Effect of Mg₂Si Phase on Extrusion of AA6005 Aluminum Alloy

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Abstract

The extrusion behavior and mechanical properties of aluminum alloys are very sensitive to the microstructure of the billets after homogenization. In order to optimize the homogenization practice for the extrusion process, the influence of different cooling rates from soaking temperature during homogenization was investigated for an AA6005 alloy. The specimens were soaked at 580°C for 8 hours, and then cooled by 3 different cooling rates: water quenching, air cooling and furnace cooling. The homogenized microstructures from specimens exhibit an increase in both amount and size of Mg2Si particles with a decreasing cooling rate. The hardness at room temperature and the flow stress at preheating temperature (450°C) and real extrusion temperatures (500-550°C) also generally decrease with a slower cooling rate. However, re-precipitation of Mg₂Si is observed upon reheating to 450 °C and dissolution is observed at 500 °C and 550 °C. A fast cooled billet is identified to have fully solutionized microstructure but requires higher extrusion pressure and is sensitive to preheating rate, while a slow cooled billet is easier to extrude but is not sufficiently solutionized after extrusion.

Introduction

Homogenization treatment for aluminum alloys is aimed to modify the as-cast microstructure of the billet so that the extrudability is enhanced in terms extrusion pressure, extrusion speed, surface finish and mechanical properties of the final products^[1]. This treatment includes a soaking procedure at an elevated temperature and a cooling step at some controlled rate.

Aluminum alloys of AA6XXX series are universally adopted in construction applications and automobile industry. As for the homogenization treatment of the 6XXX alloys, the soaking procedure is meant to dissolve the large Si and Mg containing precipitates into the Al matrix, and to transform the plate-like β - AlFeSi intermetallic particles into the more rounded α -AlFeSi particles^[2-4]. The cooling practice, on the other hand, determines the precipitation behavior of Mg₂Si, and thus has a considerable influence on the following extrusion performance of the billet and the mechanical properties of the final product, which is the subject of this paper.

Large amounts of coarse β -Mg₂Si particles tend to precipitate as a result of a slow homogenization cooling rate. During extrusion, the undissolved β -Mg₂Si particles in the billet can lead to incipient melting and surface defects, which seriously limits the extrusion speed^[5, 6]. Nevertheless, a fast-cooled billet with fully solutionized Mg and Si is not desired either, because the solid solution strengthening effect would lead to a considerable rise in flow stress during extrusion, making the billet difficult to extrude^[6-8]. Generally, such a microstructure is desired that the Mg₂Si particles mostly remain undissolved after preheating but become readily dissolved upon deformation. Therefore, a tradeoff is necessary to modify the cooling rate during homogenization. The metastable β '-Mg₂Si, resulted from some intermediate cooling rate, has been reported to be favorable for improved extrudability^[2, 9, 10].

In the current research, by adopting different cooling practices after the same soaking treatment, specimens of different microstructures and mechanical properties were obtained for an AA6005 alloy. The effects of different cooling rates on Mg₂Si precipitation, and thus on the extrudability of the specimens were investigated by measuring the flow stress at a typical preheating temperature and at the extrusion temperature range.

Experimental

The starting material was an AA6005 alloy donated by Nanshan America in the form of extrusion butts from an actual production run, with a diameter of 310mm. From these, small specimens with dimensions of $8 \text{mm} \times 8 \text{mm} \times 12 \text{mm}$ were machined for further heat treatment and mechanical testing.

Table 1. The Average Cooling Rates from 500 ℃ to 200 ℃ of Different Cooling Practices

Water Quenching	Air Cooling	Furnace Cooling
~500000°C/h	3000°C/h	130°C/h



Fig. 1 Cooling curves of the specimens after soaking at 580°C for 8 hours

During the homogenization treatment, the specimens were heated to 580 °C, isothermally soaked for 8 hours and then cooled to room temperature by: (i) water quenching, (ii) air cooling, and (iii)

furnace cooling. The temperatures of the specimens during cooling were directly recorded by thermocouples. The cooling curves are plotted in Figure 1 and the average cooling rates between 500 $^{\circ}$ C and 200 $^{\circ}$ C are listed in Table 1. Furnace cooling was intended to involve an extremely slow cooling rate, air cooling also took a rather rapid cooling rate, while water quenching served as the opposite extreme.

After homogenization, the specimens were prepared and etched with Keller's reagent according to standard methods for optical metallographic observation of the Mg₂Si particles. The microstructures of the homogenized specimens were observed under an Olympus BX41M optical microscope. Quantitative analysis was carried out for the size distribution of the Mg₂Si particles. In addition, micro-hardness tests were undertaken on the homogenized specimens using a LECO LM247AT micro-hardness tester. The Vickers hardness of the specimens at 25g under room temperature was measured.

The flow stress at preheating and extrusion temperatures was measured to examine the influence of cooling rates on extrusion behavior. The homogenized specimens underwent compression tests on MTS Insight 100SL testing system at the billet preheating temperature (i) 450 °C and the real extrusion temperatures (ii) 500 °C and (iii) 550 °C, with a heating rate of 0.7 °C/s. The temperatures of 500 °C and 550 °C represent the temperature increase during extrusion due to the work of deformation. The 0.2% engineering flow stress of the specimens at a strain rate of 0.1/min was obtained. After compression tests, the specimens were quickly quenched in water to room temperature in order to mostly preserve the microstructure at the test temperatures, and these specimens were then examined under optical microscope.

Results and Discussion

A. Homogenized Microstructure

The microstructures of the specimens subject to different homogenization cooling practices are illustrated in Figure 2. The microstructure consists of the aluminum matrix, the ironcontaining intermetallic particles and the Mg₂Si precipitates. The coarse particles distributed along grain boundaries are Al-Fe-Si intermetallic particles, which are not substantially influenced by different homogenization cooling rates. The smaller-sized particles spread within grains are Mg₂Si particles precipitated during the cooling procedure, and are significantly varied according to different cooling rates, as is revealed in the microstructures.

In the water quenched specimen, the amount and size of the Mg₂Si particles are both relatively small. During this rapid cooling process, Mg and Si are retained in the supersaturated Al matrix to a great extent and only a few Mg₂Si precipitates are observed in Figure 2(a). The small amount of Mg₂Si particles also suggests that Mg and Si are sufficiently solutionized into the matrix after the 8-hour soaking at 580 °C. For a slower cooling rate of 3000 °C/h fulfilled by air cooling, there is an obvious increase in the population of the Mg₂Si particles, and some larger sized Mg₂Si precipitates can be observed as well. The slow furnace cooling rate of 150 °C/h results in an even greater number of Mg₂Si precipitates and some larger sized particles.

The quantitative analysis of size distribution of the Mg₂Si particles, which is given in Figure 3(a), shows a tendency of coarsening with a decreasing cooling rate. It is also notable that large amounts of small sized Mg₂Si particles exist in all the three specimens. However, due to the limitation of optical microscopy, particles smaller than 0.1 μ m were not characterized in this analysis.



Fig.2 Microstructures of the homogenized AA 6005 specimens soaked at 580 °C for 8 hours with different cooling practices of (a) water quenching (~500000 °C/h), (b) air cooling (3000 °C/h) and (c) furnace cooling (130 °C/h)

For supplementation, computer simulation of the nucleation, growth and coarsening of the Mg₂Si particles is carried out with the experimental cooling rates, based on a non-isothermal precipitation model^[11], and the resulting size distribution of the Mg₂Si particles are shown in Figure 3(b). According to the simulation results, for the water quenched specimens, the Mg₂Si particle sizes mostly fall into the nanometer range, which is beyond the visibility of optical microscopy. However, for the air

cooled and the furnace cooled specimens, the bimodal distribution of Mg₂Si particle size given by simulation generally coincides with the real experiment results. For the air cooled specimen, the second peak corresponds to a particle size of 0.25 μ m from microstructure characterization, and 0.05 μ m from computer simulation. For the furnace cooled specimen, the corresponding particle sizes for the second peak are both 0.25 μ m.



Fig. 3 Size distribution of the Mg₂Si particles for AA 6005 alloy (a) from real specimens cooled from soaking temperature with different rates and (b) from simulation results with the same corresponding cooling rates

During the cooling process, the nucleation of Mg₂Si particles starts due to supersaturation at some temperature below the solvus temperature of the Mg₂Si phase. The initially nucleated particles later grow into larger sizes of micrometers, which form the second peak in the distribution curves in Figure 3. As the temperature continues to drop, the free energy for nucleation increases due to the decreasing Mg concentration in the matrix, which leads to a decrease in nucleation rate. However, on further cooling, the effect of supersaturation dominates because of the increasing undercooling. The nucleation rate starts to increase and large

amounts of clusters form during this process. These clusters have little opportunity to grow due to limited diffusion at low temperature, which is responsible for the large amounts of nanosized particles in Figure 3.

B. Hardness

The hardness test results of the homogenized specimens are shown in Figure 4. These results reflect the different concentration of Mg and Si dissolved in the Al matrix, which is consistent with the corresponding microstructures. The solution strengthening effect of Mg and Si is responsible for the significant rise in hardness as cooling rate increases. Although the difference in cooling rate between air cooling and water quenching is much greater than between furnace cooling and air cooling, the increase in hardness is not as big. This suggests that the hardness of the specimen is not very sensitive within this fast cooling range.



Fig. 4 Effect of cooling rate after soaking at 580 °C for 8 hours on the hardness of AA 6005 specimens

The higher hardness associated with the faster cooling rate potentially leads to an increase in the difficulty of extrusion. However, the hardness at room temperature alone cannot predict the corresponding extrudability at preheating and extrusion temperatures, since the microstructure of the billet may change and softening or hardening can occur as temperature increases. Therefore, the behavior of the billet at real preheating and extrusion temperatures needs to be examined.

C. Flow Stress

The influence of cooling rates on flow stress at preheating and extrusion temperatures is illustrated in Figure 5. At all test temperatures between 450 °C and 550 °C, the water quenched specimens exhibit the highest flow stress, with the furnace cooled specimens exhibiting the lowest, owning to different matrix concentrations of Mg and Si. The change in solid solution strengthening during heating can be observed by comparing the water quenched and the air cooled cases. At 450 °C and 500 °C, the flow stress of the air cooled specimens are considerably lower than that of the water quenched and air cooled specimens, while at 550 °C, the flows stress of water quenched and air cooled specimens are close enough to indicate that Si and Mg are dissolved into the matrix to

a similar degree at this temperature. On the other hand, the solution strengthening effect is not so obvious in furnace cooled specimens. Even at 550 °C, the flow stress of these specimens is still obviously lower than those of the other two specimens, which indicates that the coarse Mg₂Si particles resulted from this slow cooling rate are still not largely dissolved, which is further confirmed by microstructure examination. It can thus be argued that a slow cooling rate after soaking can produce a billet that requires relatively low extrusion pressure, but the age hardenability of the billet is deteriorated due to insufficient dissolution of Mg₂Si particles. On the contrary, a rapid homogenization cooling rate results in a billet that is already solutionized, but the billet requires extra high pressure to extrude.

D. Microstructure after High-temperature Compression Tests

The microstructures of the specimens that were water quenched after compression tests (Figure 6) are generally consistent with the flow stress results, providing a direct insight into the dissolution or precipitation behavior of the Mg₂Si particles at elevated temperatures. The general phenomenon that can be observed from the microstructures is that, with a higher test temperature, more Mg₂Si particles are dissolved during the test, and are subsequently retained in the matrix as a result of water quenching after testing.



Fig. 5 Effect of cooling rates on the flow stress of AA 6005 specimens at temperatures corresponding to the preheating temperature 450° C and extrusion temperatures 500° C and 550° C



Fig. 6 Microstructures of AA 6005 specimens after compression tests at temperatures corresponding to the preheating temperature (i) 450°C and the extrusion temperatures (ii) 500°C and (iii) 550°C, respectively; the specimens are subject to different homogenization cooling practices of (i) water quenching, (ii) air cooling and (iii) furnace cooling

For the specimens that were subject to water quenching and air cooling during homogenization, after the compression tests at 550° C and the subsequent water quenching, the Mg₂Si particles are mostly retained in the matrix, according to the resulting microstructure in Figure 6. This indicates that Mg and Si in the water quenched and air cooled specimens are largely dissolved during the compression tests at 550° C, coinciding with the similarity in the flow stress of these specimens at this test temperature. On the other hand, the dissolution of Mg₂Si particles in furnace cooled specimens is not as much as those in the other two specimens at all test temperatures from 450° C to 550° C. According to Figure 6, even after the compression test at 550° C, the Mg₂Si particles in the furnace cooled specimens are still relatively large in amount, which is consistent with the relatively low flow stress of this specimen at this temperature.

By comparing with the as-homogenized microstructure, it is also worth noting that Mg₂Si re-precipitates in the specimens during the compression tests at 450 °C , especially in the fast cooled specimens. This occurs because 450 °C is still below the equilibrium solvus temperature of 500 °C of the Mg₂Si phase, according the Al-Mg₂Si pseudobinary phase diagram^[12]. It is thus reasonable to claim that the supersaturated aluminum matrix is unstable during reheating if the heating rate is not sufficiently rapid. The sensitivity to preheating rate could be another drawback for fast cooled billets in addition to high flow stress, because a more carefully controlled heating rate is required.

Conclusions

In specimens that underwent water quenching after soaking, Mg and Si are mostly retained in the supersaturated Al matrix, with a limited number of fine Mg₂Si particles, owning to the extremely rapid cooling rate. Consequently, such specimens have a good age hardening capacity, evidenced by the small population of Mg₂Si precipitates observed after the compression test at 550 °C . However, the water quenched specimens exhibit relatively high hardness at room temperature and relatively high flow stress throughout the preheating and extrusion temperature range 450 °C-550°C, which means that extra pressure is needed to extrude such billets. Moreover, these specimens are relatively sensitive to the preheating rate because the dissolved Mg and Si are easy to reprecipitate during this process.

Specimens that were subject to furnace cooling from soaking temperature exhibit Mg₂Si precipitates greater in amount and larger in size due to the extremely low cooling rate of 130 °C/h. Although these specimens show lower hardness at room temperature and lower flow stress at preheating and extrusion temperature range, evidences are that the coarse Mg₂Si particles are still not largely dissolved even after compression test at 550 °C, leading to a decreased age hardenability of such billets.

The cooling rate of air cooling is still rather high. The microstructure, hardness and flow stress of such billets are more or less close to those of water quenched specimens. Especially when temperature is above the solvus temperature of Mg₂Si, the microstructure and flow stress of the air cooled and water quenched specimens are nearly identical.

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References

[1] Basic Metallurgy: 6000 series Extrusion Alloys, Comalco Publication

[2] Y. Birol: Metallurgical and Materials Transactions A - physical Metallurgy and Material, vol.44 (2013), pp.504-511

[3] S. Zajac, B. Hutchinson, A. Johansson, L. O. Gullman: Materials Science Technology, vol.10 (1994), pp. 323-333

[4] N. C. W. Kuijpers, J. Tirel, D. N. Hanlon, S. van der Zwaag: Materials Characterization, Vol. 48 (2002), pp. 379-392

[5] A. Jackson, T. Sheppard: Materials Science and Technology, vol.13 (1997), pp.61-68

[6] M. P. Clode, T. Sheppard: Materials Science and Technology, vol.9 (1993), pp.313-318

[7] L. Pedersen, L. Arnberg: Metallurgical and Materials Transactions A, vol.32 (2001), pp.525-532

[8] O. Reiso: Materials Forum, vol.28 (2004), pp.32-46

[9] H. Zhu, M. J. Couper, A. K. Dahle: JOM, vol.63 (2011), pp.66-71

[10] S. Zajac, B. Hutchinson, A. Johansson, L. O, Gullman: Aluminium Alloys: Their Physical and Mechanical Properties, vol.217 (1996), pp.397-402

[11] O. R. Myhr, O. Grong: Acta Materialia, vol.48 (2000), pp.1605-1615

[12] S. Li, S. Zhao, M. Pan, D. Zhao, X. Chen, O. M. Barabash, R. I. Barabash: Materials Transactions JIM, vol.38 (1997), pp.553-559