Positron annihilation spectroscopy study of gradient microstructure induced by Surface Mechanical Attrition Treatment (SMAT) in Mg

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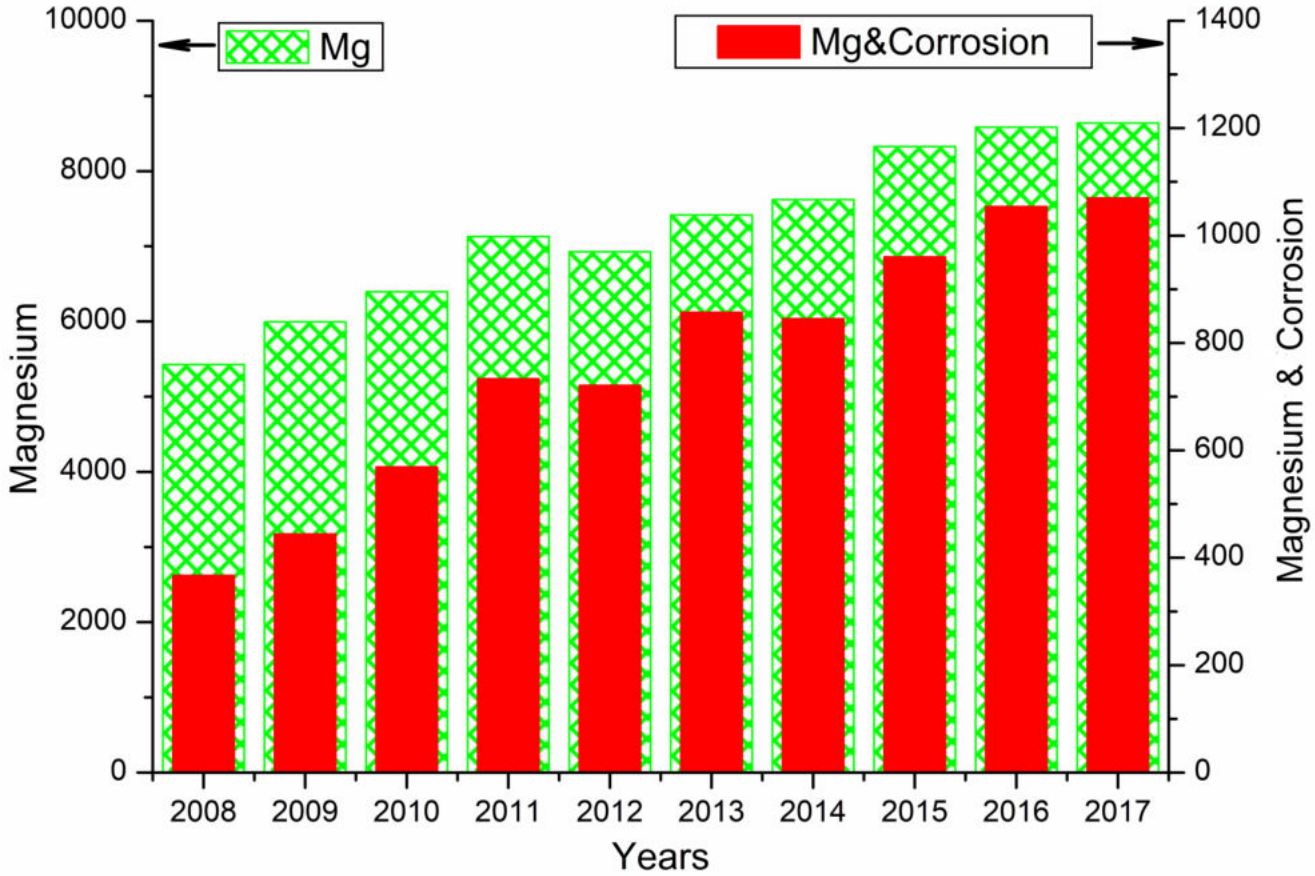
Motivation

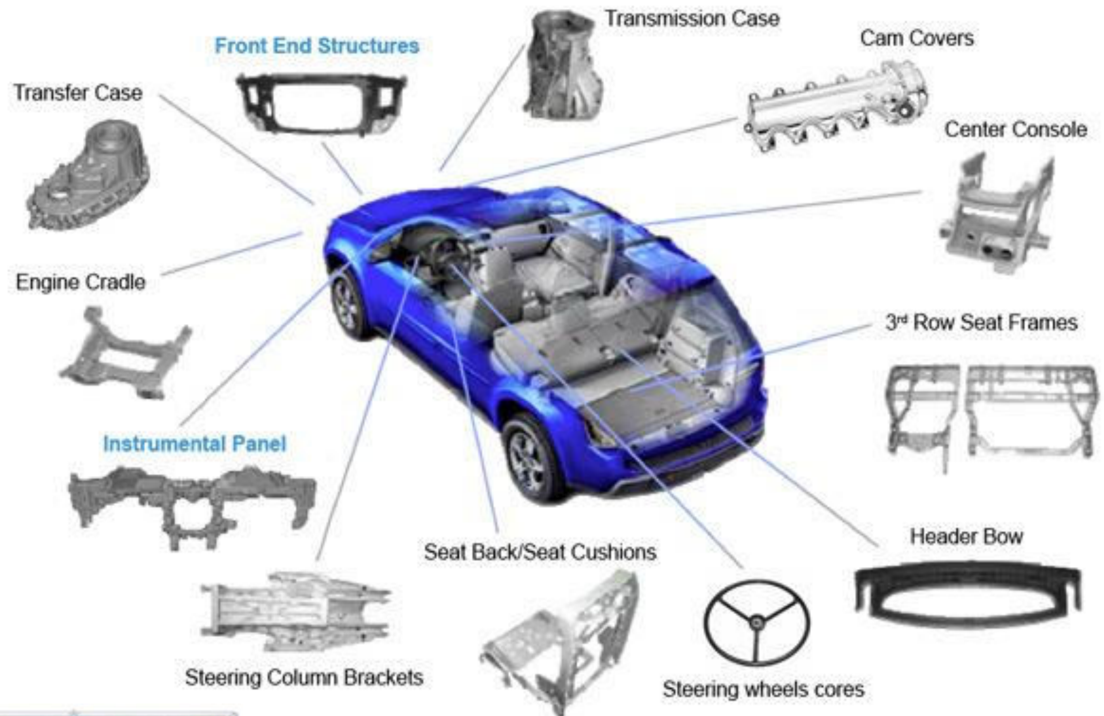


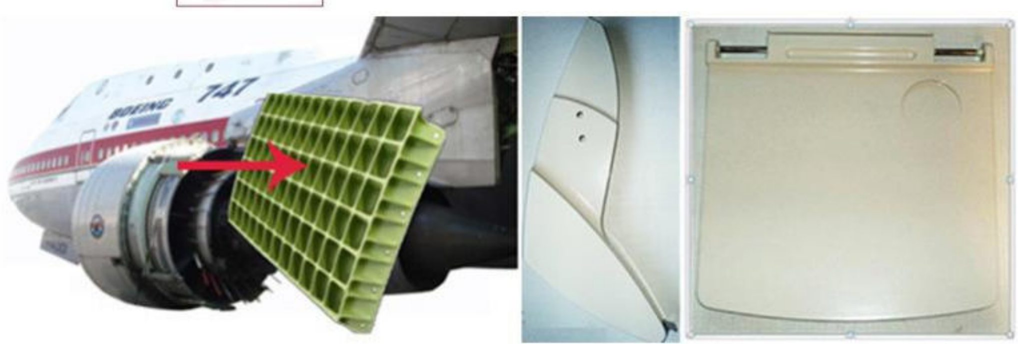
Number of scientific publications on magnesium and its alloys and the corrosion of magnesium and its alloys in 2008-2017 found in the SCOPUS database. Database searched for "magnesium" and "magnesium AND corrosion”.

Magnesium

Aerospace

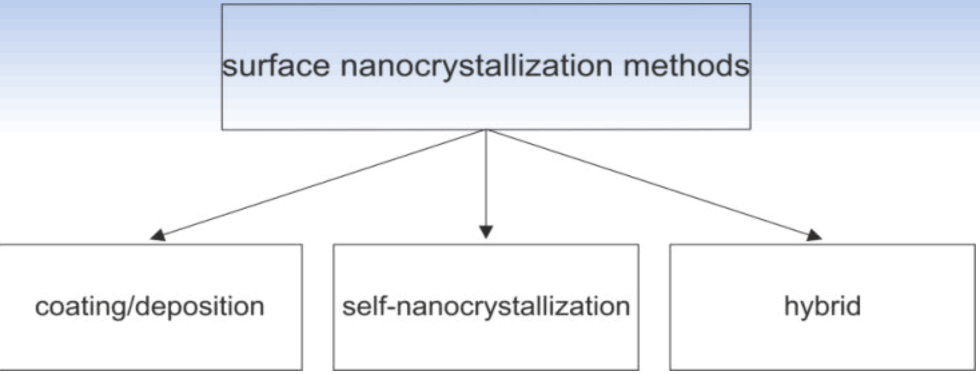
Atomobile

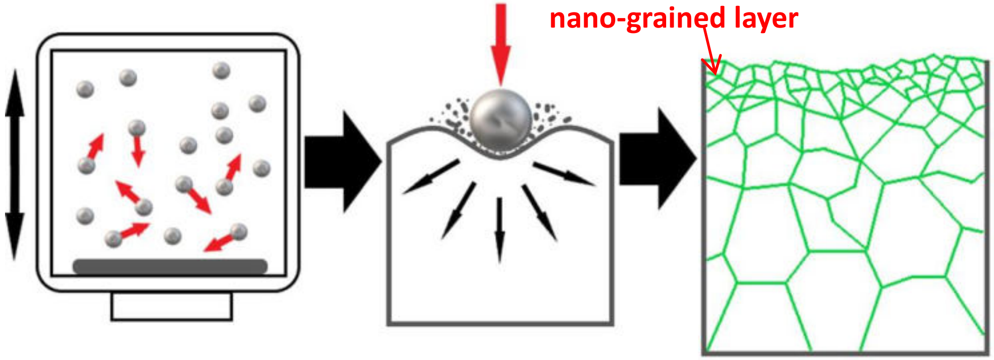




*A. Dziubińska, A. Gontarz, M. Dziubiński, M. Barszcz (2016). THE FORMING OF MAGNESIUM ALLOY FORGINGS FOR AIRCRAFT AND AUTOMOTIVE APPLICATIONS. Advances in Science and Technology Research Journal. 10. 158-168.

SMAT - Surface mechanical attrition treatment





Mg specimen after the SMAT.

Surface mechanical attrition treatment

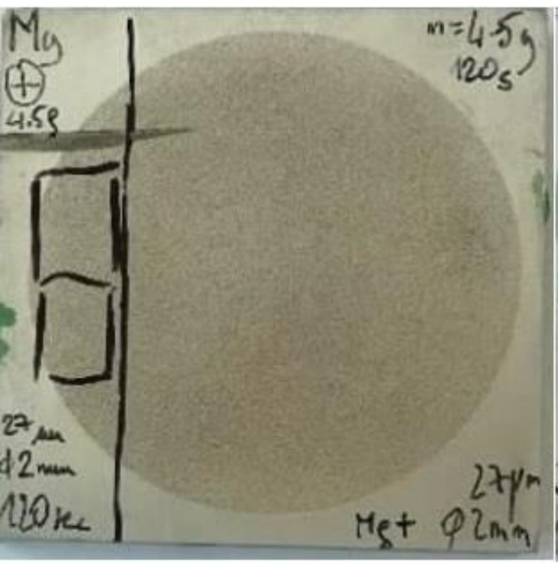
- **elimination of tensile stresses**
- **introduction of compressive stresses**
- **smoother surface finnish compared to SP**

generation of a nanocrystalline layer on the metal surface while maintaining its original chemical composition

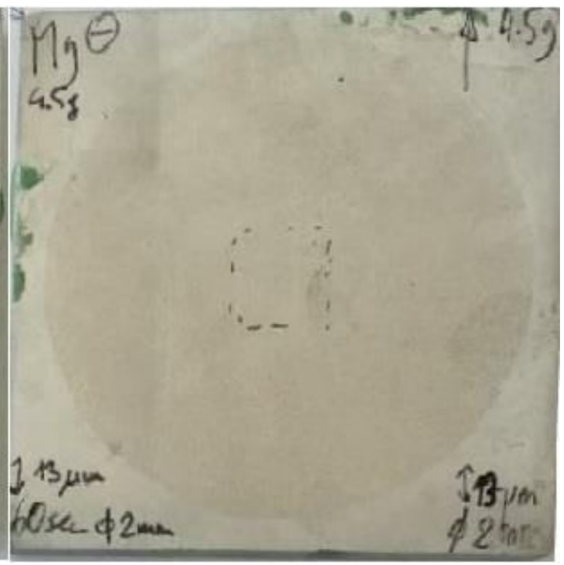
- **corrosion resistance**
- **hardness**
- **fatigue life**
- **abrasion resistance**

Sample preparation

- Commercial purity magnesium – 99.5%.
- Prior to SMAT treatment - heated at 400 °C, 30 min in vacuum.
- After the SMAT treatment, the samples were cut into pieces approximately 1x1 cm^2 in size.



Mg SMAT 120s

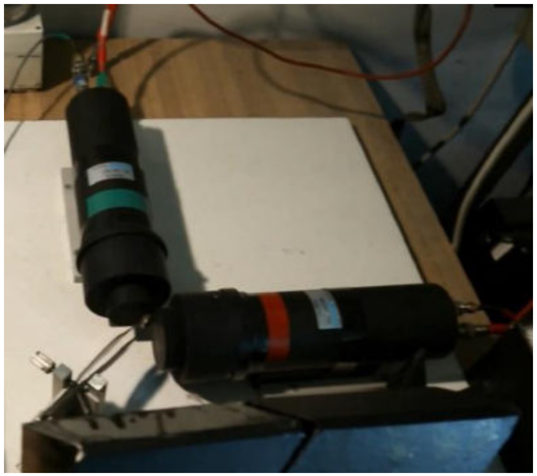


Mg SMAT 60s

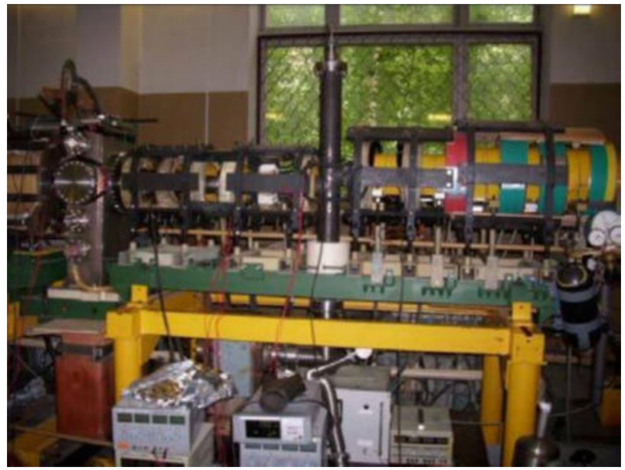
SMAT parameters:
- time 120/60 seconds,
- amplitudes 27 ± 2 µm and 13 ± 2 µm,
- frequency 20 kHz,
- 4.5 g ball weight, AISI 304L steel.

Methods

Positron annihilation spectroscopy



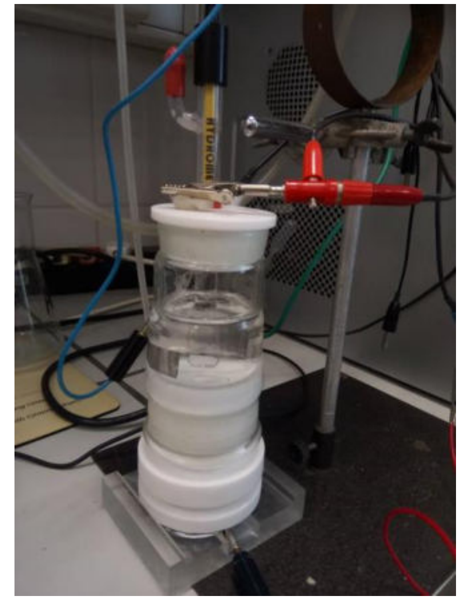
Positron lifetime spectrometer



Variable positron beam
(photography: Paweł Horodek)

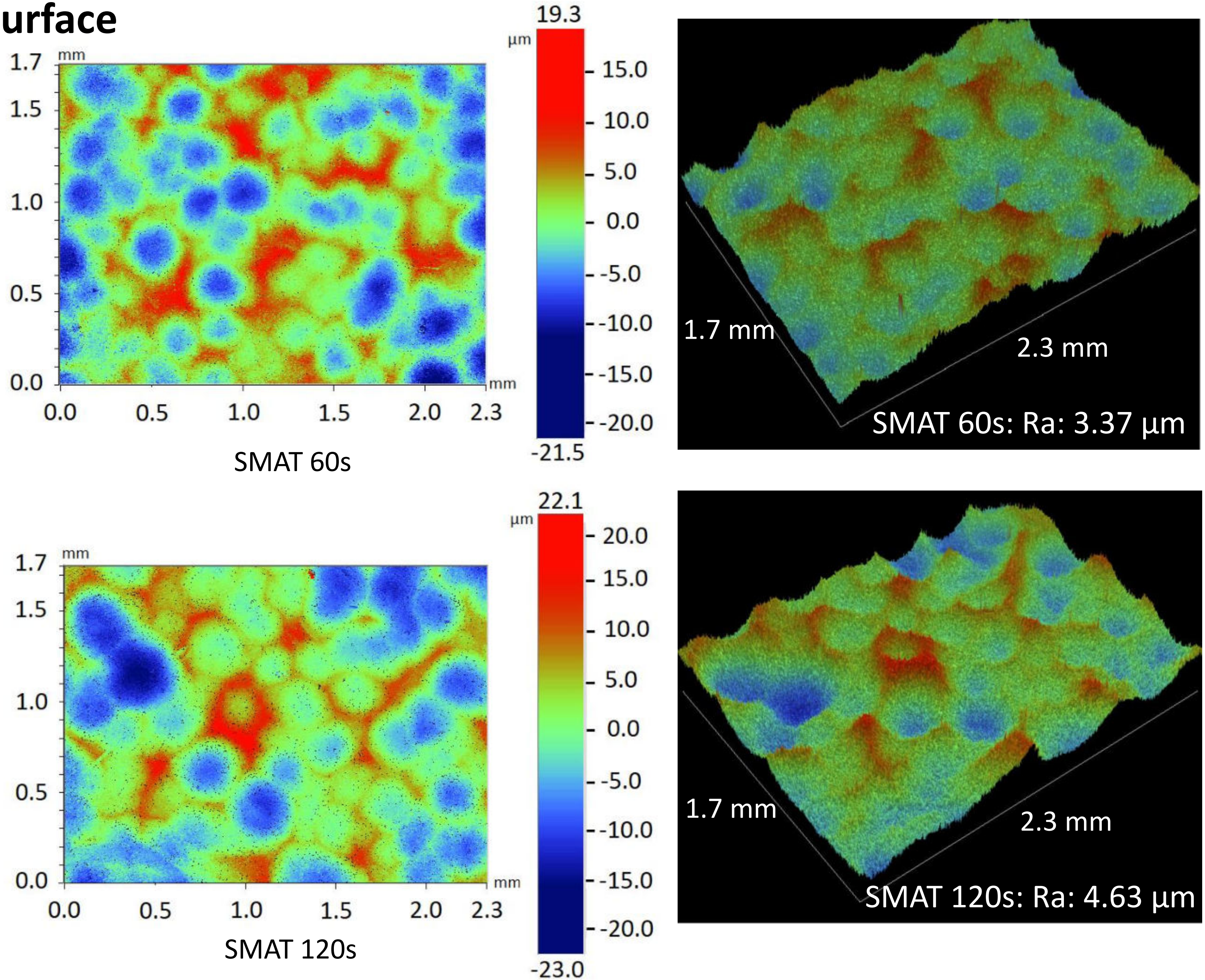
- ❑ XRD– crystallite size
- ❑ microhardness tests – mechanical properties
- ❑ EBSD – microstructure characterizatiion
- ❑ optical profilometer – surface characterization

Corrosion tests



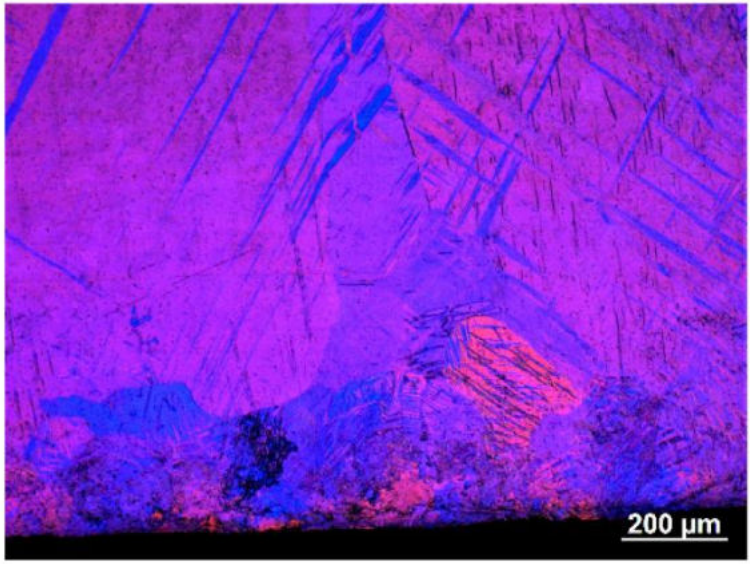
Electrochemical impedance spectroscopy

Surface

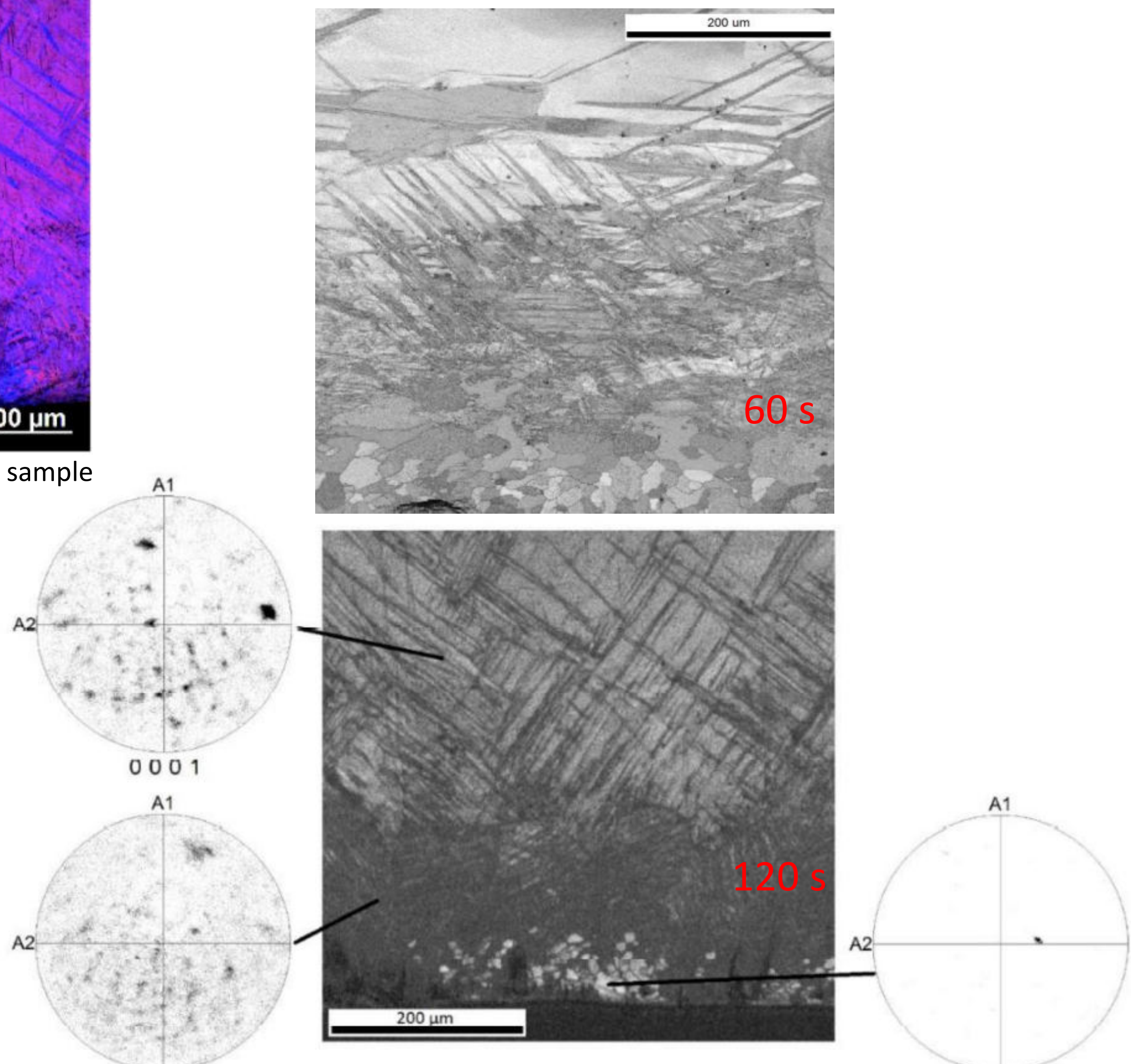


mm
1.7
1.5
1.0
0.5
0.0
0.0
0.5
1.0
1.5
2.0
2.3
mm
SMAT 60s
19.3
µm
15.0
10.0
5.0
0.0
-5.0
-10.0
-15.0
-20.0
-21.5
1.7 mm
2.3 mm
SMAT 60s: Ra: 3.37 µm
mm
1.7
1.5
1.0
0.5
0.0
0.0
0.5
1.0
1.5
2.0
2.3
mm
SMAT 120s
22.1
µm
20.0
15.0
10.0
5.0
0.0
-5.0
-10.0
-15.0
-20.0
-23.0
1.7 mm
2.3 mm
SMAT 120s: Ra: 4.63 µm

Microstructure



Optical microscopy image of the cross-section of the sample SMATed for 120 s.

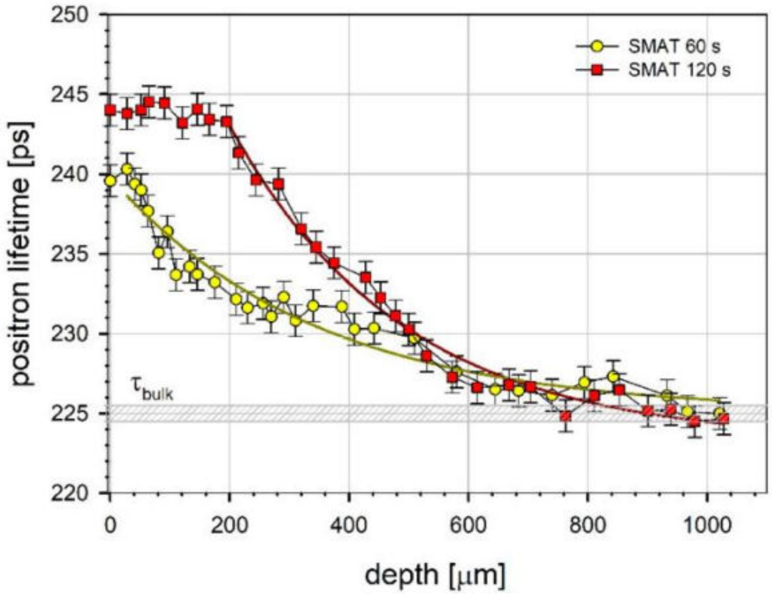


EBSD images of the cross-sectioned Mg specimens

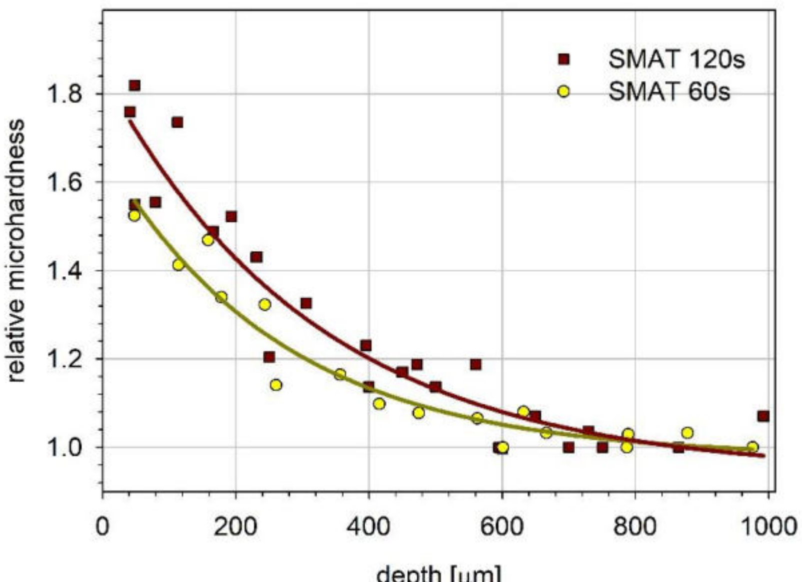
Calculated values of lattice crystallite size for magnesium samples.

Sample	Crystallite Size [nm]
Reference	205 ± 63
SMAT 60 s	38 ± 3
SMAT 120 s	37 ± 3

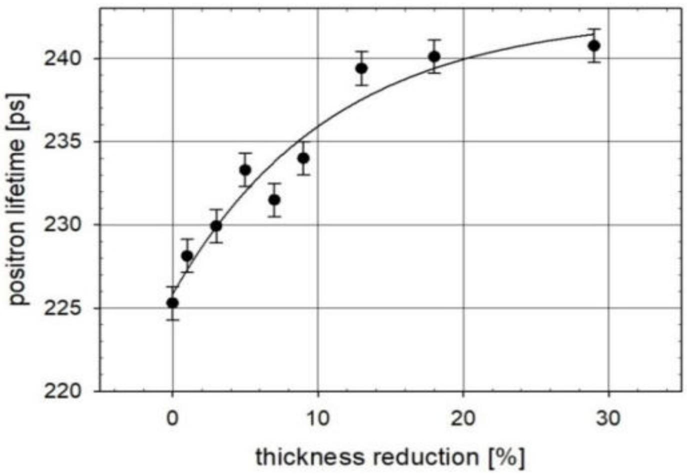
Positron lifetime measurements



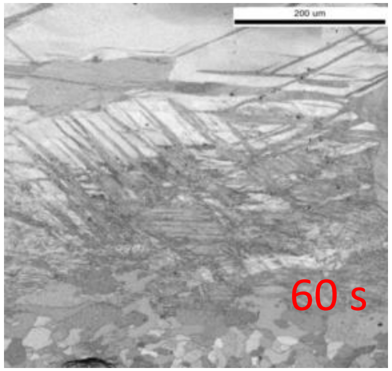
The mean positron lifetime profiles.

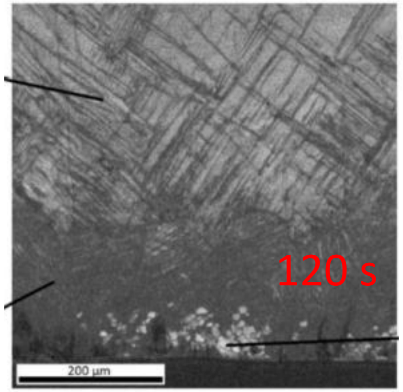


The relative microhardness of magnesium samples vs depth.

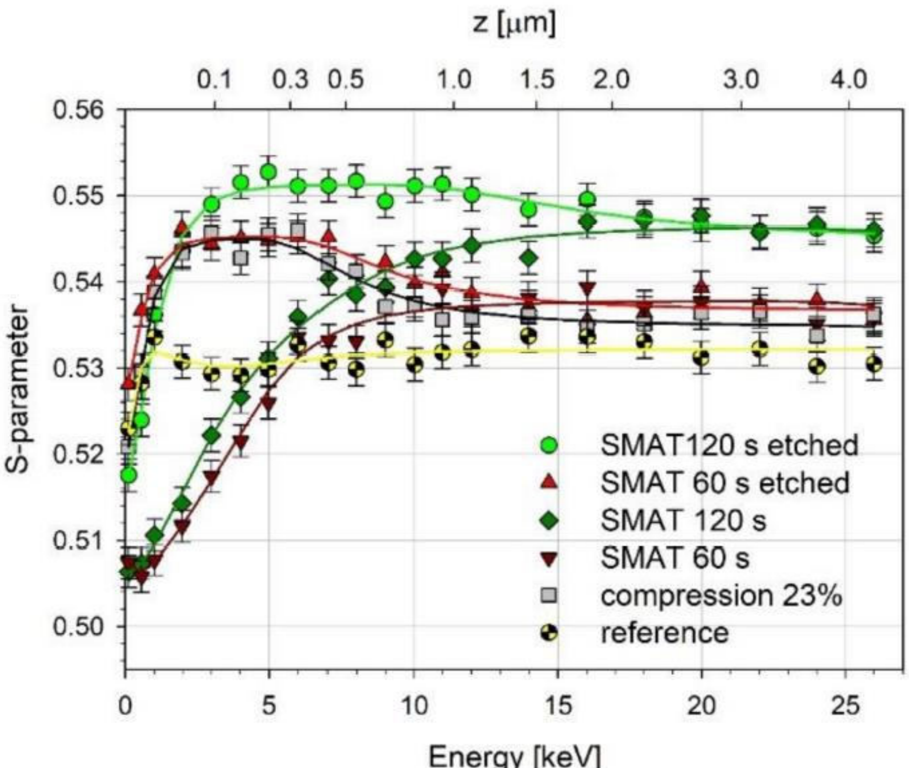


Mean positron lifetime values as a function of thickness reduction for the Mg specimen deformed using the uniaxial-hydraulic press.

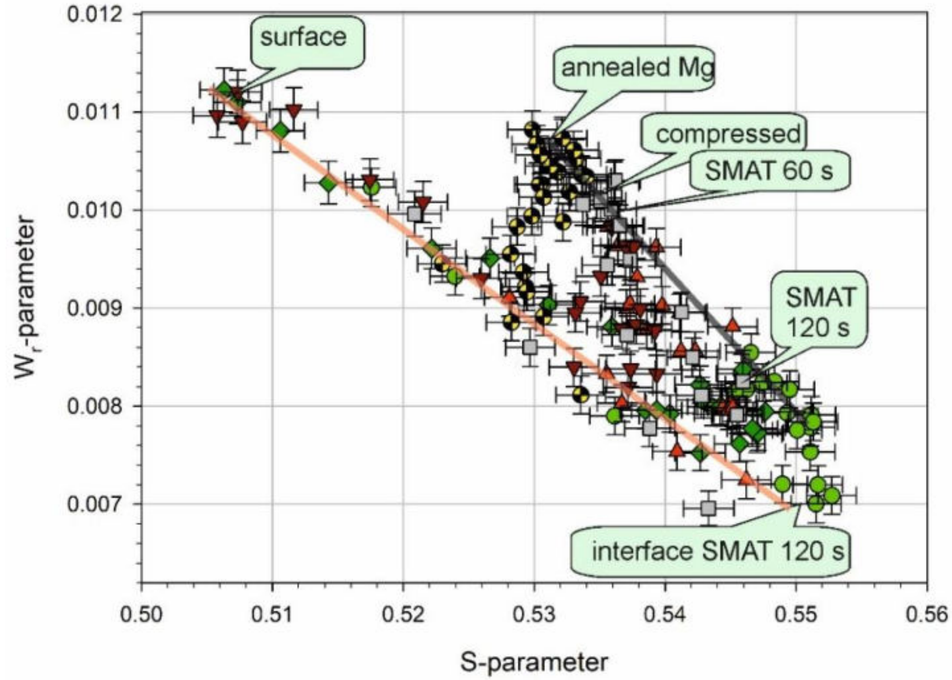




Variable energy beam measurements



The S-parameter as a function of incident positron energy (mean implantation depth)

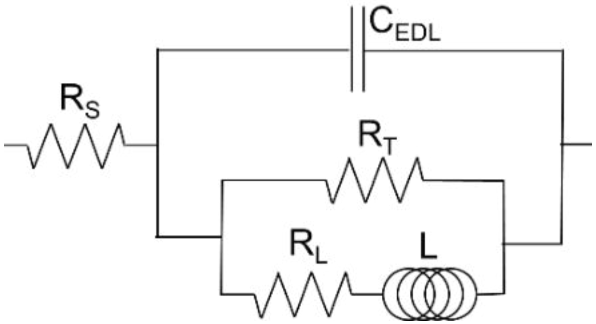


W_r-parameter as a function of S-parameter.

Results of fitting of the S(E) curves. L_{+layer} - positron diffusion length in the surface layer of the samples, *d* - thickness of this surface.

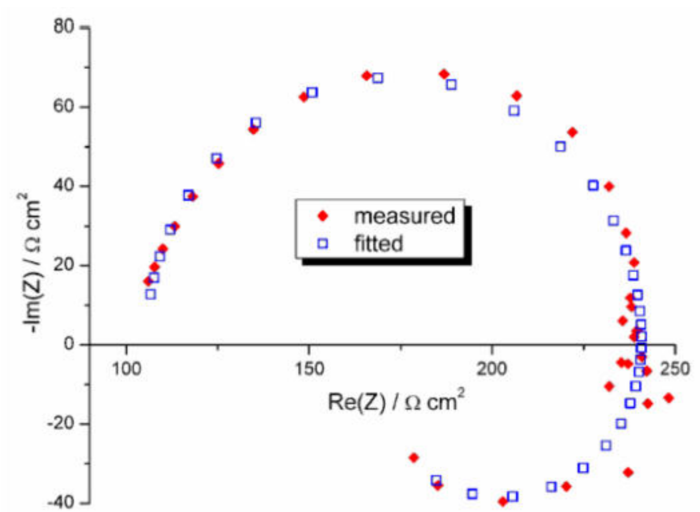
Sample	L_{+layer} [nm]	d [nm]
SMAT 60 s	74 ± 4	148 ± 9
SMAT 120 s	74 ± 2	370 ± 32
reference	21 ± 10	80 ± 43
SMAT 60 s & etched	6 ±2	320 ± 2
SMAT 120 s & etched	12 ± 1	708 ± 78
Compressed & Etched	6 ± 1	266 ± 18

Corrosion resistance



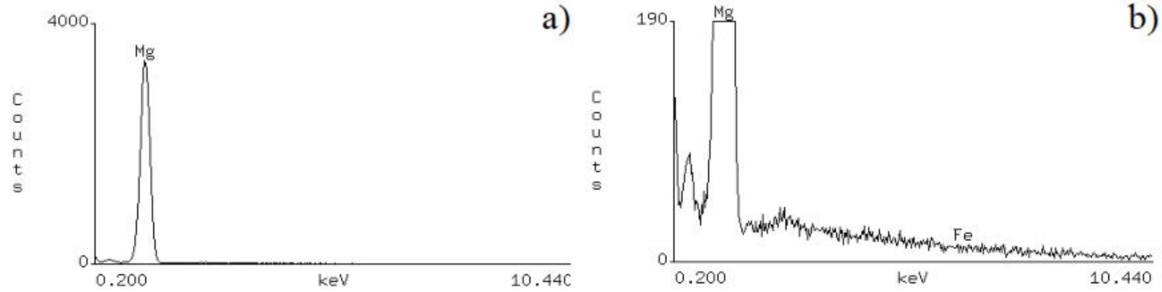
Equivalent electrical circuit applied in the interpretation of the data from electrochemical impedance spectroscopy (EIS) measurements.

$$\frac{1}{R_\mathrm{p}} = \frac{1}{R_\mathrm{T}} + \frac{1}{R_\mathrm{L}}$$

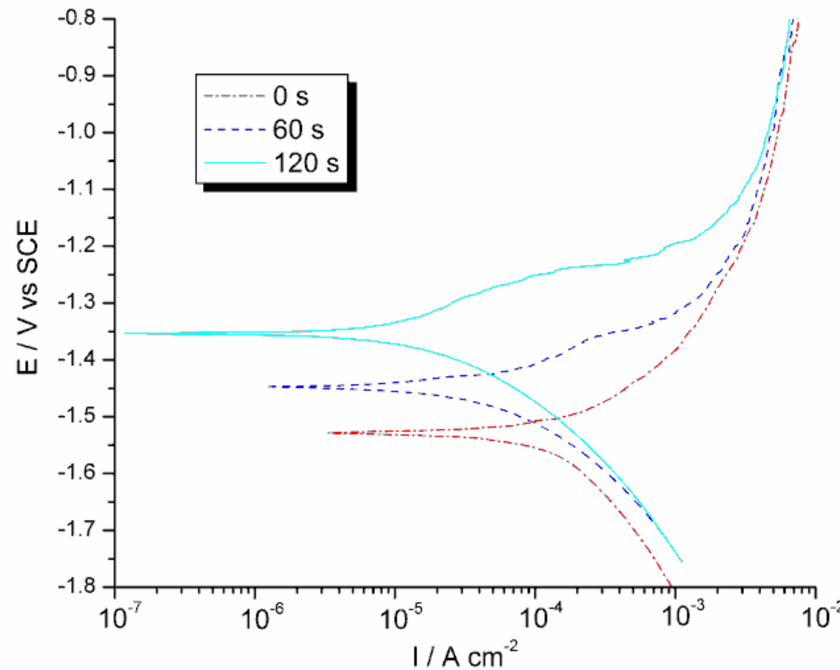


An example of the to the EIS data

$$R_{p\,\mathrm{EIS}}^{\;0} > R_{p\,\mathrm{EIS}}^{\;60} > R_{p\,\mathrm{EIS}}^{\;120}$$



The EDS spectrum for the a) sample SMATed for 120s, b) region of interest.



Linear sweep voltammetry on the Mg samples

$$E_\mathrm{corr}^0 < E_\mathrm{corr}^{60} < E_\mathrm{corr}^{120}$$

$$R_{\mathrm{p}\,\mathrm{corr}}^{\;0} < R_{\mathrm{p}\,\mathrm{corr}}^{\;60} < R_{\mathrm{p}\,\mathrm{corr}}^{\;120}$$

$$j_\mathrm{corr}^0 > j_\mathrm{corr}^{60} > j_\mathrm{corr}^{120}$$

Summary

- SMAT produced a gradient microstructure of the surface layer.

- Significant grain refinement was observed close to the treated surface. Deformation twins were created, and their density decreased with depth from the surface.

- All positrons emitted from the source, which penetrates 200 µm layer near the surface, annihilate in structure defects, which are vacancies associated to dislocations.

- The structure changes increased the susceptibility of magnesium to anodic oxidation, intensifying the formation of a hydroxide layer on the surface and, consequently, leading to better corrosion resistance.

- This is confirmed by the VEP results showing a thicker oxide layer on the surface of the sample treated with SMAT for 120 s.

- The results show that not only grain boundaries present at the surface but also other crystal defects such as dislocations and vacancies can also play a significant role in the corrosive behavior of magnesium.

THANK YOU