STUDY OF LDH GROWTH ON MAGNESIUM BIODEGRADABLE ALLOY: THE EFFECT OF MICROSTRUCTURAL CHANGES DUE TO ECAP PROCESSING

Ekaterina Pakhomova[1], Franco Bonollo[2], Alessandra Fava[3], Paolo Ferro[2], Roberto Montanari[4], Riccardo Narducci[4], Alessandra Palombi[4], Maria Richetta[4], Alessandra Varone[4]

[1] *Department of Mechanical, Chemical and Materials Engineering, University of Cagliari, Via Marengo 2, 09123 Cagliari, Italy*
[2] *Department of Management and Engineering, Padova University, Stradella San Nicola, 3, 36100, Vicenza, Italy*
[3] *ENEA, Department for Sustainability—Research Centre of Casaccia, Santa Maria di Galeria, 00123 e, Rome, RM, Italy*
[4] *Department of Industrial Engineering, University of Rome Tor Vergata, Via del Politecnico 1, 00133 Rome, Italy*

INTRODUCTION

Magnesium alloys are under intensive investigation because of their possible use in the biomedical field thanks to their good biocompatibility and mechanical properties which are similar to human bones and biodegradation. For instance, they have been used to produce a biodegradable bone fixator that does not require a second surgery. The main issue is the high corrosion rate in respect to tissue remodelling. The principal strategies to overcome this problem are: tailoring the alloy composition, inducing microstructural changes, employing surface treatments and coatings.

Several authors observed that the Layered Double Hydroxides (LDH) coatings improve the biocorrosion behaviour of the Mg-alloys and also permit drug delivery.

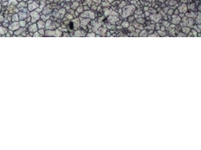
The aim of present work is to investigate how the microstructural changes induced by Equal Channel Angular Pressing (ECAP) affect the LDH films growth on the AZ31 surface.

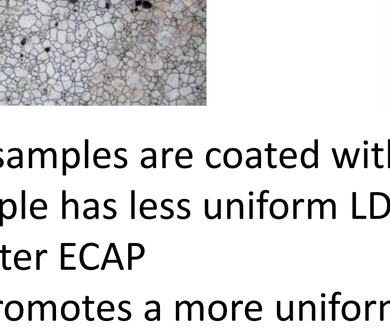
MATERIALS AND METHODS

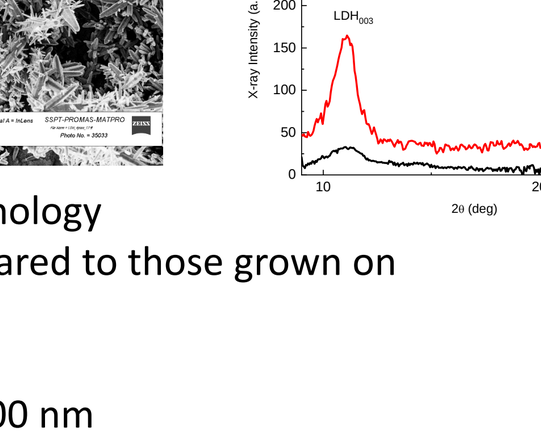
The commercial AZ31 alloy was processed by 0, 1, 2 and 4 ECAP passes.

LDH structures were grown on AZ31 samples using the co-precipitation technique. The nutrient solution was: $Zn(NO_3)_2*6H_2O$ (5 mM) and urea (15 mM) in 150 mL of distilled water. After preliminary cleaning the samples were immersed in the nutrient solutions, and kept there for 12 h at 90 C. Finally, they were cooled in the nutrient solution, extracted from the reactor, rinsed in distilled water and ethanol and air-dried.

The microstructural characterisation of the samples was performed by LM, XRD, SEM, EBSD technique.

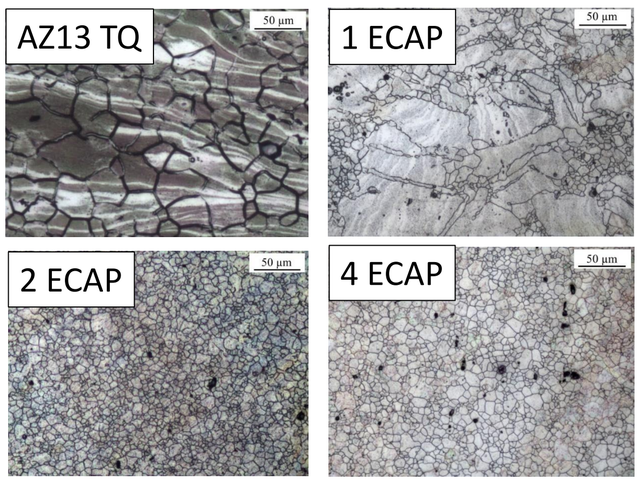




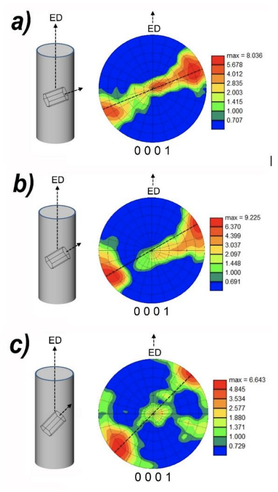


RESULTS

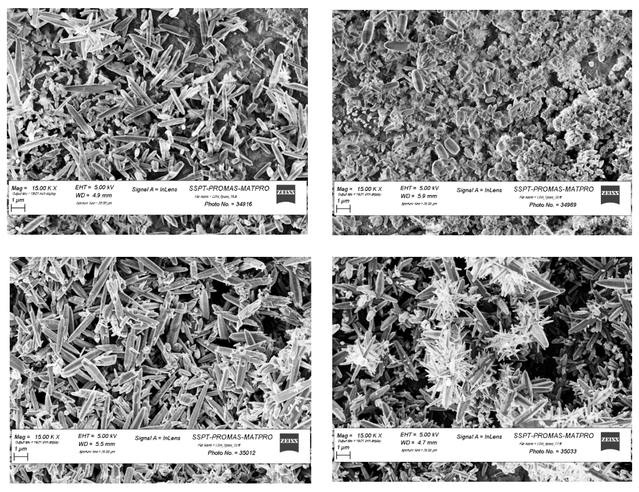
AZ31 MICROSTRUCTURE AFTER ECAP

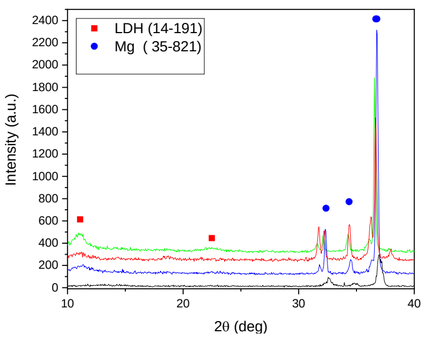


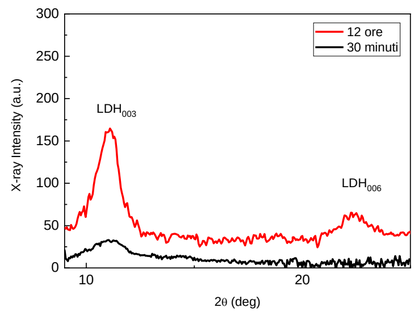
INVERSE POLE FIGURE (IPF) MAPS



12 H LDH GROWTH







- All the samples are coated with LDH crystals with an elongated and dendritic morphology
- TQ sample has less uniform LDH growth. The crystals have lager size (≥ 1 µm) compared to those grown on AZ31 after ECAP
- ECAP promotes a more uniform growth of LDH and with smaller crystals
- After 1 ECAP pass: extremely uniform distribution of LDH and small crystal size ~ 200 nm

CONCLUSIONS

The are 2 mechanism of LDH-film growth:

1. Growth by texture:

Highly packed planes have a higher activation energy for removing atoms from their surface. Basal planes parallel to the surface

- Decrease with ECAP passes

2. Growth by nucleation sites: geminated, dislocations, GB, etc.

- Increase with ECAP passes.

AZ31 TQ	ECAPed (1 run)	ECAPed (2 runs)	ECAPed (4 runs)
☒ Nucleation sites	☑ Nucleation sites	☑ Nucleation sites	☑ Nucleation sites
☑ Texture	☑ Texture	☒ Texture	☒ Texture
☒ Growth	☑ Growth	☒ Growth	☒ Growth

After 1 ECAP run both the nucleation sites and texture growth took place ➡ BEST RESULTS

Ekaterina Pakhomova, Ph.D.
Assistent Professor in Metallurgy
Department of Mechanical, Chemical and Materials Engineering,
University of Cagliari, 09123 Cagliari, via Marengo 2, Italy
ekaterina.pakhomova@unica.it