

Creep Deformation Mechanism of Al-Mn Alloy Sheets with a Wide Range of Grain Size

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Creep performance of Al-1.05mass%Mn alloy sheets with various grain size has been investigated and correlated to the microstructural parameters such as average grain size, residual solute concentration in the matrix and volume fraction of Mn-containing compounds. The obtained grain size dependence of creep characteristics (e.g. rupture time and minimum creep rate) suggests that 50-70 μ m is an optimal grain size that shows the most excellent creep performance at 523K under 45MPa. The appearance of such an optimal size could be explained by inversely changing size dependence of two creep deformation mechanisms; i.e. *the strengthening by coarse grains* due to the retained solid-solution strengthening by supersaturated Mn and *the softening by coarse grains* due to the inhomogeneous deformation of the 2mm thick sheets with few grains in the thickness direction.

Keywords: Al-Mn alloy, creep characteristics, strengthening mechanism, microstructure, grain size

1. Introduction

Al-Mn alloys have been utilized as a fin material for heat exchangers due to their high formability, moderate mechanical strength and excellent corrosion resistance. The creep performance of the alloys is also important because heat exchangers are expected to be in service at more elevated temperatures. The authors [1] reported that prolonged heat treatment at 873K before blazing (e.g. 1000hr) results in longer rupture time and smaller minimum creep rate during creep of an Al-1.05mass%Mn alloy at 473-523K. The predominant strengthening factor was found to be *the strengthening by coarse grains*, rather than by solid-solution or dispersion strengthening, in marked contrast with that of 0.2% proof stress at room temperature. However, such a prolonged heat treatment at high temperatures is industrially impractical so that different routes to fabricate coarse grained sheets; e.g. *strain-anneal grain growth treatment* and *low-reduction rolling treatment*, are worthy of being developed.

In this work, creep characteristics of Al-1.05mass%Mn alloy sheets with various grain size have been investigated, and correlated to the microstructural parameters (i.e. average grain size, residual solute concentration in the matrix and volume fraction of Mn-containing compounds) in order to make clear whether similar improvement in creep characteristics could be achieved by the coarse grained sheets fabricated through *strain-anneal grain growth treatment* and *low-reduction rolling treatment*. The grain size dependence of creep performance was also clarified in conjunction with the creep deformation mechanisms for which microstructural evolution during creep and geometric relationship between average grain size and thickness of the creep test specimens were taken into account.

2. Experimental

The chemical composition of the alloy utilized in this work is listed in Table 1. Three different routes after hot-rolling were adopted to fabricate 2mm thick sheets with various grain size; i.e. (A) cold-rolled by 70-80%, annealed at 643K for 2hr and heat-treated at 873K for 1 to 1000hr (same as the

specimens in [1]), (B) cold-rolled by 70-80%, annealed at 643K for 2hr, stretched by 2.5-10% and heat-treated at 873K for 10hr (*strain-anneal grain growth treatment*) and (C) cold-rolled by 10 or 50%, annealed at 643K for 3hr and heat-treated at 873K for 10hr (*low-reduction rolling treatment*). Hereafter, the name of each specimen is regarded as A-1000hr, B-2.5%stretch+10hr, C-50%rolling+10hr and so on. The specimens for optical microscopy were prepared either by chemical etching using 0.5% hydrofluoric acid for observation of Mn-containing compounds or by anodic oxidation using a Baker's reagent (2.7% hydrofluoroboric acid) for observation of crystal grains. Optical microstructures were observed using a microscope and quantitatively estimated through image analysis software. Residual solute concentration of i in the matrix, C_i , was estimated from electrical conductivity (in %IACS) measured by an eddy current tester through the Matthiessen's law [1]. The specimens for creep test were machined from the sheets with a gage length of 30mm and a rectangular cross section of 5mmX2mm. Creep rupture test was performed in air by lever arm creep machines at $T=523\text{K}$ under $\sigma=45\text{MPa}$. The creep strain was measured every 10s using a linear gage to an accuracy of 1/1000mm.

Table 1 Chemical composition of the alloy utilized in this work (mass%).

Mn	Fe	Si	Cu	Ti	Zn	Mg	Al
1.05	0.61	0.24	0.15	0.02	0.02	0.01	Bal.

3. Results

3.1 Optical microstructures

Fig.1 shows optical microstructures of A-1000hr, B-2.5%stretch+10hr, C-50%rolling+10hr and C-10%rolling+10hr specimens. The distribution of crystal grains is well illustrated by the difference in polarization contrast, and thus the average grain size D can be clearly quantified. From the fact that all D values of B and C specimens are much larger than D of A-1000hr specimen, it was confirmed that both of *strain-anneal grain growth treatment* and *low-reduction rolling treatment* are easier methods to fabricate coarse grained sheets.

Fig.2 illustrates optical microstructure of each specimen showing distribution of Mn-containing compounds. Equiaxed compounds are observed with diameters of $<10\mu\text{m}$ in A-1000hr and B-2.5% stretch+10hr specimens, whereas elongated compounds with several tens of μm in longer axis are inhomogeneously distributed in C-50%rolling+10hr and C-10%rolling+10hr specimens. The Mn-containing compounds were identified by X-ray diffraction (XRD) analysis to be a mixture of $\alpha\text{-Al}(\text{Mn,Fe})\text{Si}$ and Al_6Mn phases, as expected from the phase diagram of Al-Mn-Si system at 873K [2]. As for residual Mn concentration in solid solution C_{Mn} , furthermore, because the final heat treatment at 873K was identically applied to all the specimens, C_{Mn} was limited to a narrow range from $\sim 0.3\text{mass}\%\text{Mn}$ (A-1000hr) to $<0.5\text{mass}\%\text{Mn}$ (C-50%rolling+10hr and C-10%rolling+10hr).

3.2 Creep characteristics

The creep curves at 523K under 45MPa are shown in Fig.3 for A-1000hr, B-2.5%stretch+10hr, C-50%rolling+10hr and C-10%rolling+10hr specimens. It is obvious that creep characteristics of B and C specimens are inferior to those of A specimen, as indicated by shorter rupture time t_r and larger minimum creep rate $\dot{\epsilon}_{\text{min}}$ (i.e. minimum slope of tangent in secondary creep region). This suggests that *strain-anneal grain growth treatment* and *low-reduction rolling treatment* are not as effective in improving creep performance as prolonged heat treatment at 873K (i.e. A-1000hr). Fig.4 illustrates the grain size dependence of t_r and $\dot{\epsilon}_{\text{min}}$ for all the investigated specimens crept at 523K under 45MPa. It is clarified from the positions of apexes on the convex curves that 50-70 μm is an optimal grain size that shows the most excellent creep performance for the 2mm thick sheets.

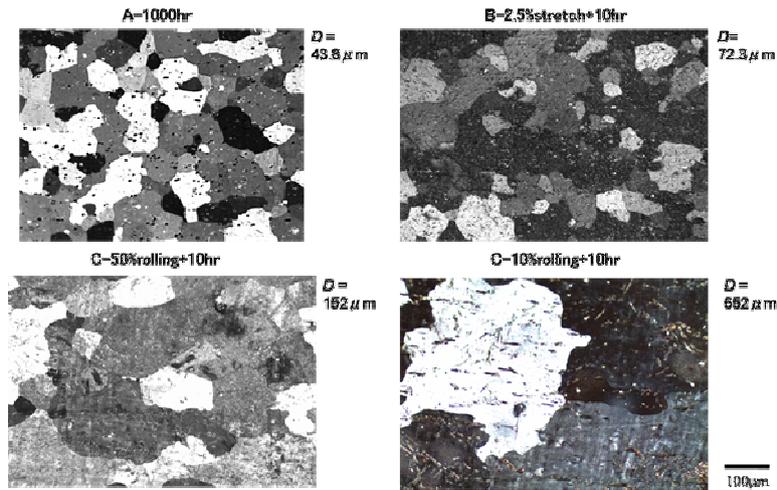


Fig.1 Optical micrograph of each specimen showing distribution of crystal grains.

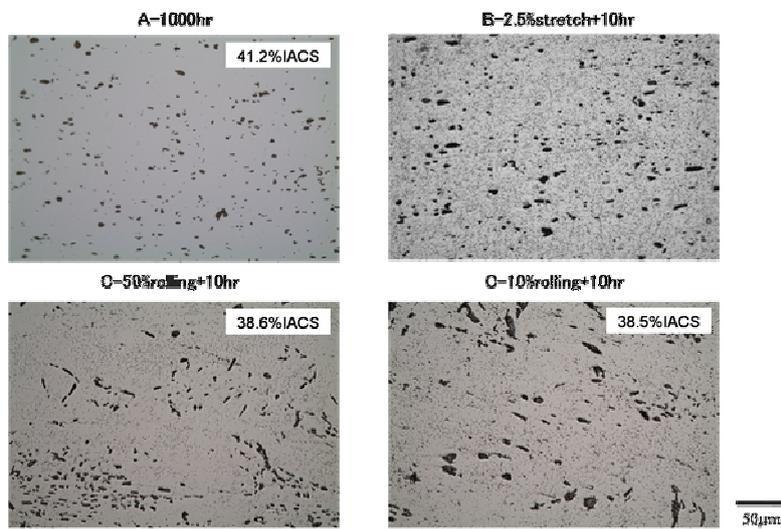


Fig.2 Optical micrograph of each specimen showing distribution of Mn-containing compounds.

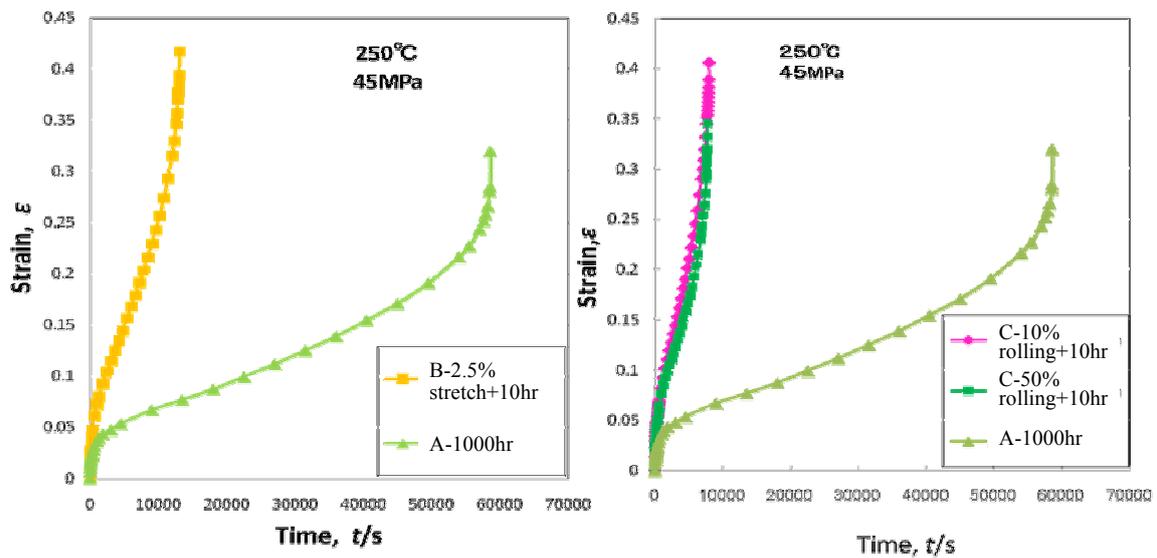


Fig.3 Creep curve of each specimen at 523K under 45MPa.

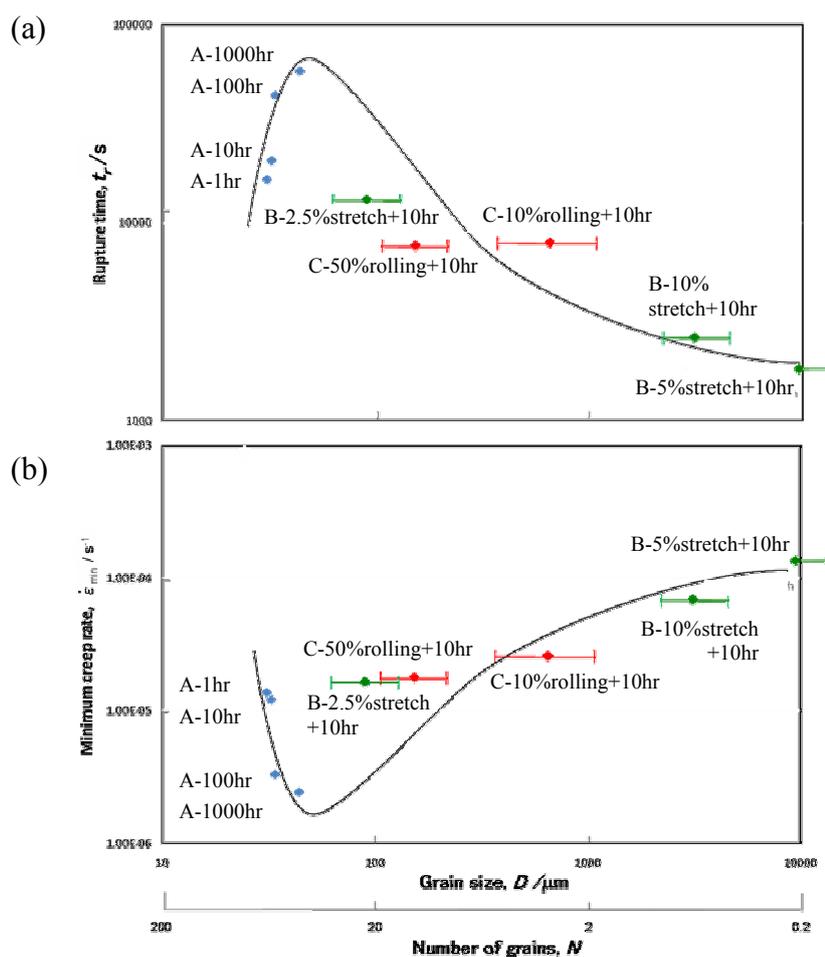


Fig.4 Grain size dependence of (a) rupture time t_r and (b) minimum creep rate $\dot{\epsilon}_{\min}$ for all the investigated specimens crept at 523K under 45MPa.

4. Discussion

Creep performance of Al-1.05mass%Mn alloy sheets was found to strongly depend on the average grain size within the investigated range of 30 μm to a few mm (Fig.4). Unfortunately, *strain-anneal grain growth treatment* and *low-reduction rolling treatment* were not effective in improving creep performance (Fig.3) although much larger grain size than that of A-1000hr specimen was successfully produced (Fig.1). However, 50-70 μm was found to be an optimal grain size that shows the most excellent creep performance, and thus becomes a target for any routes to fabricate 2mm thick sheets. The appearance of such an optimal size could be explained by inversely changing size dependence of two creep deformation mechanisms, as schematically illustrated in Fig.5.

In general, *the strengthening by coarse grains* (i.e. solid arrow in Fig.5) is believed to occur under the creep test conditions within diffusional creep or plasticity region of deformation mechanism maps [3, 4], whereas *the softening by coarse grains* (i.e. broken arrow in Fig.5) works mainly in dislocation glide region; e.g. Hall-Petch equation. The experimental conditions of this work; i.e. $T/T_m=0.56T_m$ and $\sigma/G\sim 2.1\times 10^{-3}$ (T_m : melting temperature, G : shear modulus), are likely to locate within dislocation glide region, or at least dislocation creep region, so that a new mechanism is needed to explain such a positive grain size dependence of creep performance (i.e. solid arrow in Fig.5) applicable even in the dislocation glide or dislocation creep region. Transmission electron microscopy (TEM) observation of microstructural evolution during creep suggested that large precipitates of Mn-containing compounds are formed along grain boundaries, leading to the

decreased contribution of solid-solution strengthening by Mn atoms in the matrix. This type of creep deformation mechanism will be feasible in the alloys with supersaturated solid solution at creep test temperatures, and therefore the coarse grained specimens are thought to possess few chances to form such grain boundary precipitates, resulting in the retained solid-solution strengthening by 0.3-0.5mass%Mn (Fig.2). Remember that all the specimens crept in this work possess supersaturated solid solutions because of rapid water-quenching after the final heat treatment at 873K.

On the other hand, *the softening by coarse grains* (i.e. broken arrow in Fig.5) is a result of the decreased number of grains existing along the thickness of creep test specimens. The horizontal axis in Fig.4 shows not only the average grain size D in the range of 30 μm to a few mm but also the number of grains N in the range of 66 grains to less than one grain for the 2mm thick sheets. The larger D values are, the more inhomogeneously the specimens deform because of the fewer grains in the thickness direction, resulting in the lower creep strength. By the combination of such two creep deformation mechanisms, therefore, the investigated Al-Mn alloy sheets are supposed to show the most excellent creep performance at a grain size of 50-70 μm .

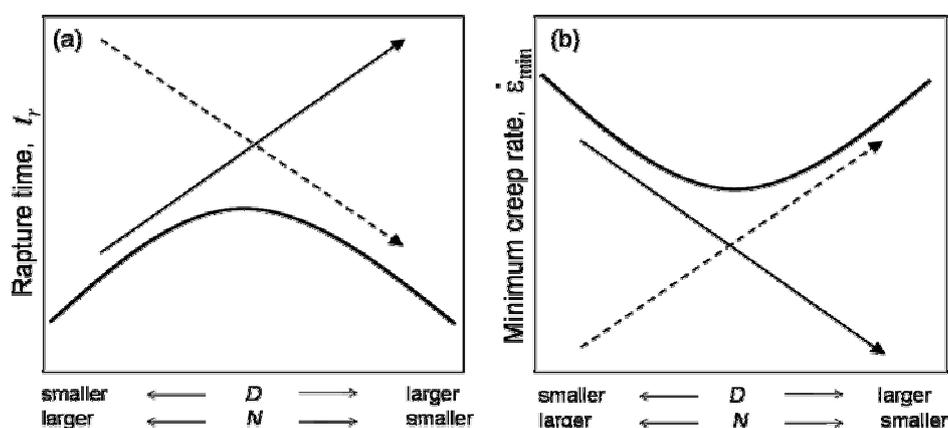


Fig.5 Schematic illustration of grain size dependence of (a) rupture time t_r and (b) minimum creep rate $\dot{\epsilon}_{\min}$ during creep of Al-Mn alloy sheets. Inversely changing size dependence of two creep deformation mechanisms is also shown by solid arrow for *the strengthening by coarse grains* or by broken arrow for *the softening by coarse grains*.

5. Conclusions

Grain size dependence of creep characteristics of an Al-1.05mass%Mn alloy has been investigated using the sheet specimens fabricated through three different routes; i.e. *prolonged heat treatment at high temperatures*, *strain-anneal grain growth treatment* and *low-reduction rolling treatment*. The optimal grain size within the investigated range of 30 μm to a few mm was found to be 50-70 μm at which the most excellent creep performance was obtained at 523K for the 2mm thick sheets. The appearance of such an optimal size could be explained by inversely changing size dependence of two creep deformation mechanisms; i.e. *the strengthening by coarse grains* which occurs in coarse grained specimens with supersaturated Mn and *the softening by coarse grains* at which the specimens are inhomogeneously deformed because of few grains in the thickness direction.

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References

- [1] S.Hirosawa, M.Ando, A.Niikura and Y.Suzuki: *Aluminum alloys, Vol.2*, Ed. by J.Hirsch, B. Skrotzki and G.Gottstein (Wiley-VCH, Weinheim, 2008), pp.1532-1538.
- [2] G.Effenberg: *COST507 Group B, Definition of thermomechanical and thermophysical properties to provide a database for the development of new light alloys, Critical evaluation of ternary systems*, 3 (1998), pp.201.
- [3] H.J.Frost and M.F.Ashby: *Deformation mechansim maps*, (Pergamon press, Oxford, 1982), pp. 26-28.
- [4] K.Kitazono: *J. Japan Inst. of Light Metals*, 59(2009), 458-463.