Experimental report

22/07/2019

Proposal:	**1-01-165**	**Council:**	10/2018
Title:	Correlation between aluminum alloys fractal creep substructure parameters and neutron diffraction peak broadening		
Research area:	Materials		

This proposal is a new proposal

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Samples: Aluminum

Instrument	Requested days	Allocated days	From	To
SALSA	4	3	17/06/2019	20/06/2019

Abstract:

The purpose of the present experiment is to investigate the evolution of dislocations substructure (sub-grain and forest of dislocations) generated during creep of aluminum alloy as a function of creep strain for further understanding the fractal nature of these substructures. These microstructural features will be investigated from the evolution of neutron diffraction peak broadening of different hkls.

Experimental Report

Experiment Title: Correlation between aluminum alloys fractal creep substructure parameters and neutron diffraction peak broadening **(REF. 01-01-165**)

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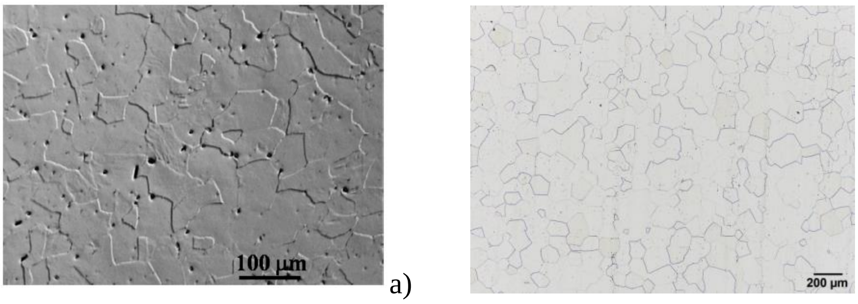
Introduction

The purpose of the present experiment was to investigate the evolution of dislocations substructure (sub-grain and forest of dislocations) generated during creep of aluminum and Al-Mg alloy respectively with strain for further understanding the fractal nature of these substructures. One of the main ideas that must be confirmed experimentally is the generation of a fractal like dislocation substructure in aluminum alloys resulting from creep strain [1,2].

Experimental method. Materials.

The samples used in the present investigation are extruded alloys and powder samples of the same alloys (same chemical composition). The initially proposed AA6061 alloy has been substituted for the experiment by pure aluminum. This decision has been taken to avoid the difficulty in the diffraction analysis introduced by the presence of a second phase (Mg_2Si) in the AA6061 alloy, which brings the precipitation process of these alloys into the problem and the associated complexity. The microstructural characterization of the materials investigated has been conducted by scanning electron microscopy and also the texture has been measured by x-ray diffraction. The typical grain size of pure aluminum and the Al-Mg alloy is shown in figure 1. The mean grain size is around 60microns for both alloys. The grains are slightly elongated along the extrusion direction. The creep samples were machined with the long axis parallel to the extrusion direction.



a) b)

Figure 1. Pure aluminum a) and Al-Mg alloy b) grain size.

The materials investigated present a different texture despite they were extruded at similar temperature and extrusion pressure conditions. In figures 2 and 3 it is shown the texture of the extruded pure aluminum and Al-3%Mg alloy (0% deformed) respectively.

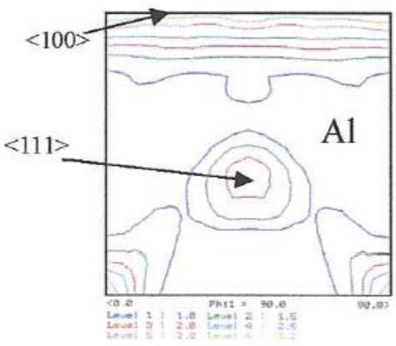


Figure 2. Texture of Pure aluminum.

The texture of the un-deformed Al-3%Mg alloy (0% strain) has been measured by neutron diffraction by tilting the sample (at 5º increments) from the radial (0º) to the axial (90º) position, for a single azimuth angle, taking advantage of the extrusion symmetry of the sample, and using the same gauge volume of 2x2x2 mm^3 (for a fixed time of 2000 sec.) that was employed to measure the variation of d_{hkl} (different hkl) with tilt angle. The integral intensity for the different hkl, and their variation with tilt angle, has been recorded, figure 3.

Figure 3. Diffracted intensity evolution of several hkl from radial to axial direction for the un-deformed Al-3%Mg alloy. These variations account for the initial texture of the alloy which is, in summary, described by a <200> and <311> fiber texture, with the fiber axes parallel to the extrusion axes direction.

The creep tests were conducted at constant stress at 573K and 21 and 29MPa. The samples were deformed up to 1, 2 and 3% for pure aluminum and 1%, 3% and 6% for Al-Mg. In order to freeze the dislocations microstructure corresponding to each strain level, the samples were cooled under the action of the applied load from 573K to room temperature.

Results

Measurements of d_{hkl} and $FWHM_{hkl}$ have been conducted for the pure Al and Al-3%Mg for different degrees of creep deformation. Due to the limited beam time assigned for the experiment, only the (111), (200) and (311) reflections have been measured. A gauge volume of 2x2x2 mm^3 for a monitor time of 2000 sec. have been using for the neutron measurements. Reflections (111) and (200) correspond to the most common ones in extruded aluminum alloys and reflection (311) reveals the average behavior of the polycrystal. Significant variations in the values of d_{hkl} for the different reflections with tilt angle have been obtained.

In the case of pure Al, the d_{hkl} hardly varies with respect to the un-deformed sample (0% strain) except for the sample of 6% that is very in the tertiary. In the case of Al-3%Mg, the d_{hkl} varies with respect to the un-deformed sample (0% strain). This information is summarized on figure 4.

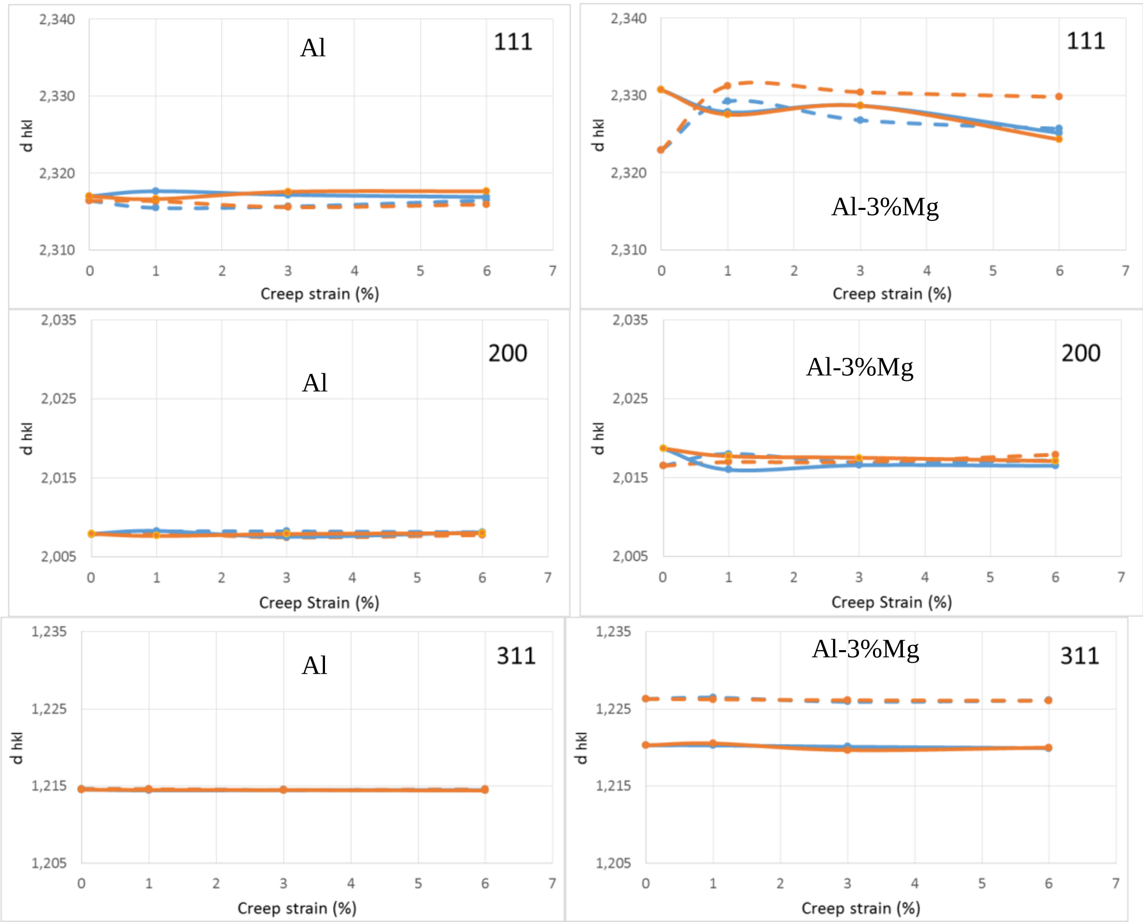


Figure 4. d_{hkl} for the pure Al and Al-3%Mg (111), (200) and (311) reflections. Axial(dashed lines) and radial directions are shown. 29MPa blue lines and 21MPa brown lines. Al plots area located to the left and Al-Mg to the right in the plot.

The evolution of the dislocations fractal substructure during creep produce in the case of Al-3·Mg a lattice parameter distortion that must result in a change in shape of neutron diffraction peaks (*e.g.*, increasing the FWHM parameter or integral area). In the case of pure Al, the FWHM is slightly reduced with respect to the sample of 0% deformation. In figure 5 the evolution of the FWHM for the materials investigated is summarized.

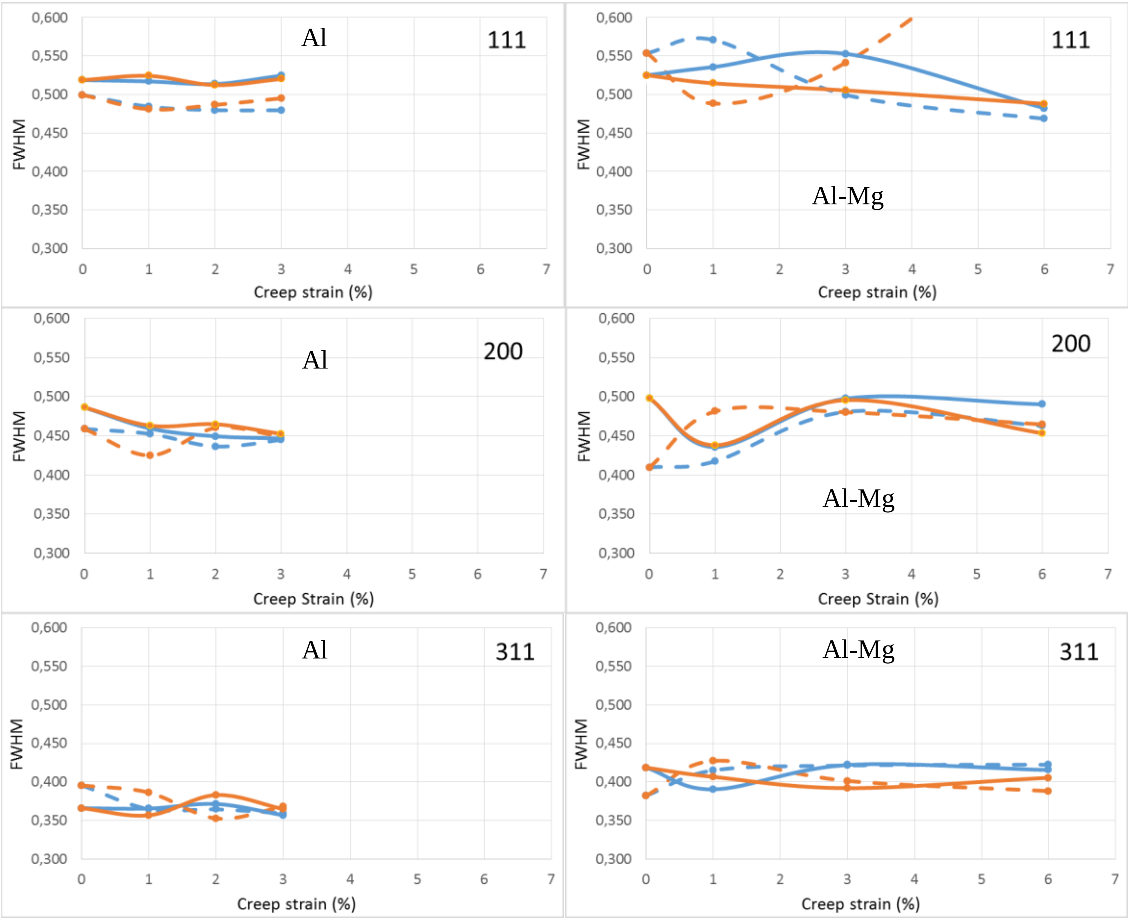


Figure 6. FWHM vs. creep strain for the materials investigated. Axial and radial directions are shown. 29MPa blue lines and 21MPa Brown lines. Al plots area located to the left and Al-3%Mg to the right in the plot. Axial components are represented by dashed lines.

Conclusions

The texture of the Al-3%Mg alloy has been measured taken advantage of the fibre texture of the extruded material.

A micro residual stress state, proportional to dhkl, generated during creep test in Al-Mg alloys have been determined using neutron diffraction experiments at SALSA instrument at the ILL.

Micro residual stresses are not developed in the pure aluminium due to the activation of the climb mechanism for dislocations movement at high temperature. The dislocation structure is represented by dislocations well organized into subgrain boundaries. The FWHM decreases with creep strain. This reflects a progressive organization of dislocations from the initial un-deformed state. On the other side, quite important micro residual stresses are developed in the Al-Mg alloy due to the activation of the slip mechanism (without climb) for dislocations movement at high temperature. The dislocation structure is represented by forest of dilocations. In this case, the FWHM increases with creep strain reflecting this messy organization of dislocations (no subgrain boundaries) in comparison to the initial un-deformed state.

Due to the low intensity signal (related to the fibre texture of the Al-3%Mg alloy investigated) of the 220 component in the axial direction of the samples, this component was not measured in the present investigation. This component must be measured to complete the experiment and check the stress equilibrium condition related to the dislocation structures generated during creep. A new proposal will be submitted with the aim of complete the study.