

# High-Resolution DIC Combined with SEM Mechanical Analysis of *In Situ* Strain and Crack Propagation in Coated AZ31 Mg Alloys for Bone Fracture Fixation

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## Summary

Biodegradable magnesium alloys are promising for biomedical applications, if their corrosion is controlled by coatings, even under load-bearing conditions. This study examines electrochemically deposited coatings on AZ31 magnesium alloy under 3-point bending (3PB) using high-resolution digital image correlation (HR-DIC) and scanning electron microscopy (SEM). *In situ* 3PB tests showed 5  $\mu\text{m}$  coatings formed more cracks, while 15  $\mu\text{m}$  coatings had fewer but larger cracks. HR-DIC identified shear strain as key in crack initiation and propagation. Despite cracking, these coatings effectively reduced corrosion.

## Introduction

Modifying the surface of Mg alloys using coatings is an efficient method for managing their biodegradation and enhancing their biological characteristics [1]. Mg alloys use in orthopedic implants, experience static and dynamic loads during their insertion into irregular bones and after implantation during walking and running [2]. These diverse loads could initiate coating cracks, followed by an accelerated corrosion and ultimately material failure. In this study, the impact of loading on crack initiation and propagation in a phosphate and fluoride enriched ceramic coating was investigated under three-point bending test.

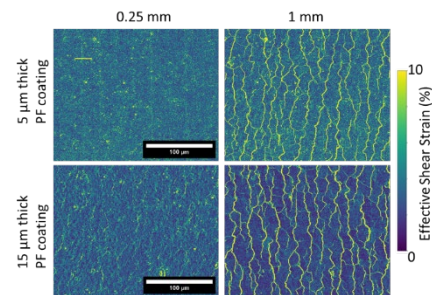
## Methods

AZ31 alloy underwent electrochemical oxidation (soft sparking ECO patented by Biocera Medical Ltd (PCT publication WO 2020049299)) to generate 5  $\mu\text{m}$  and 15  $\mu\text{m}$  thick ceramic coatings. Coated rods after 1 week in Hank's Balanced Salt Solution (HBSS), were loaded in a custom-made *in situ* three-point bending test rig from 0 to 1 mm displacement. The displacement was held constant in each step to capture secondary electron micrographs. Then, DIC analysis determined the surface cracking and strain response to these loads with a resolution of 100 nm.

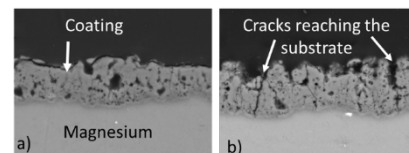
## Results and Discussion

*In situ* 3PB tests showed the initiation of cracks in the coating transversally to the longitudinal axis of the rod (Fig. 1) generally following pores. An increase in coating thickness led to the formation of wider cracks and with greater deformation more transverse cracks formed. HR-DIC

indicated that crack initiation was associated with high shear strain. There was little difference in strain and cracks on the surface of the coating after corrosion of the samples in HBSS. Cross sections of the samples show no detachment of the coating from the substrate, and by removing the load, the cracks tended to close, however under plastic deformation cracks extended to the alloys surface (Fig. 2).



**Figure 1:** HR-DIC effective shear strain maps.



**Figure 2:** Cross sections of (a) elastically and (b) plastically loaded 15  $\mu\text{m}$  coated Mg samples

## Conclusions

Coating on AZ31 Mg alloy makes it suitable for clinical applications due to a decreasing degradation rate leading to improved mechanical performance under corrosive environment. This study shows that, even beyond yield and the formation of cracks, the coating remains attached to the alloy. However, these cracks penetrate the coating and may form a route for the ingress of fluid leading to a more corrosive environment on the alloy surface highlighting the need for further research to optimize coating properties for long-term durability.

## Acknowledgments

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## References

- [1] Koo Y et al. (2017). *Mater. Sci. Eng B*, **219**: 45-54
- [2] Denkana B et al. (2013). *Procedia CIRP*, **5** : 189-195