RESEARCH REGARDING THE OBTAINING OF BULK NANOCRYSTALLINE ALUMINIUM ALLOYS

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Abstract

The aluminium alloys exhibit an outstanding industrial and scientific interest, due to their great advantages like low density, plasticity, thermal and electrical conductivity, high corrosion resistance, and not least an easy processing comparing to other metallic materials. However, the conventional aluminium alloys present low strength and hardness. A significant increase of mechanical strength and hardness was reported for nanocrystalline alloys from the Al-TM-RE family, alloys obtained by controlled crystallization of the amorphous phase, in ribbons form. This paper presents the research carried out to obtain bulk nanocrystalline aluminium alloys by copper mould casting method. The obtained alloys were structurally characterized by electron microscopy and X-ray diffraction analysis and the hardness was determined by micro-hardness tests.

Keywords: aluminium alloys, nanocrystalline structure, copper mould casting, micro-hardness

1. INTRODUCTION

The aluminium alloys dominate the major markets of the automotive, aviation, chemical and food industry with a production of over 28 million tons in 2011. The interest in these alloys is mainly due to their low density, high thermal and electrical conductivity and good corrosion resistance [1]. The limited use of conventional Al alloys, due to their relatively low mechanical resistance, has caused, on one hand, an increased research in order to improve the properties of the existing alloys and, on the other hand, the discovery of new compositions of Al based alloys with improved properties. The results obtained over the years consisted in a variety of alloys with high strength, as a result of structural hardening heat treatment [1]. Usually, the structural hardening of aluminium alloys is realized by a treatment of solution annealing followed by artificial aging. Using this complex treatment, the structural hardening is realized due to the precipitation of intermetallic phases in a metallic matrix consisting of a solid solution of aluminium [2]. The most used Al based alloys that can be structural hardened, and the inter-metallic phases which ensures the hardening process, are presented in Table 1 [1].

Tab. 1: Al based alloys that can be structural hardened

<table>
<thead>
<tr>
<th>No.</th>
<th>Al-based alloys</th>
<th>Intermetallic phases which precipitates in metallic matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Al-Cu</td>
<td>Al\textsubscript{2}Cu</td>
</tr>
<tr>
<td>2</td>
<td>Al-Cu-Mg</td>
<td>Al\textsubscript{2}CuMg</td>
</tr>
<tr>
<td>3</td>
<td>Al-Mg-Si</td>
<td>Mg\textsubscript{2}Si</td>
</tr>
<tr>
<td>4</td>
<td>Al-Zn-Mg</td>
<td>MgZn\textsubscript{2}</td>
</tr>
</tbody>
</table>

Starting from the 1990's there were synthesized metal alloys that present nano-sized crystalline phase distributed in an amorphous matrix or consist of a solid solution of aluminium. These alloys are part of the
nanocrystalline alloys. Regarding the mechanical properties, a significant improvement was noted in Al-TM-RE nanocrystalline alloys, where Al usually exceeds 85% at., and the transition metal (TM) and rare earth (RE) does not exceed 10 % at. [3,4]. These alloys were obtained by controlled crystallization of the amorphous phase [5, 6]. This method of elaboration involves two stages: 1) obtaining an amorphous alloy by melt-spinning method, in form of continuous ribbons, with a thickness of 20 - 30 μm and 2) the controlled crystallization of the amorphous phase by annealing at a temperature close to primary crystallization temperature (T_x) of the amorphous phase. The experimental research showed that the controlled crystallization of the amorphous phase has a beneficial effect on the mechanical properties [7]. For the Al-Ni-Nd system, the hardness increases dramatic compared to the amorphous state (20-40%) and to conventional Al alloys (200-300%) [4,8]. The significant increase is caused by the effect of the nanocrystalline aluminium precipitation phases or of the Al amorphous matrix, enriched in alloying elements. Based on these results, there are intensified researches on bulk nano-cristalline alloys with high mechanical properties.

2. EXPERIMENTAL PROCEDURE

The aim of this experiment was to develop 1 mm bulk nanocrystalline alloys in rod form, using the copper mould casting method. The installation used is presented in Figure 1. The casting technology involves, in a first step, the elaboration of a master alloy with a chemical composition favourable for obtaining nanostructure. The second step involves the cutting of the master alloy, in order to obtain samples weighting 4 grams, which are melted by inductive heating in a quartz crucible provided at the bottom with a ejection nozzle. By applying a pressure using an inert gas, the melted alloy is ejected from the crucible into a copper mould. We chose two technological variants: 1) copper mould casting, 2) copper mould casting with additional cooling of the mould.

![Fig. 1](image)

**Fig. 1:** Installation used for the elaboration of the alloys: 1 – copper mould; 2 – induction coil; 3 – crucible; 4 – clamping system of the crucible; 5 – Argon hose; 6 – additional cooling system for the mould

The chemical composition of the master alloy was chosen taken into account that the nano-crystallization on the phases depends mainly of the amorphization tendency of the alloy [8]. Considering the remarkable results obtained in the case of Al-TM-RE nanocrystalline alloys and previous experiments [3, 4, 5, 7], the chemical composition of the master alloy was chosen as follows: Al_{85}Nd_{8}Ni_{3}Co_{2}V_{2}. 
Using the copper mould casting method, rods of 1 mm in diameter were obtained, presented in figure 2.

Fig. 2: Al_{85}Nd_{8}Ni_{3}Co_{2}V_{2} rod obtained by copper mould casting

The rods obtained were structurally characterized by scanning electron microscopy and X-ray diffraction and were subjected to micro-hardness tests. The microscopic analysis was performed using a FEI Quanta Inspect type FP 2017/11 scanning electron microscope on metallographic samples of the master alloy and rods. The rods obtained by copper mould casting method, without additional cooling were marked with A and those obtained by additional cooling of the mould were marked with B. X-ray diffraction analysis was performed on a DRON 3 device, using the following parameters: current voltage of 33 kV, current intensity of 35 mA, a wavelength of 0.71 Å and the speed of the X-ray tube of 1 °/min. The micro-hardness tests were performed on a Wolpert 402MDV device, using a Vickers penetrator and a load of 0.3 daN. On each sample were performed 5 tests. To highlight the results, a DURALUMIN/2017 alloy was chosen as reference, which has a hardness of 110 HV0.3 after hardening and aging heat treatment.

3. RESULTS AND DISCUSSIONS

The macroscopic imagines of the rods obtained by the two technological methods are presented in Figure 3. It can be noted that when it was used the additional cooling system of the mould, the solidification of the alloy was faster, which led to a shorter length of the rod and a greater amount of material remained in the feeder - Figure 3 b.

Fig. 3: The rods obtained: a) without cooling the mould; b) with additional cooling of the mould

The microscopic analysis reveals a dendritic structure of the master alloy with a predominant mechanical eutectic mixture - Figure 4.
In the case of the rod obtained using copper mould casting (sample A) without the cooling of the mould, there is a significant grain finish, the size of the crystalline grains is around 1 ... 2 μm, Figure 5. We can state that the structure is in the microcrystalline domain.

The SEM image of the rod developed with the additional cooling of the copper mould (sample B) shows a high grain finishing. The grains have a crystalline globular form, due to the high cooling rate, and their size does not exceed 200-300nm - figure 6.

The X-ray analysis certifies the structure showed by scanning electron microscopy. The diffraction pattern of sample A (figure 7) shows the presence of multiple high diffraction peak, belonging to intermetallic compounds. The diffraction pattern of sample B (figure 8) shows only low intensity peaks belonging to an aluminium solid solution. Therefore, in the case of sample B, the cooling rate was high enough to obtain total soluble components in aluminium and a structure finishing specific to nanocrystalline structure.
The micro-hardness results are presented in table 2.

Tab. 2: Al based alloys that can be structural hardened

<table>
<thead>
<tr>
<th>Sample</th>
<th>Measured hardness, HV0.3</th>
<th>Hardness Average value, HV0.3</th>
<th>Reference hardness DURALUMIN/2017, HV0.3</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>172,7 190,5 173,8 202,3 213</td>
<td>190,4</td>
<td>110</td>
</tr>
<tr>
<td>B</td>
<td>335,9 310,3 366,6 344,8 289,5</td>
<td>329,4</td>
<td>110</td>
</tr>
</tbody>
</table>
It can be observed that the values of the hardness increase with the degree of structure finishing. It can also be noted that in the case of sample B, which has a structure that can fit in the nanocrystalline domain, the values of the hardness are about 3 times higher than the hardness of heat treated duralumin.

4. CONCLUSIONS
The Al-TM-RE alloys can be nanostructured by controlled crystallization of the amorphous phase or by the rapid cooling of the melt. The experiments had shown a high potential for obtaining these alloys in bulk products with nanocrystalline structure. The nano-crystalline structure of these alloys provides a hardness three times higher than the duralumin hardness. The copper mould casting method is a viable mode for obtaining aluminium based nanocrystalline alloy. A careful correlation between the chemical composition and technological parameters of elaboration can lead to obtaining bulk products with exceptional strength properties. The obtaining of nanocrystalline structures directly by copper mould casting, combined with exceptional mechanical properties, make these new aluminium alloys very attractive for current and future industrial applications from aviation industry, automotive, etc.

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LITERATURE