Methodology for the identification of nucleation sites in aluminum alloy by use of misorientatation mapping

S. Papadopoulou1,2, E. Gavalas1, S. Papaefthymiou2

1 ELKEME S.A., 61st km Athens-Lamia Nat. Road, Oinofyta, 32011, Viotia, Greece

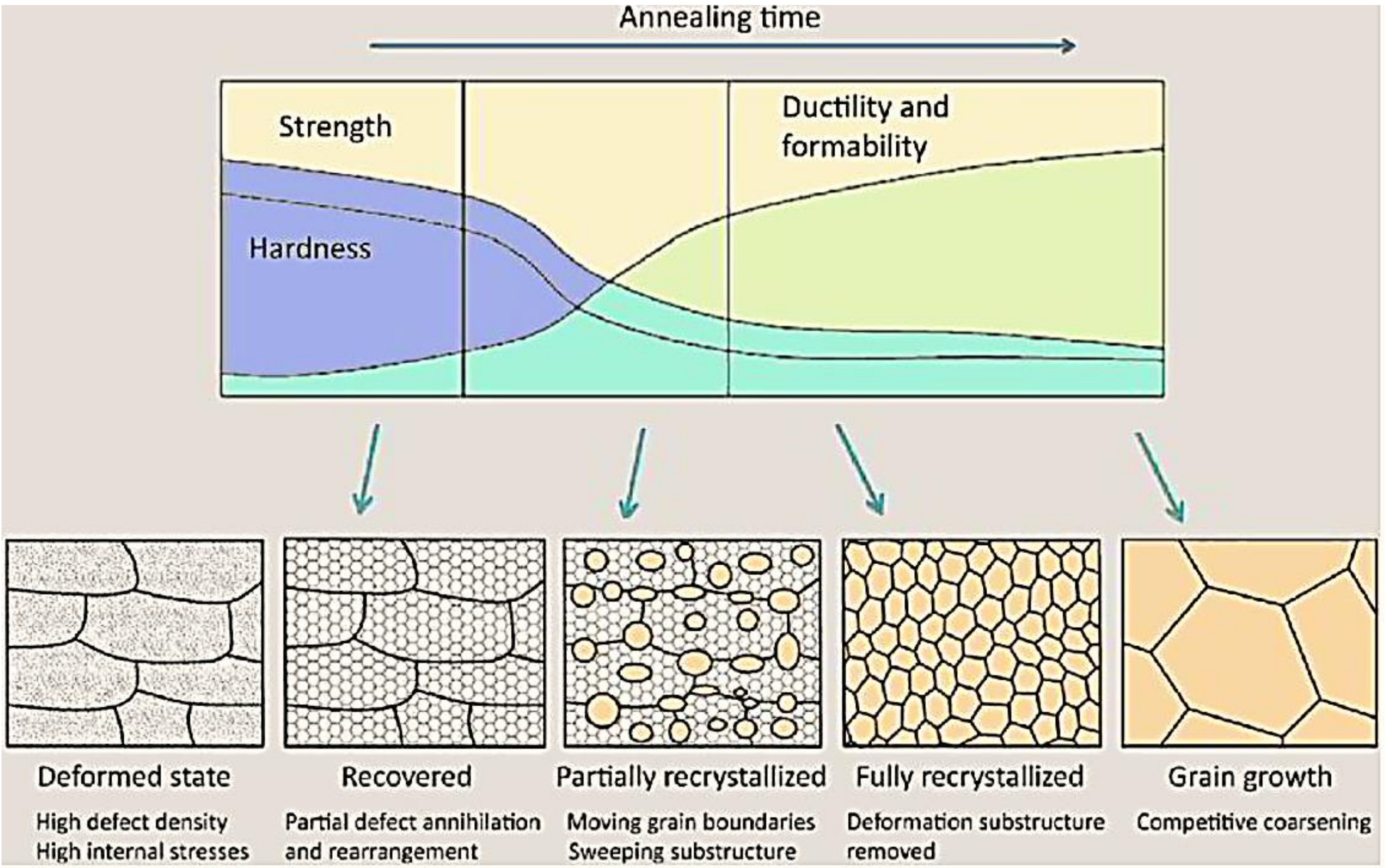
2 National Technical University of Athens, Laboratory of Physical Metallurgy, Division of Metallurgy and Materials, School of Mining & Metallurgical Engineering, 9, Her. Polytechniou str., Zografos, 15780, Athens, Greece; egavalas@elkeme.vionet.gr (E.G.); spapaef@metal.ntua.gr (S.P.)

* Correspondence: spapadopoulou@elkeme.vionet.gr; Tel.: +30 2262 60 4309

Introduction

- AA3104 is a strain-hardenable alloy having Mn as its main alloying element for the improvement of its mechanical properties and formability.
- Recovery and recrystallization processes which are present during the various thermomechanical stages are among the most important ones, yet they are not fully understood despite being critical with regards to producing suitable microstructure for several applications.

Recovery and recrystallization



1: Simulation and grain growth: basic phenomena and simulation methods, http://www.dierk-raabe.com/recrystallization-and-grain-growth-simulation/

Scope

In depth understanding of recovery and recrystallization processes regarding the possible nucleation points is required. Towards that direction EBSD measurements after various annealing processes were implemented. A processing approach considering all the possible misorientation angles and not only those exhibiting a maximum value of 5^{o} as it is used until now, was conducted in order to establish the use of this different criterium for strain analysis purposes of non-recrystallized materials as well as for the localization of possible crucial points for nucleation.

Experimental procedure

- AA 3104 sheet samples were cross-sectioned parallel to rolling direction and the specimens were cold mounted to avoid any annealing effects.
- Electron backscatter diffraction (EBSD) analysis was performed with an EDAX Hikari XP EBSD, high-speed camera, on the longitudinal cross-sections in order for the orientation of the Al crystals to be revealed as well as to determine the preferred texture components resulting from the applied manufacturing process.
- Samples corresponding to the most indicative thermal processes are presented as follows: a) hot rolled sample, b) 95% reduction cold rolled sample and the 95% reduction cold rolled sample after annealing at 250^{o}C and c) soaking time 90’, d) soaking time 100’, e) soaking time 120’ and f) soaking time 600’.
- EBSD scans were collected using a hexagonal grid with a 0.1-0.2 μm step. OIM software was used to process the retrieved EBSD results. The utilized confidence index was 0.1-0.05. IPF (inverse pole figure) diagrams and plots as well as KAM (kernel average misorientation) maps and GOS (grain orientation spread) maps were also used for the estimation of texture evolution, local plastic deformation and recrystallization percentage.

KAM maps

KAM maps are measuring the local plastic deformation by considering a pixel as a kernel and by calculating the mean deviation of orientations among this pixel and the first or second neighbors. Until now, the existing approach of KAM maps considers a maximum misorientation angle of 5^o and diminishes the relative variation through orientation averaging. Different criteria had been selected in order to find the best fit for both the cold rolled and annealed samples. The range which was selected to examine the fluctuation of KAM was 0°-65° with a 5° step. KAM was calculated considering the second neighbors and a maximum misorientation of 5° as suggested by the relevant literature, as well as setting of 65° and by comparing with the first neighbor.

Results (1)

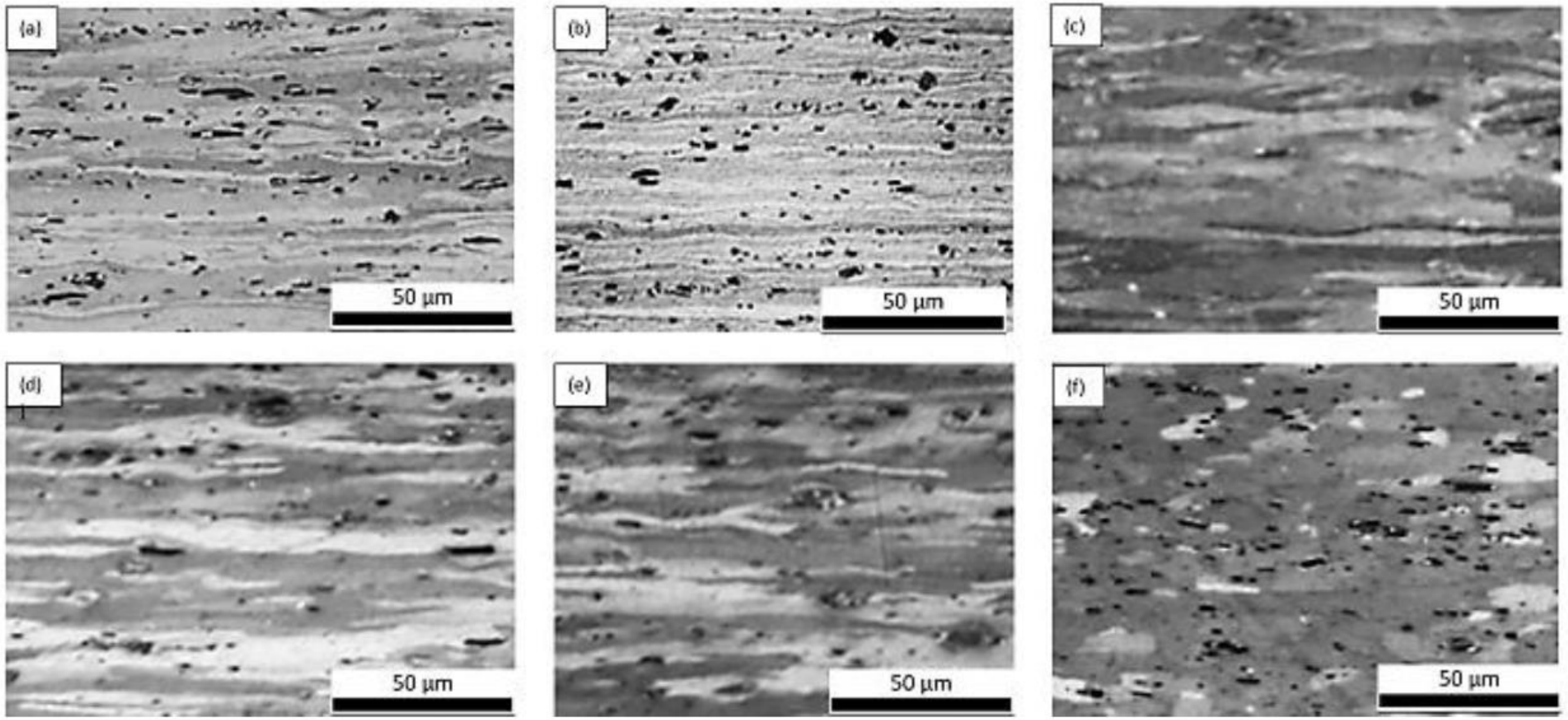


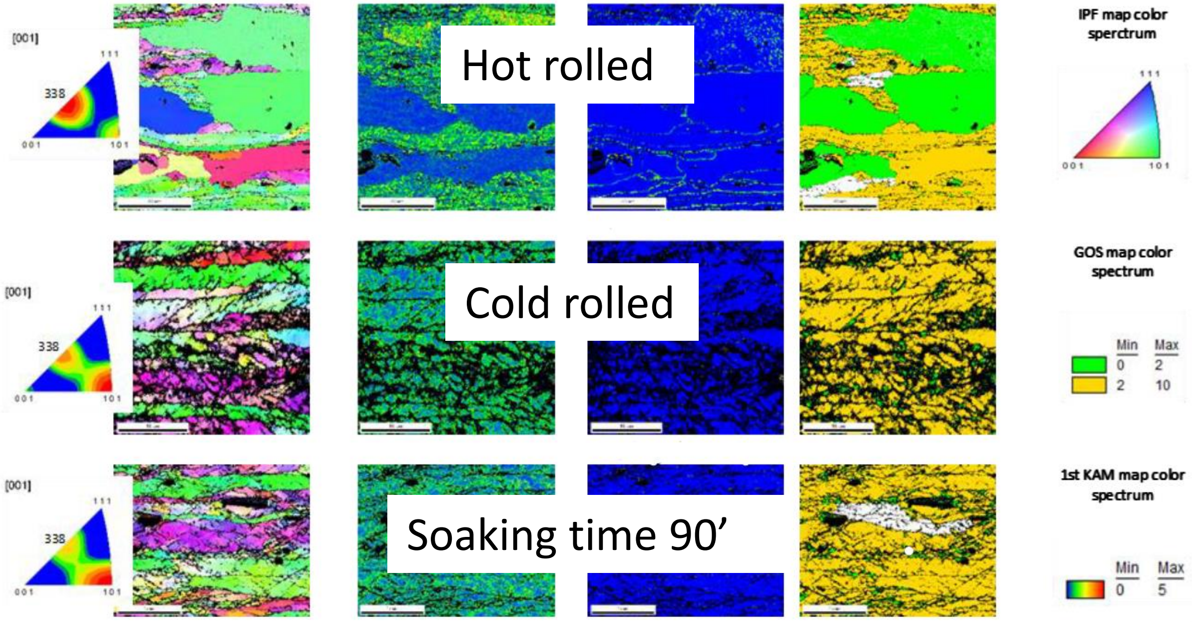
Figure 1: Optical micrographs showing a) hot rolled sample, b) cold rolled sample, c) cold rolled and annealed for 90’, d) cold rolled and annealed for 100’, and e) cold rolled and annealed for 100’ and f) cold rolled and annealed for 600’ after Barker’s etch.

Results (2)

Misorientation grain boundaries	Hot rolled	Cold rolled	Soaking time 90’	Soaking time 100’	Soaking time 120’	Soaking time 600’
2º-5º (%)	48.5	86.6	83.8	89.8%	72.4	4.9
5º-15º (%)	26.9	6.3	8.5	5.9	14.8	0.8
15º-65º (%)	24.6	7.1	7.7	4.3	12.8	94.3
KAM						
Mean KAM Angle (º)	2.2	1.1	1.3	1.6	1.1	0.7
KAM (%)	91	56.6	49.8	42.8	71.5	94.9

Table 1: Misorientation grain boundaries and KAM values measured for the examined samples.

Results (3)



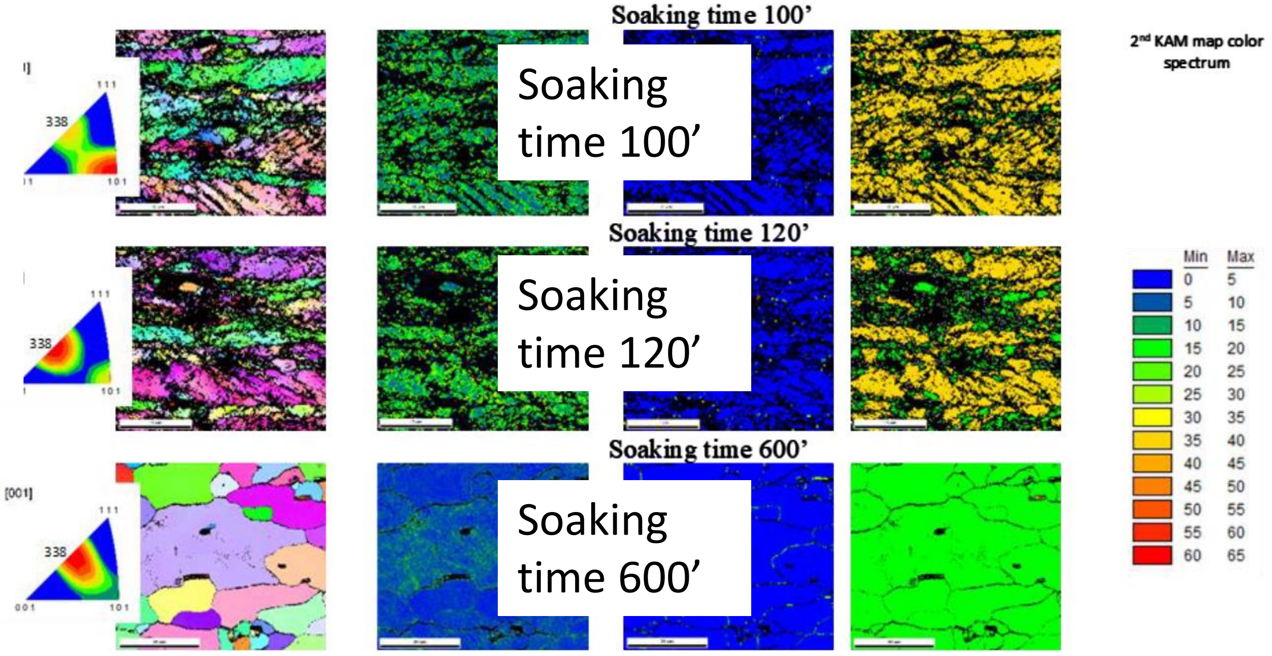


Figure 2: Indicative results of the examined samples in the form of IPF maps, KAM maps (with maximum misorientation angle 5ₒ and measuring the kernel according to the second neighbor) KAM maps (with maximum misorientation angle 65ₒ and measuring the kernel according to the first neighbor) and GOS maps.

Discussion (1)

- Determination of the sub-grain growth is challenging since the size, spread and misorientation of LAGBs are easily disturbed by a number of factors. At the same time, LAGBs exhibit reduced mobility at the recovery stage, thus contributing to the difficulties encountered towards the understanding of the roots or recrystallization [22].
- Researchers , who have tried to solve this vacancy observed that certain single crystals that formed microstructures through compression, exhibited sub-grains with a firm range of misorientation capable to provoke extensive recovery before recrystallization. The evolution of EBSD precision has also assisted in the effort for the understanding of the sub-grains kinetics. Grains separated by LAGBs are considered to be remnants of deformation when found in a recrystallized material [26]-[30]. The thermal treatment in all cases led to a high reduction in terms of HAGB fractions. The cold rolled sample exhibited a low percentage of HAGBs and dominant Cu, Brass and S oriented crystallites.

Discussion (2)

- The mean KAM angle in all annealed samples exhibited values less than 1° implying full-recrystallization as well as a homogeneous distribution of deformation within the grain while mean KAM angle values higher than 1^{o} refer to deformed grains. When KAM values vary within the grain, this is due to an inhomogeneous distribution of plastic deformation. When KAM values approach 0^{o} no significant plastic strain is detected after the thermal treatment.
- Newly formed grains and areas exhibiting high misorientation angle which are critical for the evolution of the recrystallization process and the position of possible nuclei had been recognized. It is known that sub-grains which are capable of forming new recrystallized grains are those who exhibit high misorientation angle in relation to their neighbors. High misorientation at certain regions such as HAGBs which pre-existed before the thermal treatment can be nucleation points.
- By reclaiming the retrieved results for the suggested KAM map processing, some interesting, regarding the nucleation process, points where detected and underlined in the current study.

Conclusions

- The hot-rolled sheet in the as received condition had formed orientations [101] and [338], which were maintained during all the annealing processes and coexisted with the orientations in-between before being finally restored at its initial state after the completion of recrystallization.
- The SGBs were low after hot rolling any only increased significantly after cold rolling before decreasing again after the thermal treatments.
- The increase of SGBs from the cold rolled condition until annealing with a soaking time 120’, which occurs mainly in orientations [101], allowed for the accurate definition of the completion point of recovery on the rolled 3104 sheet sample.
- No significant orientation rotations were observed during the annealing process whereas the mean KAM angle decreased during annealing.
- The mobility of LAGBs at soaking times between 90’ and 120’ indicates the evolution of the recovery process.
- The boundary mobility is found to decrease with a decreasing mean misorientation angle. The mobility of the LAGBs at the recrystallized state were found to be twelve times higher in comparison to the recrystallized state.
- The KAM approach was focused on the actual misorientation relationships within the grain as well as the detection of the possible nucleation points by use of a simpler way of evaluation, ideal for industrial applications. Multiple samples can be effectively examined in a timely manner, thus various production stages could be effectively monitored, in terms of microstructure evolution.

Indicative bibliography

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Thank you for your attention