Superplastic deformation mechanisms in fine-grained Al–Mg based alloys

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ABSTRACT

The superplastic deformation behaviour at elevated temperatures and constant strain rates of two fine-grained AA5083 type aluminium alloys was investigated. The first alloy with chromium contains $Al_6(Mn, Cr)$ particles and the second alloy without chromium has Al_6Mn particles. The effective activation energy of superplastic deformation and the activation parameters for the grain boundary relaxation was calculated. The microstructure evolution and contributions of grain boundary sliding, intragranular deformation and diffusion creep to total superplastic deformation were studied by SEM, EBSD, FIB, TEM techniques. Low values of grain boundary sliding and permanent continuous formation of sub-grain boundaries were found in both alloys. Significant dynamic grain growth during superplastic deformation and large value of intragranular deformation were found in the alloy without chromium. Intragranular deformation is not significant and the superplasticity is primarily a result of diffusion creep in the chromium containing alloy with $Al_6(Mn,Cr)$ particles.

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1. Introduction

The ability to drastically elongate (even several thousand per cent) in fine-grained polycrystalline metallic alloys has been known for many years. This quality is associated with an unusually high strain rate sensitivity to flow stress. Pearson found this phenomenon in Bi–Sn alloy in 1934 [1], and Bochvar and Sviderskaya [2] named this effect "superplasticity" in 1945. Since then, many studies of the superplasticity phenomena have been carried out because it provides fascinating opportunities for creating a complexly shaped product in one operation [3]. In spite of significant effort, there are many unanswered questions about the fundamental mechanisms responsible for the high strain rate sensitivity of superplasticity. It is well proven that a fine grain size is required to achieve high elongations, and it is a commonly accepted viewpoint that grain boundary sliding (GBS) is the fundamental microstructural mechanism of superplasticity. It is known that GBS provides 60–90% of the total elongation in the historically famous Zn–22Al alloy and in similar alloys with a eutectoid or eutectic duplex structure [4,5]. The other mechanisms or accommodation mechanisms involved in superplastic deformation are diffusion creep and intragranular dislocation sliding.

There are several experimental observations that are not consistent with the statement that the GBS is the main mechanism of superplasticity. Analysis of the Al alloys with a small volume fraction of the second phases gives inconsistent results. Portnoy [6,7] has shown an unusually weak GBS in several Al alloys (Al–Cu–Mn and Al–Mg–Mn based systems). Sothudech and Bate [8] have found a weak GBS and intragranular strain in the AA5083 alloy. According to their results, diffusion creep is the main mechanism of superplasticity for these alloys. Blackwell and Bate [9] noted a secondary role of GBS and emphasised the relevance of intragranular dislocation activity. A transition from GBS to dislocation creep was observed in Perez-Prado [10]. In the Mukherjee model [11] ledges on the grain boundary surface lead to the obstruction of GBS. Dislocations are generated at the obstructing protrusion and generated dislocations move into the grain and pile up against the opposite boundary where they are annihilated. Gifkins [12] suggested that dislocation activity is confined to a narrow mantle region close to the grain boundaries, whilst the models of Ashby and Verrall [13] are based on diffusion creep, which therefore involves no lattice dislocations. Several researchers [14] see superplasticity as involving both diffusion and dislocation mechanisms simultaneously. GBS provides equiaxed grains due to a change of neighbours. Both diffusion creep and dislocation sliding cannot ensure the conservation of the grain size and grain shape after large plastic deformations. Thus, it was concluded that the formation of transverse grain boundaries promotes the maintenance of equiaxed grains in the alloys with weak GBS.

Dynamic recovery and recrystallisation are usually observed in materials in which the microstructure, at the beginning of

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superplastic deformation, is composed of bands of similarly oriented subgrains ('Supral' type alloys). It is observed in alloys with a high Zr [15,16] or Sc and Zr content [17], due to the fine Al_3Zr or $Al_3(Zr,Sc)$ particles which pin the grain and subgrain boundaries. During superplastic deformation, the misorientation between subgrains increases and a typical microstructure of small grains with high-angle boundaries develops. At the same time, observations of dynamic recrystallisation in region II of SPD for some Al alloys with an initially completely recrystallised structure have been reported in Al [6,7,18,19] and in some other alloys [20–22]. In this group of Al alloys, for example Al–Mg–Mn and Al–Cu–Mn systems, recrystallisation occurs prior to superplastic deformation, creating a microstructure consisting of approximately equiaxed grains and fine particles [6–9]. These fine particles of the second phase control grain growth via the Zener pinning mechanism. Such particles are supposed to influence to superplastic deformation mechanisms. Formation of the dislocation walls and the transverse boundaries inside elongated grains under superplastic deformation with unusually weak GBS has been found in the Al–Cu–Mn alloy [6], with a grain size less than 10 μm, and fine particles of $Al_{12}CuMn_2$ (T-phase) of 0.2–00.5 μm in size.

Surface studies have become the most widespread method of studying the mechanisms of superplasticity since the pioneering work of Holt [23], Pearson [1] and others [12,24–28]. Surface mechanical scratches are irregular and unidirectional; thus, in most cases, this does not allow quantitative measurements of intragranular deformation. Photolithographic grids have also been used to study the creep [26]. Recently, focused ion beam (FIB) micromilling has been used to produce grids. FIB grids, used for the analyses of superplasticity for the first time in Rust and Todd's works [29–31], have several advantages when compared with mechanical scratches and other similar techniques, including an ability to produce grids of a controlled size, without plastic deformation of the surface. Some disadvantages of the FIB grids may be connected with the Ga^+ ion implantation in the Al lattice during the FIB milling and Ga segregation at the grain boundaries. According to Rust and Todd [31], implanted Ga is not substantial. It is notable that the contribution of acting mechanisms can be different at the surface and in the interior of specimens: an inevitable oxidation of Al can change this contribution. Consequently, apart from the surface studies, grain and dislocation structures studies should be involved in analysing the mechanisms of superplasticity.

The mechanical spectroscopy (MS) technique [32] is still rarely involved in the study of superplasticity. The obvious reason for that is that MS typically uses an elastic range of materials loading, in contrast with the huge plastic deformation that takes place at superplasticity. The grain boundary relaxations, which occur in pure metals as well as under the influence of solute interactions in alloys or small particles of the second phases, may originate from GB sliding [33] and GB movement in the normal direction, i.e., growth of a favourably oriented grain at the expense of an adjacent one. The first mechanism is typical for the elastic range of loading [34–37], while the second mechanism is typical for plasticity and superplasticity [38]. The dynamic IF increases rapidly at the beginning of superplastic deformation and then stays almost constant throughout the remainder of the deformation. It increases with a decrease in the measuring frequency and increases with an increase in the strain rate or applied stress. It is clear that the mechanisms of GBS in these two opposite cases are different. Nevertheless, the MS method gives some useful information about the mobility of point, linear and surface defects at the stage of anelastic and microplastic deformation.

Thus, our study is focused on the quantitative determination of the acting mechanisms of superplasticity and deformation behaviour in AA5083 type aluminium alloys with different size of particles, using various methods of surface and volume structural analysis, mechanical testing and mechanical spectroscopy analysis.

2. Experimental methods

2.1. Materials

Two alloys were used in this study; their compositions are given in Table 1. The alloys were melted in a Nabertherm S3 electric furnace using graphite-fireclay crucibles. A99 grade Al, Mg95 grade Mg, as well as Al-10%Mn and Al-10%Cr master alloys were used. The alloys were cast out into water-cooled copper moulds with dimensions of $100 \times 40 \times 20$ mm^3, where they were cooled down at the rate of approximately 15 K/s. The temperature of casting was 750 ± 10 °C. According to Thermo-Calc calculation (data base TCAL1) the liquidus temperature is 634 °C for alloy M without Cr and 641 °C for alloy C with Cr. The temperature of casting was chosen to be above the liquidus temperature of the alloys to prevent forming the primary coarse particles of Mn and Cr rich phases. All ingots were homogenised at 460 °C for 8 h to dissolve the non-equilibrium β-phase (Al_3Mg_2) and to precipitate the fine Al_6Mn and $Al_6(Mn,Cr)$ particles. Hot (80%) and cold rolling (two stages with 60% thickness reduction with intermediate anneal) were performed by means of a rolling mill with rollers of 230 mm in diameter. The hot rolling temperature was 420 ± 10 °C. The final sheet thickness was 1 mm.

Differential scanning calorimetry (DSC) was carried out in the "Setaram Labsys DSC 1600" with a heating rate of 5 K/min and a temperature range from 20 to 700 °C. The solidus temperature of both alloys is approximately 575 °C. The composition of the alloys was controlled by chemical analysis (Table 1) after homogenisation and cold rolling. Deviations from the nominal composition were 0.05 mass%, and compositions of both alloys with respect Mg and Mn were similar.

2.2. Microstructures

Microstructure observations were conducted after different stages of specimen preparation, before superplastic deformation and after different stages of deformation (from 0.1 to 1.4). Microstructure characterisations were carried out by means of an Axiovert 200MMAT "Carl Zeiss" light microscope using polarised light. Specimens were prepared by mechanical grinding and polishing: polishing in chlorine–alcohol electrolyte at 15–20 V and anode oxidising in 10% (HF in H_3BO_4). The average grain size was determined by the random secant method, using more than 200 measures in two directions: along (l) and across (s) of the rolling and tensile direction in the ls plane. Error bars were determined experimentally using the standard deviation and a confidence probability of 95%.

A Tescan-VEGA3 LMH scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS) (X-MAX80, Oxford Instruments) and with an EBSD – HKL detector (NordlysMax EBSD, Oxford Instruments) was used for the microstructural investigations. The EBSD analysis was carried out using

Table 1
The compositions (in wt%) of the alloys via chemical analysis.

Alloy	Mg	Mn	Cr	Fe	Si	Al
M	5.01	0.68	0.00	< 0.005	< 0.005	bal
C	4.92	0.62	0.28	< 0.005	< 0.005	bal