Influence of Mn in Solid Solution in Softening of AA3003 Alloy During Annealing

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<u>Abstract</u>

The present paper shows the variation in softening of 3003 alloy during annealing at final product gauge caused by fluctuation of Mn in solid solution prior to the anneal. The fluctuations of Mn in solid solution mainly are attribute in ingot preheat and/or hot rolling practice changes.

The control of Mn in solid solution prior final anneal is necessary to achieve stable mechanical properties in back annealed tempers (-H2X) of 3003 alloy.

1. Introduction

The Aluminum Association (AA) 3003 alloy, typically containing 1.1-1.5% Mn, is extensively used in mill finish sheets and also in bright finish tread plates with various patterns embossed on the metal surface by an engraved work roll. The typical tempers of 3003 alloy used for these products are the back annealed tempers designated "-H2X".

It has been reported that the recovery softening dynamics during annealing can be slowed by increasing Mn solute atoms and/or Mncontaining dispersoids. These reduce dislocation motion and delay the recovery process [1,2]. The current paper summarized the annealing curve variations observed in 3003 alloy due to different amounts of Mn in solid solution (SS) prior to final annealing.

Coils of DC-casted, AA3003 alloy material were cold rolled to the final product gauge without inter-annealing. Full width mill finish samples in the cold rolled state (-H1X) were collected at the final gauge from 5 coils in order to determine their softening curves by laboratory annealing tests. The coils No. 1 and 2 were produced by a different preheating/hot rolling practice (called process No 1) relative to the coils No. 3, 4 and 5 (process No 2).

Sheet electrical conductivity was measured in the cold rolled state and also in the lab annealed state to determine the amount of Mn in SS prior annealing and the retain Mn in SS in the laboratory annealed samples at each annealing temperature.

Grain structure analysis of the lab annealed samples was carried out in order to determine the start and finish of recrystallization process and the recrystallized grains fraction percentage at each laboratory annealing temperature

2. Softening Rate During Annealing of Final Gauge AA3003 Alloy

Laboratory annealing cycles were carried out in the final gauge sheet samples. The laboratory heating rate to the annealing temperature was kept constant in all annealing tests at 25 °C per hour, which similar to the industrial batch annealing heating rate. The soak time in all laboratory annealing tests was kept constant at 4 hours and therefore the presented annealing curves are isocronical.

Diagram 1 is a plot of the Yield Strength (YS) values versus the annealing temperature and **Diagram 2** is a plot of Ultimate Tensile Strength (UTS) versus lab annealing temperature.

The softening of the coils produced by process 2 was retarded producing higher strength values in

the recovery (linear slope area of the curve) and recrystallization regime (steep slope area of the curve) relative to the coils produced by process 1. The as-cold-rolled mechanical properties of the 5 coils are similar and therefore the 5 coils have almost the same amount of cold rolling deformation prior laboratory annealing tests.





3. Electrical Conductivity of Cold Rolled & Laboratory Annealed Samples

The Mn in SS was calculated based on the measured conductivity values by using the following equations:

Resistivity =100/conductivity = 2.65 + Mn in SS x 2.94 + Mn out of SS x 0.34 (1)

$$Total Mn = Mn in SS + out of SS$$
(2)





Diagram 3 illustrates the Mn in solute solution prior to the laboratory annealing tests (-H19 temper).

The amount of Mn in SS in the material of process 1 was about $\sim 45\%$ less compared with the material of process 2.

The large supersaturation of Mn found in the cold rolled coils of process 2 promotes significantly more precipitation of submicroscopic intermetallic particles during the heat-up of the laboratory anneal. This submicrostructure apparently retards the recrystallization process.



Diagram 4 illustrates the drop of Mn in solid solution after lab annealing versus the laboratory

annealing temperature. Coils produced by process 2 exhibits a larger drop of Mn in SS compared to the coils of process 1. This strongly suggests a higher amount of Mn-containing disperoids precipitated out of solid solution during annealing of coils produced by process 2. However, the Diagram 4 data show that coils produced by process 2 still exhibit more retained Mn in the SS after laboratory annealing relative to coils produced by process 1 after all annealing temperatures up through 330 °C.

4. Optical Microscope Examination Results of Lab Annealed Samples

Laboratory annealed samples were cross sectioned parallel to the rolling direction and examined under the optical microscope to the start and end of determine the recrystallization process. The recrystallized grains fraction for each annealing temperature was estimated. For sheet produced by process 1, the start of recrystallization process (around 10 % volume fraction of recrystallized grains) is observed at the metal temperature of 290 °C and recrystallization appeared complete at 310 °C (Fig 1-Fig 4).

For material produced by process 2, the start of recrystallization process is observed at the annealing temperature of 310 °C and the recrystallization appeared complete at metal temperature of 340 °C (Fig 5-Fig 9).

The above metallographic findings showed a delay of recrystallization in the coils produced by process 2 requiring the higher annealing temperature for the start and end of recrystallization.



Figure 1 recovered grain structure. Lab annealed at 280 °C. process 1



Figure 2 Partially recrystallized grain structure. Lab annealed at 290 °C. process 1



Figure 3 Partially recrystallized grain structure. Lab annealed at 300 °C. process 1



Figure 4 Fully recrystallized grain structure. Lab annealed at 310 °C. process 1



Figure 7 Partially recrystallized grain structure. Lab annealed at 320 °C. process 2



Figure 5 : recovered grain structure. Lab annealed at 300 °C. process 2



Figure 8: Partially recrystallized grain structure. Lab annealed at 330 °C. process 2



Figure 6 Partially recrystallized grain structure. Lab annealed at 310 °C. process 2



Figure 9 Fully recrystallized grain structure. Lab annealed at 340 °C. process 2

5. Summary and Conclusions

A delay in the recovery and recrystallization process occurred in the coils of process 2 compared to process 1 probably due to more precipitation of submicroscopic Mn-containing dispersoids and more retained Mn in SS during the annealing tests.

This delay of the recovery and recrystallization softening mechanisms observed in the final gauge material of coils produced by process 2 caused a shift of the softening curve to higher strengths relative to the softening curve of the coils of process 1.

The higher Mn in solid solution of process 2 coils, due to a different preheating/hot rolling practice compared with process 1, likely promoted more Mn-containing sub-microscopic intermetallic dispersiods to precipitate during the anneal

The annealed samples from coils of process 2 showed more Mn came out of the solid solution during recrystallization but still retained somewhat more in solution after similar annealing processes compared with the coils of process 1.

Fine dispersoid precipitation during annealing, along with higher Mn in solid solution, retards both nucleation and growth of recrystallized grains. These most likely account for why the recrystallization process started and completed at higher annealing temperatures for the coils produced by process 2.

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