Flow Stress Modelling and 3D Processing Maps of Al4.5Zn4.5Mg1Cu0.12Zr Alloy with Different Scandium Contents

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1. Introduction

High strength aluminium alloys of the 7XXX series (Al-Zn-Mg system) are widely used in aerospace, aircraft and automotive industries as structural materials [1–3]. The maximum strength of these alloys is achieved after quenching and ageing with a Zn/Mg concentration content of more than 1.0, but the corrosion resistance of this group of alloys is low [4,5]. A Zn/Mg ratio of ~1.0 or less [6] and a (Zn+Cu)/Mg ratio below 1.5 [7] provides a better combination of mechanical, corrosion and technological properties. Moreover, it is well known that minor additions of transition metals, such as Sc and Zr, to Al-Zn-Mg system-based alloys can improve mechanical and corrosion resistance properties [8–12]. Coherent Al3(Sc,Zr) dispersoids formed during ageing from an aluminium solid solution (after rapid solidification) with an L12-structure refine the microstructure and increase the recrystallisation temperature; therefore, the microstructure remains nonrecrystallised and mechanical properties remain strong up to elevated temperatures [13–17]. Due to the above, study of the hot deformation behaviour of aluminium alloys containing Al3(Sc,Zr) dispersoids is an urgent task.

Optimal technological parameters of hot deformation are important for the successful manufacture of the materials and the formation of a fine microstructure containing a minimum number of defects. Flow stress modelling and processing maps for different materials are widely used for selecting optimal parameters [18–21]. This approach was demonstrated for aluminium alloys over a wide range of strain rates and temperatures: 7075 [22], 6063 [23], 2014 [24], 5182 [25], 7085 [26], 5052 [27], and others. The models’ parameters are strictly dependent on the chemical composition of the different types of
materials, such as steel [21,28], titanium [29] and aluminium alloys [30], which is why it is necessary to determine the parameters for each composition.

In this work, the influence of Sc content of an Al4.5Zn4.5Mg1Cu0.12Zr alloy on hot deformation behaviour in the range of 300–450 °C deformation temperatures and strain rates of 0.1–15 s⁻¹ was studied and the optimal parameters of deformation were determined using 3D processing maps and microstructure analysis.

2. Materials and Methods

The chemical composition of the investigated Al4.5Zn4.5Mg1Cu0.12Zr alloys with different Sc contents is presented in Table 1. Alloys were prepared in a resistance furnace from pure Al (99.99%), Zn (99.9%), Mg (99.9%), Al-53Cu, Al-3.5Zr and Al-2Sc master alloys. The melt was poured into a water-cooled copper mould (solidification rate was about 15 K/s). Heat treatment of the ingots with a size of 20 × 40 × 120 mm³ was carried out in a Nabertherm N60/85HA furnace (Nabertherm GmbH, Lilienthal, Germany) at 450 °C for 3 h.

Table 1. Chemical composition of the investigated alloys (wt. %).

| Alloy  | Al  | Zn | Mg | Cu  | Zr | Sc |
|-------|-----|----|----|-----|----|----|
| 0.05Sc| bal.| 4.5| 4.5| 1.0 | 0.12| 0.05|
| 0.1Sc | bal.| 4.5| 4.5| 1.0 | 0.12| 0.1 |
| 0.15Sc| bal.| 4.5| 4.5| 1.0 | 0.12| 0.15|

A light microscope (LM) Carl Zeiss Axiovert 200M MAT (Carl Zeiss AG, Oberkochen, Germany) and a scanning electron microscope (SEM) TESCAN VEGA 3LMH (Tescan, Brno-Kohoutovice, Czech Republic) equipped with X-ray energy dispersive microanalysis system (Oxford Instruments Advanced AZtecEnergy, High Wycombe, UK) were used for microstructure analysis. Transmission electron microscopy (TEM) images were made using a JEM2100 (Jeol Ltd., Tokyo, Japan) microscope operated at a high voltage of 200 kV.

Compression tests were carried out using a Gleeble 3800 (Dynamic Systems Inc., Poestenkill, NY, USA) thermomechanical simulator. Cylindrical specimens with a size of 10 mm in diameter and 15 mm in height were compressed at a temperature range of 300–450 °C and strain rates of 0.1–15 s⁻¹ at a true strain value 1.0. After deformation, specimens were quenched by compressed air. Stress-strain curves were recalculated to consider friction and adiabatic heating during deformation [31,32]. Three-dimensional processing maps were constructed by B-spline interpolation using OriginLab Software.

3. Results and Discussion

3.1. Initial Microstructure Investigation

The aluminium solid solution and T-phase (AlZnMgCu) of eutectic origin were presented in an as-cast microstructure (Figure 1a). The microstructure after heat treatment consisted of an aluminum solid solution, particles of T-phase of crystallisation origin and secondary precipitates of T-phase and dispersoids of Al₃(Sc, Zr) (Figure 1b).
Figure 1. SEM-image of the alloy 0.15Sc as cast (a) and after the heat treatment (b). The images show the characteristic emissions of the alloying elements into the selected zone (the red rectangle on the microstructure) and TEM-image with secondary phases of T and Al₃(Sc,Zr).

3.2. Hot Deformation Behaviour Modelling

The typical true stress true strain curves of the investigated alloys after compression under different conditions are presented in Figure 2. It can be observed that with an increase in temperature (Figure 2a,c,e), the level of flow stress decreased, and with an increase in strain rate, it increased. Nevertheless, with an increase in the deformation rate, the difference in the level of flow stress decreased (Figure 2b,c,f), which was caused by destruction of the specimens with the accumulation of the degree of strain at a strain rate of 15 s⁻¹. Moreover, in addition to the characteristic stage of stress growth due to strain hardening (SH), the flow curves generally lacked the region characteristic for dynamic recrystallisation (DRX) with softening, and reached the steady-state stage of deformation. This means that the main controlling process of structural evolution during the growth of deformation was dynamic recovery (DRV).
The correlation between the flow stress, strain rate and temperature of hot plastic deformation is described by the Zener-Hollomon parameter $Z$ [33]:

$$Z = \dot{\varepsilon} \frac{Q}{R T}$$

(1)

where $\dot{\varepsilon}$ is the strain rate ($s^{-1}$), $Q$ is the effective activation energy of hot deformation ($J \cdot \text{mol}^{-1}$) and $R$ is the universal gas constant ($8.314 \ J \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$). The relation between $Z$ and flow stress is usually described by the power function (2) for low stresses, an exponential function (3) for high levels of stress, and a hyperbolic sinus function (4) for all ranges of stress [21,34,35]:

$$Z = A_1 \sigma^{n_1}$$

(2)

$$Z = A_2 e^{\beta \sigma}$$

(3)

$$Z = A_3 [\sinh(\alpha \sigma)]^{n_2}$$

(4)
where \( A_1, A_2, A_3, n_1, n_2 \) and \( \beta \) are experimentally-determined parameters. The value of \( \alpha \) can be determined as:

\[
\alpha \approx \frac{\beta}{n_1}
\]  

The materials constants are presented in Table 2 for the alloys with different Sc content and different strain values. The values of the effective activation energy were in the range of 161–192 kJ mol\(^{-1}\) for all compositions, which is consistent with the values obtained previously for Al-5.8Zn-2.3Mg-1.5Cu-0.21Cr (168.08 kJ mol\(^{-1}\)) [36], Al-7.3Zn-2.3Mg-0.6Cu-0.3Mn-0.11Er-0.12Zr (189.67 kJ mol\(^{-1}\)) [37] and Al–6.2Zn–0.70Mg–0.30Mn–0.17Zr (181–189.5 kJ mol\(^{-1}\)) [38].

### Table 2. The materials constants for the alloy Al4.5Zn4.5Mg1Cu0.12Zr with different Sc contents.

| \( \varepsilon \) | 0.05Sc | 0.1Sc | 0.15Sc |
|------------------|--------|--------|--------|
| \( Q, \text{kJ} \cdot \text{mol}^{-1} \) | ln\( A \) | \( n_2 \) | \( \alpha \) | \( Q, \text{kJ} \cdot \text{mol}^{-1} \) | ln\( A \) | \( n_2 \) | \( \alpha \) | \( Q, \text{kJ} \cdot \text{mol}^{-1} \) | ln\( A \) | \( n_2 \) | \( \alpha \) |
| 0.05 | 172 | 28.1 | 6.8 | 0.010 | 171 | 28.0 | 6.7 | 0.010 | 161 | 27.1 | 6.1 | 0.009 |
| 0.1 | 175 | 29.3 | 6.4 | 0.010 | 171 | 29.4 | 6.4 | 0.009 | 169 | 28.9 | 6.0 | 0.009 |
| 0.2 | 169 | 28.8 | 6.1 | 0.009 | 171 | 29.3 | 6.2 | 0.009 | 164 | 28.4 | 5.6 | 0.008 |
| 0.3 | 165 | 28.7 | 6.0 | 0.008 | 172 | 30.1 | 6.2 | 0.008 | 164 | 28.7 | 5.5 | 0.008 |
| 0.4 | 162 | 28.6 | 6.0 | 0.008 | 171 | 30.2 | 6.0 | 0.008 | 166 | 29.2 | 5.5 | 0.008 |
| 0.5 | 167 | 29.5 | 6.1 | 0.008 | 174 | 30.9 | 6.1 | 0.008 | 169 | 29.9 | 5.5 | 0.008 |
| 0.6 | 172 | 30.4 | 6.1 | 0.008 | 179 | 32.0 | 6.2 | 0.008 | 173 | 30.9 | 5.6 | 0.008 |
| 0.7 | 180 | 31.9 | 6.2 | 0.008 | 186 | 33.6 | 6.3 | 0.007 | 179 | 32.3 | 5.6 | 0.008 |
| 0.8 | 184 | 32.5 | 6.2 | 0.009 | 189 | 34.5 | 6.4 | 0.007 | 185 | 33.5 | 5.6 | 0.008 |
| 0.9 | 186 | 32.8 | 6.2 | 0.009 | 192 | 35.1 | 6.4 | 0.007 | 183 | 33.0 | 5.3 | 0.008 |

The comparison between experimental and calculated values of stress is shown in Figure 3a. As one can see, the dispersion of the stress values was significantly higher at high values. The accuracy of the strain-compensated model was quantified by additional experiments for the alloys with different Sc content at different temperature-strain rate conditions: 0.05Sc—375 °C and 0.05 s\(^{-1}\); 0.1Sc—425 °C and 1.5 s\(^{-1}\); 0.15Sc—325 °C and 0.8 s\(^{-1}\). The average absolute relative error (AARE) determined accordingly [39] was 5.0% (Figure 3b).

### Figure 3. The comparison between experimental and calculated values of stress (a) and model validations using additional experiments at different temperature and strain rate conditions (b): alloy 0.05Sc—375 °C and 0.05 s\(^{-1}\); alloy 0.1Sc—425 °C and 1.5 s\(^{-1}\); alloy 0.15Sc—325 °C and 0.8 s\(^{-1}\).

#### 3.3. 3D Processing Maps Establishment

Processing maps are important tools for analysing hot deformation parameters. They are based on the dynamic material model (DMM) [40] and usually established by combining two diagrams: power dissipation and flow instability [18,23,41]. According to DMM, the power dissipated consists of the content term \( G \) representing the power dissipated by...
plastic work (most of it converted into heat), and the cocontent term \( J \) (consumed by the evolution of the material microstructure) [40]:

\[
P = σ\dot{ε} = G + J = \int_0^ε σ d\dot{ε} + \int_0^σ \dot{ε} dσ
\]  \hspace{1cm} (6)

If temperature and strain are constant, the flow stress is given by

\[
σ = K ε^m
\]  \hspace{1cm} (7)

where \( K \) is the material constant and \( m \) is the strain rate sensitivity factor, which is related to \( G \) content and \( J \) cocontent [19,42]:

\[
m = \frac{∂J}{∂G} = \frac{ε∂σ}{σ∂ε} = \frac{∂lnσ}{∂lnε}
\]  \hspace{1cm} (8)

\( J \) co-content can be written as

\[
J = \int_0^σ \dot{ε} dσ = m + 1 \sigma \dot{ε}
\]  \hspace{1cm} (9)

The maximum possible dissipation is reached when \( m = 1 \), so the \( J \) co-content reaches a maximum, which implies \( J_{\text{max}} = (σ\dot{ε})/2 = P/2 \).

The power dissipation efficiency \( η \) can be calculated by as [18]:

\[
η = \frac{J}{J_{\text{max}}} = \frac{2m}{m + 1}
\]  \hspace{1cm} (10)

Optimal hot deformation parameters are characterised by higher a dissipation efficiency \( η \) and a flow stability \( ξ_P > 0 \), which is calculated by the following [42]:

\[
ξ_P = \frac{∂ln\left(\frac{m}{m+1}\right)}{∂lnε} + m > 0
\]  \hspace{1cm} (11)

Areas with negative \( ξ_P \leq 0 \) are characterised by adiabatic shear bands, slip localisation [43] and crack formation based on grain boundary cavitation [32], dynamic strain ageing, kink bands, mechanical twinning and flow rotations [42].

Three-dimensional processing maps were established by combining power dissipation efficiency and flow stability diagrams at different strain values (Figure 4). As one can see, the optimal hot deformation parameter regions are in the field of red regions of power dissipation efficiency and the areas of flow stability (marked as S). For alloy 0.05Sc these optimal parameters were a temperature range of 400–450 °C and a strain rate of 0.1–1 s\(^{-1}\) at the beginning of deformation, and 380–420 °C and strain rates lower than 1 s\(^{-1}\)—at high strain values. For the 0.1Sc alloy, the optimal temperature range primarily decreased up to 360–400 °C with the same strain rates (0.1–1 s\(^{-1}\)) at all stages of deformation. Furthermore, for the alloy with 0.15% Sc, the optimal temperature range was 360–450 °C during the first stages and near 360 °C at the end of deformation, with the lowest strain rates (near 0.1 s\(^{-1}\)) for all stages. The specimens deformed with parameters at the lowest power dissipation efficiency (green areas) and flow instability regions (450 °C and 15 s\(^{-1}\), for example) had cracks on the surface, as shown in Figure 4.
The typical microstructures of specimens after deformations at different conditions are shown in Figure 5. Primarily, the microstructures after all deformation conditions were non-recrystallised, and elongated deformed grains were present. However, upon deformation at rates of 5 and 15 s$^{-1}$ and temperatures of 450 °C, the microstructure was partially recrystallised, but there were also microcracks along the grain boundaries. Therefore, the main mechanism of energy dissipation during deformation was DRV. Under those deformation regimes, where DRV did not have time to occur, cracks formed and the specimens were destroyed. Thus, the optimal deformation parameters were a temperature range 350–400 °C and strain rates of 0.1–1 s$^{-1}$ for the 0.05Sc and 0.1Sc alloys, and 0.1 s$^{-1}$ for the 0.15Sc.

Figure 4. 3D processing maps for Al4.5Zn4.5Mg1Cu0.12Zr alloys with different Sc contents, and images of 0.1Sc alloy specimens after deformations at 450 °C/15 s$^{-1}$ (with cracks) and 400 °C/0.1 s$^{-1}$ (without cracks).

Figure 5. LM-images of the microstructures of the alloy 0.1Sc after deformation at different conditions.
4. Conclusions

The hot deformation behaviour of Al4.5Zn4.5Mg1Cu0.12Zr alloys with different scandium contents were investigated at a temperature range of 300–450 °C and strain rate intervals of 0.1–15 s⁻¹. The materials constants of a flow stress model based on the Zener-Hollomon parameter were determined (AARE was no more 5.8%). Three-dimensional processing maps were established by combining the power dissipation efficiency and flow stability diagrams. Based on processing maps analysis and the microstructures investigations, the optimal deformation parameters were determined as a temperature range of 350–400 °C and strain rates of 0.1–1 s⁻¹ for the alloys with 0.05% and 0.1% Sc, and 0.1 s⁻¹ for the alloy with 0.15% Sc. These ranges were characterised by the highest dissipation efficiency in the range of 30–35% and a positive flow stability value.

Author Contributions: Conceptualisation, M.G.K. and A.V.P.; methodology, A.Y.C.; validation, M.G.K.; investigation, R.Y.B. and M.V.G.; writing—original draft preparation, M.G.K. and A.Y.C.; writing—review and editing, M.G.K.; supervision, A.N.S. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Russian Science Foundation (Project №20-79-00305).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data is contained within the article.

Conflicts of Interest: The authors declare no conflict of interest.

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