Experimental report

25/08/2021

Proposal:	**1-01-178**	**Council:**	10/2019
Title:	Correlation between aluminum alloys fractal creep substructure parameters and neutron diffraction peak broadening on (220 planes)		
Research area:	Materials		

This proposal is a continuation of 1-01-165

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Samples: Creep tensile samples of pure Al and Al-3.85%Mg

Instrument	Requested days	Allocated days	From	To
SALSA	3	1	08/09/2020	10/09/2020

Abstract:

The purpose of the present experiment is to complete a previous one that was carried out at SALSA instrument in June 2019. The aim of the experiment was to investigate the evolution of dislocations substructure generated during creep of aluminum alloy as a function of creep strain for further understanding the fractal nature of these substructures. The microstrains in connection with the dislocation structure introduced during creep deformation were investigated using the planes 200, 111 and 311 in the axial and radial directions of the samples. The planes 220 were not measured in the previous experiment because of the texture effect, also present in the 111 planes, thus hindering us for a proper measurement in the time available. The idea is to use the information provided by neutron diffraction peak shape to complete the description of the evolution with creep strain of dislocations substructures in Al-Mg alloy and pure aluminum for the plane 220 in order to complete the previous results for the planes 200, 111 and 311. In pure Al, the microstrains are relaxed during creep and the dislocations structure is similar in all grains. This behavior is opposite in Al-3.85%Mg

Experimental Report

Experiment number 01-01-178

Experiment title Correlation between aluminum alloys fractal creep substructure parameters and neutron diffraction peak broadening on (220 planes) ***CONTINUATION*** of experiment 01-01-165

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Introduction

The purpose of the present experiment is to complete a previous one that was carried out at SALSA instrument in the ILL in June 2019. The aim of the former experiment was 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. The microstrains in connection with the dislocation structure introduced during creep deformation were investigated using the planes *200, 111* and *311* in the axial and radial directions of the samples. The planes *200* and *111* lead usually to the highest intensity for the axial direction in extruded aluminum alloys. The *311* plane represents the isotropic response of the material for the present case and this explains its importance. The planes *220* were not measured in the previous experiment because the texture effect, also present in the *111* planes, strongly reduced the statistics, thus hindering us for a proper measurement in the time available.

The idea of this proposal is to use the information provided by neutron diffraction peak shape to complete the description of the evolution with creep strain of dislocations substructures in Al-Mg alloy and pure aluminum for the plane *220* in order to complete the previous results for the planes *200*, *111* and *311*. In pure Al, the microstrains are relaxed during creep and the dislocations structure tends to homogenize in all grains. This behavior is opposite in the Al-Mg alloy.

Experimental method. Materials.

In the present experiment, two different aluminum alloys will be investigated. On one side, pure commercial aluminum which develop subgrains during creep. On the other side an Al-Mg alloy that form forest of dislocations, or very small subgrains of fractal nature according with the SSTC model. Cylindrical tensile samples of 10mm length and 3mm diameter have been deformed up to four different levels: 0, 1, 2 and 3%. The microstrain and FWHM of these samples have been measured successfully for the planes *200, 111* and *311* in a previous experiment Ref 01-01-165. Two different experimental conditions during creep have been selected: 21MPa and 29MPa (at 300ºC). In this experiment, broadening of *220* reflections of the deformed samples will be characterized. Also the axial and radial residual stress will be calculated. Scanning a gauge volume of 2×2×2 mm^3 as in the previous experiment will be used which will allow obtaining a good peak statistics for times around 1 hour. Aluminium reference powders will be stored in vanadium cans of the same size as alloy bulk samples.

Neutron diffraction measurements will be performed on SALSA diffractometer. Based on previous experience (experiment reference 01-01-165), we plan to count on average about 1h per scanned peak considering the strong texture effect for this plane and 2h for the powder samples to provide high statistics. Considering the combination of: 2 alloys, 4 deformation degrees (0, 1, 2 and 3%), 2 creep conditions (21MPa/300ºC and 29MPa/300ºC), 2 reflections (*220* and *111)* and 1h per scanned peak, the TOTAL measuring time for the experiment is 32 hours. The 111 reflection was measured in the previous experiment but it is necessary to improve the measurements including a new calibration for this specific plane. The initial set-up of the experiment and also the diffractometer rotation motions for the different reflections/samples must be also considered. The **output** of the experiment is supposed to be principally:

- A detailed **and complete** (including all the important families: *200, 111, 220* and *311)* knowledge of the peak broadening parameter (*e.g.* FWHM or peak integral area) evolution with creep strain for aluminium alloys that correlates with the dislocations substructure. This study will assess the idea of the fractal nature of creep dislocations structure in aluminium alloys.

Implemented results will serve for the on-going article preparation and its final submission to a peer review journal in collaboration with SALSA scientists.

The creep tests were conducted at constant stress. The experimental details are provided in a previous work [3]. The samples were deformed up to 1, 2 and 3% for pure aluminum and 1%, 3% and 6% for Al-Mg. This difference is due to the very high strain rate found in the aluminum alloy in the tertiary stage (after 4-5% strain at

573K). 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. In this way, it is assumed that the *ex-situ* neutron diffraction measurements were conducted on samples with the dislocation structures developed during creep deformation.

Results

In this proposal it is intended to explain the evolution with creep strain of dislocations substructures in pure aluminum and Al-Mg from the information provided by neutron diffraction peak data. The strains studied cover from initial un-deformed condition (0% strain) and strains belonging to the primary, secondary and tertiary stages. It is supposed that the dislocations structure evolves smoothly during all creep stages.

<u>Variation of d_{hkl} and FWHM as a function of the creep strain.</u>

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), (220) and (311) reflections have been measured.

A comparison of the reflections (111), (200) and (311) for the experiments 01-01-165 and 01-01-178 reveals significant variations in the values of 2θ, figure 1.

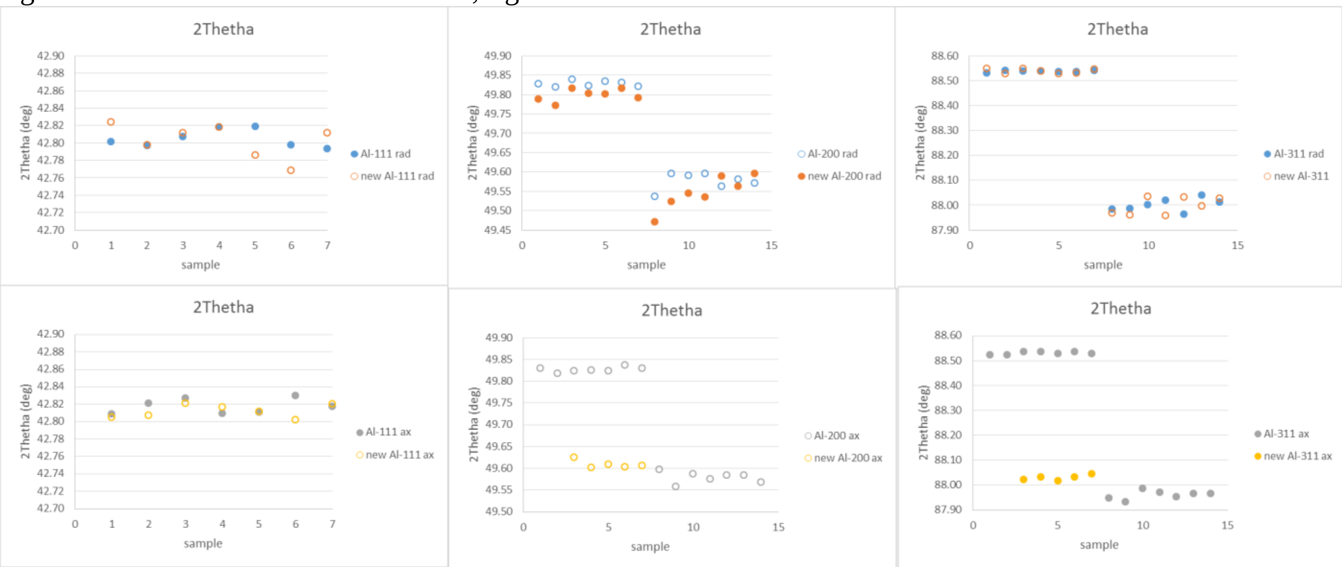

Figure 1.Variation of 2θ, for the (111), (200) and (311) peaks of the Al and Al-Mg alloy samples.

In the same way, comparison of the reflections (111), (200) and (311) for the experiments 01-01-165 and 01-01-178 reveals significant variations in the values of FWHM, figure 2.

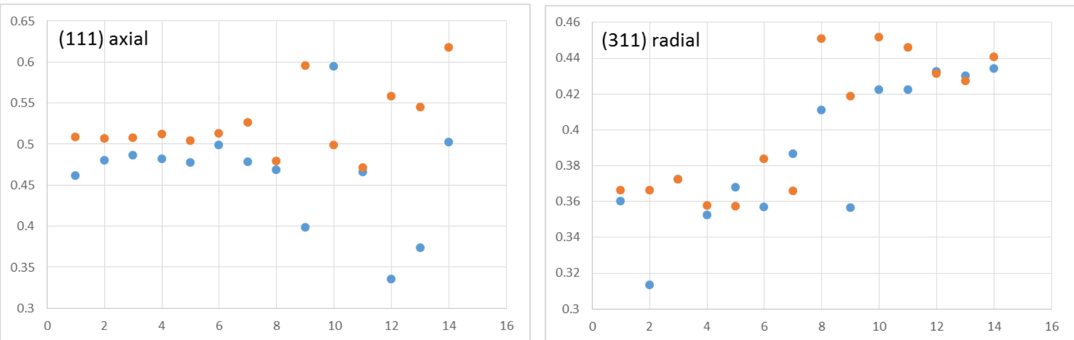

Figure 2.Variation of FWHM, for the (111), (200) and (311) peaks of the Al and Al-Mg alloy samples.

These differences have been associated, in principle, with the different gauge volume used in both experiments because the wavelength correction was done properly to compare both experiments.

The results found in the present experiment are not conclusive. This is due to the fact that the residual stresses are not equilibrated, Table 1.

Sample	111	200	220	311
1	-126.2	54.8	4.3	-6.0
2	-30.1	108.5	-23.4	13.8

3	-100.5	165.4	19.0	29.2
4	-119.8	234.0	-7.6	38.6
5	8.4	232.7	1.7	48.8
6	89.6	194.5	27.1	45.9
7	-100.0	269.7	-30.1	32.2

Table 1. Radial component of the residual stress, in MPa, for the Al alloy.

Conclusions

In the present experiment, the same samples in a previous experiment and 01-01-165 including reflection (220) on Al and Al-Mg creep samples have been measured and compared.

Significant differences have been found both in the microstrain and in the FWHM. These differences are mainly associated with differences in the volume gauge. In this second experiment, the gauge volume was decreased to 2*2*2*2mm3 to prevent the gauge volume from leaving the sample during the measurements. In addition, the residual stresses were found to be non-equilibrium in some of the samples studied.

For the aforementioned reasons, no definite conclusions on the state of residual micro stresses and the FWHM that develops in creep-deformed Al and Al-Mg samples can be drawn from the data of this experiment.