Proceedings of the 12th International Conference on
Aluminium Alloys, September 5-9, 2010, Yokohama, Japan

pp. 253-258

Effect of Homogenization Practice and Mn Content on the High Temperature Constitutive Behaviour of AA3xxx Aluminum Alloys

A.D. Kubiak[1], W.J. Poole[1] M.A. Wells[2] and N. Parson[3]

[1] Department of Materials Engineering, The University of British Columbia, 309-6350 Stores Rd., Canada, V6T 1Z4
[2]Department of Mechanical and Mechatronics Engineering, University of Waterloo, 200 University Avenue West Waterloo, Ontario, N2L 3G1, Canada
[3]Rio Tinto Alcan, Arvida Research and Development Centre (ARDC), 1955 Mellon Blvd, Jonquière, Québec, G7S 4K8, Canada

Various AA3xxx alloys (e.g. AA3003, AA3102) were homogenized in the temperature range of 500 - 600°C for various times to produce different volume fractions and distribution of dispersoids in the microstructure. After the homogenization treatment, the materials were compression tested in the temperature range of 300 to 600 °C with strain rates in the range of 10^{-1} to 10 s^{-1} to simulate hot extrusion conditions. The flow stress model based on the work of Chen and Kocks was found to well describe the experimental results for the steady-state flow stress. It was observed that the steady state flow stress was dependent on the manganese solid solution level and the distribution of the dispersoids. Finally, a series of trials was conducted on a fully instrumented extrusion press to determine the effect of homogenization treatment on the extrusion force. It was found that the extrusion force could be correlated very well with the steady-state flow stress values predicted by the Kocks/Chen model. A simple relationship was developed to predict extrusion pressure based on the prior homogenization treatment.

Keywords: *Homogenization, AA3xxx alloys, manganese, constitutive equation, extrusion, Gleeble 3500.*

1. Introduction

During complex manufacturing routes such as those used to manufacture heat exchanger tubes from AA3xxx series alloys, it is increasingly important to develop quantitative knowledge of the microstructure evolution at each stage of the process as well as the inter-dependencies between the processes. For example, a change in the homogenization parameters for an AA3xxx alloy (with the same chemistry) alters the microstructure which may in turn influence the flow stress and hence the extrusion pressure. An important aspect in being able to do this is understanding the range of microstructures that can be produced during homogenization of AA3xxx alloys and how these starting microstructures respond during subsequent deformation.

An important question that has received little attention in the literature is the effect of microstructure on the high temperature flow stress which is relevant to large strain deformation processes such as extrusion or rolling. In the current study the effect of homogenization practice and Mn level (0.26 – 1.27) on subsequent flow stress behaviour of AA3xxx alloys was studied. In addition, a constitutive model was developed to represent this behaviour using the frame-work for a physically constitutive model successfully developed by Kocks and Chen [1,2].

2. Experimental

The materials used in this study were laboratory DC cast extrusion billets with a diameter of 100mm. Three alloys were investigated: AA3102, an intermediate Mn composition alloy, and AA3003. As-cast billets were provided by the Arvida Research and Development Center (ARDC) of Rio Tinto Alcan located in Quebec, Canada. The chemical composition of the alloys studied is given in Table 1. Laboratory samples for homogenization and subsequent deformation were taken from the as-cast billet at a sub-surface location to avoid the area of inverse segregation. The compression samples

Table 1 Chemical composition of alloys used in experiments (wt %).

Alloy	Mn	Fe	Si	Ti	Al
AA3102 (Low Mn)	0.26	0.50	0.10	0.02	bal.
Med Mn	0.75	0.52	0.10	0.02	bal.
AA3003 (High Mn)	1.27	0.54	0.10	0.02	bal.

were taken along the axial direction of the billet such that the compression direction was oriented parallel to the casting direction.

Homogenization treatments were done for a variety of temperatures (500-630 °C) and times (1-24 hrs) to create a range of starting microstructures for compression testing. In addition some as-cast samples were tested with no homogenizaiton treatment. All homogenization heat treatments were performed with a heating rate of 150˚C/h and a heating rate of 50˚C/h for the last hour (to model industrial practices). Samples were water quenched after soaking.

Electrical conductivity (converted to resistivity) was measured intermittantly during each homogenization treatment to infer changes in the microstructure during homogenization. These measurements were made using a Sigmatest® 2.069 with an 8 mm probe. Compression tests were done using the Gleeble™ 3500 thermal-mechanical simulator at the University of British Columbia. Compression samples were heated at 5°C/s and held at the deformation temperature for 3s. Deformation occurred at temperatures of 300-600°C and strain rates of 0.1, 1, and 10/s.

In order to validate the constitutive equation developed, extrusion trials were performed using an instrumented research extrusion press at the ARDC. Billets with a range of homogenization conditions and starting chemistries were induction heated and trials were performed at 400°C and 550°C. The die was preheated to a temperature of 450°C and 480°C for the 400°C and 550°C extrusion trials respectively. In all trials the extruded strip had an I-beam profile with an extrusion ratio of 130. The extruded strip had an exit speed of 1.8 m/s and was water quenched after extrusion.

3. Results and Discussion

3.1 *Microstructure evolution during homogenization*

The evolution of resistivity (normalized based on the starting resistivity) during a 600°C homogenization for the three alloys used in this study is shown in Figure 1. The alloys show little change in resistivity when heated from room temperature to 300°C. Resistivity quickly decreases to a minimum at around 520°C and then increases. Resistivity then begins to decrease upon the completion of heating. During soaking resistivity initially decreases quickly but then levels off. The resistivity curves for the high Mn and medium Mn alloys are similar while the low Mn alloy resistivity curve has a noticeably different shape, increasing with soak time.

Resistivity measurements and optical micrographs for the three alloys suggest that homogenization at high temperatures (such as 600°C) over long periods of time allows both short and long range diffusion to occur. For all three alloys, the constituent particles grew and coarsened with soak time. Dispersoids were observed for the high Mn and medium Mn alloys and were seen to precipitate, then coarsen and dissolve with soak time. No dispersoids were observed during homogenization for the low Mn alloy, but the resistivity results from Figure 1 suggest that dispersoid formation does take place but that is not visible via optical metallography. The nominal Mn content of the alloy had an effect on the constituent particle morphology and the amount of dispersoids observed at a given soak time. An increase in the nominal Mn concentration appeared to give a larger number of constituent particles and dispersoids. The amount of Mn in solid solution slowly decreases with soak time as shown by the slow decrease in resistivity; however, this was not observed for the low Mn alloy. The low Mn alloy experiences an increase in resistivity with soak time which would suggest that the

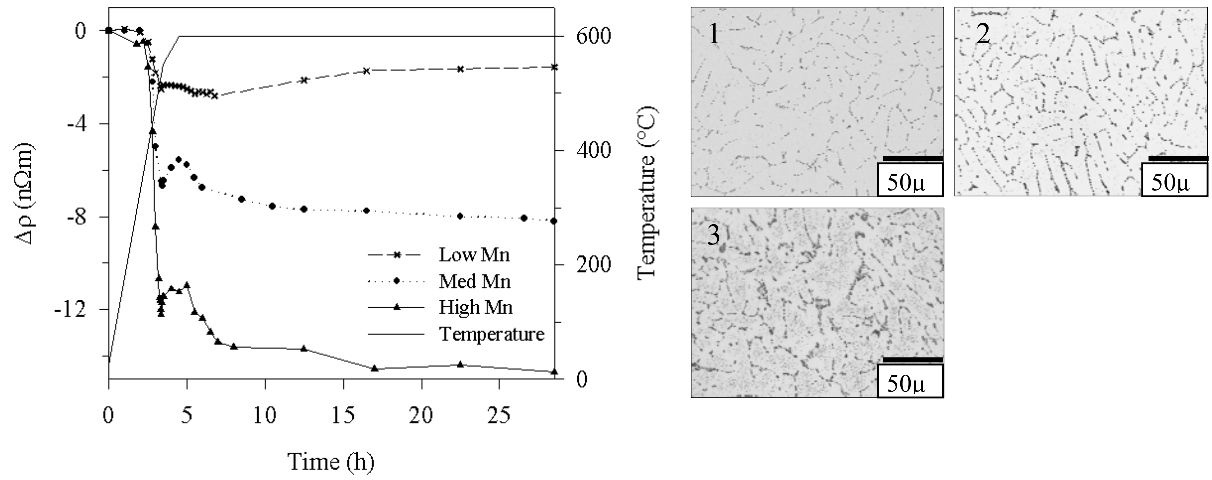

Figure 1 Measured resistivity during homogenization of alloys. Base values for Low, Med, and High Mn alloys are: 37.6, 49.5, and 61.9 nΩ·m, respectively. 8 hr soak microstructure: 1) Low, 2) Med, and 3) High Mn.

amount of Mn in solid solution is increasing, however, this seems unlikely. At the current time, the resistivity behaviour of the low Mn alloy cannot be explained and future work will need to be done to determine the cause of this phenomenon.

3.2 *Compression Test Results*

Compression test results for the high Mn alloy deformed at 500°C with a strain rate of 1/s are shown in Figure 2. Homogenization treatments can be seen to decrease the flow stress substantially as compared to the as-cast alloy without any homogenization. The 500°C (8 hr) homogenization treatment has a significantly higher flow stress than the other conditions, with values closer to the as-cast state. Under these deformation conditions, the 600°C (24 hr) has the lowest flow stress. At large strains the 550°C (8 hr), 600°C (24 hr), and 630°C (8 hr) flow stress curves converge while the 500°C (8 hr) remains relatively unchanged.

The flow stress was averaged between 0.4 and 0.6 strain to give the steady state flow stress or flow stress for each compression test. It was found that an increase in homogenization temperature and/or time may decrease flow stress values however this is not always the case. A complex relationship between the evolution of homogenization microstructure and the corresponding effect on flow stress values was seen to exist.

3.3 *Constitutive Model*

The Kocks and Chen [1,2] physically-based constitutive model applies to the viscous dislocation motion regime, characterized by a class of materials and conditions distinguished on the microscopic scale by the continuous motion of dislocations accompanied by diffusion of solute, i.e..

$$\dot{\varepsilon} = A\left(\frac{\sigma}{\mu}\right)^n \frac{\mu b^3}{kT}\exp\left(-\frac{Q_D}{RT}\right) \qquad (1)$$

The activation energy for diffusion of Mn, Q_D, was taken as 211.4 kJ/mol. [3]. The high Mn alloy flow stress results plotted using the Kocks-Chen constitutive model are given in Figure 3. Model fitting parameters (A and n) were found using a least squares fit to the measured data. Table 2 lists the calculated n and A parameters determined for each alloy and homogenization condition.

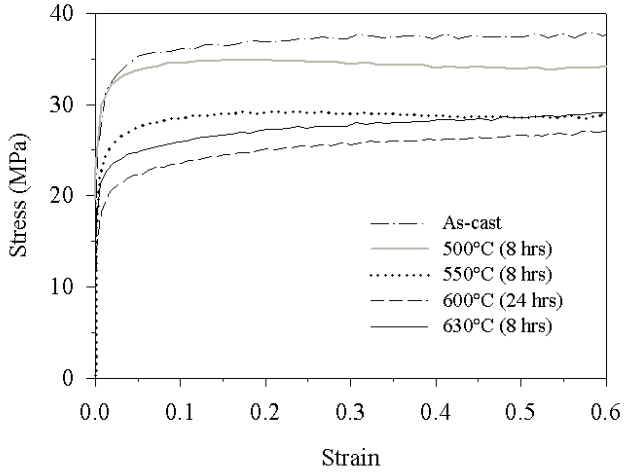

Figure 2 Comparison of homogenization treatment flow stress results for the high Mn alloy with a deformation temperature of 500°C and a strain rate of 1/s.

The model is valid over the temperature range of interest in this study (400 to 600°C) and can be extended to temperatures of approximately 350°C (for a strain rate of 1/s).

Table 2 Calculated stress exponent values for each alloy studied.

Homogenization Treatment		Low Mn		Medium Mn		High Mn	
Time (hr)	Temp (°C)	n_{flow}	A (s^{-1})	n_{flow}	A (s^{-1})	n_{flow}	A (s^{-1})
As-Cast	N/A	7.0	$9.10\text{x}10^{32}$	8.0	$8.84\text{x}10^{34}$	8.1	$4.53\text{x}10^{34}$
8	500	8.3	$1.05\text{x}10^{37}$	9.2	$1.07\text{x}10^{39}$	10.5	$4.87\text{x}10^{41}$
8	550	8.0	$2.04\text{x}10^{36}$	8.6	$5.72\text{x}10^{37}$	9.7	$1.04\text{x}10^{40}$
1	600	7.2	$6.28\text{x}10^{33}$	8.0	$6.07\text{x}10^{35}$	8.8	$3.67\text{x}10^{37}$
24	600	7.0	$3.15\text{x}10^{33}$	7.3	$1.38\text{x}10^{34}$	8.1	$1.06\text{x}10^{36}$
8	630	7.0	$2.27\text{x}10^{33}$	7.3	$9.00\text{x}10^{33}$	8.0	$3.41\text{x}10^{35}$

A comparison of the effect of homogenization treatment on the constitutive behaviour (flow stress) for the high Mn alloy is shown in Figure 3. As can be seen, the as-cast material has the highest flow stress values whereas the material given the 600°C (24 hr) homogenization has the lowest flow stress. Assuming that flow stress and extrusion pressure have a direct relationship, the model can compare relative extrusion pressures for the homogenization treatments. For each deformation temperature, flow stress appears to initially decrease with increasing homogenization temperature but subsequently increases or levels off.

Homogenization conditions where dispersoids were observed/expected ($8.6 \leq n \leq 10.5$) have different *n* values and cannot be directly compared to the solid solution curves. Quantifying the effect of dispersoids on flow stress requires further experimental measurements of dispersoid number densities and sizes. The physical basis of the Kocks Chen model requires stress exponent values of or close to 3. Stress exponent values determined in this study do not satisfy the model microstructure component, making the model empirical and suggesting an alternative deformation mechanism(s) to that experienced by the 5182 alloy in the Kocks and Chen study. However, application of the physically based model to this study does provide a good mathematical representation of the experimental data.

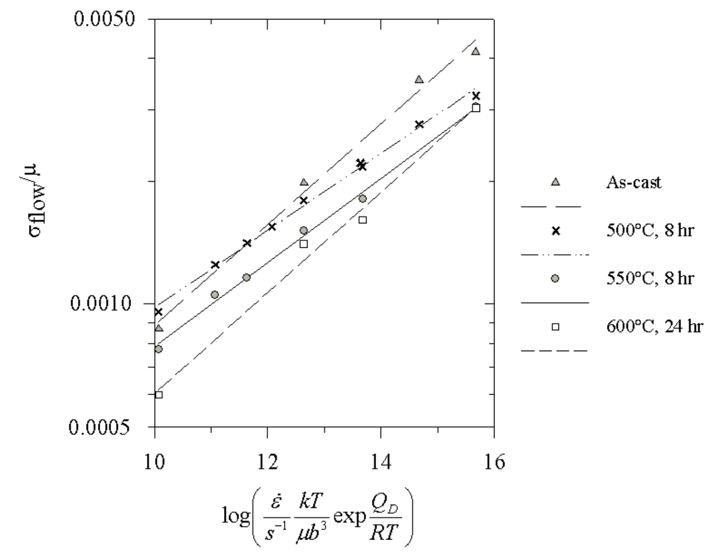

Figure 3 Constitutive model predictions (lines) using values given in Table 2 as compared to measurements (symbols) for the high Mn alloy for a range of homogenization treatments.

3.4 *Extrusion Trials*

A series of extrusion trials were run using the instrumented press at the RioTinto Alcan research centre in Jonquiere, Canada. Billets with different starting chemistries and homogenization treatments were extruded at a mean strain rate of 4/s at two different extrusion temperatures (400°C and 550°C). It is expected that there would be a direct relationship between the measured extrusion force and the material flow stress, hence knowledge of the constitutive behaviour and how the material flow stress varies as a function of chemistry and homogenization heat treatment is critical. A comparison of the measured extrusion force as compared to the calculated flow stress for these trials in shown in Figure 4. As shown in Figure 4, there is a linear relationship between the extrusion force and material flow stress. As expected, the largest effect on extrusion force occured as a result of the change in extrusion temeprature (400°C (open symbols) versus 550°C (closed symbols)). Alloy chemistry also had a significant effect on the extrusion pressure as evidenced by the range of flow stresses/extrusion forces at each extrusion temperature.

Referring to Figure 4, the biggest effect on extrusion force, for the conditions studied, is the extrusion temeprature followed by the Mn level and finally the starting microstrucutre as determined by the homogenization condiditon. The effect of homogenizaiton condiditon is shown by the variation in the data for a given Mn level and extrusion temperature, with the as-cast alloy typically exhibiting the highest extrusion force and the alloy homogenized at 600°C for 24 hrs showing the lowest. The calculated flow stress data correlates directly to the extrusion force measured during the industrial trials validating the previous assumption that the effect of homogenization on extrusion forces/pressures can be quantified by the effect homogenization has on flow stress values.

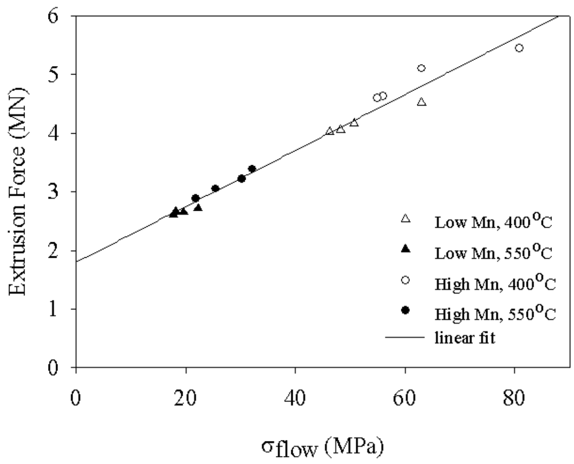

Figure 4 Measured extrusion force as a function of calculated material flow stress.

4. Conclusions

1. An increase in homogenization soak time increases constituent particle size and decreases number density; the observation was independent of alloy Mn content. A decrease in alloy Mn content decreased dispersoid number density (at a given soak time) as did an increase in soak time.
2. Flow stress results can be accurately described using the physically-based constitutive model at temperatures and strain rates in the industrial extrusion range (400 to 600°C). The effect of homogenization and alloy content (Mn) on constitutive behaviour was quantified.
3. The starting microstructure of the material prior to hot deformation has a significant impact on the flow stress. Microstructures with a large density of dispersoids increase stress exponents and flow stress values, consistent with climb controlled plasticity. In the absence of large dispersoid densities, flow stress values increase linearly with estimated Mn in solid solution.
4. The effect of homogenization on extrusion pressure can be quantified using flow stress results allowing the constitutive model to be used in optimizing the extrusion process.

Acknowledgements

The authors would like to gratefully acknowledge the financial support of NSERC and Rio Tinto Alcan.

References

[1] U.F. Kocks and S.R. Chen. *Aspects of high temperature deformation and fracture of in crystalline materials,* Ed. Y. Hosoi, H. Yoshinaga, H. Oikawa and K. Maruyama, (Japan Institute of Light metals, 1993), pp. 593-600.

[2] S.R Chen, M.G. Stout, U.F. Kocks, S.R. MacEwen, and A.J. Beaudoin. *Hot Deformation of Aluminum Alloys Ii*, Ed. T.R. Bieler, L.A. Lalli and S.R. MacEwen (TMS, 1998) pp. 205-216.

[3] Q. Du and A. Jacot. Acta Mater. 53, (2005) 3479-3493.