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Secondary precipitation during homogenization of Al-Mg-Si alloys: Influence on high temperature flow stress

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ABSTRACT

In the automotive industry Al-Mg-Si alloys are often chosen for high strength extruded profiles. However, the production of such profiles can be challenging for high alloy contents. Among several processing steps, the homogenization heat treatment before extrusion is readily accessible to modifications to improve high temperature formability. In this work, the influence of five different homogenization variants on the flow stress of AA6082 at elevated temperatures was assessed by compression testing in a deformation dilatometer at 450 °C, 480 °C, and 510 °C. The observed differences in flow stress were interpreted with regards to the microstructure. Samples from billets homogenized at higher soak temperatures exhibited lower hot flow stresses in subsequent dilatometer testing, indicating better processability. Since the decrease in flow stress is approximately constant at all test temperatures, it is suggested that it is due to a lower number density of relatively temperature-stable α-Al(Mn,Fe)Si dispersoids formed during homogenization at higher temperatures; these dispersoids are stable at all three test temperatures. In contrast, the influence of a cooling rate variation was relatively minor and diminishes at higher test temperatures since Mg-Si precipitates – whose size and number density is affected by changes of the cooling rate – are less stable at the higher test temperatures.

1. Introduction

Aluminium alloys of the 6xxx series offer a good combination of specific strength and processability. For applications that require an ultimate tensile strength above 300 MPa, such as frame rails in cars, AA6082 is frequently used. However, the extrusion of this alloy can be challenging due to its high content of Mg and Si; the influence of Mg and Si levels on the achievable extrusion speed was shown by Reiso [1].

Extrudability denotes the suitability of a material for the extrusion process. For good extrudability, surface finish problems should not occur and the deformability of the material at the chosen extrusion temperature should allow for high extrusion speeds [2]. Temperature, time, and heating/cooling rates of billet homogenization are important and readily accessible parameters that can be altered in a wide range with the goal to achieve a good combination of extrudability and artificial ageing response for a certain alloy.

One metallurgical phenomena occurring during homogenization of AA6082 is the transformation of primary, plate-like β-AlFeSi particles into multiple α-Al(Mn,Fe)Si particles which exhibit more rounded edges and therefore are less detrimental to the surface quality and the mechanical properties of the extrudate. This transformation is greatly accelerated by Mn and higher homogenization soak temperatures [3,4].

Additional to this phase transformation, dispersoids of the α-Al(Mn,Fe)Si type or, when the alloy contains Cr, α-Al(Fe,Mn,Cr)Si are formed. These dispersoids are usually of 10 nm to a few hundred nm in diameter and their direct influence on mechanical properties is generally regarded as minor. However, the dispersoids can improve fracture toughness and increase resistance to recrystallization [5,6]. High resistance to recrystallization is important in extrusion since the resulting non-recrystallized laminar microstructure is beneficial to the mechanical properties of the profiles [7]. For many applications of AA6082, it is therefore preferable to achieve a high number density of dispersoids in a certain size range, maximizing the Zener forces. Below, the term dispersoids is only used to denote secondary precipitates of the α-Al(Fe,Mn)Si type.

It is generally desired that primary Mg_2Si particles be fully

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dissolved during homogenization to allow for a maximum artificial ageing response of the finished extrudate. When the billet is cooled at rates relevant for industrial practice, Mg and Si re-precipitate to a certain extent. The distribution of Mg and Si, i.e. the amount in solid solution and the number and size distribution of Mg-Si precipitates is a critical factor regarding extrusion pressure, surface quality and artificial ageing potential [1]. It was suggested that the fraction of Mg and Si in solid solution should be as low as possible to avoid solid solution hardening detrimental to productivity – for an overview on research leading to this assumption see the review by Zhu et al. [8] which covers the role of Mg-Si precipitates over the whole process chain.

A low matrix content of Mg and Si can be achieved by a low cooling rate allowing for near-complete precipitation. However, when the cooling rate is too low, the re-precipitated Mg_2Si-particles are too large to be fully dissolved during the subsequent extrusion process, leading to incipient melting, poor surface finish, or low mechanical properties of the final product. The influence of the cooling rate remains controversial with some studies reporting improved extrudability after water quenching and other studies finding no influence of the cooling rate on extrusion breakthrough pressure [8].

When Mg and Si precipitate in a way that they can be fully dissolved during preheating or extrusion, the constituent Mg and Si from the dissolved particles add to the amount of Mg and Si in solid solution. It is, however, possible to design the cooling rate and the extrusion process in a way that the particles do not dissolve during pre-heating but only when the material briefly reaches a higher temperature during deformation. Such an approach was successfully demonstrated by applying a two-step homogenization treatment to achieve a fine distribution of β′ [9]. However, such a treatment may be too complex for the industrial practice.

Here we present an investigation of the influence of five different homogenization heat treatment regimens with industrial relevance on the high temperature flow stress of AA6082. We studied the influence of three different homogenization soak temperatures; for one temperature, the influence of the heating and cooling rate was also explored. The observed differences in high temperature flow stress are explained with regards to the microstructure investigated by differential scanning calorimetry (DSC), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). SEM was used to assess the number density and size of Mg-Si-precipitates after cooling from homogenization temperature, and TEM was used to quantify number density and size of the dispersoids. Furthermore, TEM was used to clearly identify different types of Mg-Si phases. The findings regarding the number density and size of dispersoids and Mg-Si precipitates, respectively, and their contribution to hot flow stress at different temperatures allow for the better understanding and improvement of industrial extrusion and rolling processes of high strength Al-Mg-Si alloys.

2. Experimental

Billets of AA6082 with a diameter of 254 mm were direct chill cast and cut to a length of 600 mm. The exact alloy composition was determined by optical emission spectrometry and is Al – 0.81% Si – 0.70% Mg – 0.44% Mn – 0.22% Fe – 0.05% Zn – 0.02% Cr – 0.02% Ti (all values in wt%). The billets were homogenized in an industrial homogenization oven using different time/temperature regimens given in Table 1. Samples for SEM were electropolished using an electrolyte of methanol and nitric acid [10]. SEM investigations were performed on a Zeiss Ultra Plus 55 field emission gun SEM equipped with a GEMINI in-lens detector for secondary electrons (SE). The accelerating voltage was 15 kV and the working distance was between 5–10 mm.

TEM thin foils were either prepared by electropolishing at 8 V, −10 °C and a flow rate of 4 in a Struers Tenupol-5 using the nital electrolyte for 1–2 min, or thinned by ion-milling in a Precision Ion Polishing System (PIPS) at 4.5 V for about 10 h. TEM investigations were carried out on an FEI Tecnai F20, equipped with an X-FEG field emission gun with operation voltage at 200 kV, and an FEI Tecnai G20 with a CeB_6 emitter and operation voltage at 200 kV. The characterization of the different precipitates was done by means of TEM bright field, high resolution TEM (HRTEM), selected area electron diffraction (SAED) patterns, energy dispersive x-ray spectroscopy (EDX), and electron energy loss spectroscopy (EELS).

Table 1
Homogenization regimens.

Name	Heating rate [K h^{-1}]	Homogenization soak temperature [°C]	Holding time [h]	Cooling rate [K h^{-1}]
Hom530	120	530	4	300
Hom530_fast_heating	480	530	4	300
Hom530_fast_cooling	120	530	4	800
Hom550	120	550	4	300
Hom580	120	580	4	300

For the calculation of number densities of dispersoids, these particles were manually count in three micrographs per sample. Assuming spherical dispersoids, the number density was calculated as follows: $number\ density = N/[A(t + \bar{d})]$. In this equation, N is the number of dispersoids, A is the area, t is the thickness of the foil as measured by EELS, and $\bar{d}$ is the mean diameter of the dispersoids which was added to the foil thickness to account for dispersoids which are visible but whose centre lies outside of the foil. The mean diameters of dispersoids were calculated after measuring the diameter/diagonal of at least 65 dispersoids exhibiting good contrast in different micrographs.

Vickers micro-hardness (HV0.1) was measured five times per sample using a load of 100 g. Conductivity measurements were conducted using a Fischer Sigmascope SMP10.

Compression tests were performed using a Bähr DIL805 A/D deformation dilatometer. Cylindrical specimens with a diameter of 5 mm and a length of 10 mm were machined from the homogenized and as-cast billets. The specimens were heated to either 450 °C, 480 °C, or 510 °C at a heating rate of 15 K s^{-1} and then deformed at a rate of 1 s^{-1}. The test temperatures were chosen due to their relevance to the industrial extrusion process. Measurements were performed in triplicate.

DSC experiments were carried out using a Netzsch DSC 204 F1 differential scanning calorimeter with a heating rate of 10 K min^{-1}.

3. Results

3.1. Microstructural investigation

The precipitation of Mg-Si precipitates resulting from the different homogenization regimens were investigated by SEM; representative micrographs for each variant are given in Fig. 1. It can be seen that a higher soak temperature leads to a coarser precipitation of Mg-Si precipitates (i.e., fewer, larger precipitates) while a higher cooling rate leads to a higher number density of smaller precipitates.

For the variants Hom530 and Hom580, the Mg-Si phases prevalent were identified by TEM. The larger precipitates with sizes of roughly 1–3 µm (Fig. 2a) can be clearly identified as the β Mg_2Si equilibrium phase by means of EDX and SAED. Furthermore, smaller needles with lengths in the range of approximately 5 nm up to 400 nm are visible in the micrographs. By indexing fast Fourier transforms (FFT) of HRTEM micrographs, the needles were identified as β″ phases, as shown in Fig. 2b, c, and d for a relatively large β″ needle in Hom530. The crystal structure that was used for simulating the FFT and SAD patterns was: Mg_5Si_6, C2/m, a=1.516 nm, b=0.405 nm, c=0.674 nm, beta=105.3° [11]. The composition of the phase was measured by EDX an can be quantified with 44 at% Mg and 56 at% Si. The numerous precipitates smaller than roughly 20 nm visible in Fig. 2(b) are also β″ as confirmed