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Effect of Heating Rate on Recrystallization of Twin Roll Cast Aluminum

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The effect of heating rate on precipitation and recrystallization behavior in twin roll cast (TRC) AA3105 has been investigated by three different means: conventional air furnace, controlled infrared, and lead bath heating. Experimental results showed that as-recrystallized grain size decreased and became more equiaxed as the annealing heating rate increased. These results were explained *via* time-temperature-transformation (TTT) curves for both dispersoid precipitation and recrystallization. With the faster heating rate, recrystallization could occur before precipitation of Mn present in the unhomogenized TRC samples. At a heating rate of 50 °C/s, the material underwent grain growth after recrystallization at 500 °C. No sign of grain growth was observed in materials annealed with lower heating rates, 3 °C/s, 0.5 °C/s, and 0.01 °C/s, due to greater dispersoid precipitation.

I. INTRODUCTION

TWIN roll casting (TRC) is an efficient and economical process for producing aluminum sheets directly from molten metal.[1–3] The process involves feeding the melt directly between water-cooled rolls where solidification occurs, producing 4- to 10-mm-thick sheets that can be coiled and later cold rolled to finished thickness. Advantages of TRC over conventional direct chill (DC) casting include reduced scale of operation, avoidance of ingot scalping, homogenization, and hot rolling, with their additional costs. Although the process is predominantly used for low alloys, it can also be used for higher alloys, but with some problems. In AA3105, the rapid solidification and subsequent cooling with no homogenization retain considerable manganese supersaturation. The supersaturated manganese can form fine α-Al(Mn,Fe)Si dispersoids during the imposed annealing after cold rolling to final thickness. These particles inhibit recrystallization by Zener pinning of subgrain boundaries, resulting in a coarse, elongated grain structure[4,5] giving material with poor formability.[6–8] The object of the study was to find a way to overcome the problem of the large grain size produced by the new process of twin roll casting with its inherent Mn supersaturation.

In DC castings, the problems of heavy dispersoid precipitation on recrystallization annealing are avoided by always applying an expensive predeformation homogenization anneal lasting many hours at elevated temperatures. The TRC can potentially avoid this expensive processing step if the major problem of inhibited nucleation and, thus, a large resulting grain size on annealing cold rolled nonhomogenized metal can be avoided. The present study was designed to examine as quantitatively as possible the conditions needed to allow recrystallization to a fine grain size in such nonhomogenized material.
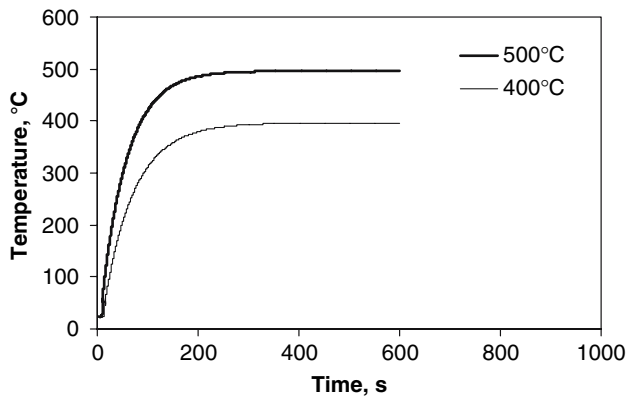
Prior studies have shown that a higher annealing temperature produces a finer, more desirable recrystallized grain structure.[9] However, the heating rate of material placed in a conventional air furnace at temperature varies with both furnace temperature and time, as shown in Figure 1(a). As a result, in a conventional air furnace, the effect of annealing temperature on recrystallization is combined with the effect of heating rate; *i.e.*, higher furnace temperatures produce higher workpiece heating rates and the two effects on recrystallization are thus difficult to separate.

Infrared (IR) heating provides an excellent means for investigating the effect of heating rate on precipitation and recrystallization in a material with supersaturated matrix.[10] The IR furnace employs a high intensity tungsten filament to transfer radiant energy to the part. The extremely high source temperature of the tungsten filament leads to high thermal transfer and extremely fast heating.[11] The low thermal mass of the tungsten filament gives good control of the heat output and process temperature. Full output can be obtained within seconds of applying power. Also, power can be turned
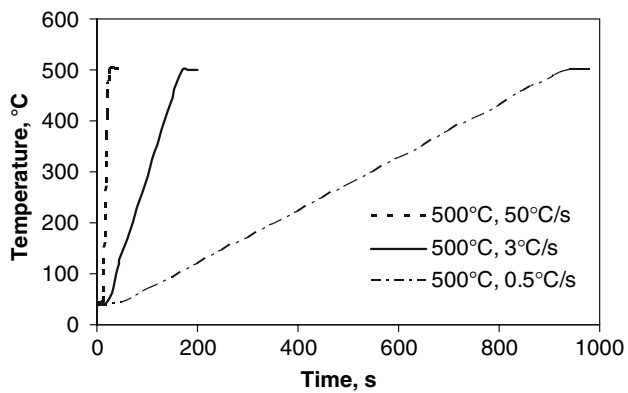
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Fig. 1—Heating curves for TRC AA3105 after 90 pct cold rolling: (a) conventional air furnace and (b) infrared furnace.

Element	Mn	Mg	Fe	Si	Cu
Wt pct	0.59	0.44	0.55	0.28	0.04

precipitation, respectively. Average grain size and grain aspect ratio were measured *via* the mean linear intercept length in both the longitudinal and transverse directions. All optical micrographs were taken on the longitudinal cross section, parallel to the normal direction (ND) and rolling direction (RD). Electrical conductivity (EC) was used to calculate the change of manganese in solid solution, *i.e.*, the evolution of dispersoid precipitation. The calculation procedure has been described elsewhere.[12]

III. RESULTS AND DISCUSSION

Figure 2 shows the grain structure for TRC AA3105 annealed to full recrystallization in an air furnace at 500 °C and 400 °C, respectively. At 500 °C, the material exhibits a finer recrystallized grain structure than at 400 °C, as shown in Figures 2(a) and (b). This apparent effect of recrystallization temperature on grain size is, our results suggest, mainly the effect of anneal heating rate. Figure 3 shows the as-recrystallized grain structure for material annealed in a lead bath at 500 °C for 1 second after achieving the temperature. The estimated heating rate for the lead bath anneal is approximately 100 °C/s, resulting in a much finer and more equiaxed grain structure than with the air furnace anneal, as shown in Figure 2(a).

Dispersoid particles, either pre-existing at a spacing of the subgrains or precipitating during heating or forming at the annealing temperature, are known to interact with coarsening or coalescence of subgrains that form the nuclei for recrystallization.[5,13–16] Slower heating should allow more dispersoid precipitation on subgrain boundaries, preventing nucleation of recrystallization and resulting in a coarser recrystallized grain size. Typical scanning transmission electron microscopy (STEM) (Figure 4) shows the interaction between precipitates and subgrain boundaries at the early stage of recrystallization in air-furnace-annealed TRC AA3105. It is clear that dispersoids preferentially formed along dislocations and subgrain boundaries, whose subsequent motion will be inhibited by these fine particles.

Figure 5 shows the time-temperature-transformation (TTT) diagram determined for 90 pct cold-rolled TRC AA3105, overlaying the start and finish of recrystallization with the times for different amounts of dispersoid precipitation at different temperatures, the latter determined from EC measurements. The early stage of precipitation, arbitrarily chosen as 10 pct removal of total manganese supersaturation, was obtained *via* infrared annealing with the fastest heating rate of 50 °C/s to minimize the precipitation during heating. The curve for nearly complete precipitation, *i.e.*, removal of 80 pct of manganese supersaturation, was

off almost immediately at the end of the process. Thus, the heating rate of the studied material with a thickness of approximately 500 μm can be well controlled and is independent of temperature, as shown in Figure 1(b). In the present study, the microstructural change on annealing cold-rolled TRC AA3105 is investigated at different controlled heating rates to different annealing temperatures to determine the relative kinetics of Mn precipitation and recrystallization.

II. EXPERIMENTAL

The TRC AA3105 was produced by Alcoa (Pittsburgh, PA), with the chemical composition given in Table I. The material, in the as-TRC state of 5-mm thickness, was cold rolled 90 pct in thickness prior to annealing. Three of the highest constant heating rates, 50 °C/s, 3 °C/s, and 0.5 °C/s, were achieved in an electric IR furnace at the Materials Processing Laboratory, Oak Ridge National Laboratory. The slowest constant heating rate, 0.01 °C/s, was obtained with a conventional programmable air furnace. For comparison, materials were also annealed at different temperatures in a conventional air furnace or in a lead bath. The latter gave the highest heating rate investigated; however, the rate is not linear nor readily controlled. Optical and electron microscopy were used to examine the recrystallized grain structure and dispersoid
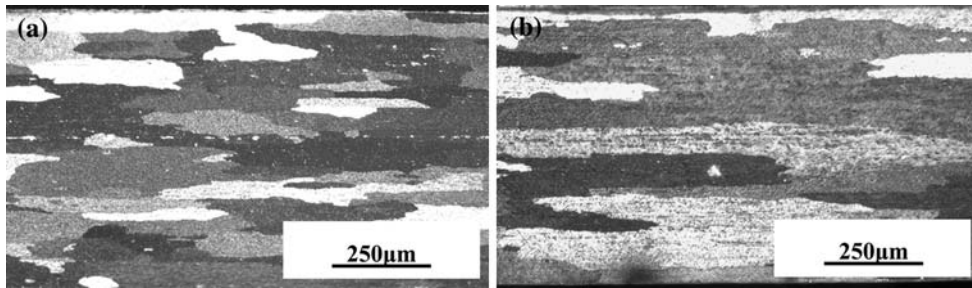
Fig. 2—As-recrystallized grain structure for material air furnace annealed at different temperatures: (a) 500 °C for 5 min and (b) 400 °C for 8 h.
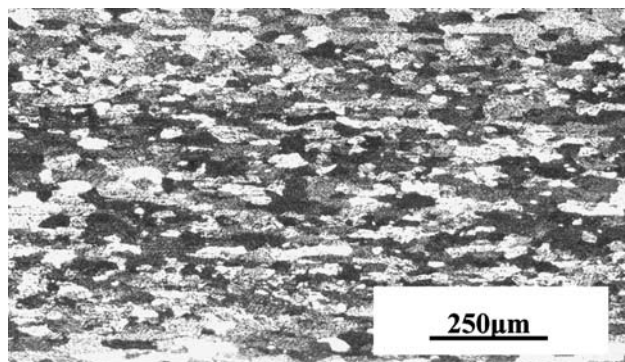

Fig. 3—As-recrystallized grain structure for material lead bath annealed at 500 °C for 1 s.
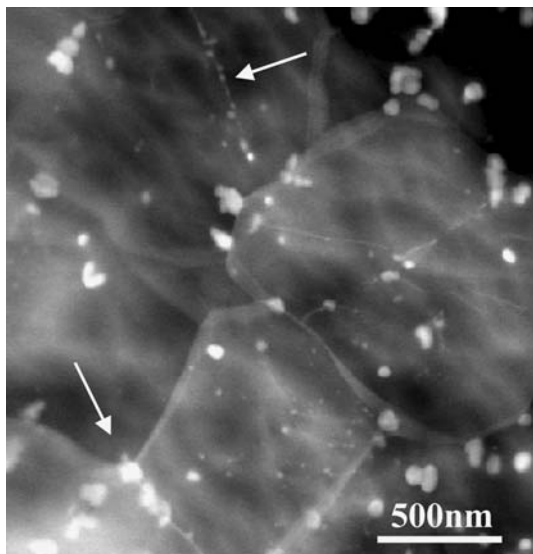

Fig. 4—STEM image of early recrystallization in TRC AA3105 air furnace annealed at 375 °C for 30 min. Note that dispersoids tend to form along subgrain boundaries and dislocations.
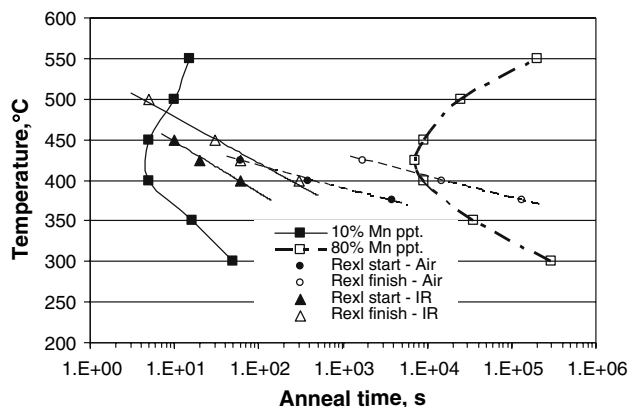

Fig. 5—Recrystallization and precipitation TTT curves for TRC AA3105 after 90 pct cold rolling. The 10 pct manganese precipitation curve was obtained *via* infrared heating of 50 °C/s. The 80 pct precipitation curve was obtained using conventional air furnace heating, because the heating time was negligible compared to the isothermal anneal time.

obtained for materials annealed in a conventional air furnace. For these air-furnace-annealed specimens, the effect of heating on the amount of precipitation is negligible, because the time spent during heating was less than 5 pct of the isothermal holding time.

The start and finish of recrystallization were followed *via* optical microscopy for both air furnace and the

50 °C/s infrared anneal. A faster heating rate to temperatures allowed rapid recrystallization prior to formation of many inhibiting dispersoids and resulted in increased recrystallization kinetics and a finer recrystallized grain size. Conversely, a slower heating rate allowed significant dispersoid precipitation prior to the start of recrystallization, greatly hindering nucleation of recrystallization and resulting in slower kinetics (Figure 5) and a much coarser grain size (Figure 6). This result is most clearly seen for the samples recrystallized at 400 °C in the air furnace. These samples took over 200 seconds to reach temperature (Figure 1(a)), while the sample in the IR furnace took only 8 seconds to reach 400 °C on heating at 50 °C/s (Figure 1(b)). The IR sample started recrystallization after about 70 seconds and recrystallization was complete after about 300 seconds, at which time recrystallization in the air furnace sample, also at 400 °C, had not yet started (Figure 5). Even in the IR furnace, at 50 °C/s, some significant Mn precipitation, about 10 pct, had occurred at 400 °C after about 5 seconds. In the samples annealed at 400 °C, 80 pct Mn precipitation was achieved after about 10,000 seconds. This was about the same time as the *completion of recrystallization* in the sample annealed in the air furnace. On heating to 500 °C at 50 °C/s, recrystallization was completed in 5 seconds in the IR furnace, before even 10 pct Mn precipitation was seen.
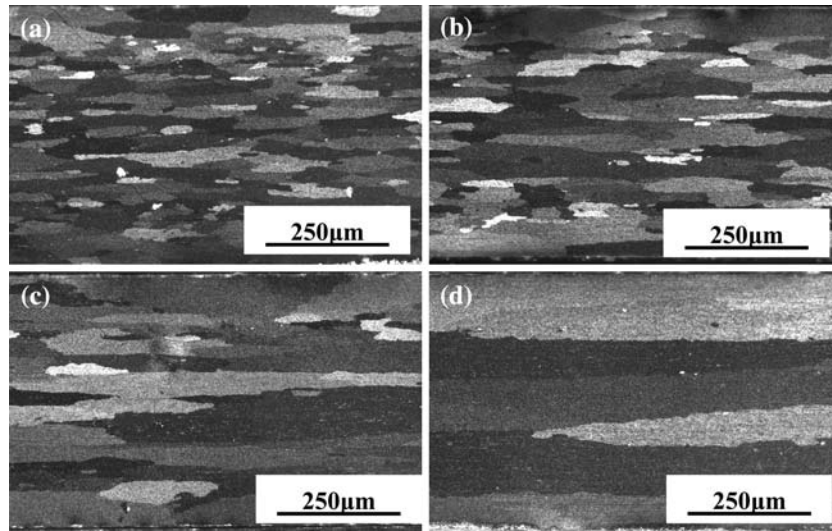
Fig. 6—As-recrystallized grain structure for material IR annealed at 500 °C with different heating rates: (a) 50 °C/s, (b) 3 °C/s, (c) 0.5 °C/s, and (d) 0.01 °C/s. Note that the grain size increases as the anneal heating rate decreases.

The displacement to the longer times for conventional heating is thus clearly due to the greater dispersoid precipitation during slow heating to temperature. The lower slope with slow heating is, as reported by Koster,[17] due to the progressively greater amount and finer dispersion of precipitates forming at lower temperatures with decreased heating rate. These finer precipitates have been shown to lead to slower recrystallization, mainly by inhibition of the nucleation of the new grains, leading to the larger recrystallized grain size.[13] Recrystallization is made even slower by the greater recovery allowed by longer times before recrystallization starts.

The rapid, controlled heating rate of IR annealing enabled separation of the effects of heating rate and annealing temperature on recrystallization. Figure 6 shows the as-recrystallized grain structure, i.e., prior to any grain growth, for material IR annealed with different controlled heating rates. In these micrographs, together with Figures 2(a) and 3, the increase in grain size with decreased heating rate, even at the same annealing hold temperature, is visually apparent. Figure 7, including both the lead bath anneal and the IR treatments with different hold temperatures, illustrates these effects more clearly. It is apparent that the average recrystallized grain size markedly decreased as the heating rate increased. For the fastest heating rate, approximately 100 °C/s in the lead bath, the material was heated to target temperature within 5 seconds. As a result, very little manganese precipitation occurred, less than 0.05 wt pct Mn, i.e., 10 pct removal of the total supersaturation of approximately 0.5 wt pct, as shown in Figure 5. As the heating rate decreased, the material spent a longer time heating through the lower temperatures, resulting in greater precipitation. These concurrently forming precipitates tended to pin subgrain boundaries and inhibit the nucleation process during recrystallization,[13] as shown in Figure 4.
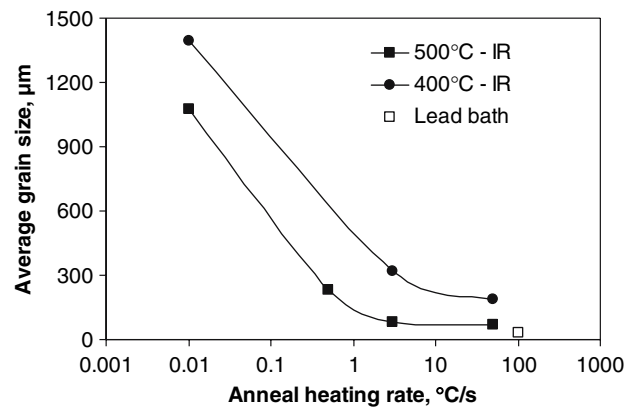

Fig. 7—Effect of anneal heating rate and temperature on recrystallized grain size. The grain size was estimated by averaging the mean linear intercept length in both the longitudinal and transverse directions of the strip shown in Figs. 3 and 6.

Figure 7 also shows that for a constant IR heating rate, a lower annealing hold temperature also resulted in coarser grain size. This effect was more predominant at the higher heating rates and relatively smaller at the lowest rates. At the highest rate of 50 °C/s, the grain size at 400 °C was a factor of 1.7 larger than that at 500 °C, while for 0.01 °C/s, the difference was only a factor of 0.3. Again, the cause of this grain size increase with decreased hold temperature, even at the same heating rate, is apparently due to the greater precipitation at the lower hold temperatures, near the nose of the precipitation transformation curve. For practical purposes, however, this effect of temperature on recrystallized grain size is secondary to that of the anneal heating rate.

Figure 8 shows the effect of the heating rate and hold temperature on the grain aspect ratio in the IR and lead-bath-heated materials. As with grain size, the aspect ratio decreases markedly with increased heating rate and
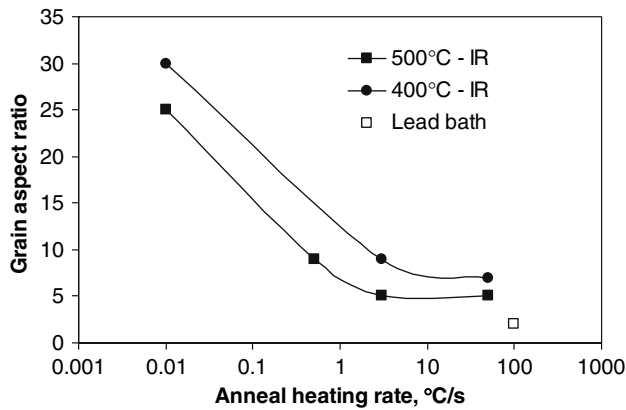
Fig. 8—Effect of anneal heating rate and temperature on grain aspect ratio, computed as the ratio of the mean linear intercept length in the longitudinal and transverse directions of the strip.

higher hold temperature. The increased grain aspect ratio with decreased heating rate is most likely due to the concurrent precipitates preferentially formed at higher angle grain boundaries, which tend to orient themselves parallel to the rolling plane. Doherty[18,19] has previously analyzed this phenomenon. After heavy rolling, materials exhibit pancaked microstructures with high-angle boundaries, for instance, the prior grain boundaries and transition bands, drawn out in the RD and lower angle subgrain boundaries in the ND. Based on both tension and energy models, Doherty analytically showed that the anisotropically deformed microstructure results in an elongated recrystallized grain structure even though the stored energy, the driving force for recrystallization, is isotropic. His idea was that a recrystallization boundary moving in the normal direction would spend only a very short time experiencing the high driving forces of the high-angle boundaries and most of the time experiencing only the low driving force of the low-angle boundaries. Boundaries migrating in the rolling direction will, however, experience a constant medium driving force. In addition, in samples undergoing precipitation at the slower heating rates, the higher angle boundaries in the deformed state are likely to be more potent nucleation sites for dispersoid precipitation, tending to give planar sheets of precipitates on the high-energy boundaries parallel to the rolling plane.[20] Such sheets of dispersoids will accentuate the already anisotropic recrystallization in the studied material. At the very highest heating rate, in molten lead, which took place with negligible precipitation of Mn, not only was the grain size finer still but the grain shape was almost equiaxed (Figures 3, 7, and 8).

Although rapid heating rates are very desirable for reducing recrystallized grain size, it was observed that the avoidance of dispersoid precipitation, beneficial to recrystallization, also allows rapid grain growth after annealing. Figure 9 shows the material annealed at 500 °C with the 50 °C/s heating. It is apparent that grain growth has occurred while holding at temperature for a short period of time, from 5 seconds to 5 minutes. However, grain growth was not observed in material annealed at the lower heating rates, 3 °C/s, 0.5 °C/s, and 0.01 °C/s, even at extended times. These observations suggest that the grain growth at 500 °C is likely to be due to the absence of precipitates during recrystallization in the most rapidly heated material. At lower heating rates, the material had more time to precipitate enough dispersoids to pin the grain boundaries, on slow heating through the temperature range with the fastest precipitation rates, 400 °C to 450 °C (Figure 5). As a result, the as-recrystallized grain structure was stable in material annealed with the slower heating rates, as shown in Figure 10.

The observed fine recrystallized grain size followed by grain growth in material annealed at the highest heating rate indicates optimum industrial production routes to achieve fine-grained TRC 3105 sheet. For instance, heavy cold rolling and rapid heating to a high temperature in a continuous annealing line followed by rapid cooling to room temperature would produce a fine-grained supersaturated alloy, desirable for H temper material. Following the rapid heating with partial cooling and coiling at a temperature at the nose of the TTT curve for precipitation would produce a fine-grained nonsupersaturated alloy, if preferred.

With the insight gained from this study, it appears that the problem of recrystallization to a large grain size with the TRC alloy can be avoided by controlling the relative kinetics of two processes: (1) recrystallization and (2) precipitation of the Mn dispersoids. The TRC avoids the expensive process of lengthy homogenization to precipitate the Mn dispersoids before hot rolling to
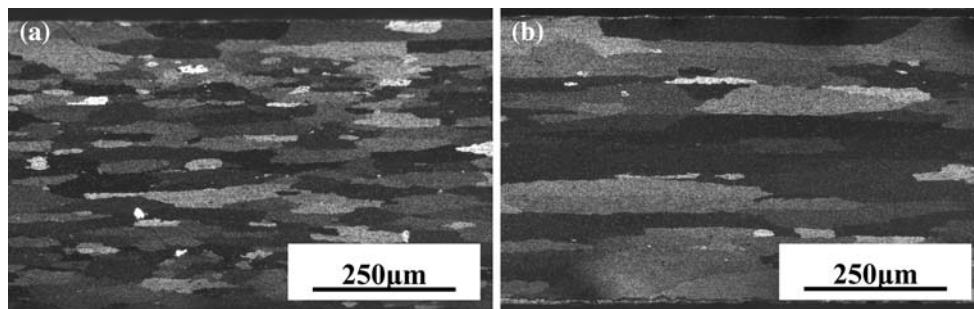

Fig. 9—Grain structure in TRC AA3105 annealed at 500 °C with 50 °C/s heating rate: (a) 5 s and (b) 5 min hold at temperature. Grain growth occurred during isothermal dwell at temperature.
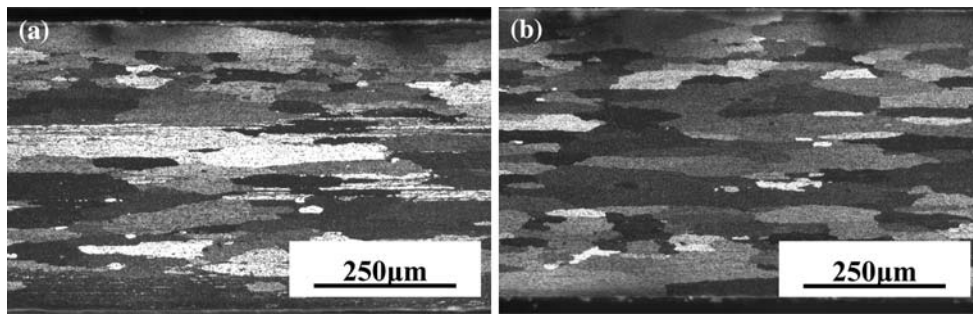
Fig. 10—Grain structure in TRC AA3105 annealed at 500 °C with 3 °C/s heating rate: (*a*) 5 s and (*b*) 5 min hold at temperature. Note that the material has not fully recrystallized yet after 5 s. No apparent grain growth during dwell at temperature.

the TRC thickness. Pre-precipitation of Mn in DC material, giving coarser dispersoids than seen in the TRC samples (Figure 4), allows recrystallization to be less inhibited than with the slowly heated TRC samples.[13,17] However, with the procedure suggested here, rapid annealing of the cold-rolled strip to 500 °C followed by rapid cooling appears to allow the possibility of very fine and nearly equiaxed grain structures that are expected to show the highest formability. In preparing this article for publication, the authors were surprised to find that there appears to be little published information on the empirically well-established need for a fine grain size to achieve good formability in aluminum sheet products. Therefore, quantitative investigations of this relationship would appear to be necessary. The understanding gained from the present study should allow the use of the TRC process for this and other high alloyed aluminum alloys that would also form fine precipitates from the rapidly cooled supersaturated TRC material.

IV. CONCLUSIONS

The following conclusions can be drawn from this study on TRC AA3105.

1. For conventional air furnace heating, annealing at a higher temperature results in a finer recrystallized grain structure. This observed effect is mainly, but not exclusively, due to the effect of a faster heating rate as a result of higher set point temperature than the effect of the anneal temperature itself.
2. An increased anneal heating rate results in a finer recrystallized grain structure and decreased grain aspect ratio, due to reduction of dispersoid precipitation.
3. The recrystallized grain size increases somewhat with decreased annealing hold temperature, even for the same heating rate. The magnitude of this effect increases with increased heating rate and is secondary to the heating rate effect.
4. Grain growth is observed after recrystallization at 500 °C, with the fastest 50 °C/s heating, again due to avoidance of dispersoid precipitation. No grain growth occurred in material annealed at the slower heating rates, 3 °C/s, 0.5 °C/s, and 0.01 °C/s.

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