RECRYSTALLIZATION OF COMMERCIAL TWIN-ROLL CAST ALUMINUM 3003 ALLOY DEFORMED UNDER COLD-WORKING CONDITIONS.

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Abstract

The effect of the combined action of cold deformation prior to homogenization on the annealing response of a cold rolled commercial 3003 aluminum alloy produced by twin roll casting, has been investigated by means of optical microscopy, scanning and transmission electron microscopy and mechanical testing. It has been determined that the deformation-homogenization step applied to the material gives rise to significant morphological changes of large intermetallic particles and their distribution. The annealing response of the material at 673 K has been found to be significantly dependent of the strain applied to the alloy. Particularly, it has been observed that for cold reductions less than about 50%, a marked interaction between restoration (recovery and recrystallization) and precipitation processes takes place, possibly due to the formation of small dispersoids from remaining supersaturation in the deformed matrix.

Introduction

In recent years, the industrial practice related to the manufacture of thin products for domestic purposes, from twin-roll cast (TRC) commercial aluminum alloys, have explored the application of combined deformation and homogenization processes to improve the annealing response of such materials subsequent to cold-rolling. This, has been particularly true for those alloys that are well known to have anomalous annealing behavior due to the marked interaction that could arise between recrystallization, precipitation and segregation effects, depending upon the deformation and annealing conditions. 3003 aluminum alloy constitutes one of such materials, since the important content of alloying elements present such as Mn, Fe and Si promotes the occurrence of such phenomena. The combined action of deformation and homogenization processes on the reduction of the Mn supersaturation in 3004 aluminum alloy, has already been investigated by Merchant et al. (1). After homogenization at 813 K for 18 hours, these authors observed that the alloy supersaturation determined by electrical resistivity measurements, was significantly reduced for cold reductions greater than about 40%. The present investigation has been conducted in order to study the annealing response of a commercial 3003 aluminum alloy manufacture by a TRC process, deformed by 50% thickness reduction prior to homogenization at 863 K for 8 hours and subsequently cold rolled in different degrees before annealing isothermally at 673 K.

Experimental techniques

The present investigation has been conducted with samples of a commercial twin-roll cast (TRC) aluminum 3003 alloy produced as coil of 8 tons. weight and dimensions: 6 mm thickness and 1400 mm width. The material has been provided by C.V.G. ALUMINIO DEL CARONI S.A.- DIVISION GUACARA and its chemical analysis is presented in Table I.

Table I. Chemical analysis of the investigated alloy, wt. %.

Al	Mn	Fe	Si	Cu	Ti	Zn	Mg	Cr
Rem.	1.0	0.67	0.28	0.1	0.01	0.005	0.004	0.00 1

Cold rolling of small samples was carried out in an experimental 20 H.P. two-high reversible rolling mill employing rolls of 175 mm diameter and a peripheral speed of approximately 67 mms^{-1}. Initially, all the samples were cold-rolled to 50% thickness reduction, homogenized at 863 K for 8 hours and air cooled. Subsequently, the alloy was cold rolled in three different degrees: 25, 50 and 75% thickness reduction, from which tensile, hardness and metallographic samples were machined. Annealing of the deformed material was carried out in a salt bath furnace at a constant temperature of 673 K, for different time periods ranging between 10 and 180000 s approximately. The specimens were subsequently water quenched in order to retain the high temperature microstructure. All the metallographic analyses were performed on the plane defined by the short transverse direction and the rolling direction. Optical metallography was conducted under conditions of polarized light by means of electroetching the specimens in a solution of 52 ml HF (48%) and 973 ml distilled water, employing a stainless steel cathode and a potential of 22 volts. Scanning electron microscopy studies were conducted on a HITACHI S-2400 scanning microscope equipped with a KEVEX-IV X-ray energy dispersive spectroscope unit. Transmission electron microscopy (TEM) analyses were carried out on a 125 KV HITACHI H-500 microscope. Tensile tests were carried out on a 10 ton. universal testing equipment employing specimens machined according to the ASTM E-8 standard, at a constant cross-head speed of approximately 0.42 mms^{-1} in a range interval of 0-1000 Kg. All tensile strength (U.T.S.) values here referred to, correspond to a mean of at least three tests for every condition.

Experimental results and discussion

As-cast material

The as-cast alloy showed a typical metallographic microstructure composed of columnar grains, characteristic of TRC materials. It has been observed that at the center of the sample the grains present a higher aspect ratio than near the edges, where the grain structure shows a distinctive pattern making an angle of approximately 45° to the surfaces. The orientation of the grains is observed to change progressively from surface to center where the grain pattern is seen to align along the casting direction. The different features characteristic of TRC aluminum alloys have been discussed previously by Strid (2,3) who has pointed out that the resulting grain structure is associated to the dynamic transition from the solidification stage to the subsequent hot-working condition to which the material is subjected during fabrication. Thus, the more elongated grain structure observed towards the edges of the sample could be related somehow both to the higher solidification rates and the more severe deformation pattern induced in the material. The analysis carried out by SEM techniques revealed the presence of a large volume fraction of second-phase particles densely distributed in the matrix and also on the grain boundaries, as shown in Fig. 1a. Numerous colonies of dendrite-like morphology particles such as those depicted in Fig. 1b, were observed throughout the matrix, combined with smaller round and elongated particles.

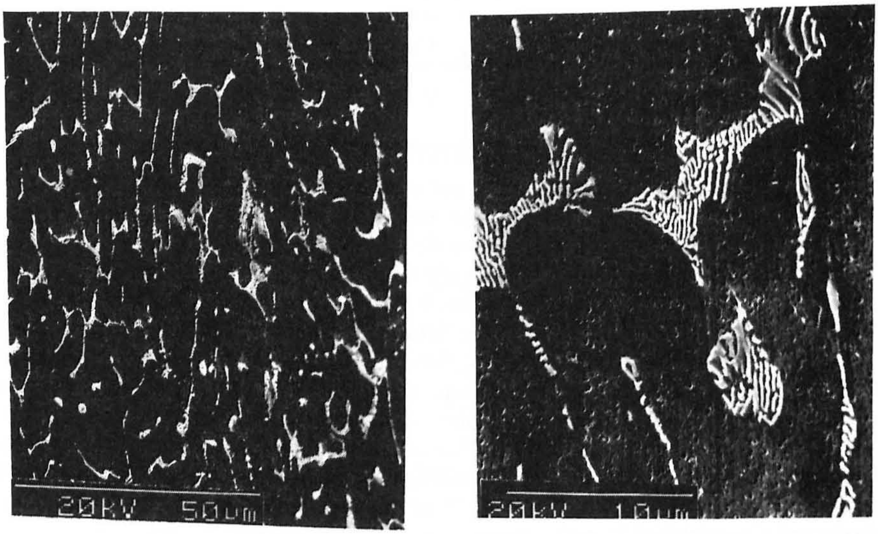

Figure 1. (a) SEM micrograph of the as-cast sample showing the presence of second-phase particles. (b) Details of several colonies of dendrite-like morphology particles combined with aligned stringers of smaller particles.

However, towards the edges of the samples no such colonies were observed but only small elongated particles aligned in the roll-casting direction, which could possible be associated to the difference in cooling rate that takes place throughout the thickness of the specimen. All the EDS analyses conducted on different particles allowed to determine the presence of Al, Fe, Si and Mn with Fe/Si and Fe/Mn ratios between 2-3 approximately. Figures 2a and b, on the other hand, illustrate different features of the microstructure of the as-cast material observed by means of TEM techniques. Small particles of different sizes ranging approximately between 0.4-1 μm, finely distributed throughout the as-cast matrix, were observed all over the sample. Some of them were also found to interact with grain boundaries. The as-cast material was subsequently cold-rolled to 50% thickness reduction before the homogenization treatment. As expected, the SEM observations revealed the fragmentation and partial redistribution of the second phase particles earlier referred to, as shown in Figs. 3a and b.

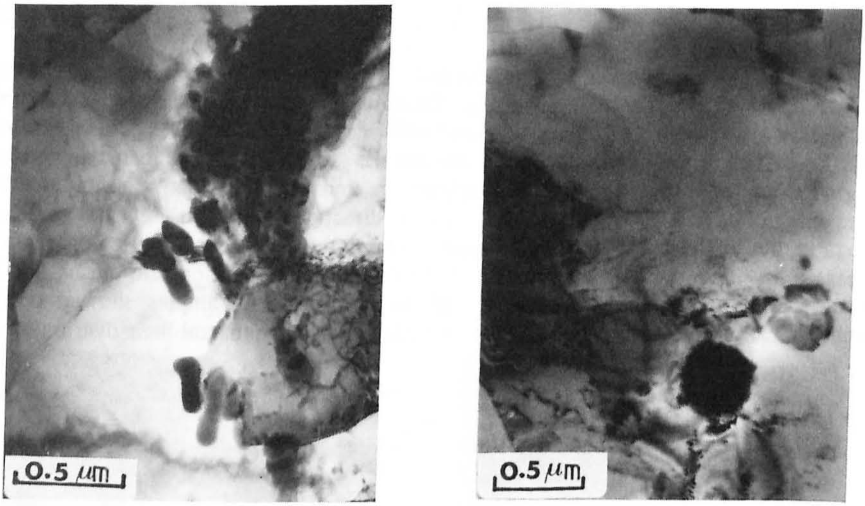

Figure 2. TEM micrographs of the as-cast sample showing the presence of dislocations arrangements and small particles.

Thus, it can be noticed the initial deformation process applied to the material gives rise to some morphological changes particularly in relation to the dendrite-morphology particles, although the fragmented phases are observed to remain close together rather than redistribute more homogeneously throughout the deformed matrix.

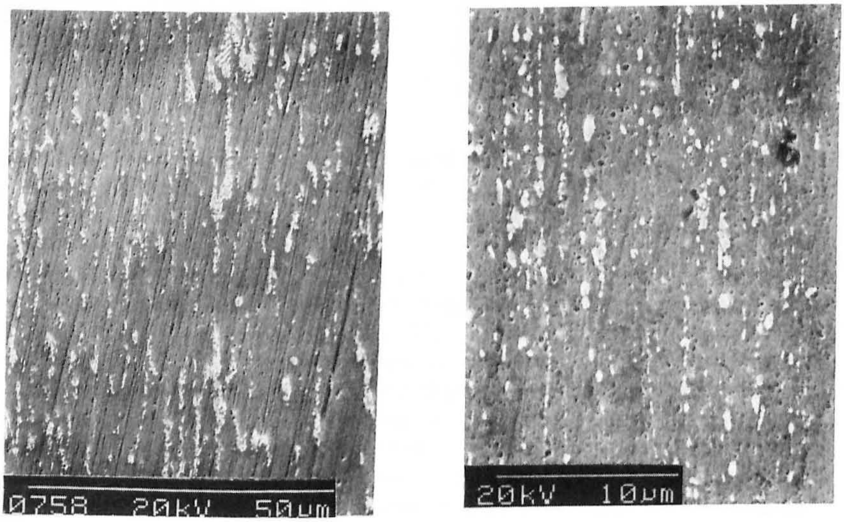

Figure 3. SEM micrograph corresponding to the as-cast and deformed material. (a) Center. (b) Edge of the sample.

Homogenized material.

After cold rolling, the material was homogenized at 863 K for 8 hours. The metallographic analysis of the homogenized samples revealed a fully recrystallized microstructure composed of elongated grains with a much higher aspect ratio near the edges of the specimen than in the center line. These grain morphology indicates that the growth rate of the recrystallized grains is higher along the rolling direction than in the short transverse direction, possibly due to the alignment of the particles along the rolling direction. Also, it reveals that solute supersaturation effects near the edges are considerably more pronounced in comparison with the center of the strip, as a consequence of the differences in cooling rate during TRC. As shown in Figure 4a, the SEM analyses of the homogenized samples showed profound morphological changes in relation to the intermetallic constituents and their distribution.

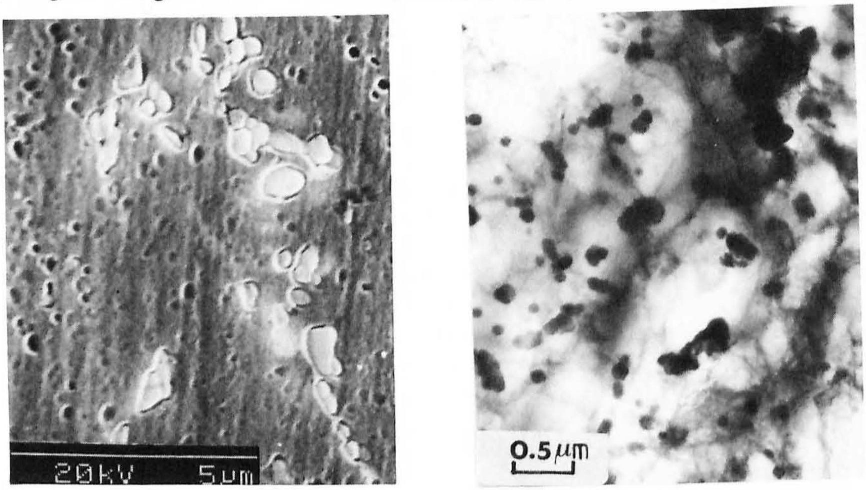

Fig. 4. (a) SEM micrograph corresponding to the deformed and homogenized sample showing the coarsening of second phase particles. (b) TEM of the same material illustrating the precipitation of small particles.

The colonies of dendrite-like morphology particles were completely eliminated and the previous particles distribution was noticed to be replaced for a more homogeneous one composed of larger globular phases. The EDS analyses carried out in a large number of particles after homogenization revealed a significant enrichment in Mn, Fe and Si, which reveals a depletion of the supersaturated as-cast matrix. As pointed out by different authors (4,5), the diffusion of these alloying elements to the large intermetallic particles is likely to give rise to the partial transformation the $Al_6(Fe,Mn)$ particles into $Al_{12}(Fe,Mn)_3Si$ and to a reduction of the content of transition elements within the matrix. The TEM micrographs of the homogenized material obviously showed a marked reduction of the dislocation density and the presence of small homogeneously distributed particles of sizes ranging between 0.1-0.3 μm approximately and different morphologies, as observed in figure 4b.

Restoration kinetics.

The annealing response of the homogenized material after cold rolling in different proportions was evaluated by means of a restoration index determined from the changes in mechanical properties with annealing time. Such an index has been defined assuming the validity of a linear law of mixtures based on the equal strain distribution model between the restored and unrestored fractions present during annealing after cold-rolling:

$$X_R = \frac{P_0 - P_i}{P_0 - P_f}$$

Therefore, the partially restored material is considered equivalent to a massive two-phase aggregate composed of a "soft" (restored) and a "hard" (unrestored) fractions. P_0 represents either mechanical property (VHN or UTS) measured in the as-deformed condition, P_i the value of the property at any intermediate annealing time and P_f the property after sufficiently long annealing times have taken place, such that no more changes are observed if annealing is continued. Figure 5 illustrates the changes of the fraction restored defined for tensile strength with annealing time when the material has been cold rolled by 25, 50 and 75% thickness reduction.

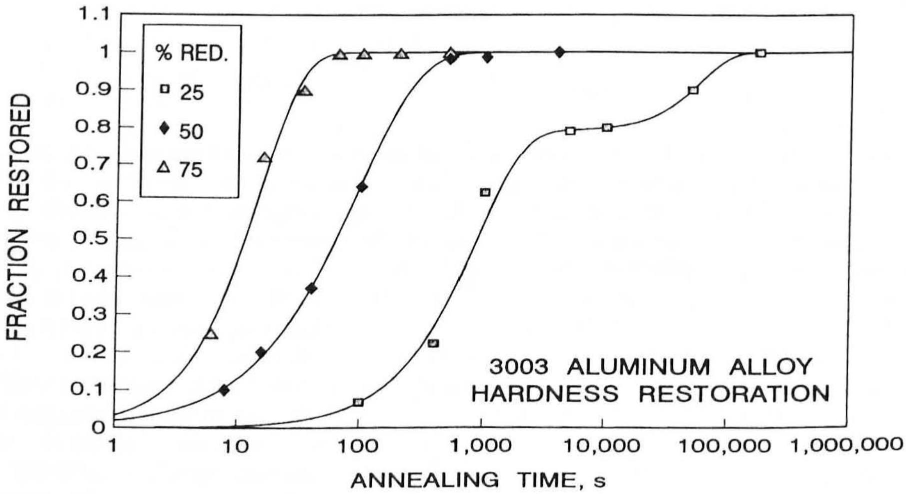

Fig.5. Fraction restored versus annealing time at 673 K for different initial deformations.

As it can be observed, for cold rolling reductions of 50 and 75% the restoration curves present typical sigmoidal shapes characteristic of nucleation and growth processes, although it is understood that the restoration curve involves phenomena both of recovery and recrystallization. Therefore, under these conditions a Johnson-Mehl-Avramy-Kolmogorov (JMAK) equation of the form:

$$X_R = 1 - \exp(-kt^n)$$

can be used to describe the change of X_R with t. The change in the JMAK exponents as the amount of cold work increases could be partially explained in terms of the operation of different nucleation sites as the strain increases. It is broadly accepted that for moderate deformations grain boundaries play a predominant role as nucleation sites for new recrystallized grains. However, as the applied strain increases second-phase particles become more important as nucleation agents for recrystallization with a consequent increase of the JMAK exponent. Also, recovery effects are likely to be somewhat diminished as the strain applied to the material increases. The other important aspect to be noticed in relation to this figure is that for 25% thickness reduction the restoration of the alloy follows an anomalous path where a well defined plateau is achieved after about 10000 s of annealing time. This phenomenon is quite likely to be associated with the interaction between restoration and precipitation processes and the formation of small dispersoids which hinder the nucleation of new recrystallized grains and their growth throughout the deformed matrix. Thus, it can be observed that under the present deformation and annealing conditions, the restoration processes can only continue to operate after about 10000 s of annealing time and full restoration can be achieved after approximately 100000 s. The description of the change in the restored fraction with annealing time can be accomplished by means of a convolution of two JMAK equation of the form:

$$X_R = f[1 - \exp(-k_1 t^{n_1})] + (1 - f)[1 - \exp(-k_2 t^{n_2})]$$

where f represents the fraction restored achieved in the first stage of the curve which in the present case is observed to be about 0.8, whereas k_1, n_1 and k_2, n_2 the constants of the JMAK equations for the description of the first and second restoration regimes respectively. All 5 constants that appear in the above equation can be determined by means of non-linear regression analysis applied to the experimental data. During the industrial processing of 3003 TRC aluminum alloys, the as cast coil is cold rolled to about 90% thickness reduction before annealing at approximately 673 K. Therefore, as far as deformation effects are concerned, in theory full restoration of the material should be achieved before the occurrence of any precipitation and a smooth restoration behavior with a high JMAK exponent should be expected. However, it must also be taken into account the fact that in the industrial practice, annealing after cold rolling takes place under non-isothermal conditions, since the annealing temperature is achieved in a period of 8-10 hours. Therefore, significant recovery and precipitation effects might become operative giving rise to important changes in the annealing response of the material.

Conclusions

The combined effect of deformation prior to homogenization produced significant changes in the morphology and distribution of the intermetallic particles initially present in the alloy. The colonies of dendrite-like morphology particles were observed to disappear after the homogenization stage, together with the coarsening and enrichment of the resulting particles, and a partially depletion of the as cast matrix in the main alloying elements. The annealing response of the material has been determined to depend upon the prior amount of cold work applied to the alloy. For thickness reductions less than about 50%, significant segregation and precipitation effects are expected to occur concurrently with the restoration mechanisms. For higher cold rolling reductions, a smooth restoration behavior has been observed indicating that full restoration can be achieved before the onset of precipitation of secondary particles from the remaining as cast matrix, and a single JMAK equation can be use to describe the restoration data.

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Acknowledgments

The present investigation has been carried out with the financial support of the Venezuelan National Council for Scientific and Technological Research (CONICIT) through the projects S1-2580 and RP-II-C-135, and the financial support of the Scientific and Humanistic Development Council of the Central University of Venezuela. The assistance of Mrs. Sonia Camero and Mrs. Joslinda Arraiz in the electron microscopy studies is gratefully acknowledged. M. Escorche and Y. Pérez also acknowledge the bursary given to them by C.V.G. ALUMINIO DEL CARONI S.A.-DIVISION GUACARA during the conduction of the present investigation.