31.0 ACCUMULATIVE ROLL BONDING OF AL AND TI SHEETS TOWARD LOW TEMPERATURE SUPERPLASTICITY

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31.1 Project Overview and Industrial Relevance

Accumulative roll bonding (ARB) is a severe plastic deformation technique used to produce ultra-fine grain material by introducing large plastic strains within a material [31.1]. The surfaces of two sheets are prepared in a particular manner before being 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 sample's original dimensions after each roll bonding cycle [31.1]. The ARB process is largely different from conventional rolling processes in that heavy single-pass reductions are conducted without lubrication. This introduces redundant shear into the surface of the rolled material, which is introduced through the material's thickness with subsequent roll bonding cycles [31.2].

The attraction to ARB lies in the ability to produce ultra-fine grained material with conventional processing equipment. With the accumulation of large strains, dislocation cell structures form within the material that further develop into refined grains [31.3-4]. Significant grain size strengthening is observed after one or two ARB cycles, which tends to reach a plateau after around 6 cycles [31.3]. Grain sizes as small as 280 nm have been obtained in Al 5083 after 5 cycles [31.5]. Ultra-fine grains produced by ARB can also lead to superplastic responses at lower temperatures. Superplastic elongations above 200% have been exhibited by ARBed Al 5083 at strain rates of 10^{-3} s^{-1} and temperatures as low as 200 °C [31.5]. In comparison, superplastic Al 5083 produced with conventional processing methods typically requires temperatures of 500 °C and strain rates of no more than 10^{-3} s^{-1} to produce elongations of 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.

31.2 Previous Work – Development of ARB Process & Understanding Bonding Mechanisms

Previous work included the development of tooling and procedures needed to replicate an accumulative roll bonding process at Colorado School of Mines (Mines). Rolling mill edge guides were designed and machined to ensure both sheets of material enter the mill in a straight orientation. A digital load cell data acquisition system was installed and configured to measure rolling loads in real-time. This real-time rolling load data can also be used to see how process parameters, such as rolling temperature and friction conditions, affect rolling loads, and to ensure the capacity of the mill is not exceeded.

A specific surface preparation procedure was developed based on literature review and initial experiments to ensure adequate bonding is obtained. The passivation layer found on aluminum alloys prohibits bonding between two sheets during cold rolling. To circumvent this issue, sheets can be wire brushed prior to rolling, which creates a very brittle surface layer on top of the preexisting passivation layer [31.6]. This brittle wire-brushed surface layer, which cannot elongate as much as the underlying material, fractures during rolling to expose the virgin material beneath it, thus allowing bonds to form [31.3]. Bonding that occurs during high temperature rolling is less understood. Studies of aluminum alloys have shown wire-brushed surface layers to exhibit comparable ductility to the underlying material when roll bonded at 300 °C, while also producing higher bond strengths [31.7]. Some studies attribute the enhanced bond strength to diffusion between fine-grained surface material produced by wire brushing [31.7], whereas others attribute it to enhanced ductility of the underlying material at elevated tempeartures which can more easily flow between fractures in the surface layer to form bonds [31.8]. Nevertheless, roll bonding at higher temperatures has been shown to increase bond strength over cold rolling.

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During accumulative roll bonding, high temperature rolling is advantageous in that it enhances thermal energy available for bonding and reduces flow stress of the material. Conversely, high temperature rolling can also lead to rapid recrystallization and grain growth, negating accumulated strain and grain refinement in the material. Thus, "warm rolling" is often practiced either at or just below the recrystallization temperature to achieve the benefits of enhanced bonding in conjunction with reduced flow stress. In the case of Al 5083, preheating at 200 °C for 5 minutes is common practice, although preheating as high as 300 °C has shown to still provide enhanced mechanical properties after 6 cycles [31.9].

31.3 Recent Progress

31.3.1 Modification of ARB Process to Reduce Edge Cracking

Previous roll bonding trials of Al 1100 highlighted edge cracking that becomes increasingly severe at higher strain levels. During these trials, two sheets were sheared to the same width, stacked, and loosely constrained by lateral edge guides while entering the mill. Although the edge guides aided in maintaining proper alignment as the material entered the roll gap, the sheets still had freedom to move laterally with respect to each other, resulting in a portion of one sheet overhanging the other sheet. During high reduction rolling passes, this overhanging portion of material is not compressed and therefore does not elongate longitudinally with the bulk of the material. As a result of this tensile stress state, severe edge cracking of the overhanging material occurs, an example of which can be seen in **Figure 31.1a**.

A revised method was employing to mitigate material overhang during rolling. This method consisted of drilling small holes into the sheets prior to wire brushing and binding the sheets tightly together with thin copper wire, as shown in **Figure 31.2a**. The combination of lateral edge guides and wire binding has significantly reduced the amount of edge cracking due to material overhang. With proper material constraint, cracking largely occurs due to lateral spreading of the material with high reduction deformation passes, which can be seen in **Figure 31.1b**. This issue of reducing lateral spread is one of the main topics to investigate for the next reporting period. Possible techniques to be explored include altering the roll surfaces to increase friction and rolling strips with sacrificial material on each side to further constrain the strips in the roll gap and reduce lateral spreading.

31.3.2 5 Successful Cycles of Al 5083

After demonstration of a successful ARB process with Al 1100, preliminary trials were conducted on 1 mm sheets of Al 5083, as shown in **Figure 31.2b**. If kept below the static recrystallization temperature, preheating before rolling is thought to provide multiple benefits to the bonding process, including reduced flow stress of deformation and enhanced bond strength. Reducing the flow stress is critical for being able to roll bond samples with large, single-pass reductions. Preliminary experiments at Mines have shown that preheating at 250 °C for 5 min sufficiently reduces the flow stress of the material to process 50 mm wide strips of material within the load capacity of a 50-ton laboratory scale rolling mill.

With use of shearing, wire brushing, stacking, binding and preheating sheets prior to each roll bonding step, as many as five subsequent cycles of ARB have been conducted on Al 5083. It is worth noting that significant edge cracking still exists after the second ARB cycle. Inspection of edge cracks indicate that both sheets were well aligned in the rolling process with help of the copper wires, which suggests material overhang does not appear to be the primary cause of edge cracking. Measurements of sheet width before and after rolling show that about 5 % lateral spreading occurs on single-pass 50% reductions at 250 °C. This lateral spreading leads to longitudinal tensile stresses in the edges of the material, which leads to edge cracking.

31.3.3 Preliminary Microstructural Evaluation

Microstructural techniques for grain size determination were replicated using both ARBed Al 1100 and Al 5083. As grain contrast is difficult to achieve in aluminum alloys with conventional etching, techniques such as anodizing, electron backscatter diffraction and transmission electron microscopy were used to characterize heavily deformed microstructures.

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Electrochemical anodizing with fluoroboric acid, known as Barker's Reagent, creates a layer of aluminum oxide on the surface of samples that creates an interference pattern when viewed under polarization light. As the cells of aluminum oxide grow based on the orientation of the originating surface grain, different cell orientations create different interference patterns, which lead to colorful grain contrast. This technique can be used to view bulk deformation structure and grain sizes on the scale of about 10 μm.

Transmission electron microscopy (TEM) and electron backscatter diffraction (EBSD) are more suited to obtaining grain size for submicron sized grains. TEM micrographs for Al 1100 and Al 5083 subject to 8 and 5 cycles respectively are shown in **Figure 31.3.** From these micrographs it can be seen that grain sizes on the scale of about 200 nm to 500 nm have been achieved through ARB. An inverse pole figure map was collected on 8 cycle Al 1100 using a 20 kV accelerating voltage; the raw map with an average confidence index (CI) of 0.09 is shown in **Figure 31.4a** prior to any post-processing. A post processing filter was applied to remove low confidence points – those with a confidence index less than 0.1 – and is shown in **Figure 31.4b** to give an average CI of 0.24. It can be seen that the grain interiors are indexed with a high level of confidence, whereas the regions near grain boundaries, which remain unclear, are not indexed as easily.

Similar EBSD scans were attempted on 5 cycle Al 5083, but the majority of scanned points produced multiple Kikuchi patterns. Although EBSD is a surface measurement technique, there is a finite interaction volume of the material that produces the Kikuchi patterns at each scanned location. As the grain size of 5 cycle Al 5083 is smaller than that of the 8 cycle Al 1100, it is possible that submicron sized grains beneath surface grains are also contributing to Kikuchi patterns, which is preventing proper indexing. Other analysis techniques that use thin foil samples, such as transmission Kikuchi diffraction (TKD), will be investigated in the next reporting period to see if better results can be obtained for quantifying grain size.

31.4 Plans for Next Reporting Period

Successful accumulative roll bonding of Al 5083 up to five cycles has proven the developed ARB process to be feasible and repeatable. Additional steps will be taken to in the upcoming months to further this project, including:

- Experiment with different friction conditions and constraint mechanisms to reduce edge cracking in ARBed sheets by reducing the amount of lateral spreading.
- Conduct high temperature tensile testing of conventionally superplastic and ARBed 5083 to demonstrate low temperature superplasticity.
- Explore transmission Kikuchi diffraction (TKD) as a means of quantifying ultrafine grain structures.
- Begin experimenting with titanium alloys, including commercially pure titanium and Ti 6Al-4V.

31.5 References

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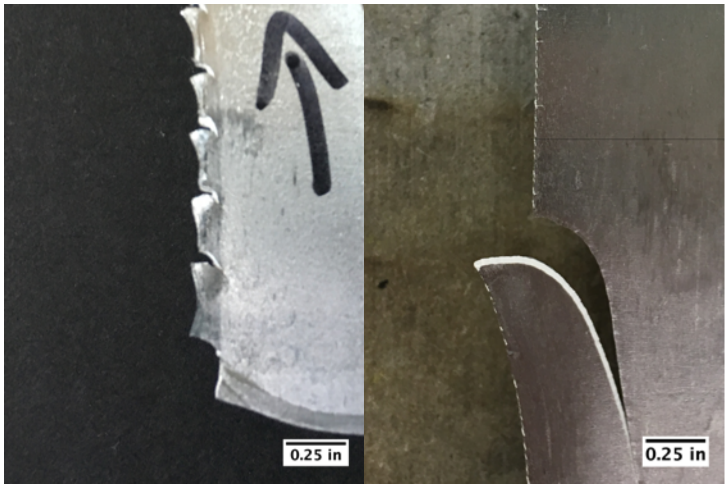
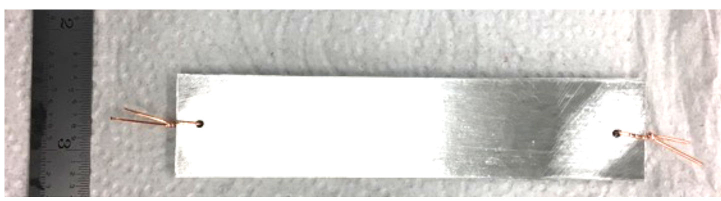
31.6 Figures and Tables

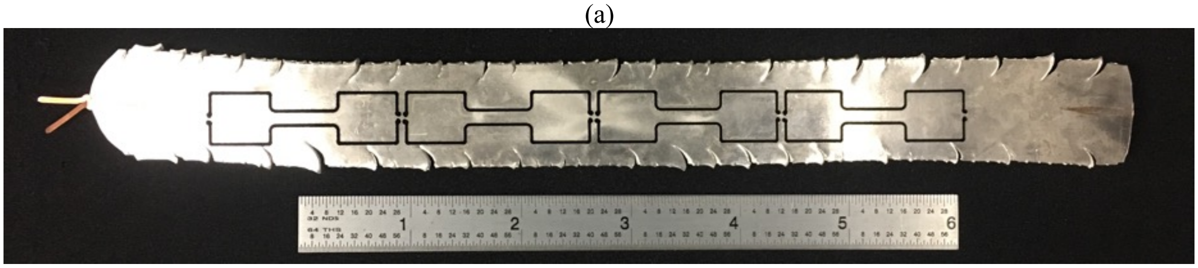


(a) Al 2024 after 2 ARB Cycles (b) Al 5083 after 3 ARB cycles

Figure 31.1: Edge cracking mainly caused by material overhang (a) and lateral spreading (b). In (a), the edge of underling material can be seen by faint line at the tip of all cracks. In (b), material overhang cracking was reduced by using copper binding wire to constrain sheets together; cracking instead resulted from tensile stress state that develops along the edge due to high lateral spreading. Rolling direction is bottom to top in both images.



(a)



(b)

Figure 31.2: Al 5083 before (a) and after (b) 5th ARB cycle at 250 °C. The use of copper binding wire is shown in (a), which led to reduced material overhang cracking in (b). Scale bars are in inches.

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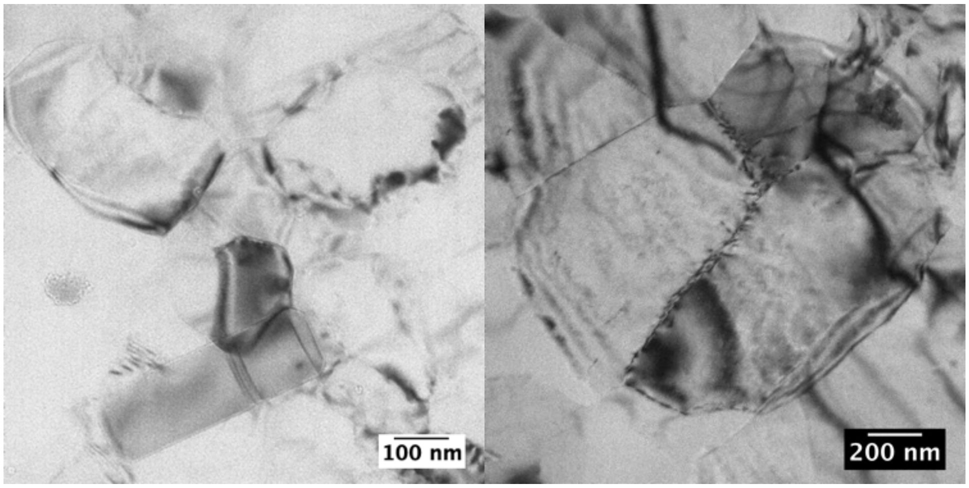
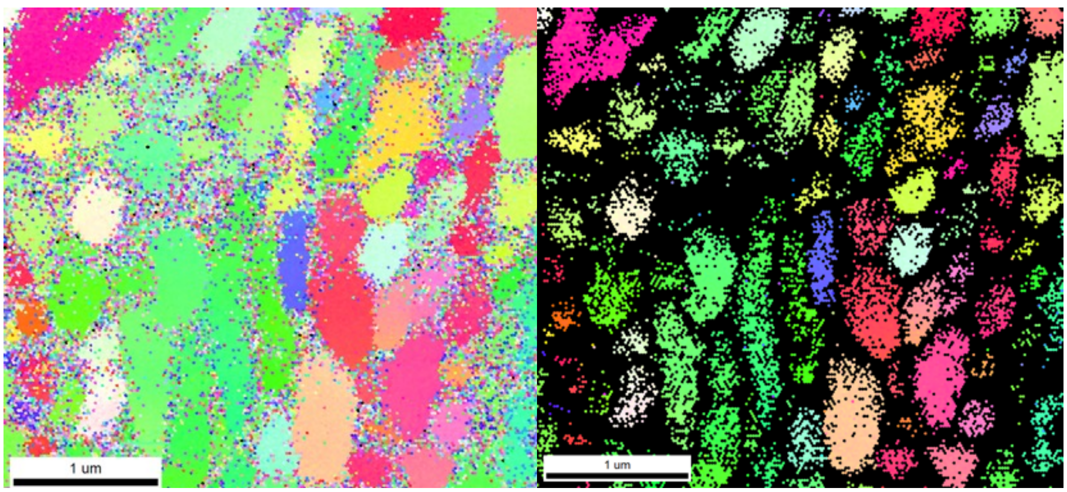


(a) (b)

Figure 31.3. TEM micrographs showing submicron sized grains in (a) 8 cycle Al 1100 rolled at room temperature and (b) 5 cycle Al 5083 rolled at 250 °C.



(a) (b)

Figure 31.4. Inverse pole figure maps of 6 cycle Al 1100 rolled at room temperature. (a) shows raw data from EBSD scan, while (b) shows the same data filtered to remove points with a confidence index less than 0.1. It should be noted that grain interiors are indexed with high confidence.

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