix generated by milling prior to forging and retained by the high volume fraction of dispersoids present.
- The reduction of hardness of the forged materials even after 1000h at 425°C is attributable to the stability of the dislocation structure in spite of a small increase in dispersoid size during this prolonged heat treatment.

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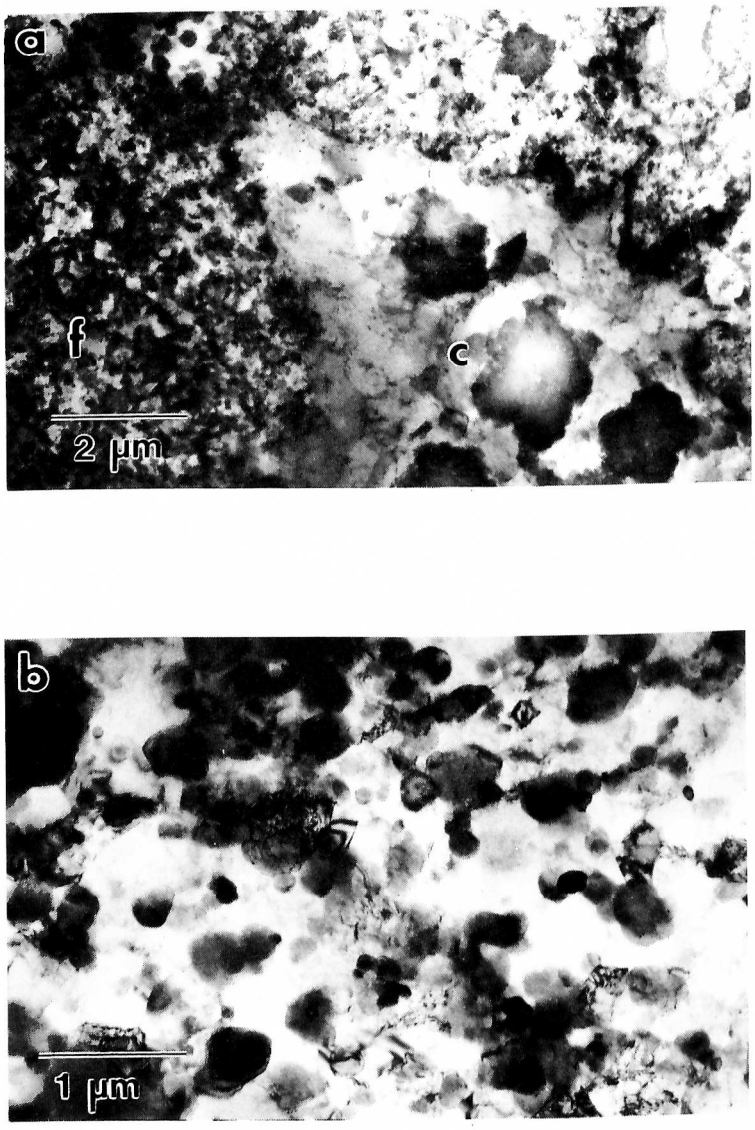


Figure 1 TEM micrographs showing (a) binodal microstructure of E400 exhibiting coarse (c) and fine (f) dispersoids compared with (b) more uniform F400, both as-consolidated.

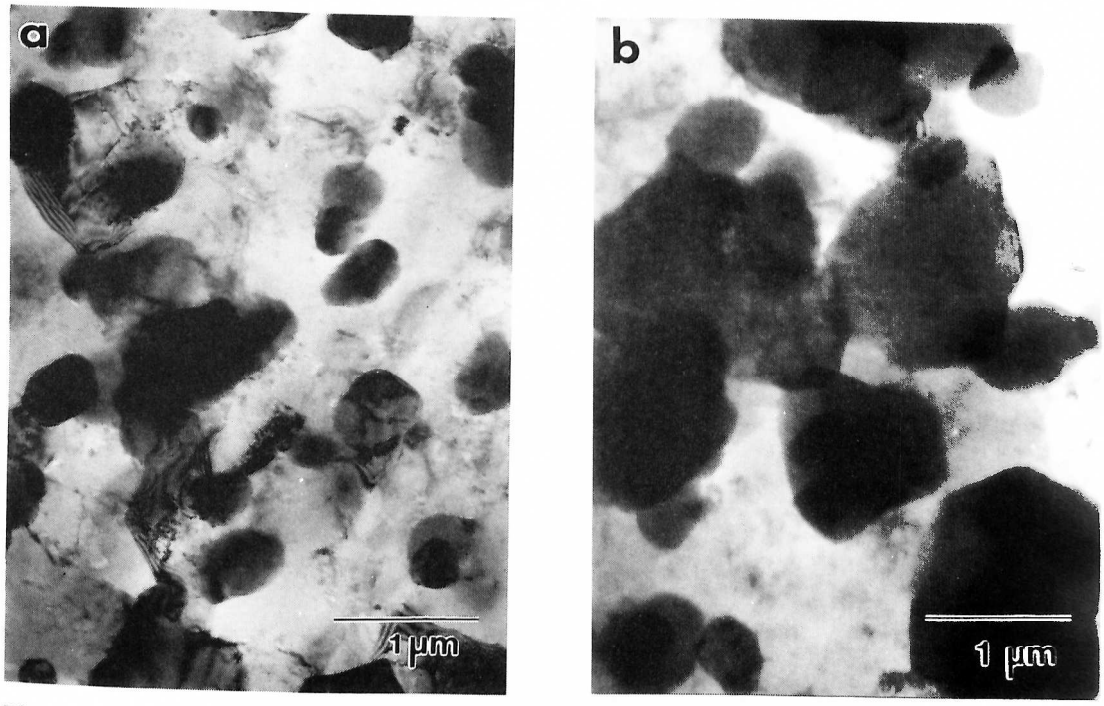


Figure 2 - TEM micrographs showing αAlMnSi dispersoids in F495 (a) as-forged and (b) after 3100h at 425°C.

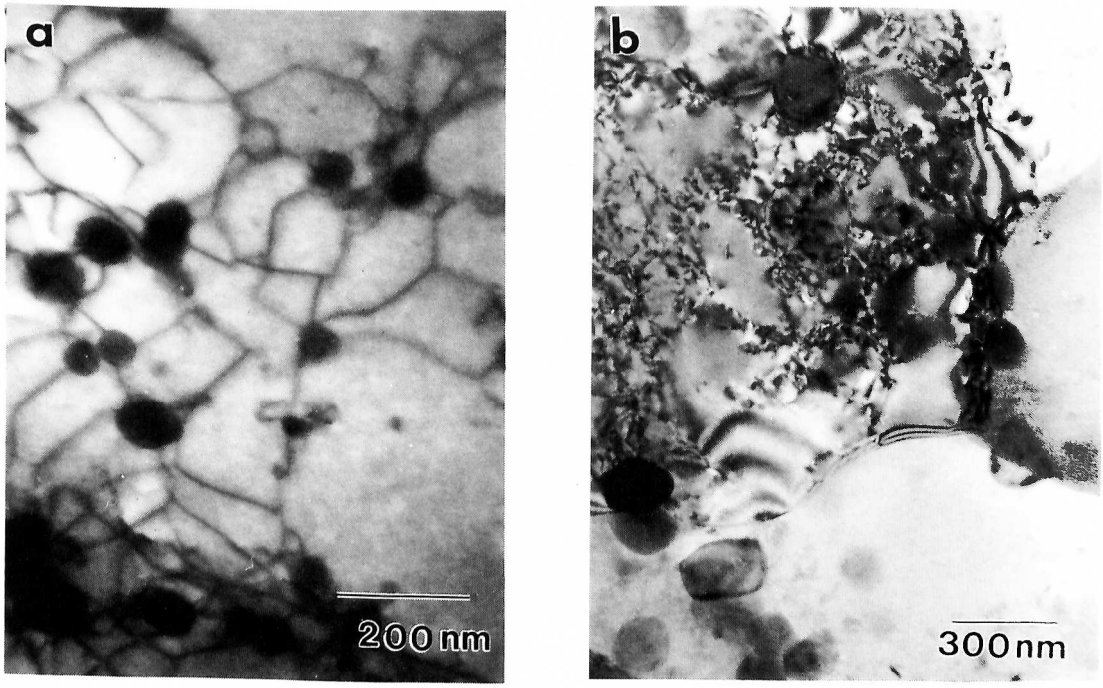


Figure 3 - TEM micrographs showing dislocations in (a) E400 after 100h at 425°C and (b) F495 after 1000h at 425°C.