Exploring the effects of repetitive corrugation and strengthening on alloy A5083 using X-ray diffraction techniques

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Abstract. Aluminum alloys are highly versatile materials and their behavior can be improved by severe plastic deformation. X-Ray Diffraction (XRD) is a key tool for studying the microstructural changes and the evolution of the crystalline structure during this process. This combination of processing techniques helped the development of high-performance aluminum alloys for various industrial applications. The objective was to determine a heat treatment to promote a new crystallographic texture of AA5083 under a severe plastic deformation process. The determination of this thermal ratio, as well as the crystallographic characterization of the material, were studied through XRD, thermo-diffraction, Rietveld analysis, and pole figures (PF). The results showed that using a partial recrystallization heat treatment combined with the RCS process favors obtaining a characteristic recrystallization and deformation texture (*cube* and *brass* component). Furthermore, the evolution of diffraction peaks intensities at different temperatures confirm that recrystallization takes place during the process. Finally, the phases present in the A5083 alloy were determined by XRD.

Introduction

Aluminum alloys are widely used materials in various industries due to their excellent combination of properties, including high strength, lightness, and corrosion resistance. These alloys can further improve their characteristics through the process of severe plastic deformation, which allows them to obtain a refined microstructure and superior mechanical properties. The X-Ray diffraction technique plays a fundamental role in the study of the structural changes that occur during Severe Plastic Deformation (SPD) of aluminum alloys[1].

SPD involves applying large deformations to a material in one or several stages, generating a high density of crystalline defects, such as dislocations and subgrains. This process leads to significant changes in the microstructure of the material, including the formation of subgrains of reduced size, a higher dislocation density, and greater uniformity in the distribution of alloying elements[2]. These microstructural changes directly impact the mechanical properties of aluminum alloys, improving their strength and ductility.

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XRD is an experimental technique used to analyze the crystalline structure of materials as well as phases of the sample. The diffraction pattern contains valuable information about the spatial arrangement of the atoms in the sample and the structural changes that occur during severe plastic deformation[3].

By studying aluminum alloys by XRD before and after severe plastic deformation, changes in the intensity and position of the diffraction peaks can be observed. These changes indicate alterations in the crystalline structure, such as the appearance of new phases, the formation of dislocations, and the generation of crystalline defects. In addition, XRD also makes it possible to determine important structural parameters, such as grain size and dislocation density[4].

The combination of severe plastic deformation and XRD provides a deep understanding of the microstructural mechanisms that govern the behavior of aluminum alloys during plastic deformation. This is essential for designing and developing alloys with improved properties for specific applications, such as the aerospace, automotive, and construction industries.

Material and methods

The starting material was a commercial AA5083 sheet of 1.2 cm in thickness. The nominal chemical composition is shown in Table 1. Table 1. Composition of the studied AA5083 alloy.

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Element	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
% wt	0.40	0.40	0.10	0.4	4.00	0.05	0.25	0.15	Balance

First, a stress relief heat treatment was carried out at 200°C for 1h and cooled in the air, to ensure homogeneity in the material to be worked on and eliminate any residual stress present. Following this, the material was rolled at 2.7 cm/min to reach a thickness of 1 mm and achieve the first crystallographic texture of the material.

As the first preparation step for any mechanical forming process, a stress-relieving heat treatment is performed to ensure homogeneity in the material to be worked on and eliminate any residual stress.

The second step is the most important for obtaining a crystallographic texture. In order to achive this, a matrix with a sinusoidal profile with an amplitude of 2mm and a period of 16mm was used. Samples were rotated 90° from the normal direction as shown in Fig. 1.



Fig. 1. Procedure to carry out one pass of RCS.

XRD, thermo-diffraction, and Rietveld analysis will be performed at each stage of the process. Finally, it was determined a pole figure record using Rigaku X-ray diffraction equipment, observing the families of crystalline planes {200}, to validate the new crystallographic texture.



Fig. 2. Heating chamber for thermo-diffraction.

Results and discussion

XDR coupled with a heating chamber allows to identify the crystalline phases and the microstructural changes, indicating the texture of the material, based on the change in peaks width and their intensity.



Fig. 3. Diffractograms of the peak (200) at different temperatures for the rolled A5083 alloy.



Fig. 4. Diffractograms of the peak (200) at different temperatures for the A5083 alloy subjected to 2 steps of the RCS process.

Figures 3 and 4 show the peaks associated with the plane (200), which was chosen for having a good balance between intensity and sensitivity to the effect of temperature. It is observed how the increase in temperature shifts the diffraction peaks to the left regardless of their previous state of deformation. This is due to the thermal expansion that occurs in the material, which causes an increase in the lattice parameter "a" [5]. In addition, in Fig. 3, an increase in the intensity of the peaks is observed, which indicates a texturing of the material. This texture needs a heat treatment before of the RCS process to favor the formation of a new texture.



Fig. 5. Thermal profiles of the peak intensity (200), for the alloy A5083, subjected to the rolling process and two steps of RCS.

Fig. 5 shows in more detail the favoring of a crystallographic texture over the material, where there are marginal changes in the intensity of the peaks from 32 to 220°C; from 280°C it increases significantly, which is an indicator that from that temperature, a new preferential texture is favored[6].

The Crystallography Open Database (COD)[7] was used to identify the characteristic peaks of the aluminum phase, as well as the presence of precipitates. The data was processed with the PDXL2 software, showing the presence of Al, Al₃Mg₂, MgZn₂, y Mg₂Si.



Fig. 6. X-ray diffractogram of the rolled A5083 alloy, refined with the Rietveld method.



Fig. 7. X-ray diffractogram of the A5083 alloy subjected to two RCS steps, refined with the Rietveld method.

Figures 6 and 7 show the phases observed in the A5083 alloy, with a slight shift of the peaks corresponding to the (313) and (240) planes to the left.

With the help of the Rietveld refinement, the analysis of micro-deformations and reduction in grain size can be quantitatively calculated (see Table 2).

Table 2. Rietveld refinement for allow A5083 subjected to the rolling process and 2RCS						
	Table 2.	Rietveld refinen	nent for allov	A5083 subject	ted to the rolling	process and 2RCS.

Rolled		2RCS			
Apparent Grain Size [A]	1334.91	Apparent Grain Size [A]	1499.64		
Micro-deformations	1.79E-03	Micro-deformations	1.21E-03		
Chi2	1.990	Chi2	0.890		

Once the sample has been subjected to two RCS steps, the microstrains in the crystal lattice decrease due to the high amount of shear stresses to which the material is subjected. This is evidenced in the Rietveld refinement showed in Table 2.

With this information, a Partial Recrystallization Heat Treatment (PRHT) was carried out, which was adapted to promote the formation of a new crystallographic texture; the following thermal cycle was proposed:

- Heat the alloy from room temperature at a rate of 15°C/min until reaching a solubilization temperature of 400°C, and hold for 60min.
- Heat the alloy from the temperature of 400°C at a rate of 15°C/min until reaching a solubilization temperature of 500°C; hold for 30min.
- Cool in the air.



Fig. 8. Proposed Partial Recrystallization Heat Treatment (PRHT).

Finally, with an established thermal treatment, pole figures were made to observe the crystallographic texture changes qualitatively.





Fig. 9. Evolution of the Pole Figures of the planes (200), of the AA5083 subjected to a process to observe the microstructural evolution.

As shown in Fig. 9, introducing a suitable heat treatment (PRHT) between the rolling process and RCS can encourage the formation of a new crystallographic texture.

In this case, the starting point is a beta fiber (rolled), which is maintained during the PRHT, and when subjected to the RCS process, it changes to a component with a typical recrystallization texture (*cube*) coexisting with a typical component of shaping processes (*brass*).

The formation of textures in aluminum alloys produced by thermo-mechanical processes, including SPD processes, is a very striking and novel field of knowledge that is currently being studied[8]. It is intended to continue with the study of this work to finish explaining the phenomena of texturing that were produced.

Conclusions

- According to crystallographic analysis, a partial recrystallization heat treatment was determined to obtain a bimodal texture.
- The Al, Al₃Mg₂, MgZn₂, and Mg₂Si phases were identified with the help of XRD.
- A Rietveld analysis was quantified and determined the severity of the RCS process in the micro-deformations, as well as evidenced by the texturing of the material when subjected to this process.
- It was possible to obtain a characteristic crystallographic texture of recrystallization (*cube* component) with another characteristic of mechanical processes (*brass* component) for the A5083 alloy.

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