31.0 ACCUMULATIVE ROLL BONDING OF ALUMINUM SHEETS TOWARD LOW TEMPERATURE SUPERPLASTICITY

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This project initiated in Fall 2017. The research performed during this project will serve as the basis for a Ph.D. thesis program for Brady McBride.

31.1 Project Overview and Industrial Relevance

Accumulative roll bonding (ARB) is a severe plastic deformation technique used to produce ultra-fine-grained material by introducing large plastic strains via rolling [31.1]. The surfaces of two sheets are wire-brushed, stacked and roll bonded together in a conventional rolling mill [31.1]. After rolling, the material is sectioned in half and the process is repeated. A single-pass, 50% rolling reduction is commonly employed to ensure adequate bonding and to retain the original dimensions after each cycle [31.1]. The ARB process is largely different from conventional rolling processes in that heavy, unlubricated, single-pass reductions are used. This imparts redundant shear into the surface of the rolled material which is introduced through thickness with subsequent roll bonding cycles [31.2]. The combination of redundant shear and large rolling reductions ultimately lead to grain refinement.

ARB is attractive in its ability to produce ultra-fine-grained material with conventional processing equipment while maintaining consistent sample geometry. With the accumulation of large strains, dislocation cell structures form within the material that further develop into refined grains [31.3]. Ultra-fine grains (~250 nm) produced after 5 cycles of ARB in Al 5083 have exhibited tensile elongations in excess of 200% for strain rates of 10^{-3} s^{-1} at 200 °C [31.4]. In comparison, superplastic deformation of Al 5083 produced with conventional processing methods typically requires temperatures of 500 °C and strain rates above 10^{-3} s^{-1} to produce elongations around 300%. Enhanced superplasticity provided by the ARB process would be beneficial to superplastic sheet forming operations where reduced temperatures and/or increased strain rates could lead to cost savings and reduced die wear. Additionally, ARB processing may produce sheets that retain a submicron grain structure after forming operations, leading to increased strength in the final part.

31.2 Previous Work – Production of Samples and Characterization of ARBed Microstructure

Previous work for this project has focused on the bulk production and microstructural characterization of samples produced by ARB. One hundred tensile specimens of Al 5083 were produced using the same ARB processing method, which included preheating pairs of 1 mm sheets of material at 250 °C and roll-bonding them together with a single 50 % reduction pass. This process was repeated for 5 ARB cycles. Preheating was used to encourage bonding and reduce flow stress, but was restricted to only 5 minutes to avoid recovery and recrystallization. Issues with edge cracking were mitigated by use of rolling in sacrificial constraint frames as detailed in previous reports and publications [31.5].

The microstructures achieved after each ARB processing cycle were characterized using electron backscatter diffraction (EBSD) and are summarized in **Figure 31.1**. The grain size decreases with each subsequent cycle. After 5 ARB cycles the grain size approaches 250 nm and the high angle grain boundary (HAGB) fraction saturates around 65 % [31.6]. Grains also become highly elongated after the 4th and 5th cycle. Due to the saturation of grain size and HAGB fraction, 5 ARB cycles were chosen to create the baseline microstructure for this project to be tested for superplasticity with different parameters, including strain rate, temperature, and static annealing. This starting microstructure is henceforth referred as the *ARBed* microstructure.

31.1 Previous Work – PhD Proposal

Preliminary results from previous reporting periods were compiled into a document that served as a formal PhD proposal for this project. The following research hypotheses were formed from this information:

1. Thermomechanical processing by means of severe plastic deformation and static heat treatments can be used to produce a submicron grained microstructures conducive for superplasticity.
2. Deformation mechanisms (i.e. grain boundary sliding (GBS), dislocation creep) can be selectively activated in a submicron microstructure through proper selection of strain rate and deformation temperature to encourage superplastic flow.

3. The combination of severe plastic deformation processing and deformation mechanism selection provides enhanced uniaxial superplastic behaviour over conventionally processed material.

31.3 Recent Progress

31.3.1 Static Annealing Trials on ARBed Microstructure

Static annealing trials were conducted on the ARBed microstructure to determine microstructural stability. Prior to tensile testing for superplasticity, samples are preheated at the deformation temperature for 15 minutes to thermally equilibrate with the furnace and tooling, during which time significant recovery or recrystallization may occur; it is therefore imperative to understand the microstructural evolution that takes place during static annealing. Microstructural evolution was characterized via EBSD for samples statically annealed in an air furnace for 15 minutes, and the results are summarized in **Figure 31.2.** Grain boundary maps obtained from the transverse direction are shown in **Figure 31.3.**

There appears to be a transformation that occurs upon heating 250 °C, where elongated grains evolve into near-equiaxed grains. Similar microstructural changes have been observed from the transverse direction in severely deformed Al 1100 and Al-3%Mg at temperatures of 225 and 275 °C, respectively [31.6, 31.7, 31.8]. This evolution of grain morphology has been described as "grain growth accompanied by dislocation recovery", consistent with broad continuous peaks as observed with differential scanning calorimetry (DSC) [31.6,31.7,31.8]. The absence of distinct nucleation and growth events is thought to be due to the stability of high angle grain boundaries produced by severe plastic deformation, which were formed by "in-situ" dynamic recrystallization [31.9].

The thermal stability of severely deformed Al 5083, differing from Al-3%Mg with the addition of dispersoid-forming Mn, has not been comprehensively studied. Annealing of severely deformed Al as observed via TEM from the normal direction has been described as showing a reduction in dislocation density within grain interiors and grain boundaries, with only minimal grain growth [31.4,31.10]. The planar cross-section does not necessarily capture the elongation of grains in the rolling direction and may not accurately describe the 3-dimensional grain morphology. The annealing results presented in **Figures 31.2 and 31.3** show that grain morphology varies markedly with static annealing as viewed in the longitudinal plane. It is believed that a form of continuous recrystallization, as described previously with Al 1100 and Al-3%Mg, occurs near 250 °C, albeit with slightly different onset temperatures due to the presence of grain-boundary-pinning dispersoids.

The thermal stability of microstructure is pertinent in optimizing superplasticity. Recrystallization or grain growth of preferential orientations may lead to significant texture changes in the material that affect deformation mechanisms. As an example, GBS may be encouraged if the majority of grains are not oriented preferentially for slip in the tensile direction. Similarly, grain growth is to be avoided if GBS is to be desired as the ideal deformation mechanism. Some studies have suggested GBS may occur at temperatures as low as 200 °C assuming grains are equiaxed with sufficient HAGB character [31.11].

Additional analysis will be conducted to determine the microstructural changes that occur during static annealing of the ARBed Al 5083 microstructure. DSC will be used to assess the thermal activation of mechanisms for microstructural evolution. X-ray diffraction will be used to quantify the evolution of texture components and reduction of strain with static annealing. Time-permitting, bulk texture techniques, such as neutron diffraction, will be conducted via the HIPPO diffractometer at Los Alamos National Laboratory (LANL) to assess bulk texture. Collectively, this information paired with transmission electron microscopy (TEM) will be used to make conclusive arguments about the microstructural evolution.

31.3.2 First Round of Tensile Tests

An initial round of tensile testing was conducted to evaluate strain rates and temperatures of interest. This round of testing included strain-to-failure and strain-rate-jump tests conducted with strain rates between 5 x 10^{-4} and 5 x 10^{-3} s^{-1} and temperatures between 225 and 275 °C. Tensile elongations to failure are summarized in **Figure 31.4**. Tensile elongations are highly dependent on tensile testing parameters, with elongations near or above 200% for temperatures and strain rates at or below 250 °C and 1 x 10^{-3} s^{-1}, respectively. Based on the static annealing trials summarized in **Figure 31.2**, it is evident the grain growth exhibited at 275 °C is not conducive for superplasticity with the testing parameters used. These results are similar to those conducted from tensile testing of severely warm rolled Al 5083 by

Hsiao and Huang, who concluded that grain boundary sliding was disrupted by a bimodal grain size distribution resulting from incomplete grain growth [31.11].

The samples tested at 225 and 250 °C exhibited the highest tensile elongations and were further investigated with strain-rate-jump tests. The flow curves and strain rate sensitivity values are summarized in **Figure 31.5**. For samples tested at 225 °C, strain rate sensitivity values stabilized just above 0.4, suggesting grain boundary sliding ($m \approx 0.5$ [31.12]) with accommodation as the primary mode of deformation. This is contrary to the samples tested at 250 °C, where the strain rate sensitivity values continuously decreased from above 0.5 to around 0.3, representative of grain boundary sliding followed by dislocation creep, respectively [31.12]. By comparing the flow curves in **Figure 31.5** with the static annealing data in **Figures 31.2** and **31.3** it appears that grain growth reduces the tendency for grain boundary sliding, thus leading to lower strain rate sensitivities. The greater elongation exhibited at the higher strain rate ($1\text{x } 10^{-3}\ s^{-1}$) suggests competition between deformation rate and grain growth. Further characterization via TEM is needed to draw conclusions about the observed tensile properties, and TEM foils have been prepared from interrupted strain tests conducted to strains of 0.35 and 0.75 for future analysis.

The results herein illustrate how superplastic elongation depends on temperature, strain rate and microstructural stability. It is has become apparent that microstructural evolution occurs around 250 °C to produce near-equiaxed submicron grains, while extensive grain growth occurs with prolonged exposure at or above 250 °C. A requirement for GBS is a homogenous microstructure of small, equiaxed grains [31.12]. In the future, it may be advantageous to use the microstructural evolution which occurs around 250 °C to form equiaxed grains from the ARBed microstructure, and then tensile test at lower temperatures to avoid grain growth. It has been suggested that GBS may operate at temperatures as low as 200 °C in submicron grained aluminum [31.11].

31.3.3 Plans for Next Reporting Period

For the next reporting period, the following items will be pursued:

- Complete DSC analysis on ARBed microstructure to further investigate recrystallization phenomena.
- Analyze microstructure of sampled strained to true strains of 0.35 and 0.75 to compare with observed tensile strain rate sensitives and flow curve behavior.
- Investigate two-step testing procedure: annealing around 250 °C and tensile testing at lower temperatures.

31.4 References

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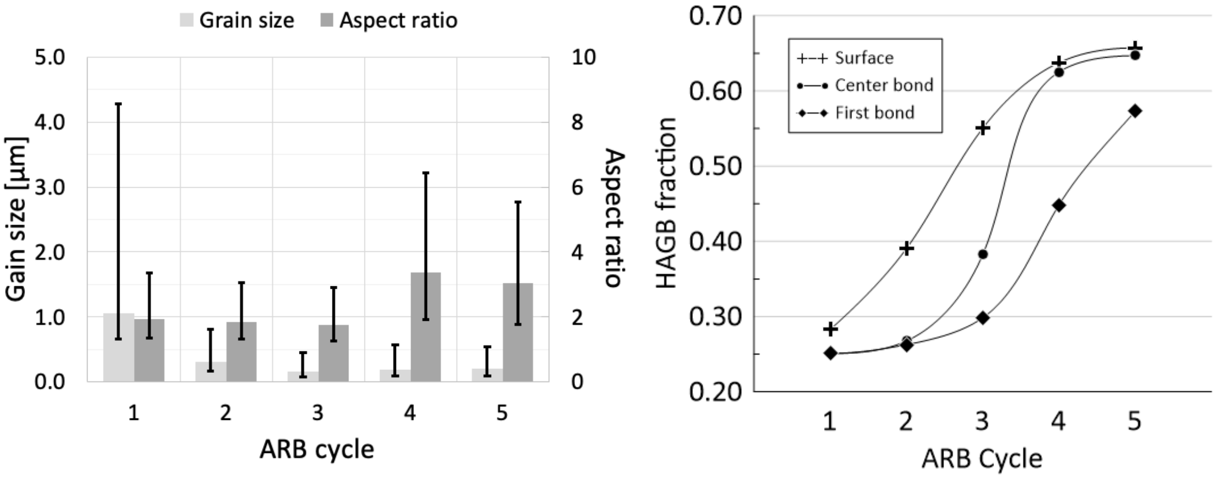
31.5 Figures and Tables



(a) (b)

Figure 31.1: Summary of (a) grain size, aspect ratio and (b) HAGB fraction obtained from EBSD of cross-sections containing the RD and ND for consecutive ARB processing cycles at indicated locations through the sheet thickness. Error bars represent the median 80% of data. HAGBs are defined as misorientations greater than 30°. Grain size reported using an equivalent area ellipse method, with the primary ellipse axis reported.

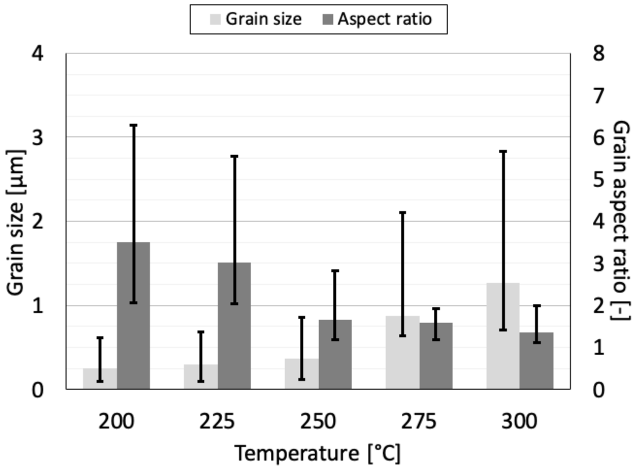


Figure 31.2: Summary of grain size and aspect ratio obtained using EBSD of longitudinal sections of ARBed samples after static annealing for 15 minutes. Error bars represent the median 80% of data. At 250 °C grains are submicron and equiaxed.

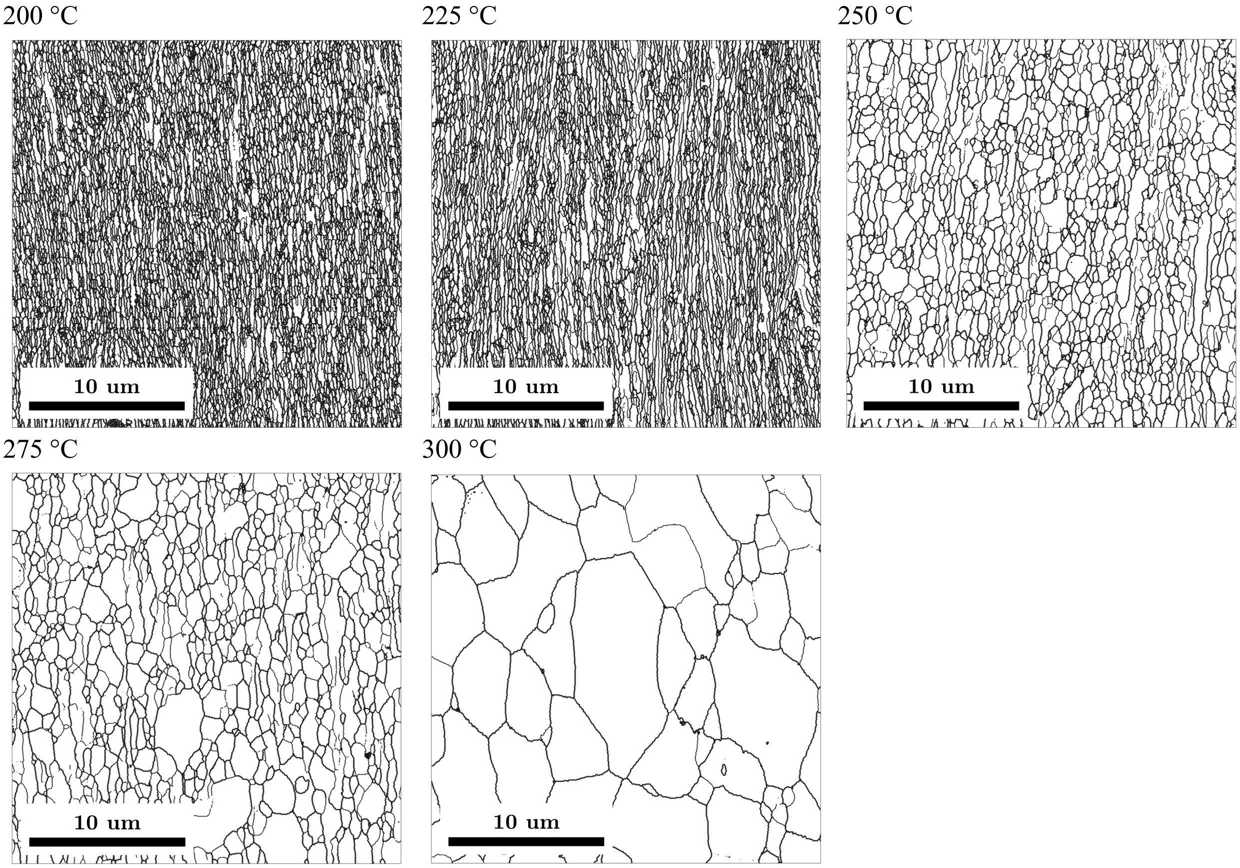


Figure 31.3: Grain boundary maps obtained via EBSD of ARBed microstructure after static annealing for 15 minutes at the indicated temperatures. Dark lines represent HAGB with misorientations greater than 30°, light lines represent low angle grain boundaries (LAGB) with misorientations less than 30°.

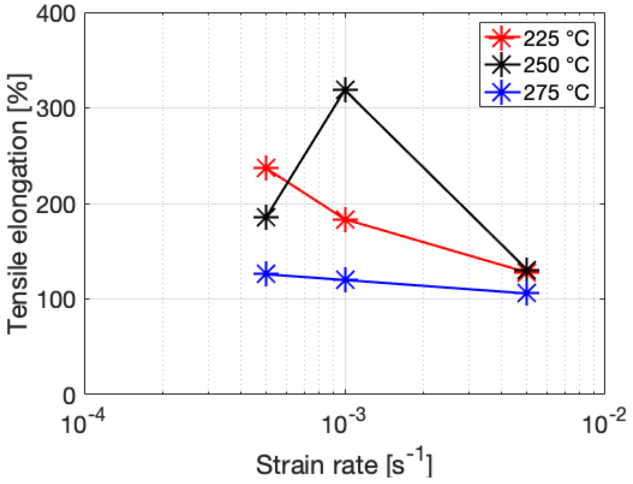
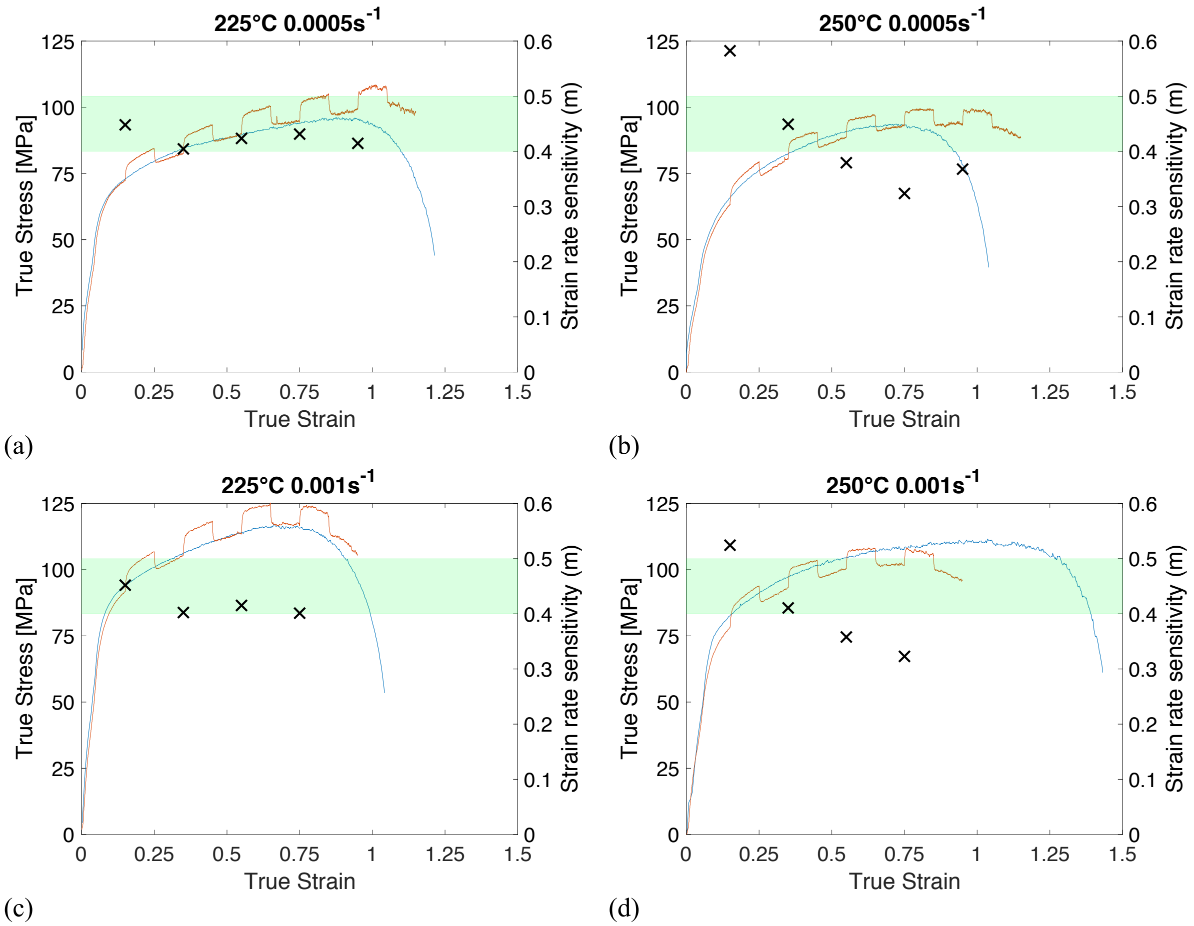


Figure 31.4: Total uniaxial tensile elongation for as-ARBed microstructures tested at indicated strain rates and temperatures. All samples were preheated for 15 minutes at the testing temperature prior to testing. All testing was conducted at constant strain rates.



(a) (b) (c) (d)

Figure 31.5: Flow curves and strain rate sensitivity values for uniaxial tensile tests conducted on ARBed samples at 225 °C (a,c) and 250 °C (b,d) for different strain rates. True stress and true strain values are obtained assuming uniform elongation with volume constancy per ASTM E2448. The blue curves represent strain-to-failure at the indicated strain rate and the orange curves represent strain-rate-jump tests to determine strain rate sensitivities. Strain rate sensitives were determined by systematically increasing and decreasing the strain rate by 20 % from the nominal strain rate at different strain levels and are overlayed as a function of strain with markers. The shaded green area represents deformation commonly attributed to grain boundary sliding ($m \approx 0.5$) [31.12].