RECRYSTALLIZATION OF COMMERCIAL TWIN-ROLL CAST ALUMINUM-IRON-SILICON ALLOY HOMOGENIZED AT 853 K

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Abstract

The effect of the homogenization treatment conducted at 853 K on the recrystallization and restoration behavior of a commercial twin-roll cast aluminum-iron-silicon alloy deformed under cold-working conditions and subsequently annealed has been investigated. The as-cast and homogenized material has been cold rolled to approximately 50% thickness reduction and subsequently annealed at two different temperatures, which has allowed to follow the microstructural evolution and the changes in the mechanical properties during such a treatment as a function of annealing time. From the microstructural point of view it has been shown that the homogenization treatment gave rise to the total dissolution of the colonies of dendrite-like morphology particles of AlFeSi present in the alloy. It has also been determined that the restoration kinetics of this material remains nearly unchanged in relation to that of the as-cast material, possibly due to the reduction of the accumulated strain by the occurrence of recrystallization and the increase of the initial grain size during homogenization prior to cold rolling. It is believed that mayor differences in the restoration kinetics of both materials arise from precipitation effects in the as-cast alloy annealed at 598 K.

Introduction

One of the most distintive microstructural features of twin-roll cast (TRC) commercial aluminum alloys is the presence of colonies of large dendrite-like morphology particles with typical sizes ranging between 10-30 μm, which are formed during casting of the alloy. As it has been pointed out by before (1), Fe and Si have both partition coefficients less than unity and therefore segregate to the liquid between dendrite arms during solidification. Accordingly, primary particles of the type Al_3Fe, α-AlFeSi, β-AlFeSi and also metastable AlFeSi and AlFe particles may be formed during casting of an Al-rich commercial alloy, depending on the casting conditions (2,3). For example, in TRC processes solidification rates are much higher than in direct-chill casting (DC-casting) and could achieve values up to about 10^2-10^4 $^\circ Cs^{-1}$ (4,5). Therefore, marked microstructural differences are expected to arise between both commercial processes. The elevated cooling rates characteristic of TRC processes give rise to a significant supersaturation of alloying elements in solid solution. Subsequent decomposition of such solutions during annealing after cold deformation brings about the massive precipitation of small dispersoids that exert a marked influence on the recrystallization and annealing behavior of the material. In general, it is believed that homogenization treatments conducted in the alloys of the Al-Fe-Si systems below 673 K have no effect whatsoever on the distribution of Fe, although the precipitation of Si might be promoted. However, at temperatures about 873 K the Si level in the solid solution could increase near the value achieved in the as-cast condition whereas Fe might precipitate on grain boundaries. Therefore, it would be expected that homogenization treatments conducted under these conditions would give rise to the dissolution of the colonies of dendrite-like morphology particles present in these materials and to a more homogeneous distribution of the AlFeSi particles. Thus, a research study has been carried out in order to investigate the effect of a high temperature homogenization treatment on the recrystallization kinetics and on the recrystallized grain size of a commercial AlFeSi alloy deformed under cold-working conditions and subsequently annealed.

Experimental techniques

The present investigation has been conducted on samples of a commercial TRC Al-0.56% Fe-0.40% Si alloy whose detailed chemical analysis is reported 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 alloy has been provided by C.V.G. ALUMINIO DEL CARONI S.A. - DIVISION GUACARA in the as-cast condition in the form of strip of about 6 mm thickness and 1400 mm width. Smaller samples of about 300x80x6 mm were machined from the as-cast strip and subsequently homogenized at 853 K for 5 hours at a heating rate of about 0.024 Ks^{-1} until the homogenization temperature was achieved. After homogenization the samples were air-cooled to room temperature. The homogenized material was cold-rolled to 50% thickness reduction by means of an experimental two-high fully instrumented reversible rolling mill of 20 HP capacity, at a mean peripheral roll speed of 0.04 ms^{-1}, using a pair of rolls of about 175 mm diameter. Tensile and metallographic specimens were subsequently machined from the cold-rolled samples and annealed afterwards in a salt bath furnace at two different temparatures: 598 and 648 K, for different time intervals ranging between 10 and 50000 s. After annealing, the samples were water quenched in order to retain the microstructure developed at high temperatures. The material was thoroughly characterized from the microstructural and mechanical point of view in every step of the investigation. Metallographic samples were mounted, ground and mechanically polished according to the standard procedures. Etching was carried out electrolitically using a solution of 10 ml HF (48%) and 90 ml destilled water and a stainless steel cathod. The optical examination of the samples was conducted under conditions of polarized light, on the plane defined by the rolling direction and the short transverse direction. The recrystallized grain size was determined as the geometric mean of the values corresponding to the mean linear intercepts measured along the two directions above mentioned. Scanning electron microscopy (SEM) techniques were employed in the analysis of size, shape, distribution and chemical composition of second-phase particles present in the as-cast, homogenized and deformed and annealed samples. Such a study was carried out in a HITACHI S-2400 microscope equipped with a KEVEX IV X-Ray energy dispresive spectrometer (EDS), at a constant potential of 20 KV. The samples were previously etched in a solution of 98 ml HF (48%) and 2 ml destilled water. Transmission electron microscopy (TEM) analyses of the samples were conducted on a HITACHI HU-12 microscope at a constant potential of 125 KV. Final thinning of foils employed in the observations was achieved with a double jet technique employing a solution of 80% vol. methanol and 20% perchloric acid at 243 K. Tensile tests of cold-rolled and partially annealed samples were conducted on a standard universal testing machine of 10 ton. capacity, employing specimens machined according to the ASTM E8 standard, oriented along the rolling direction. The tests were carried out at a constant cross-head speed of 0.167 mms^{-1}. Ultimate tensile strength (UTS) values reported correspond to a mean of 3 tests. Vickers hardness tests were conducted on a standard equipment using a load of 5 Kg. Every Vickers hardness number (VHN) value reported corresponds to the mean of at least 10 measurements conducted on the same plane where microstructural observations were carried out.

Experimental results and discussion

As-cast material

Metallographic observations of the microstructure of the alloy in the as-cast condition revealed an elongated structure composed of columnar grains. In the middle plane of the sample such grains are observed to be oriented in the cast direction and to present a lower aspect ratio. Towards the edges of the sample the grains present an orientation near 45° with the surfaces of the specimen and a higher aspect ratio, which gives the appearance of a higher accumulated strain in these areas. The mean grain size determined was about 24 μm. The SEM observations conducted on these samples revealed the existence of a large volume fraction of second phase particles located on the grain boundaries and the matrix of the alloy, as shown in Fig. 1a. Large colonies of dendrite-like or eutectic-like particles of sizes ranging between 10-30 μm approximately were particularly noticeable, together with smaller globular and plate-like morphology particles. The EDS analyses revealed that the matrix was mainly composed of Al and Fe, whereas the particles were found to be composed of Al, Fe and Si in different proportions. Plate-like particles part of the colonies above referred to, were found to have a ratio of Fe/Si of approximately 1. Globular particles, on the other hand, were observed to have a higher ratio Fe/Si somewhat greater than about 2 and also a higher content of Fe and Si. TEM observations of the as-cast alloy revealed the presence of both groups and isolated second-phase particles of sizes ranging between 0.5-1 μm mostly of rounded shapes and also a well defined dislocation substructure and dislocation networks inside the cells, as shown in fig. 1b. As it has been pointed out before (4,5), during TRC processes a dynamic transition between a solidification reaction and a hot-working condition takes place. Therefore, well defined subgrains and dislocation cells are expected to form together with a recovery-like dislocation network in the subgrains interior.

Homogenized material

Homogenization of the as-cast alloy gave rise to a significant reduction of both hardness and tesile properties. It has been observed that the VHN decreased from about 34 to 22 $Kgmm^{-2}$, whereas the UTS values also decreased from 123 to 91 MPa.

Fig. 1. (a) SEM micrograph of the as-cast material showing dendrite-like morphology particles. (b) TEM of the same material showing both particles and dislocation arreangements.

It is a well known fact that among many other effects, high temperature homogenization treatments give rise to a significant depletion of the matrix supersaturated solid solution and also to a marked reduction of the dislocation density through recrystallization processes, both of which contribute to the reduction of the mechanical properties of the material. The metallographic examination of the homogenized alloy showed a completely different microstructure indicating that recrystallization and grain growth had occurred during the high temperature treatment. The grains were observed to remain elongated although the aspect ratio was found to be higher near the borders of the sample than in the middle plane. The mean grain size was determined to be about 63 μm. The elongated recrystallized grain grain morphology is associated with the distribution of second-phase particles which aling themslves mostly along the roll-casting (RC) direction. Therefore, the growth rate in the ST direction is somewhat slower than in the RC direction which gives rise to an aspect ratio greater than 1. However, the effect is much more marked near the edges of the sample than in the center. The homogenization treatment gave rise to a significant change in the distribution and morphology of the intermetallic particles and to the complete elimination of the colonies of eutectic-like morphology particles present in the as-cast condition, as it can be observed in fig. 2a. It is believed that it is due to the disolution and subsequent precipitation of Fe and Si, of pre-existing second-phase particles. The dominant morphology has been observed to change to globular and plate-like, and also the distribution throughout the matrix was noticed to be more uniform. The EDS analyses of the matrix showed again the presence of Al and Fe although a reduction in this element was observed in comparison with the as-cast matrix. Also,the elemental analyses of the particles revealed the presence of Al, Fe and Si although the proportion of these last two elements was found to be much higher than before, indicating a significant enrichment of the particles during homogenization. For example, the ratio of Fe/Si in plate-like particles was found to increase to about 2.8, whereas for round-like particles it was determined to be range between 2.2-3. Examination of the homogenized samples by means of TEM techniques revealed a signifinat change in the substructure, as observed in fig. 2b. In some areas, subgrains and cells were observed to almost disappear, remainig only some dislocation bundles associated with relatively large second-phase particles.

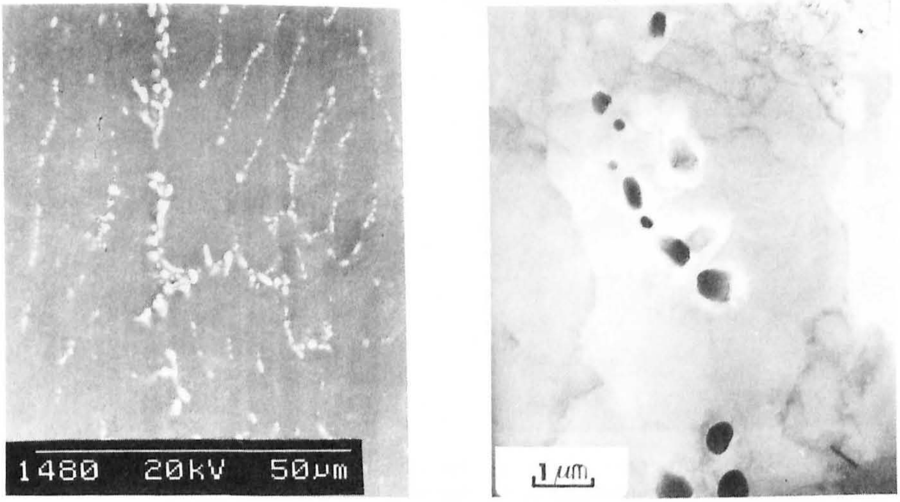

Fig. 2. (a) SEM micrograph of the homogenized alloy showing the resulting particle distribution. (b) TEM micrograph of the same alloy showing significant changes in the dislocation substructure.

In other areas subgrains were still observed but with a much lower dislocation density in their interior, indicating that a profound change in the dislocation structure had taken place possibly as a consequence of a continuous recrystallization reaction.

Cold-rolled and annealed material

The distribution of second-phase particles resulting from high temperature homogenization was also modified during cold-rolling. Such particles were observed to aling in the rolling direction whereas some of them also fractured during deformation. As expected, TEM analyses of deformed samples showed a significant increase in the dislocation density. Large dislocation tangles associated to groups of particles were observed together with a primitive cell structure in evolution. However, the dislocation distribution was also seen to be quite heterogeneous with the presence of areas of high and low dislocation density close together. The microstructure of the material observed after cold-rolling was significantly altered during subsequent annealing both at 598 and 648 K, although some microstructural features from the as-cast condition were inherited throughout thermal and mechanical processing. At both temperatures the microstructural analysis revealed the evolution of the deformed and elongated grains until the deformed matrix was completely replaced by a new strain-free recrystalized structure. However, at 598 K the first recrystallization signs were observed at about 1000 s of annealing time starting at the center of the specimen. After 100000 s, the microstructure is completely recrystallized but with a much larger grain size and a greater aspect ratio towards the borders of the sample than in the middle. The mean recrystallized grain size was determined to be about 123 μm. These microstructural differences are believed to be related fundamentally to the solidification and deformation conditions that take place during TRC. Higher solidification rates near the surfaces of the material bring about a higher solute supersaturation of the matrix. Therefore, decomposition and precipitation reactions during annealing subsequent to cold-rolling are expected to be more pronounced in these areas and to slow down the recrystallization kinetics. At 648 K the deformed alloy begins to recrystallize at a more shorter time of about 40 s and as expected the recrystallized grain size was observed to be much smaller, approximately 58 μm. The TEM observations conducted in the deformed and annealed samples allowed to follow the dislocation substructure evolution of the material during annealing. Even after 16 s of annealing time at 598 K some areas of the sample revealed a significant reduction of the dislocation density and sharpening of the dislocation cells. As annealing progresses much cleaner dislocation cells were observed together with the precipitation of fine rod-like particles. However, despite the fact that under the optical microscope the sample sems fully recrystallized after long annealing times, the TEM observations revealed the existence of sharp dislocation cells even after annealing at 100000 s, together with a large volume fraction of precipitates. Such a dislocation network indicates that recrystallization as a discontinuous reaction has not taken place all over the specimen and that small precipitates formed during annealing contribute to the stabilization of the dislocation substructure. At 648 K the dislocation substructure is observed to be completely removed after sufficiently long annealing times and that the amount of small precipitates formed during annealing is significantly less than at 598 K. It is believed that under these conditions recrystallization occurs in a relatively short time period before precipitation reactions become operative.

Restoration and recrystallization kinetics

The recrystallization kinetics of the material has been followed through the restoration of the mechenical properties determined during annealing after cold-rolling. Therefore, a restoration 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:

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

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. The analysis of the restoration curves obtained from hardness measurements by means of a Johnson-Melh-Avramy-Kolmogorov (JMAK) kinetics indicated that the exponents of the relationship fluctuate around a value near 0.38 for the data at 598 K, whereas at 648 k it was found to be about 0.50. As illustrated in Fig. 3 the JMAK model seems to describy reasonably well the experimental data although the exponents determined are somewhat low.

For comparative purposes, the above figure also presents the hardness restoration results previously determined for the as-cast alloy deformed and annealed in the same conditions. Several interesting aspects are worth to comment in relation to this figure. Firstly, it is observed that the homogenization treatment does not give rise to an acceleration of the restoration kinetics, in contrast to the results reported for the same alloy homogenized at 653 K.

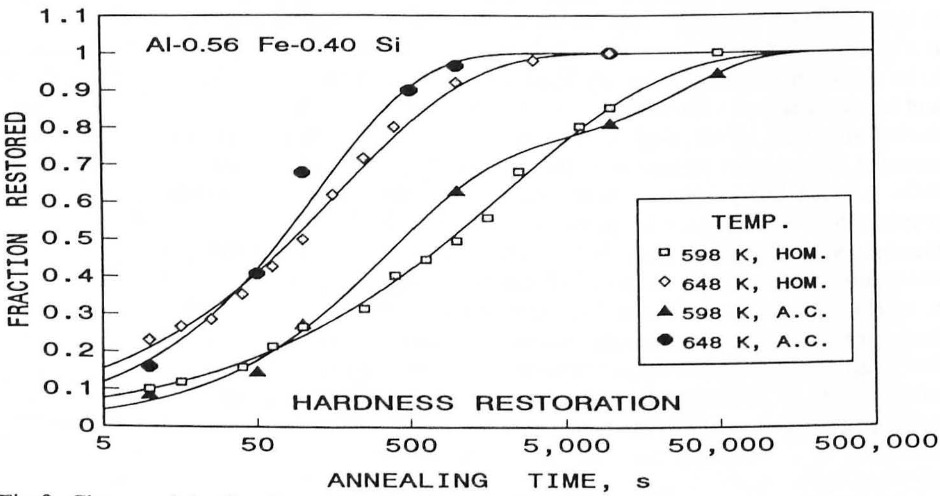

Fig.3. Change of the fraction restored with annealing time at two different temperatures.

In fact, it is noticed that at both annealing temperatures, the kinetics is sligthly slower than for the as-cast material, except for the curve at 598 K and fractions restored higher than about 0.7. On the other hand, both restoration curves for the homogenized alloy are seen to be quite smooth which allows the use of a single JMAK equation for their description, contrary to the curve at 598 K for the as-cast material. These results tend to indicate that the homogenization treatment has effectively reduced the precipitation of fine particles from the as-cast supersaturated matrix even at low annealing temperatures. However, it also has produced many other microstructural changes that have affected the stored energy of the material and have therefore modified the restoration kinetics. The depletion of the as-cast matrix in elements such as Fe and Si would expect to lead to an acceleration of the restoration kinetics due to the elimination of segregation and precipitation effects that would hinder the nucleation and growth of new recrystallized grains. Nevertheless, the homogenization treatment also affects two more important parameters that play an important role in the amount of stored energy available for the restoration process. Firstly, the reduction of the dislocation density of the material through recovery and recrystallization processes, a fact that has clearly been shown to take place in the present case. Secondly, an increase of the recrystallized grain size of the material previous to the cold rolling stage. It has been recognized that under conditions of moderarte deformations, such as those involved in the present work, grain boundaries play an important role as nucleation sites for recrystallization. Therefore, it would be expected that an increase in this parameter would also slow the restoration kinetics of the homogenized alloy. In summary, the reduction of the segregation and precipitation effects achieved through homogenization would be opposed, in terms of the acceleration of the restoration kinetics, by a reduction of the accumulated strain in the material and the increase of the initial grain size prior to cold rolling. A crude estimate of the activation energy of the process or processes involved in the changes of this mechanical property with annealing time, based only two different temperatures allowed to determined a mean value of approximately 149.6 $KJmol^{-1}$. Although it is only an approximation, the value calculated is quite similar to the usually accepted value of 156 $KJmol^{-1}$ for the activation energy for self-difussion of aluminium and aluminium alloys.

Conclusions

The homogenization treatment applied at 853 K gives rise to the complete elimination of the colonies of dendrite-like morphology particles present in the alloy in the as-cast conditions and to a significant depletion of the supersaturated as-cast matrix in Fe and Si. As a result, the decomposition and precipitation effects present in the as-cast alloy have virtually been eliminated even at low annealing temperatures. However, the restoration kinetics of the homogenized alloy is observed in general to remain nearly unchanged in relation to the as-cast material, possibly due to the reduction of the accumulated strain by the occurrence of recrystallization and the increase of the initial grain size prior to cold rolling. Mayor differences in the restoration kinetics of both materials arise from precipitation effects in the as-cast alloy annealed at 598 K.

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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. B. Fajardo and J.V. Valera 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.