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The Effect of Alloying on the Structure Formation during Compaction of Microcrystalline Aluminum-Silicon Alloys

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Abstract

The microcrystalline structure of aluminum-silicon alloys is obtained when high cooling rates (more than 10^4 $K.s^{-1}$) are applied, which results in a highly non-equilibrum state in the form of suppersaturate solid solution. A product is obtained in the form of ribbons which are less than 100 μm thick. These fine ribbons are usually subjected to consolidation by cold isostatic compaction followed by hot extrusion at relatively high temperatures (above 400°C), during which phase transformations (decomposition of the supersatursted solid solution) and coarsening of the structure occur and this results in deterioration of the properties. The purpose of recent work is to study the structure formation at lower temperatures. These data will allow the development of technologies that retain as much as possible the finegrained two phase structureas after the applied heat treatment.The microstructures of the alloys are examined with a Reichert MeF2 optical microscope and the average area of the silicon particles (S, μm^2) is determined as a measure of the structure dispersion. Particular stages in structural change are determined, both by X-ray analysis of crystal lattice parameters of the alluminium solid solution, and by the Perkin-Elmer DSC-2 Differential Scanning Calorimeter, with transient heating. X-ray tests are performed with a powder diffractometer DRON-3 (CuK_αfiltered emission, scintillation registrtion, continuous recording on a chart band).The lattice parameter variatiosn are used to examine the kinetics of structural changes in the microcrystalline state. The resulting curve shape suggests that the lattice parameter follows a parabolic dependence. A value of 94.6 $kJ.mol^{-1}$is obtained for activation energy of the decomposition of the solid solution at lower temperatures which was explained with acceleration of the Si diffusion process, due to the defects in the structure of the aluminum matrix.

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In the case of high temperature annealing at 400-500°C the activation energy of the process is 135kJ.mol-1 which was explained with the decomposition of the supersaturated solid solution. Coarsening process can thus be devided into two stages. During the first stage the particles reach size of several tens of nm. During the second stage, the average size of the silicon phase is in the micronial area. The temperature efect requires special measures to reduce the microstructure coarsening. One of the possible ways is via an additional alloying which is object of this investigation.

Keywords: Rapid Crystalization; Microcrystalline Alloys; Aluminum-Silicon Alloys; Alloying.

1. Introduction

Microcrystalline alloys are of great interest because of their unique physical and technological properties due to their extremely small grain size. The synthesis of materials with microcrystalline structure can be achieved in two ways. The first involves the application of significant plastic deformation at relatively low temperatures. The second consists of microcrystalline structure obtaining by non-equilibrum crystallization. This allows a unique structural state with improved strength and plasticity to be achieved.

The rapid crystallization of microcrystalline alloys allows the creation of a new generation of high strength alloys whose strength is comparable to that of titanium. Whereas, the maximum strength of conventional aluminum alloys does not exceed 550 MPa, titanium starts with a strength level from 750 MPa. Rapidly crystallized aluminum alloys allow filling the gap between aluminum and titanium. In addition to strength, other properties such as thermal expansion, wear resistance and high-temperature strength can be significantly improved [1]. Among various rapidly solidified alloys hyper-eutectic AlSi alloys have attracted with growing interest due to their excellent properties, such as light-weight, good wear resistance, and low coefficient of thermal expansion (CTE) [12].

The recent achievments in the field of rapid solifidication of metal alloys is characterized by widening of the range of new alloys with structure and composition outside standard foundary and deformble alloys. Of particular importance is the possibility of processing of the aluminum scrap [2].

However, the preparation of a microcrystalline structure of aluminum alloys requires the application of high cooling rates of $\geq 10^4 K.s^{-1}$, which results in alloys in a highly non-equilibrum state in the form of supersatureted solid solution of alloying and inpurity elements. This leads to a significant improvement in the homogeneity of the composition and the structure of the obtained microcrystalline alloy. It is characterized by reduced grain, colony, dendrite and individual phase sizes, which also determines the increased mechanical and anti-corrosion properties [3].

However, the product obtained in the rapid crystallization is usually in the form of ribbons less than 100 μm thick. This requires further consolidation of the ribbons via heat treatment at elevated temperatures in view of the industrial application of the material. Typically, consolidation is achieved by hot pressing or hot extrusion at relatively high temperatures (≥ 400°C) in which phase transformations (decomposition of the supersatursted solid solution) and coarsening of the structure occur and this results in deterioration of the properties. The

microstructure and mainly the local clustering of Si phase during heat treatment play an important role in determining the mechanical properties because fracture initiates at the clusters and grows rapidly through the matrix [13]. Isothermal annealing of Al-12wt%Si alloy ribbons showed that the microstructure features of rapid solidification disappeared when the annealing temperature was above 250°C [14]. Their results indicated that the microstructure of rapidly solidified alloy was less stable. However, most of the rapidly solidified Al alloys are needed to be consolidated at the temperature above 250°C to obtain near full density [15]. Si particles grow predominantly via precipitation of the solute Si from the supersaturated solid solution below 300°C while coarsening is the predominant mechanism at higher temperatures [14]. It is also observed that with increasing annealing temperature, second phase (Si) particles become coarser and coarser. The microstructure reveals that silicon particles are spherical and randomly distributed in the aluminum matrix. This is due to outward diffusion of Si from a supersaturated solid solution of Si in Al. This causes nucleation of a separate Si phase and as the annealing continues more and more Si comes out of the supersaturated solid solution. This continuous supply of Si during annealing causes growth and coarsening of Si nuclei into spherical Si particles. By increasing annealing temperature coursing of Si-particles take place [16].

Preliminary study showed that rapid solidified microcrystalline AlSi alloy has a typical hypoeutectic structure with Si particles segregated on dendrite boundary [17]. The size of the particals is in the range of 5-6 nm. This structure guarantees mechanical characteristics suitable for further plastic deformation treatment. In the compaction process, the ribbons undergo a thermal action, resulting in a substantial increase in Si particles size. The coarsening process of the structure can be divided into two stages: during the first particles reach the size of several tens of nm. During the second stage, the average size of the silicon phase is in the micronial area which deteriorates the properties of alloy. So the purpose of recent work is achieving of an essential reducing of structure coarsening during consolidation process by additional alloyng of AlSi11 and hence deriving alloys with improved mechanical and operational properties.

2. Materials and Methodology

Microcrystalline ribbons of alloy with composition closed to AlSi11 are obtained in a one wheel facilityforplaner flow casting (PFC) used in our previous studies [4].The contact surfaces of cooling wheel were made of copper. The strips have width of 7-12 mm and a thickness of 50-100 μm.

Compacting of the metal strips was carried out in two stages:

1. Cold isostatic compaction at a specific pressure of 600-650 MPa to blanks with a diameter of Ø 40 mm and a density of 70-75% of the theoretical one for these alloys.
2. Final compaction to massive blanks by hot extrusion. For this purpose a die with its own heater and the possibility for changing the degree of reduction is used. The extrusion was carried out at 723K and a reduction degree of 1/12.

The heat treatment of the ribbon samples was carried out in a laboratory electrical resistance furnace with low thermal inertia and high precision of addjustment. The rapid reach of the operating temperatures (up to 500°C

for 60 s) was facilitated by the small mass of the tested samples. They were packed in aluminum foil and were in good contact with the NiCr-Ni thermocouple.

The microstructures of the strips after annealing as well as the compacted alloys were examined with a Reichert MeF2 optical microscope. The average area of the silicon particles (S, μm^2) was determined as a measure of the structure dispersion.

Particular stages in structural change were determined, both by X-ray analysis of crystal lattice parameters of the alluminium solid solution, and by the Perkin-Elmer DSC-2 Differential Scanning Calorimeter, with transient heating. X-ray tests were performed with a powder diffractometer DRON-3 (CuK_αfiltered emission, scintillation registrtion, continuous recording on a chart band). The lattice cell parameters of the elemental cell of Al solid solution were determined by the angular position of the α_1 component ($\lambda CuK\alpha 1 \alpha 1$ = 1.54056 Å) of the line (422) recorded at an angular velocity of 1/8 [$^o 2\theta min^{-1}$]. Polycrystalline silicon standard with lattice parameter a_{et} = 5.43088Å was used as an external standard. The analysis of the effects of microdeformations and the dimensions of coherent scattering blocks on the widths of the diffraction lines was performed by Hall's relation [11].

$$Cos\theta\ (\Delta 2\theta)/\lambda = 1/<L> + \Delta a/a.2sin\theta/\lambda \qquad (1)$$

where: λ is the wavelength, $\Delta 2\theta$ is the semi-width of the diffraction line, θ is the Breg angle, <L> is the average size of the coherent scattering blocks, $\Delta a/a$ is the relative microdeformation.

The profiles of the diffraction lines (111), (200) and (220) recorded using CuKβ radiation were investigated. The choice of radiation was made in order to avoid systematic errors in the determination of $\Delta 2\theta$, which normally arise when using CuKα1, α2 doublet radiation.

The degree of supersaturation of the aluminum matrix was determined by the concentration dependence of the lattice parameter a = f (C_{Si}) of the Al-solid solution in the ribbons. Data from other authors was used for the calibration curve [5].

3. Experimental Results and Discussion

In the present study the influence of the alloying of "basic" aluminum alloy (AlSi11) with 0.6% Mg and with 0.2% Mg - 0.025% Sr was studied. It has been found that the addition of the alloying elements to the basic alloy has an impact on the above mentioned two coarsening stages. During the first stage, the influence on the temperature is less pronounced but the rate of desintegration process of the supersaturated matrix is higher. In Figure 1 the data from the changing of the lattice parameter with temperature are compared. The incleanation of the curves demonstrates the disintegration rate of matrix depending on temperature rise. In the "pure" and alloyed microcrystalline alloys the maximal rate apears at almost the same temperature. Differences in rates of degradation are attributed to differences in supersaturation of alloys and the parallel formation of $MgSi_2$ phase which interfere with the onset and growth of the secondary silicon phase. On the other hand, the solubility of the admixtures in the solid solution, i.e. their equilibrium concentration increases with temperature rise. This is particularly evident in Figure 1 of the alloy parameter data of the investigated alloys above 300 °C (curve for

alloy with Mg).

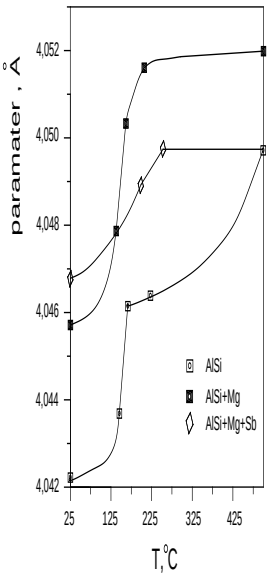

Figure 1: Change of latice parameter of Al matrix after isothermal annealing

The final structure is formed at higher processing temperatures in which the second stage proceeding is the coaresning of the microstructure. The kinetics of this process is demonstrated by the dependence of the average area of Si particles on the time determined for AlSi microcrystalline alloys with different elements additives. Table 1 gives the activation energies [Q, kJ.mol-1] for several Al-Si alloys. The activation energy of the structure coarsening during second stage is influenced by the presence of alloying elements and the final structure of the extruded samples in the presence of additives is finer than that of the "pure" alloy - Table 1 and Figure 2.

Table 1

Alloy	AlSi11	+Mg0.6	+Mg0.2Sr0.025
Q, kJ.mol^{-1}	135	106	216
Mean size of Si phase in extruded samples, μm^2	1.0	0.8	0.9

A comparison with the published in literatute data give a reason to suppose that in the "pure" AlSi microcrystalline alloys the transfer of Si to the growing particles perfoms by a diffusion mechanism. The alloying elements present in alloys change the mechanism, and it can be supposed that the surface processes become a rate determining step in the process of coarsening. Structure change performs with different values of activation energy.

The first or the second stage is more important for the influence of the alloying elements on shaping the final structure - this is a question that requires further studies. In our previous work [17] we have shown that in the presence of microcomponents, despite the higher rates of the structure change, determining for the finer

structure (compared to the pure “binary” alloy), is the transition state of the silicon phase. The latter is obviously determined by the low temperature stage - the decomposition of the saturated solid solution.

After compacting the massive specimens of alloy with magnesium have a finer structure, despite the fact that the structure coarsening is accelerated. This confirms the determining influence of the first stage in the structure formation. Thus, it is expected that at all studied high temperatures, the structure of the pure alloy will be coarser than that of the additionally alloyed alloys. This is supported by Figure 2 a, b, c whre the structures of compacted samples of AlSi alloys with and without magnesium are shown.

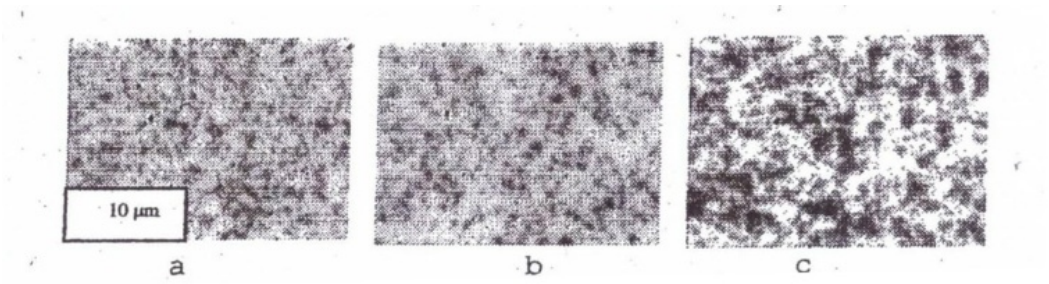

Figure 2: Compacted alloys a) Al-Si+Mg, b) Al-Si + Mg + Sr, c) Al-Si

The results of the present investigations have found a place in the development of technology for the prfduction of blanks of Al-Si-Mg alloys with a super-fine grain structure. From these blanks are produced sliding bearings, distinguished by increased wear resistance.

4. Conclusions

The rapid ctystallized microcrystalline AlSi alloys have a typically subeutectic structure with Si paticles 5-6 nm in size separated at the dendrite boundaries. This structure guarantees high mechanical properies after subsequent plastic deformation. In the compaction process, the ribbons undergo a thermal effect, resulting in a substantial coasrsening of Si particles. The process of structure corsening can be divided into two stages: through the first particles reach several tens of nm. In the second stage, the average size of the silicon phase fall into the micronient range. The influence of temperature on the described structural changes supposes nessesity of special measures to reduce the microstructure coarsening.

In order to limit this drastic increase in the silicon phase, it is necessary to select the possible low temperatures of hot extrusion. Their establishment requires a study of the kinetics of the processes of the structure's growth in both stages. It has been shown that the X-ray analyis is a convenient method to study this kinetics during the first stage. Through this analysis we can also look at the influence of the alloying elements on the structure formation. The study of structure change and plasticity on the output microcrystalline ribbons allows selection of an optimal temperature of hot extrusion. It is supposed that with higher extrusion forces the initial fine grain structure can be kept to a great degree.

The results obtained from the studies were applied in the production of sliding bearings with increased wear

resistance.

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