RECRYSTALLIZATION OF COMMERCIAL TWIN-ROLL CAST Al-Fe-Si ALLOYS PREHEAT TREATED AT 653 K.

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Abstract

Recrystallization studies have been conducted on a commercial twin-roll cast Al-0.56 Fe-0.40 Si alloy, previously homogenized at 653 K for 5 hours, by means of restoration of its mechanical properties, optical microscopy and scanning and transmission electron microscopy techniques. It has been determined that the pre-heat treatment applied to the as-cast material gives rise to a significant acceleration of the restoration and recrystallization kinetics. Comparison of the restoration behavior of the homogenized material with that of the initial as-cast alloy indicates that although precipitation effects are still present, larger fractions restored can be achieved before any interaction between restoration and precipitation takes place. In spite of the fact that the homogenization treatment gives rise to a marked depletion of the as-cast matrix in Fe and Si, it has been found that the thermal cycle has not been enough to eliminate altogether both precipitation and segregation effects during annealing subsequent to cold rolling. From the viewpoint of modelling, it has been determined that the interaction between the restoration and precipitation processes precludes the use of the classical form of the Johnson-Mehl-Avramy-Kolmogorov (JMAK) relationship to describe the restoration behavior. However, an integration or convolution of two JMAK type equations can be satisfactorily used for this purpose.

Introduction

It is a well established fact that the homogenization treatment of commercial aluminum alloys and particularly the dilute alloys corresponding to the Al-Fe-Si, Al-Mg, Al-Mn and Al-Mg-Mn systems play an extremely important role in the annealing response of these materials. Therefore, in the past few years a number of research studies have been conducted in order to investigate the different mechanisms and complex phenomena involved in such a treatment and its effect on the recrystallization behavior of commercial aluminum alloys deformed both under cold and hot working conditions (1-3). Particularly in relation to this aspect, Merchant and Morris(1) have conducted a research study on the annealing response of 5005 and 3004 aluminum alloys by means of a comprehensive texture evolution analysis which included the investigation of textures before and after annealing, kinetics of the (200) cube component and 45 to 0/90° deg earing transition. This study allowed to examine the effect of supersaturation and precipitation on the annealing behavior. In relation to the effect of the homogenization treatment on the annealing response of the strip cast alloy investigated, it has been concluded that multistep homogenization results in a more effective depletion of the matrix concentration initially supersaturated with alloying elements such as Mn, Fe and Si which might interfere with the cube texture generation. Also, this work has pointed out the fact that unhomogenized alloys or insufficient matrix solute depletion due to incomplete homogenization could lead to the continuous precipitation of fine secondary particles during subsequent annealing and to the consequent interference between recrystallization and precipitation. Although it has been pointed out that usually soft aluminum alloys employed in the manufacture of household foil are not homogenized as part of its thermomechanical process (2), TRC alloys as well as strip cast alloys contain most of the alloying elements in solid solution. The present research work has been carried out in order to examine the recrystallization behavior of a commercial TRC Al-Fe-Si alloy which has been homogenized at a relatively low temperature (653 K) and to compare its annealing response with that of the material in the as-cast condition.

Experimental techniques

The present investigation has been conducted on samples of a commercial TRC Al-0.56% Fe-0.40% Si alloy whose chemical analysis is presented in Table I.

Table I. Chemical composition of the experimental alloy, wt %.

Fe	Si	Mn	Zn	Cr	Cu	Al
0.56	0.40	0.01	0.004	0.003	0.01	REM

The material has been manufactured by C.V.G. ALUMINIO DEL CARONI S.A.-DIVISION GUACARA and was provided in the as-cast condition in the form of strip of approximately 6 mm thickness and 1400 mm width. The material has been homogenized in an electric furnace at a temperature of 653 K for 5 hours. However, in order to simulate the actual industrial conditions, the homogenization temperature was achieved at a heating rate of about 1.09 $Kmin^{-1}$, in such a way that homogenization conditions were reached after 10 hours heating. The homogenized samples were subsequently cold-rolled in a fully instrumented experimental rolling mill to 50% thickness reduction at a peripheral roll speed of about 0.04 ms^{-1}, using a pair of rolls of 175 mm diameter approximately. Both the as-cast and cold-rolled material were thoroughly characterized by means of optical and electron microscopy techniques as well as tensile and hardness testing conducted on machined specimens. In order to follow the microstructural evolution of the material a number of samples for tensile and hardness tests as well as suitable metallographic specimens were also machined from rolled strips and subsequently annealed in a salt bath furnace for different time periods ranging between 10 and 50000 s at temperatures of 598 and 648 K. Metallographic samples corresponding to the plane defined by the short transverse and rolling directions were mounted and prepared following standard metallographic procedures before anodizing in a solution of 52 ml HF (48%) and 973 ml distilled water employing a stainless steel cathode, for optical observation under cross polarizers. The recrystallized grain size was reported as the geometric mean of the mean linear intercepts determined along the short transverse and rolling directions. Scanning electron microscopy analyses were conducted on a HITACHI S-2400 microscope equipped with a KEVEX IV EDX detector, using a constant potential of 20 KV. Transmission electron microscopy studies were carried out on a HITACHI HU-12 microscope using a constant potential of 125 KV. Tensile tests were conducted on a standard universal testing machine with specimens prepared according to the ASTM E8 standard. Testing was carried out at a constant crosshead speed of 0.167 mms^{-1} and a load range of 0-2000 Kg. Vickers hardness tests were performed on previously conditioned samples using a standard equipment with an applied load of 3 Kg. Tensile results represent a mean value of at least three tests for every condition, whereas hardness measurements correspond to the mean value of at least ten measurements.

Experimental results and discussion

As-cast material

The metallographic analysis of the as-cast samples revealed a microstructure composed of highly deformed grains and the existence of a strain gradient from the surface to the central line of the specimens. In this particular area the deformed grains present a lower aspect ratio and many deformation bands within some of the grains can also be observed. As it has been pointed out before, (4,5) the TRC process involves a dynamic transition from a solidification reaction to a subsequent hot rolling condition in the solid state, which possibly could account for the microstructural aspects described that indicate the existence of typical recovery features. The SEM observations conducted on these samples indicated the presence of large second-phase particles densely distributed throughout the matrix, as observed in Fig. 1a. It was particularly noticeable the existence of large dendrite-like morphology particles of irregular shapes and different sizes ranging between 10-20 μm. A large number of smaller isolated rounded and elongated particles of the order of 1-2 μm could also be observed. The EDX analyses conducted on such particles determined that they were mainly of the type AlFeSi, which could correspond to any of the different phases that present a similar morphology to the one above indicated, as has already been discussed by different authors (6). The TEM observations carried out conducted on foils of the as-cast material indicated the existence of dislocation networks and cell substructures with a relatively high dislocation density in the interior, Fig. 1b, and also many small second-phase particles and precipitates distributed within the matrix and in some cases associated with grain boundaries. Dendrite-like morphology particles were also observed associated to dislocations tangles.

Homogenized material

After the homogenization or pre-heat treatment at 653 K the microstructure of the samples showed some coarsening of the elongated grains in the ST direction, although the microstructural gradient already discussed for the as-cast samples was still present. Of course, as a consequence of the heat treatment it was observed a reduction of the mechanical properties of the material. The VHN showed a decrease from about 35.6 to 32.5 $Kgmm^{-2}$, whereas the UTS were observed to diminish from approximately 131 MPa to 118 MPa, which indicates that such a treatment gives rise to a moderate reduction of the dislocation density as well as a decrease of the supersaturation of Fe and Si through precipitation processes. Also, it was expected to observe changes in the morphology of second-phase particles present in the alloy. Particularly, as shown in Fig. 2a, the dendrite-like morphology particles were noticed to coarsen as a result of the heat treatment as well as the smaller particles. The TEM analyses of the samples showed a significant reduction of the dislocation density and the formation of a well defined substructure with a lower dislocation density in the interior of the subgrains, Fig. 2b. Also a large number of fine particles and dispersoids were observed some of which were associated with grain boundaries. Isolated large second-phase particles similar to those which are seen to compose the dendrite-like morphology particles were also noticed to be distributed throughout the matrix.

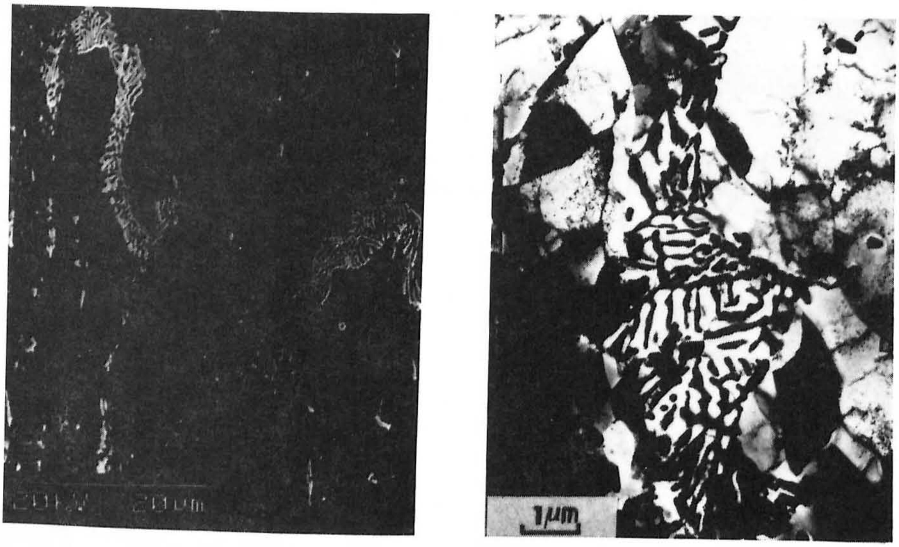

Fig. 1. (a) SEM micrograph corresponding to the microstructure of the as-cast alloy. (b) TEM observation of the as-cast material showing the presence of particles and dislocations arrangements.

Microstructural evolution during annealing

The microstructural analysis of the deformed and annealed samples revealed that at both annealing temperatures the initial deformed matrix is essentially replaced by the nucleation and growth of a new set of undeformed grains. However, at 598 K some deformed grains were still visible in different areas of the sample even after annealing for 10000 s. The microstructure that results after long annealing periods at this temperature is observed to be composed of coarse grains with an elevated aspect ratio, which indicates the slower growth rate in the ST direction in comparison with the rolling direction. At 648 K resulted a much finer microstructure and the deformed matrix was niticed to be totally consumed. In general, the recrystallized grain size was found to be more coarser near the edges of the specimens in comparison with the center. Therefore it is believed that towards the upper and lower surfaces of the strip the segregation and precipitation effects become much more pronounced which brings about a reduction of the recrystallization kinetics due to the interaction between the recrystallization fronts and precipitates. The net effect is the coarsening of the final recrystallized grain size near the edges of the sample. Of course, this phenomenon is accentuated at 598 K where the Zener drag is large and the recovery and precipitation effects are more marked. As expected, the mechanical properties of the material are quite sensitive to the microstructural changes and therefore they have been used to follow the microstructural evolution. From the curves that describe the change of both VHN and UTS with annealing time, a restoration index was defined based on the model of equal strain distribution in a massive two-phase aggregate according to which the fraction restored is related to the mechanical properties of the deformed and annealed material by means of a simple law of mixtures:

$$P_i = (1 - X_r)\,P_0 + X_r P_f \qquad \textbf{(1)}$$

In the above eqn. P_i, P_0 and P_f represent the values of the mechanical properties for the partially annealed, deformed and fully restored samples respectively, and X_r the fraction restored.

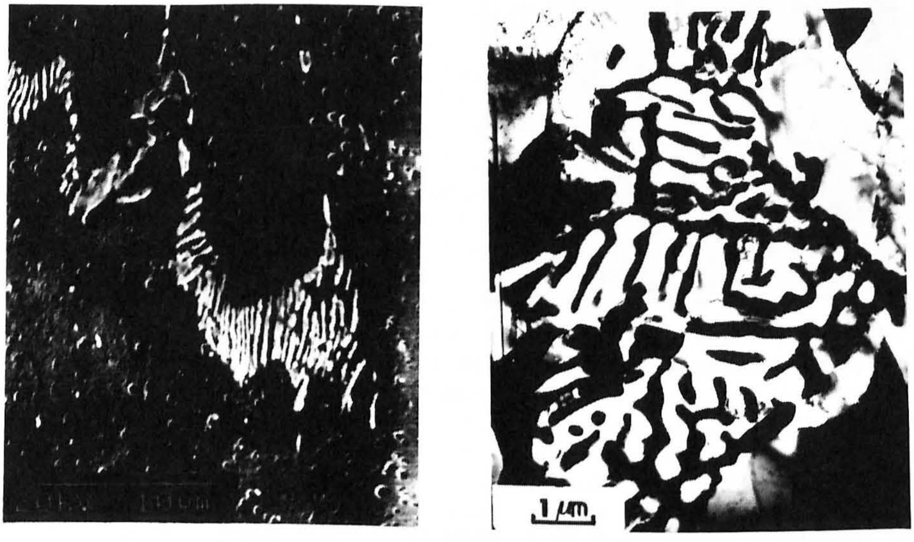

Fig. 2. (a) SEM micrograph showing second phase particles after homogenization. (b) TEM micrograph of the homogenized material.

In relation to the change of X_r defined from hardness measurements, with annealing time it has been noticed that at 598 K the curve presents a rather abnormal shape, as shown in Fig. 3. The smooth behavior that can be appreciated up to a fraction restored of about 0.9 is drastically altered showing a marked retardation of the restoration process. This anomalous behavior precludes the use of the classical form of the JMAK equation to describe the change of the fraction restored with annealing time. However, this behavior can be described satisfactorily by means of a convolution or integration of two JMAK equations of the form:

$$X_r = 1 - f_1 \exp(-K_1 t^{n_1}) - (1 - f_1) \exp(-K_2 t^{n_2}) \quad (2)$$

where f_1 represents the fraction restored associated to the change in the restoration behavior, and K_1, n_1, K_2 and n_2 different constants of the JMAK expressions corresponding to every "regime" of the curve. All these constants have been determined by means of a least-square non-linear regression model applied to the experimental data. It has been determined that in this particular case $f_1=0.91$, $K_1=9.68 \times 10^{-3}$, $n_1=1.11$, $K_2=1.41 \times 10^{-6}$ and $n_2=1.72$. It can be clearly appreciated that the behavior described is very similar to that reported before for the same alloy without any homogenization treatment, included in the figure for comparative purposes. However, two marked differences could be pointed out.

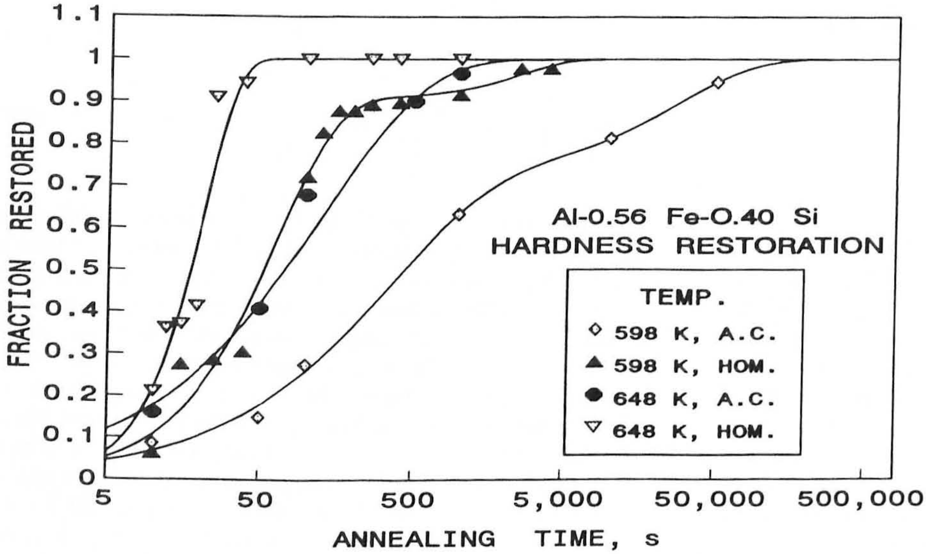

Fig. 3. Change of the fraction restored with annealing time at two different temperatures.

Firstly, the fact that the homogenization treatment gives rise to a pronounced acceleration of the restoration kinetics, promoting a clear shift of the curve towards the left. Secondly, possible precipitation effects for the homogenized alloy are observed to begin when a relatively higher restored fraction has been achieved in comparison with the unhomogenized material. Therefore, under the same conditions of cold deformation and annealing, the homogenized alloy restores faster than the unhomogenized one, although it is clearly observed that treatment applied has not been sufficient to deplete the as-cast matrix of Fe and Si to such an extend as to avoid completely precipitation effects at relatively low annealing temperatures. In relation to the restoration behavior at 648 K it can be appreciated that the curve corresponding to the homogenized alloy is also significantly displaced towards shorter annealing times in relation to the unhomogenized material and that a fully restored condition is achieved in a relatively short annealing period. Under these annealing conditions both restoration curves present a smooth behavior indicating that the restoration process and therefore the recrystallization of the materials occurs at a much earlier time than any precipitation reaction. In this particular case the change of the fraction restored with annealing time can be satisfactorily described by means of the JMAK equation and JMAK exponents of about 1.90 and 0.64 can de determined for the homogenized and unhomogenized alloys respectively. Although the experiments on the homogenized alloy have been conducted only at two different temperatures, the restoration curves determined from changes of VHN with annealing time can be employed to carry out a crude estimation of the activation energy of the process or processes involved, that could be compared with the values earlier reported for the unhomogenized alloy. The classical restoration studies conducted on deformed and annealed materials usually define a restoration time, typically the time required to achieved 50% restoration. This parameter is subsequently used in the determination of the constants involved in the simple parametric relationships that express the restoration time in terms of the different variables of interest , e.g. strain, strain rate, annealing temperature, etc. However, this limitation can be avoided by defining a normalized restoration time :

$$t' = \frac{t_{X_r}}{\{\ln[\frac{1}{(1 - X_r)}]\}^{(1/\bar{n})}} \tag{3}$$

In this equation t_{Xr} represents the time to achieve a particular fraction restored X_r and $\bar{n}$ the mean value of the JMAK exponent. The graphical representation of this normalized time in a classical Arrhenius plot allows the determination of the activation energy form the slope of the straight line obtained. Thus, for the hardness data it was determined a mean value of Q of about 105 $KJmol^{-1}$ for different fractions restored ranging between 0.1-0.9. In relation to these results several comments could be made. Firstly, it is important to mention that the Q value derived from the VHN data up to a restored fraction of 0.9 does not change markedly with fraction restored due to the fact that as it was pointed out earlier the precipitation effects are appreciated to be present for fractions greater that about this value. Therefore, if Q were determined for fractions between approximately 0.9-0.99 a larger value would be expected. Secondly, the Q value determined fairly agrees with those values earlier reported for the unhomogenized alloy although it is clear that in the present case it is only a crude approximation over a limited temperature interval.

Conclusions

The homogenization treatment conducted at 653 K has given rise to a marked increase of the restoration and recrystallization kinetics of the investigated alloy, in comparison with the behavior displayed for the as-cast material under similar deformation and annealing conditions. Although precipitation effects are still present during annealing at 598 K, larger restored fractions can be achieved before the interaction between these processes takes place. The homogenization treatment also brought about a depletion of the as-cast matrix in Fe and Si and an important reduction of the dislocation density of the initial material, which gave rise to a marked decrease of the properties related to its mechanical strength. The interaction between the restoration and precipitation processes during isothermal annealing, precludes the use of a classical JMAK type equation to describe the restoration behavior. However, the phenomenon can be satisfactorily modelled by means of a convolution or integration of two different JMAK equations taken into consideration the fractions restored associated to every "regime" of the restoration curve. Similarly to the behavior reported for other commercial aluminum alloys, the recrystallized grain size has been observed to decrease as the annealing temperature increases.

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