Grain Structure Evolution in 7000 Series Aluminum Redraw Rod for Aerospace Applications

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Industrial Sponsors: Orin Reininga, Craig Evans

Abstract: The project investigates the annealing process of a precipitation hardening aluminum redraw rod produced by Vista Metals. After extrusion and rolling, annealing is used to restore ductility of the finished product. However, peripheral coarse grains can form at excessively high annealing temperatures, leading to inconsistencies in the product's mechanical properties. The effect of both hold time and temperature on this process was investigated through microscopy and hardness testing, with hold time standing out as the more significant variable.

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Background & Objective

Motivation: Grain defects were observed in Vista's hard alloy redraw rod after the product is extruded, rolled, and annealed. Specifically, the occurrence of blown-out grains that inconsistently compromise different sections of the product was identified as the primary issue.

Two primary microstructural challenges:

1. Continuous dynamic recrystallization (C-DRX)
   - At high temperatures and strains, sub-grain boundaries absorb dislocations and increase disorientation [1].
   - This occurs during hot rolling.
2. Peripheral grain growth (PGG) (Fig. 1)
   - Abnormally large grains along edges of sample (peripheral coarse grains, PCGs).
   - Compromise mechanical integrity.
   - Occur dynamically during extrusion or statically during annealing [1].

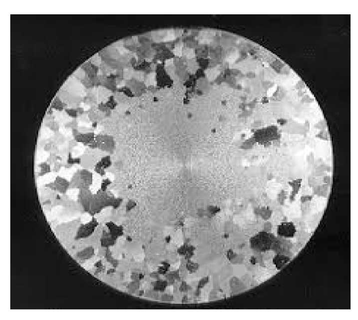

Figure 1. Example of Peripheral Coarse Grains [1]

Objective: We aim to aid with the optimization of Vista Metals' current heat treatment procedure by testing variations in their annealing parameters and tracking the evolution of peripheral grain growth through microscopy, while investigating changes in the uniformity of their product's mechanical properties through microhardness testing.

Methodology

Annealing: The purpose of annealing is to ensure that the product becomes fully recrystallized while preventing undesired grain growth. Hardness testing can help determine whether the final stage, grain growth, has been reached, at which point the material becomes "dead soft", and uncontrolled grain growth occurs.

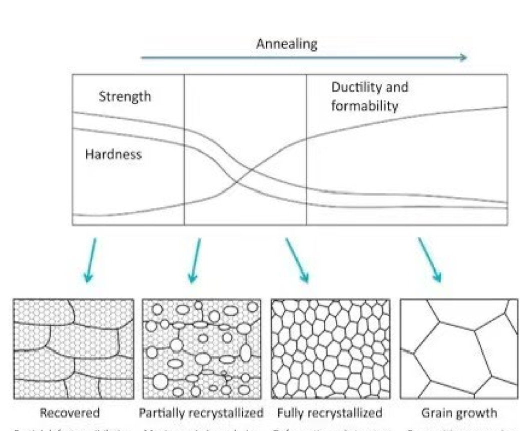

Figure 2. Schematical diagram illustrating the most generic steps during recovery, static primary recrystallization, and grain growth [2]

Table 1. Table shows the 2 rounds of experiments done.

<table>
<tr><th></th><th colspan="5">Round 1 - Time Variation</th></tr>
<tr><td>Hold Temp [F]</td><td colspan="5">670</td></tr>
<tr><td>Hold Time [min]</td><td>15</td><td>30</td><td>60</td><td>120</td><td>240</td></tr>
<tr><th></th><th colspan="5">Round 2 - Temperature Variation</th></tr>
<tr><td>Hold Temp [F]</td><td>620</td><td>670</td><td>720</td><td colspan="2">770</td></tr>
<tr><td>Hold Time [min]</td><td colspan="5">360</td></tr>
</table>

Experimental Design: The experiment was designed to observe microstructural progression during annealing, relating to temperature and time. A heat-up rate of 4°F per minute was used across all rounds of annealing. Head and middle samples were included in each set, as defects occurred most often in head samples, and least in middles.

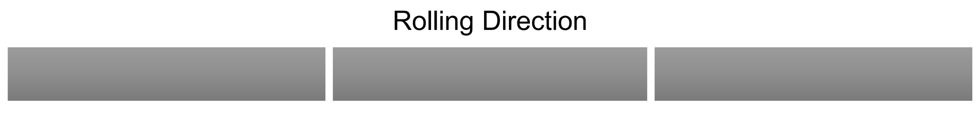

Figure 2. Diagram shows the sections taken out of the drawn rod based on the order at which it was drawn. The heads came out first and tails last.

Microscopy: Samples were polished up to colloidal silica and electrochemically etched with Barker's Reagent [4]. Imaging was done using polarized light to more clearly see grain contrast. Due to time constraints, microscopy was only done on Round 2, acting as anchor points for the progression of grain growth after recovery.

Hardness: Hardness testing was done across all samples, providing a proxy variable on annealing progression. The Vickers hardness method was used, with 18 indents taken moving from sample edge to center. As samples anneal, hardness is expected to decrease as heavily cold worked grains are replaced by new grain growth. With peripheral grain growth, differences in hardness between the edges and centers of the rods may appear [5].

Electropolishing & Microscopy

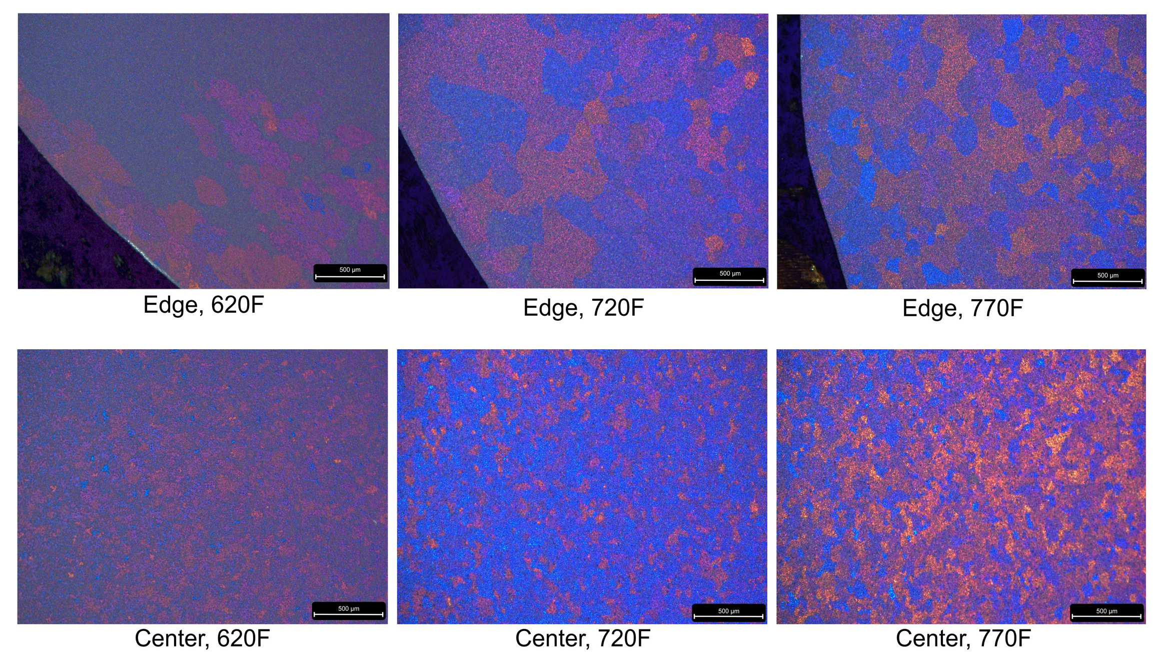

Figure 3. Micrographs of head section samples, all with a hold of 6 hours, at 50x.

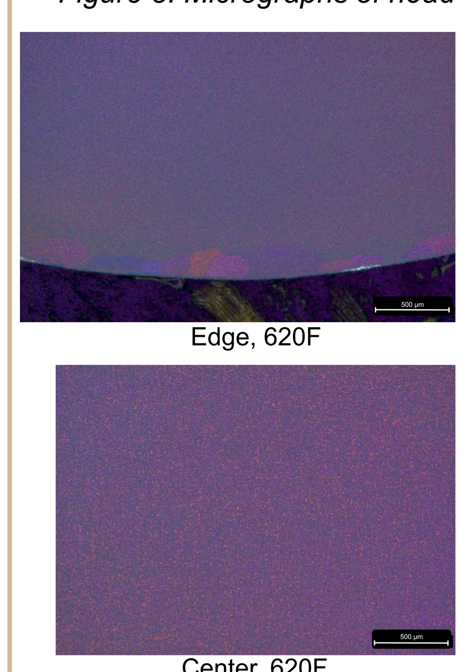

Figure 4. Micrographs of middle section samples, 6 hours, at 50x.

Large peripheral coarse grains can be seen along the edges of all samples, particularly the 720°F samples. The 770°F samples seem to have smaller grains overall. At 620°F, recrystallization and grain growth have begun, but not uniformly. Large grains are present at the edges, and no grains can be seen between the edge and the small, center grains. The 620°F samples also display the greatest difference between middle (Fig. 4) and head samples (Fig. 3), with significantly more grains present in the head.

Hardness Testing

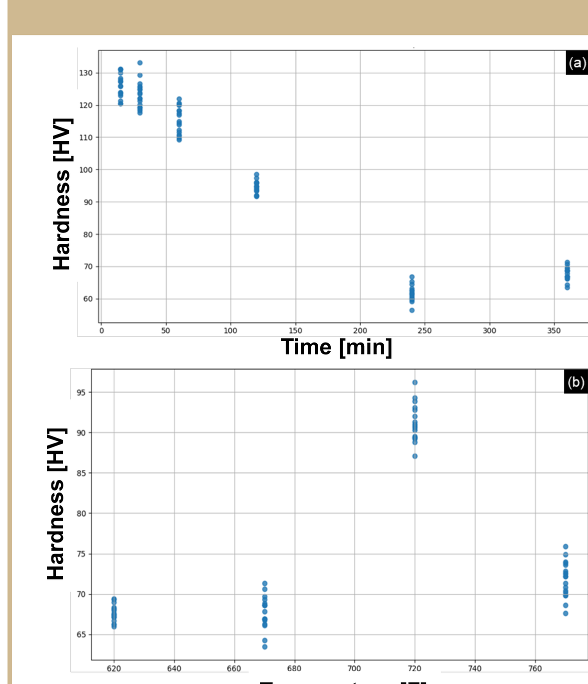

Figure 5. Graphs depicting change in head hardness data over (a) hold time variations and (b) hold temperature variations.

Hardness testing revealed that a four hour hold time led to a significant drop in hardness, indicating a "dead-soft" condition. A slight increase in hardness was noted between the 4 hours and 6 hours hold times. Contrary to initial expectations, peak hardness occurred in the 720°F samples, rather than the 770°F.

Annealing revealed strength differences between head and middle samples that originate from rolling, as seen in the unequal control sample hardness (Fig. 6). Earlier recrystallization and age hardening in head samples – especially at 620°F – lead to higher hardness, with a 95% confidence interval showing greater variation in the 6 – hour sample.

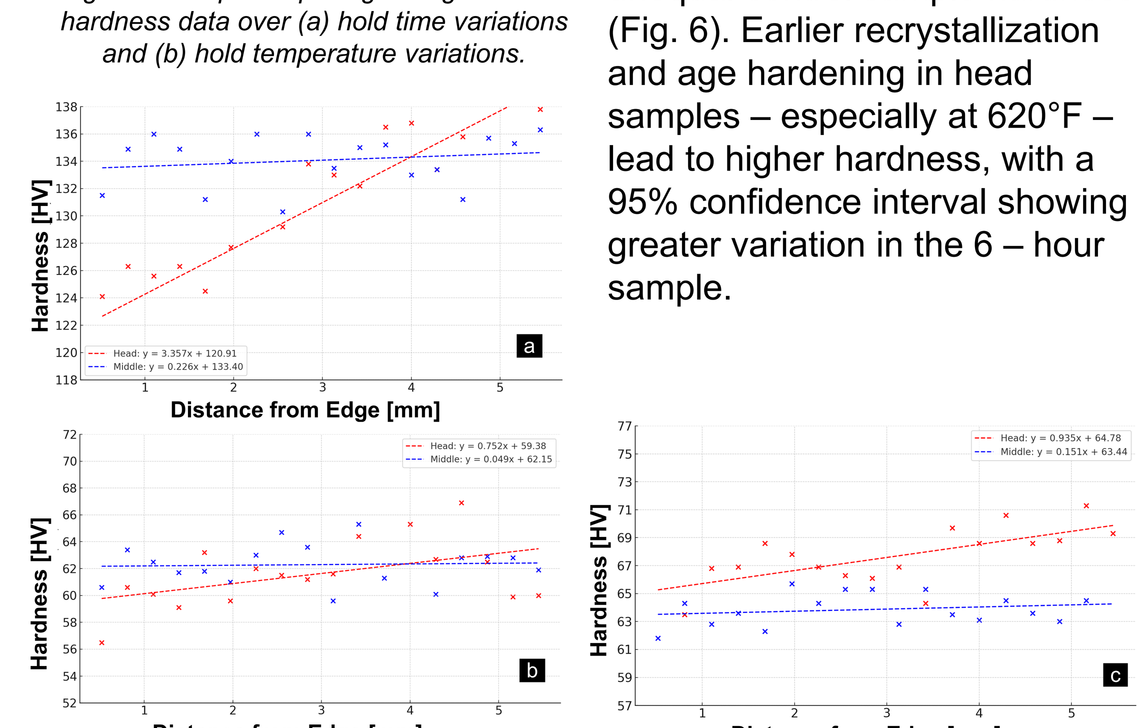

Figure 6. Hardness change trends from sample edge to center of head (red) and middle (blue) sections for (a) non-heated samples, (b) samples heated for 4 hours at 670F and (c) samples heated for 6 hours at 670F.

Table 2. Average hardness values for non-annealed controls, and both rounds of annealing.

<table>
<tr><th colspan="7">Average Hardness [HV]</th></tr>
<tr><td rowspan="2">Controls</td><td>Head</td><td colspan="5">130.93</td></tr>
<tr><td>Middle</td><td colspan="5">134.08</td></tr>
<tr><td rowspan="3">Round 1 - Time</td><td>Hold Time [min]</td><td>15</td><td>30</td><td>60</td><td>120</td><td>240</td></tr>
<tr><td>Head</td><td>125.33</td><td>123.08</td><td>115.33</td><td>94.54</td><td>62.04</td></tr>
<tr><td>Middle</td><td>129.29</td><td>124.94</td><td>121.49</td><td>92.91</td><td>62.53</td></tr>
<tr><td rowspan="3">Round 2 - Temp</td><td>Hold Temp [F]</td><td>620</td><td>670</td><td>720</td><td colspan="2">770</td></tr>
<tr><td>Head</td><td>67.61</td><td>67.41</td><td>90.77</td><td colspan="2">71.83</td></tr>
<tr><td>Middle</td><td>66.24</td><td>64.13</td><td>82.37</td><td colspan="2">68.61</td></tr>
</table>

Discussion & Results

Edge vs Center: Differences between edge and center are present after rolling (Fig. 6). After samples hit 'dead-soft', trends became less conclusive. The 620°F micrographs (Fig. 3) show PCGs at the onset of grain growth, which remain at higher temperatures. Due to data spread, any trends between hardness and grain size are inconclusive. It's unclear why 770°F grains are smaller than 720°F, since higher temperatures result in larger grains [5]. PCGs are likely unavoidable, but 4 hours of annealing at 670°F may minimize mechanical inconsistencies.

Head vs. Middle: Recrystallization and grain growth seem to begin earlier in head samples (Fig. 3-4). They likely retain greater cold work after rolling, possibly due to low or inconsistent mill temperatures. That additional cold work lowers the recrystallization temperature as dislocations release stored energy and speeds up annealing [6]. A 4-hour hold time resulted in comparable hardness values, confirmed by a 95% t-test. Above 6-hours, particularly at high temperatures, head samples are significantly harder. This could be a result of age hardening, as solute particles nucleate and grow to strengthen the material [2]. Initially higher cold work in head samples propagates throughout the annealing process as both recrystallization and age hardening begin earlier.

Hardness: The hardness trends for Round 1 match the expected progression of annealing (Fig. 7). Age hardening could potentially explain the Round 2 hardness trends seen in Figure 5B. Comparatively, at 670°F, age hardening may not have had a significant effect yet, while at 770F, particles may be too large to increase strength.

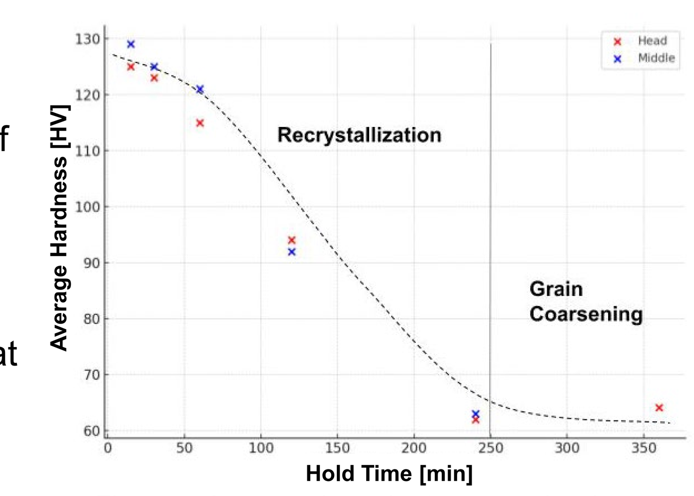

Figure 7. Annotated graph of average hardness vs. hold time at 670F

Hardness data is also consistently scattered when applying a 6 hour hold time, indicating the need for a shorter treatment to avoid entering the grain coarsening stage of the process.

Conclusions

Challenges: Due to the periodic nature of the issue, it is possible that the rods selected for testing did not contain the drawing-related defects responsible for the mechanical issues. Additionally, age hardening, a phenomenon observed in certain metallic alloys, may have influenced some samples that were not tested immediately that remained in storage for an extended period.

Recommendations: It is recommended that the annealing time be reduced to approximately 4 hours, as dead-softness is achieved earlier than the current duration. Beyond this point, the grain structure becomes less uniform.

Future Work: Further investigation should have additional microscopy samples to more clearly identify the boundary at which peripheral grain growth initiates. To improve the clarity of the hardness-time relationship, further time variation experiments should be done to build a library of Hardness vs. Time curves, allowing for a more precise characterization of the trend. Moreover, testing must be performed immediately after annealing to minimize the influence of age hardening, thereby preserving the accuracy and reliability of the results

Acknowledgements & References

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