Experimentally quantifying critical stresses associated with basal and prismatic slips in Zn and Zn-Ag alloys using *in situ* micropillars compression

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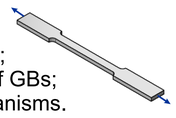
MOTIVATION & OBJECTIVES

* **Zinc** (Zn) has been considered a novel promising material for bioresorbable medical implants.
* **Poor strength and brittleness of** as-cast **pure Zn require alloying with other elements** and **plastic deformation** for grain size refinement to enhance mechanical properties (Fig. 1).

* Mechanical properties testing:

→ in the macroscale

The strengthening effect is affected by:
- grain size;
- texture;
- phase composition;
- fraction and type of GBs;
- deformation mechanisms.



→ in the microscale

Individual strengthening effect can be distinguished:
- size effect;
- solid solution strenghtening.

Besides, one deformation mechanism can be activated within a single grain, so critical resolved shear stresses in a specific slip system can be measured.

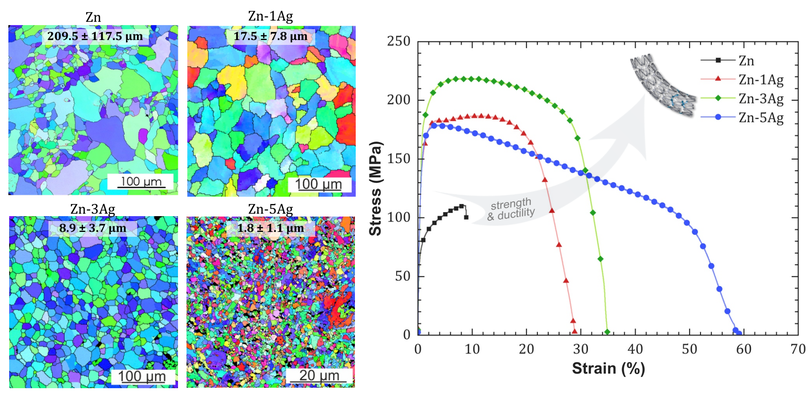


Fig. 1. Effect of Ag additions and hot extrusion on the microstructure and mechanical properties of Zn alloys

MICROPILLARS FABRICATION

* The samples of the **Zn-xAg alloys (x = 0 ÷ 2.21 at.%)** were fabricated by casting and annealing.
* Grains with the highest Schmid Factor for **basal and prismatic slip system** were selected for the micropillars fabrication.

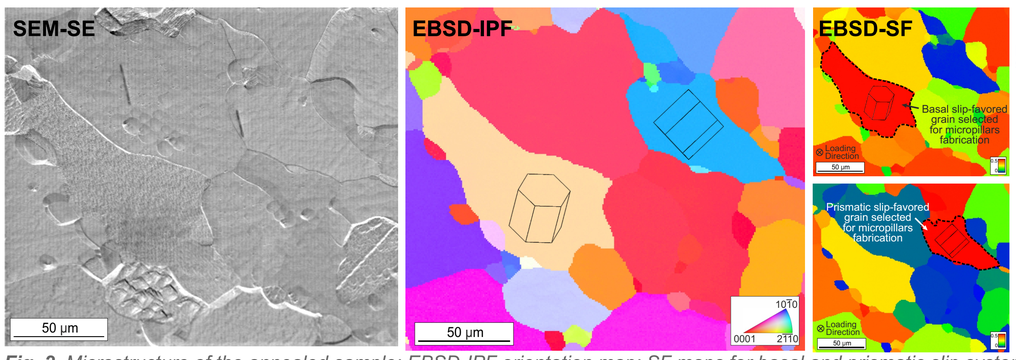


Fig. 2. Microstructure of the annealed sample; EBSD-IPF orientation map; SF maps for basal and prismatic slip system; angles considered in the calculation of the Schmid factor.

* Micropillars were fabricated by multi-step Ga^+ focused ion beam (FIB) milling operated at 30kV and beam currents from 4.5 nA to 40 pA.
* A micropillar **diameter** between **3 µm and 9 µm** had been chosen based on the studies [1-2]: **transition from twinning to dislocation slip** dominant deformation mechanism occurred within this grain size range.

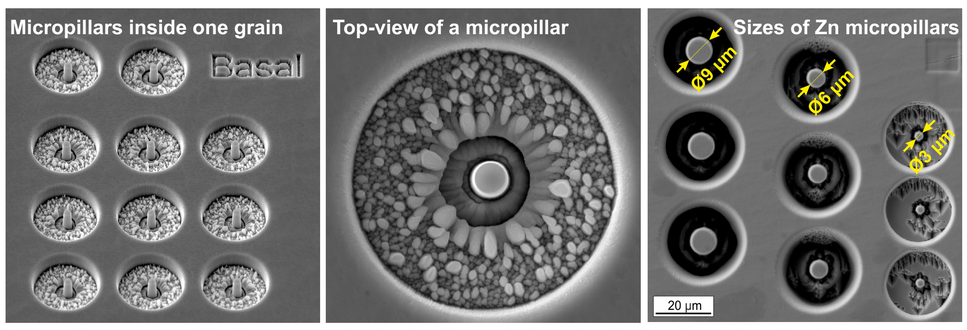
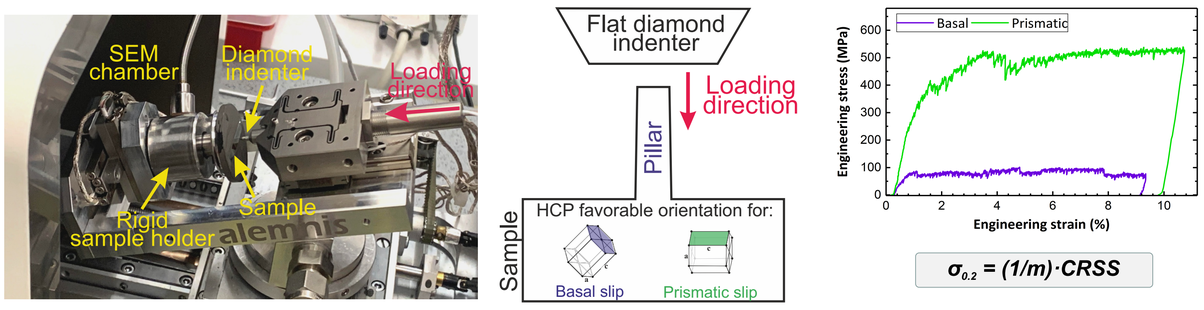


Fig. 3. Micropillars prepared for the compression tests withing single grain with basal- or prismatic- slip favoured orientation

IN SITU MICROPILLARS COMPRESSION

* Micropillars compression tests were perfomed using flat punch installed in Alemnis Standard Assembly ***in situ* nanoindentation system** (Fig. 4) up to ~10% of deformation
* Different-sized **pure Zn** micropillars **were deformed at 10^{-3} s^{-1}** to investigate **the *size effect* on CRSS**
* **Zn-xAg** micropillars (ø3 µm) **were deformed at 10^{-4}, 10^{-3}, 10^{-2} s^{-1} to measure the effect of Ag on CRSS**
* **According to the Schmid Law**, **CRSS** can be caluclated based on the yield stress ($\sigma_{0.2}$) determined from the stress-strain curves and Schmid Factor (***m***) calculated for a single grain and particular slip system



$\sigma_{0.2} = (1/m) \cdot CRSS$

Fig. 4. Setup for the in situ micropillars compression and the example of stress-strain curves

FUTURE PERSPECTIVES

- Current studies provide fundamental knowledge about possibilities of Zn solid solution strengthening.
- Calculated CRSS can be further implemented in crystal plasticity models, as input data, for designing fabrication processes of bioresobable implants requiring specific mechanical properties.
- Investigated here Zn-Ag system can be used as an antibacterial thin coating on the biomedical devices, such as orthopedic implants.
- The knowledge gained in this research will be translated into the design of novel Zn-based porous metamaterials with tailored microstructure and tunable properties for potential bioresobable maxillofacial implant applications.

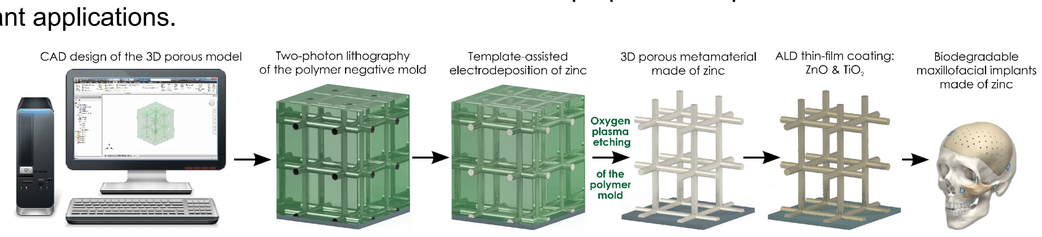


Fig. 5. Process diagram for fabrication of coated Zn-based metamaterials

SIZE EFFECT IN PURE Zn

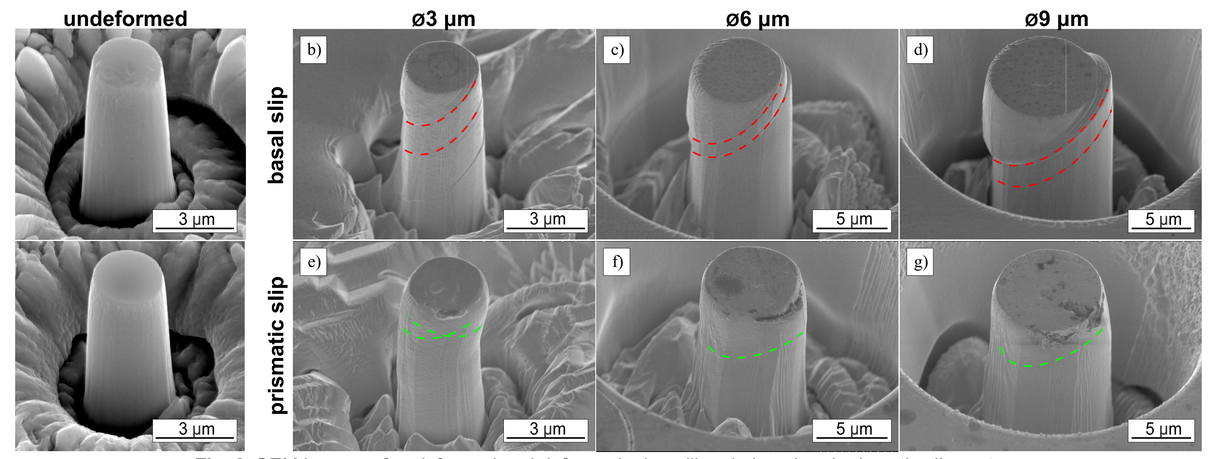
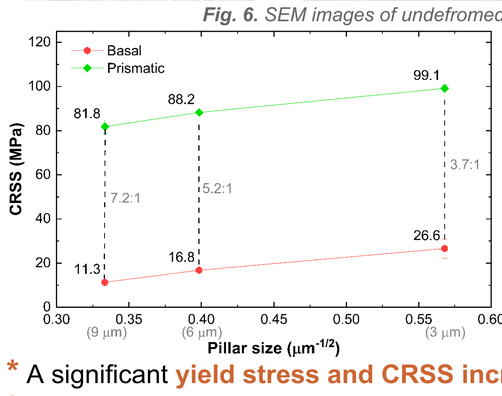


Fig. 6. SEM images of undefromed and deformed micropillars in basal and prismatic slip system;



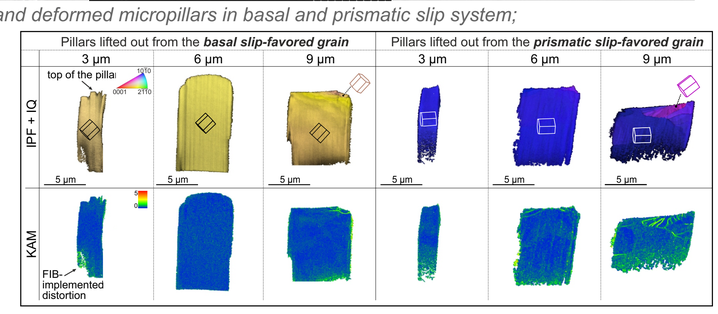


Fig. 7. The effect of Zn pillar diameter on CRSS in basal and prismatic slip systems. EBSD analysis in cross-section of deformed micropillars.

* A significant **yield stress and CRSS increase** with the micropillar **diameter reduction** was observed.
* The deformation in the **basal slip** system produced **slip traces, distinctly separated in 3 µm** pillars and **localized for 9 µm** pillars.
* **Prismatic slip traces on two slip planes** were observed in **3 µm** pillars and **only one** can be seen **in 6 µm** and **9 µm** pillars.
* A **uniform orientation within 3 µm pillars deformed** up to 10% was observed, while **in bigger pillars a localized deformation** occurred on the top of the pillar, resulting in a lattice rotation.

SOLID SOLUTION EFFECT IN Zn - xAg ALLOYS

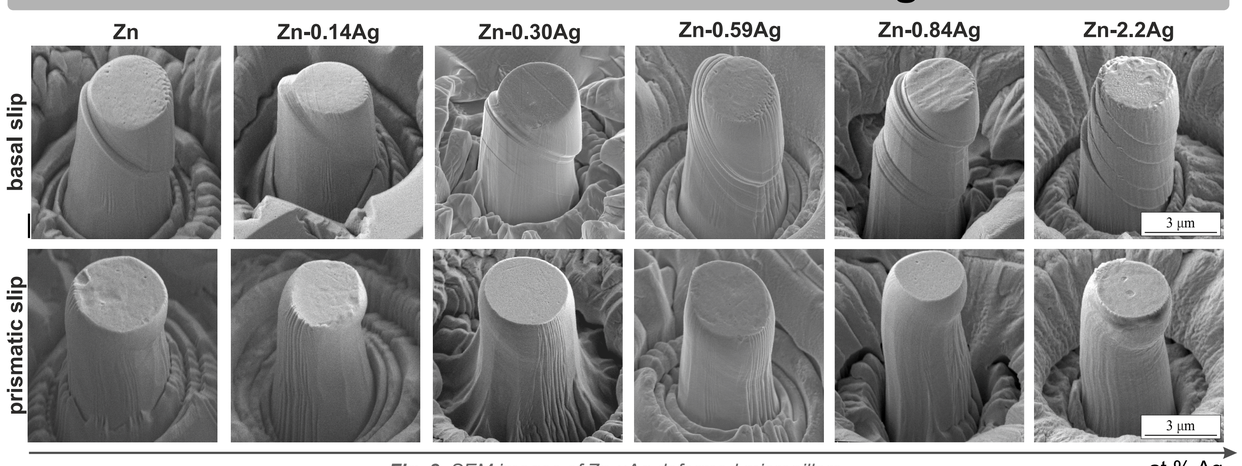


Fig. 8. SEM images of Zn-xAg deformed micropillars

* Ag content is increasing →
* the recrystallization at RT is suppresing →
* dislocation density is increasing →
* the probability of finding dislocation source is increasing →
* Change in basal slip character from localized deformation in pure Zn to uniform in the Zn-2.2Ag alloy. Prismatic slip takes place in two favorable planes resulting in buckling than localized pure shearing.

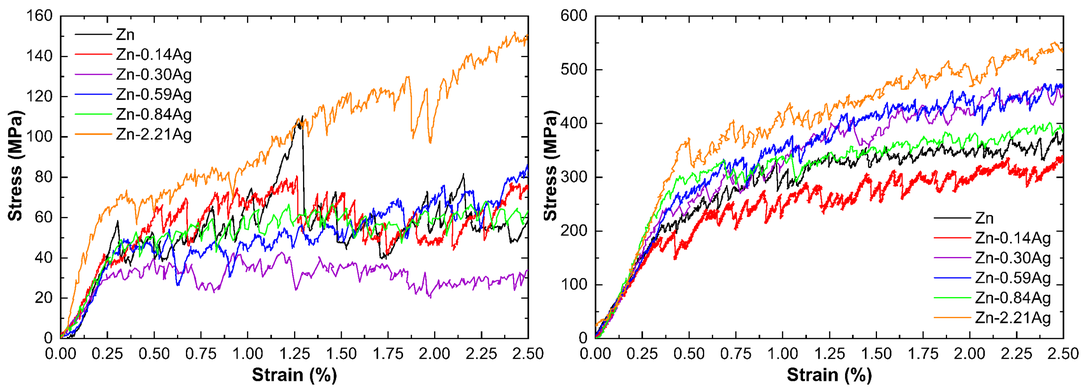


Fig. 9. Compression stress-strain curves of pure Zn and Zn-Ag alloys

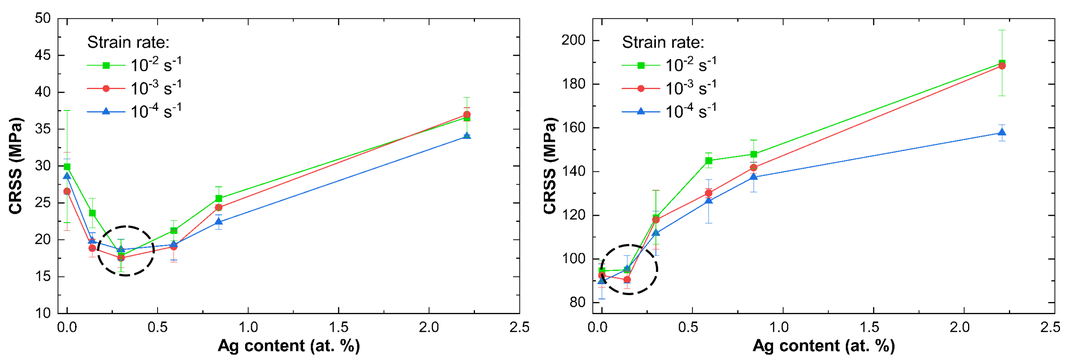


Fig. 10. The effect of Ag content on CRSS in basal and prismatic slip systems at various strain rates

* **Strain rate sensitivity**: **no** significant **effect in basal slip system**, while in **prismatic slip system**, pronounced **effect** was seen **above 0.59 at.% Ag**.
* **Strengthening effect: small Ag** additions result in **decrease** of both **$CRSS_B$** (up to 0.3 at.% Ag) and **$CRSS_P$** (up to 0.14 at.% Ag) while **further increase in Ag** content, **increase the CRSS** in both cases.
* According to [3] initial drop and further incease of CRSS with Ag additions result from increasing dislocation density in the micropillars. The transition in strenghtening source is expected as follows: dislocation starvation → single-source strengthening → exhausted hardening → forest hardening.

References:
[1] M. Wątroba, et al., Fine-tuning of mechanical properties in a Zn–Ag–Mg alloy via cold plastic deformation process and post-deformation annealing, Bioact. Mater. 6 (2021) 3424–3436.
[2] W. Bednarczyk, M. Wątroba, et al., Abnormal grain growth in a Zn-0.8Ag alloy after processing by high-pressure torsion, Acta Mater. 207 (2021) 116667.
[3] J.A. El-Awady, Unravelling the physics of size-dependent dislocation-mediated plasticity, Nat. Commun. 6 (2015) 5926.