Investigation of the tensile response and the microstructural evolution during hot drawing of multi-layered, ultrafine-grained AA6014 sheets by thermomechanical analyzes

B Zettl*, M Merklein

1 Institute of Manufacturing Technology (LFT), Friedrich-Alexander-Universität Erlangen-Nürnberg, Egerlandstr. 13, 91058 Erlangen, Germany

* Author to whom any correspondence should be addressed: bastian.zettl@fau.de

Abstract. Mechanical strengthening of sheet material can be realized by accumulative roll bonding, which belongs to the severe plastic deformation processes. Beside fine-grain hardening also work and precipitation hardening is responsible for the rise of material strength. However, this is also accompanied by a significant decrease in ductility. Thus, the need for an enhancement of ductility is essential for a sufficient formability in later applications and was already introduced by tailored heat treatments. The challenge, however, is to realize a process temperature that leads to a degradation of dislocations and dissolution of MgSi-precipitations, without a recrystallization of the fine-grained microstructure to coarse grain sizes. In order to identify a suitable temperature range to avoid recrystallization, hot forming experiments are carried out at successive temperatures from 20 to 300 °C. Tensile specimens are drawn with the thermomechanical simulator Gleeble 3500 (Dynamic Systems Inc.) aided by the strain measurement system Aramis (GOM GmbH). The aim is to investigate the dependence of the mechanical properties from the forming temperature in order to identify a temperature range, in which high strength with simultaneous enhanced ductility is maintained.

1. Introduction

Beside alloying and heat treatment strategies, inducing high degrees of plastic deformation can lead to strengthening of aluminum alloys. These methods are summarized under the term of severe plastic deformation (SPD) [1]. Belonging to the SPD processes, accumulative roll bonding (ARB) is suitable for producing large-scaled sheets with an increased level of strength compared to the initial material condition [2]. For the investigated wrought aluminum alloy AA6014, beside the hardening effect due to the MgSi-precipitations, also fine-grain as well as work hardening are of an important role in the context of ARB. This is due to the accumulation of the deformation degree by repeated rolling which leads to a high concentration of dislocations and subsequently to the formation of subgrain boundaries. With an increasing misorientation angle, the subgrain boundaries evolve to regular grain boundaries which results in an ultrafine-grained (UFG) microstructure [3]. The fine-grain hardening effect itself can be explained by the Hall-Petch relation in Equation 1 [4].

\[
YS = \sigma_0 + k_y/d^{1/2}
\]  

(1)
YS is the yield strength, $\sigma_0$ and $k_y$ are material constants and $d$ is the average grain diameter. In the term of lightweight applications, it is favorable to establish UFG as the main hardening factor because the high density of grain boundaries results in more possibilities of dislocation gliding mechanisms and thus, to an overall better material formability [5]. Nevertheless, the combination of the hardening effects resulting from ARB lead to a significant decrease of ductility and thus, formability of the multi-layered sheets. In previous investigations, a short-time local heat treatment strategy was already proven to be beneficial for a better local formability of multi-layered AA6016 sheets [6]. The observed softening of material is attributed to the dissolution of the MgSi-clusters. It was shown that the mechanical properties depend from both the maximum temperature as wells as the temperature holding time.

In general, the temperature induced strength decrease for precipitation- and UFG-hardened aluminum alloys can be explained by three basic mechanisms. Firstly, the degradation of grain boundaries by recrystallization (reduction of fine-grain hardening effect). Secondly, the annihilation of dislocations by annealing (reduction of work-hardening effect) and lastly, the already mentioned dissolving of MgSi-clusters (reduction of precipitation-hardening effect) [7]. Thus, the aim of this investigation is to determine the possibility of an enhancement of ductility by introducing elevated temperatures during forming by thermomechanical analyzes. It is objected to identify a temperature range in which the UFG microstructure is maintained but softening mechanisms, like the dissolving of precipitations and the annihilation of dislocations, are already triggered.

2. Experimental setup

2.1. Material
The investigated material is a precipitation-hardening wrought alloy of the Al-Mg-Si system and is used under the numerical designation AA6014. Aluminum alloys of the 6000 series are already used in structural and outer skin lightweight applications for example in the automotive sector, due to their very good formability, excellent corrosion resistance and high hardening potential [8]. One reason for this are the finely distributed MgSi-precipitations which lead to higher resistance against dislocation movement. In Table 1 the chemical composition of the AA6014 aluminum Ac-PX by Novelis is shown. The sheets have a thickness of 1 mm and are solution annealed at 520 °C for 1 h in an air circulation chamber oven in order to accomplish a finely and homogeneously distribution of the MgSi-phase.

Table 1: Chemical composition in weight percentage of Novelis Ac-170 PX [9].

| Element | Si     | Fe    | Cu    | Mn    | Mg    | Cr    | Zn    | Ti    | Others. | Unit |
|---------|--------|-------|-------|-------|-------|-------|-------|-------|---------|-------|
| Concentration | 0.3 – 0.6 | 0.35  | 0.25  | 0.05 – 0.2 | 0.4 – 0.8 | 0.2   | 0.1   | 0.15  |          | Wt. % |

2.2. Accumulative roll bonding
The ARB process for the production of multi-layered sheets consists of the process steps of a surface treatment, the stacking of sheets, the cold rolling and a cutting operation. In Figure 1 the cyclic ARB process chain is depicted schematically.

![Diagram of ARB process chain](image)

Figure 1: Process steps for cyclic accumulative roll bonding according to [2].
The surface treatment is done by manual wire brushing with the handheld PolyPTX 800 (Eisenblaetter, Germany) with a rotating brush roller in rolling direction of the sheets. The brush has a diameter of 100 mm, a width of 70 mm and curled stainless steel filament. In past investigations it was shown that it provides a high level of roughness and a removal of the oxide layer which both are necessary for the bonding by cold welding in the later rolling step [10]. After the brushing treatment of two sheets, they are stacked together with their roughened side in contact. In the third step of ARB the actual bond formation by cold rolling is realized. For this, the two-layered sheet packages are transferred to an industrial sized rolling stand (Gerd Wolff GmbH, Germany). The targeted relative pass reduction is 50% which enables a constant sheet thickness of the multi-layered sheets compared to the initial material. The width of the rolled sheets also remains the same whereas its length doubles due to the halving of the sheet thickness. The rolling velocity was set to 3 m/min at a rolling pressure starting at 80 bar for the first rolling cycle. Finally, a laser cutting operation is done to cut the twice as long sheets in order to be able to stack them in the following ARB cycle. In this investigation up to four ARB cycles are carried out in order to produce 16-layered sheet material with a thickness of 1 mm. The number of rolling cycles is further referred as \(N\). Thus, N0 stands for the initial sheet material and N4 for the 16-layered sheets after four ARB cycles.

2.3. Tensile tests at elevated temperatures

In order to characterize the hot forming properties of multi-layered ARB sheets, tensile tests from 20 °C up to 300 °C were performed on the thermomechanical simulator Gleeble 3500 (Dynamic Systems Inc., USA) parallel to the rolling direction (RD). The aim is to identify a certain temperature range in which a good compromise between the strength and the ductility of the multi-layered sheets is apparent. The tensile tests at elevated temperatures are performed in accordance to DIN EN ISO 6892-2. For the investigation, tensile specimens were cut out of the 16-layered sheets by fine EDM in order to reduce effects on the mechanical properties through heat input (e.g. by laser cutting) or work hardening (e.g. by regular milling). Through the combination of a statistical pattern on the specimens surface and the optical strain measurement system Aramis (GOM GmbH, Germany), the actual strain during forming can be measured via the digital image correlation (DIC) method. The elevation of temperature is done by resistance heating and the controlling of the actual temperature of the specimen is realized via type K thermocouples which are attached in the horizontal and vertical centre of the measuring area in a prior welding step. In Figure 2 the experimental setup for thermomechanical characterization is shown.

![Experimental setup for the thermomechanical analyses.](image)

The temperature window for the hot tensile tests ranges from 80 to 300 °C with an heating rate of 10 K/s respectively and incremental temperature steps of 20 °C. The dwell time for the targeted temperature is 5 s. Thus, twelve different tensile tests are performed. The temperatures and the corresponding strain rates as well as recording frequencies for the DIC-method are depicted in Table 2. The frequency determines the number of images which are recorded during testing and thus, the temporal resolution of the image correlation. To eliminate the influence of the strain rate on the mechanical properties as far as possible, the traverse speed is adjusted. Thus, the otherwise increasing strain rate because of the elevation of temperature can be maintained constant at a value of 0.0067 1/s over the entire temperature range.
Table 2: Parameter values for tensile tests at elevated temperatures.

| Temperature in °C | Crosshead speed in mm/s | Targeted strain rate in 1/s | Frequency in Hz |
|-------------------|--------------------------|----------------------------|-----------------|
| 80, 100, 120      | 2.14                     | 0.0067 ± 0.01              | 50              |
| 140, 160          | 1.50                     | 0.0067 ± 0.01              | 50              |
| 180, 200          | 1.15                     | 0.0067 ± 0.01              | 50, 20          |
| 220, 240          | 1.00                     | 0.0067 ± 0.01              | 20              |
| 260, 280, 300     | 0.75                     | 0.0067 ± 0.01              | 20              |

3. Results and discussion

For the metallurgic analysis, images of the grain-structure were recorded by electron microscopy. In Figure 3 the derived grain sizes are illustrated. An ARB characteristic grain-structure is formed, which is defined by severely elongated grains in rolling direction. Already after two ARB repetitions (N2) the grain length is over 200 µm, starting from mean grain sizes around 20 µm in the initial condition (N0). The elongation is even more pronounced at higher number of ARB cycles. A different grain size distribution is observed considering in direction perpendicular to rolling where the ARB characteristic grain refinement is evident. Starting from mean grain sizes of 15 µm in the initial state (N0), grain sizes decrease to a mean size of only 600 nm after four rolling cycles (N4). This represents a decrease in grain size by 96 % from N0 to N4 in total. The number of samples tested is depicted as small letter n in the upcming diagrams.

As already mentioned in the state of art, this leads to a fine-grain hardening of the AA6014 alloy which results in an increase of the overall material strength according to equation 1 (Hall-Petch). One possibility to vary the actual ductility of metals is the elevation of temperature during forming. It can lead to dynamic recovery or to recrystallization which both enhances the materials formability.

In order to analyze this possibility for roll bonded AA6014, the mechanical properties of the yield strength YS, the ultimate tensile strength UTS, the uniform elongation UE and the total elongation TE are determined in correlation to the actual testing temperature. In Figure 4, the stress-strain curves, which are derived from the tensile tests at elevated temperatures as well as the associated mechanical properties of the yield strength YS, the ultimate tensile strength UTS, the uniform elongation UE and the total elongation TE are depicted in relation to the testing temperature for the 16-layered AA6014 specimens.
Regarding the exemplary stress-strain curves depicted in Figure 4 a), it is evident that there is a general tendency to a decreasing strength but increasing ductility level with higher temperatures during tensile tests as expected for hot forming of aluminum. Special characteristics show up when looking at the temperatures between 120 °C and 180 °C where the stress strain curves are rather similar regarding the general level of material strength (YS and UTS) as well as the total elongation TE.

Looking at the mean values for both the yield strength YS and ultimate tensile strength UTS in Figure 4 b), it is apparent that the yield to tensile strength ratio YS/UTS maintains a high value of over 0.94 in the entire temperature range. This is an indicator for the low plastic formability of the investigated 16-layered AA6014 sheets after four rolling cycles even at higher temperatures. Overall, there is a maximum in strength at 20 °C and in comparison only a minor decrease in strength up to 100 °C. Analyzing the mean value for UTS in the temperature range from 120 °C to 180 °C, it is evident that UTS declines in comparison to the average level in the temperature field from 80 to 100 °C by a relative value of 14 %. Focusing on the temperature range from 120 °C to 180 °C itself, a tendency to a strength plateau is apparent. Thus, only a minor decline in UTS from 120 °C to 180 °C with a relative value of 8 % is present. Regarding the strength properties at temperatures from 200 °C to 300 °C, a nearly linear decline of UTS and YS is evident. More concretely, with every temperature increment of 20 °C a drop in both yield as well as tensile strength of 24 MPa in average occurs.

Thus, it is assumed, that the thermal stability of the UFG microstructure decreases starting at 200 °C which is in accordance to previous investigations by Sharma et al. [11]. A distinct contribution in the strength increase with higher temperatures is attributed to the already identified dissolving of MgSi-clusters [6]. Regarding the rather short temperature holding time of 5 s in combination with forming times of 2 to 3 s it is suspected, that no pronounced recrystallization is possible. Geiger et al. [7] observed that even for a 256-layered ARB sheet of the relatively similar aluminum alloy AA6016, only a minor increase in grain size is apparent at holding times between 5 to 10 s at 300 °C. Thus, it is assumed that the major contribution for strength decline is initiated by the precipitation solution as well as the relaxation of internal stresses by dislocation annihilation and rearrangement at elevated temperatures. Overall, UE lies on a very low level. Taking the standard deviations into account, UE only surpasses the 1 % mark in the temperature range from 120 to 200 °C. Thus, it is noticeable that this local plateau of ductility lies in the same temperature window in which the strength values show their slowest rate of decline. Considering the total elongation TE, an almost linear progression with increasing temperature
is apparent. Pronounced ductile necking occurs only at temperatures above 140 °C where TE exceeds the 3 % mark and thus, shows a relative increase in ductility of 42 % in comparison to TE at 80 °C. All strain values were corrected in consideration of the coefficient of thermal expansion for aluminum.

4. Conclusion and outlook
In the scope of this work, UFG sheet material processed by accumulative roll bonding the wrought aluminum alloy AA6014 was characterized by thermomechanical analyzes. The aim was to identify a favorable temperature range in which the materials ductility can be enhanced without lowering the strength level significantly. It was observed that in a temperature range from 120 to 180 °C a slower decline in both yield strength YS as well as ultimate tensile strength UTS is evident and a relatively high strength level of over 250 MPa can be maintained until 180 °C. Additionally, the uniform elongation UE shows the highest values in this range of temperature. In combination with a total elongation TE over 3 % for temperatures equal or higher than 140 °C the proposed temperature range for forming 16-layered AA6014 lies between 140 and 180 °C. It is assumed that in the range of these temperatures no distinct recrystallization occurs, which is in accordance to previous investigations regarding UFG aluminum alloys of the 6000 series. In future investigational work, the assumptions based on the microstructural mechanisms need to be analyzed more specifically. For this, the method of laser-ultrasonic studies can be applied. It allows in-situ measurements of metallurgical processes like recrystallization, grain growth and phase transitions and thus, a more detailed insight on the temperature driven microstructural changes in UFG materials which could be accompanied by metallographic analysis to validate the method.

5. Acknowledgements
The authors gratefully acknowledge the German Research Foundation (DFG) for funding the research project 392174229 with the title “Improvement of the application characteristics of multi-layered sheet material for forming technology produced via accumulative roll bonding” at the Friedrich-Alexander-Universität Erlangen-Nürnberg.

6. References
[1] Azushima A, Kopp R, Korhonen A, et al. Severe plastic deformation (SPD) processes for metals. CIRP Annals 2008; 57(2): 716–35.
[2] Tsuji N, Saito Y, Utsunomiya H, Tanigawa S. Ultra-fine grained bulk steel produced by accumulative roll-bonding (ARB) process. Scripta Materialia 1999; 40(7): 795–800.
[3] Hansen N, Mehl RF, Medalist A. New discoveries in deformed metals. Metallurgical and Materials Transactions A 2001; 32(12): 2917–35.
[4] Hall EO. The Deformation and Ageing of Mild Steel: III Discussion of Results. Proc. Phys. Soc. B 1951; 64(9): 747–53.
[5] Borodachenkova M, Wen W 2017 High-Pressure Torsion: Experiments and Modeling. In: Cabibbo M, editor. Severe Plastic Deformation Techniques (London: InTech).
[6] Merklein M, Vogt U. Enhanced Formability of Ultrafine-Grained Aluminum Blanks by Local Heat Treatments. KEM 2009; 410–411: 169–76.
[7] Geiger M, Merklein M, Vogt U. Aluminum tailored heat treated blanks. Prod. Eng. Res. Devel. 2009; 3(4-5): 401–10.
[8] Bloeck M 2012 Aluminium sheet for automotive applications. In: Rowe J, editor. Advanced Materials in Automotive Engineering (Sawston: Woodhead Publishing) pp 85–108.
[9] Herrmann J 2020 Kumulatives Walzplattieren: Bewertung der Umformeigenschaften mehrlagiger Blechwerkstoffe der ausscheidungshaerbaren Legierung AA6014. Erlangen: FAU Univ. Press.
[10] Jamaati R, Toroghinejad MR. The Role of Surface Preparation Parameters on Cold Roll Bonding of Aluminum Strips. J. of Materi Eng and Perform 2011; 20(2): 191–7.
[11] Sharma SK, Hassan MS, Kumar BS. Annealing Response of Aluminum Alloy AA6014 Processed By Severe Plastic Deformation. International Refereed Journal of Engineering and Science (IRJES) 2017; 6(7): 59–65.