(ILM Joint Usage/Research)

# Institute of Light Metals (ILM) Joint Usage/Research Grant Report in FY 2025

2026/05/15

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| Principal investigator | Affiliation | Costa Rica Institute of Technology Center for Materials Research and Extension |
| | Job title | Associate Professor |
| | Name | Jorge M. Cubero-Sesin |
| Collaborated researcher of ILM | Affiliation | Magnesium Research Center |
| | Job title | Professor |
| | Name | Zenji Horita |
| Title of the joint research | Nanostructured Mg-6Al-3Ca-0.02Mn-0.008Be and Mg-1Ca-0.5Zn alloys processed by High Pressure Torsion and High Pressure Sliding to fabricate orthopedic prostheses. | |
| Joint research Program<br>※check the box | ☐ Program for Joint Usage / Research Centers (JURC)<br>※ Program for International JURC<br>※ Program for providing samples and materials<br>※ Program for using ILM facilities for sample analysis and characterization | ☐ Focused themes<br>☐ Transportation<br>※ Biomaterials<br>☐ Bridge/building materials<br>☐ Kink strengthening<br>☐ Independent research theme |
| Name of joint usage apparatus | • Vickers Microhardness.<br>• Electrochemical Polishing (ECO-POL).<br>• Scanning Electron Microscope (SEM).<br>• Transmission Electron Microscope (TEM).<br>• Electron Backscatter Diffraction (EBSD). | |
| Total amount of grant | Travel expense ( 300,000 JPY) | Consumable Fee ( JPY) |

**Research Results** ※Please describe following three items briefly.

【The major results】

This research investigates the effect of severe plastic deformation (SPD) by equal-channel angular pressing (ECAP) and high-pressure torsion (HPT) on the microstructural and mechanical properties of two magnesium alloys: Mg-6Al-3Ca-0.02Mn-0.008Be and Mg-1Ca. The alloys were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), and Vickers microhardness measurements to evaluate the influence of processing on phase constitution, grain refinement, and strengthening mechanisms.

The Mg-6Al-3Ca-0.02Mn-0.008Be alloy exhibits an α-Mg matrix strengthened by Ca- and Al-rich intermetallic phases, mainly $Mg_2Ca$, $Al_2Ca$, and $(Mg,Al)_2Ca$. XRD analysis revealed that these phases remain stable after SPD processing, indicating that neither HPT nor ECAP processing induces phase transformations. However, HPT produced significant peak broadening, reflecting severe grain refinement, increased dislocation density, and higher lattice strain. In contrast, ECAP preserved sharper diffraction peaks due to dynamic recovery at elevated temperatures, although lower processing temperatures promoted additional refinement.

SEM observations confirmed that the as-cast alloy consists of eutectic intermetallic networks distributed along grain boundaries. After HPT processing, these networks fragmented into submicrometric particles that became homogeneously dispersed within the α-Mg matrix. A progressive refinement from the center toward the edge of the disk was observed, leading to an ultrafine and homogeneous microstructure.

The microhardness results highlight the different strengthening capabilities of the SPD routes. The as-cast alloy exhibited an average hardness of 73.25 ± 4.09 HV, while the first and second ECAP routes showed similar values of 72.94 ± 1.15 HV and 70.98 ± 1.09 HV, respectively, due to the competition between strain hardening and dynamic recovery. Processing through the third ECAP route increased the hardness to 76.22 ± 0.47 HV. In contrast, HPT processing produced a substantial increase in hardness, reaching 105.69 ± 4.13 HV, demonstrating its superior ability to generate ultrafine grains, high dislocation densities, and a refined distribution of intermetallic particles.

The Mg-1Ca alloy represents a simpler Mg-Ca system, in which the limited solubility of calcium promotes the formation of $Mg_2Ca$ precipitates. XRD analysis identified α-Mg and $Mg_2Ca$ in both the extruded and HPT-processed conditions. After HPT, the α-Mg peaks broadened significantly and decreased in intensity, indicating severe grain refinement and the accumulation of microstrain. A slight reduction in the intensity of the $Mg_2Ca$ reflections suggests partial dissolution and redistribution of calcium into the α-Mg matrix.

EBSD analysis demonstrated that both HPT and ECAP processing refine the grain structure and weaken the strong basal texture characteristic of the extruded alloy. HPT produced the most pronounced effect, generating ultrafine grains with a narrow size distribution and promoting the conversion of low-angle boundaries into high-angle grain boundaries. Furthermore, HPT significantly reduced the basal texture, indicating grain rotation and activation of non-basal slip systems. Although ECAP processing also refined the microstructure, the resulting grain-size distribution remained broader, particularly at higher processing temperatures, where dynamic recovery limited fragmentation.

The microhardness behavior of Mg-1Ca followed a similar trend. In the as-extruded condition, hardness values ranged from 55 to 60 HV. HPT processing produced the largest strengthening effect, increasing hardness progressively toward the disk edge and reaching approximately 80-85 HV. In contrast, ECAP generated more homogeneous hardness profiles but lower values than those obtained after HPT, confirming that the accumulated strain and grain fragmentation are less pronounced.

Overall, both alloys benefit from severe plastic deformation, which promotes grain refinement, modifies crystallographic texture, and enhances mechanical properties. Among the investigated routes, HPT proved to be the most effective technique, producing the highest degree of microstructural refinement and the greatest improvement in hardness.

【Future Prospects】

Future work will focus on the electrochemical characterization of both alloys through electrochemical impedance spectroscopy, mass-loss measurements, and hydrogen evolution tests in environments representative of their intended applications. For the Mg-1Ca alloy, corrosion studies will be performed in simulated body fluids, such as phosphate-buffered saline (PBS) and Hanks' solution, to evaluate its degradation behavior under physiological conditions. In contrast, the Mg-6Al-3Ca-0.02Mn-0.008Be alloy will be investigated in environments that reproduce its potential service conditions, particularly considering its enhanced high-temperature performance and creep resistance. These studies will provide a deeper understanding of the relationship between microstructure, degradation mechanisms, and application-specific performance.

【Concrete results】

International conference presentation : Oral presentation at the 34th International Materials Research Congress (IMRC 2026), Cancún, Mexico. Title: "Bioabsorbable Mg-1Ca alloy processed by severe plastic deformation: microstructural, mechanical and electrochemical behavior".

Notes

・Please use the form and submit to the URL provided in the email by Friday, May 15, 2026.
・The joint research report will be published in the ILM joint research report (annual report) and will be available on our website. Therefore, please prepare the contents for public release accordingly.
・Please add pages, if needed.