Experimental measurements of critical resolved shear stresses in pure Zn and Zn-Ag alloys using micropillars compression

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MOTIVATION & OBJECTIVES

* **Zinc** (Zn) has been recently considered as a novel promising material for bioresorbable medical implants *(Fig. 1)*.

* **Poor strength and brittleness of** as-cast pure Zn require **alloying with other elements** and **plastic deformation** resulting in grain size refinement aimed at mechanical properties enhancement *(Fig. 2)*.

* Mechanical properties testing:

→ at the macroscale

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

→ at 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.
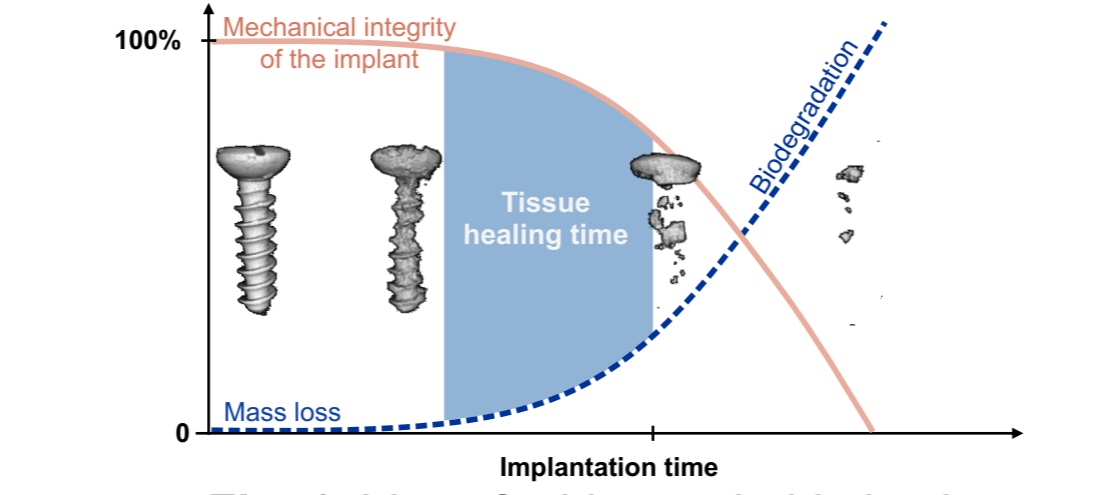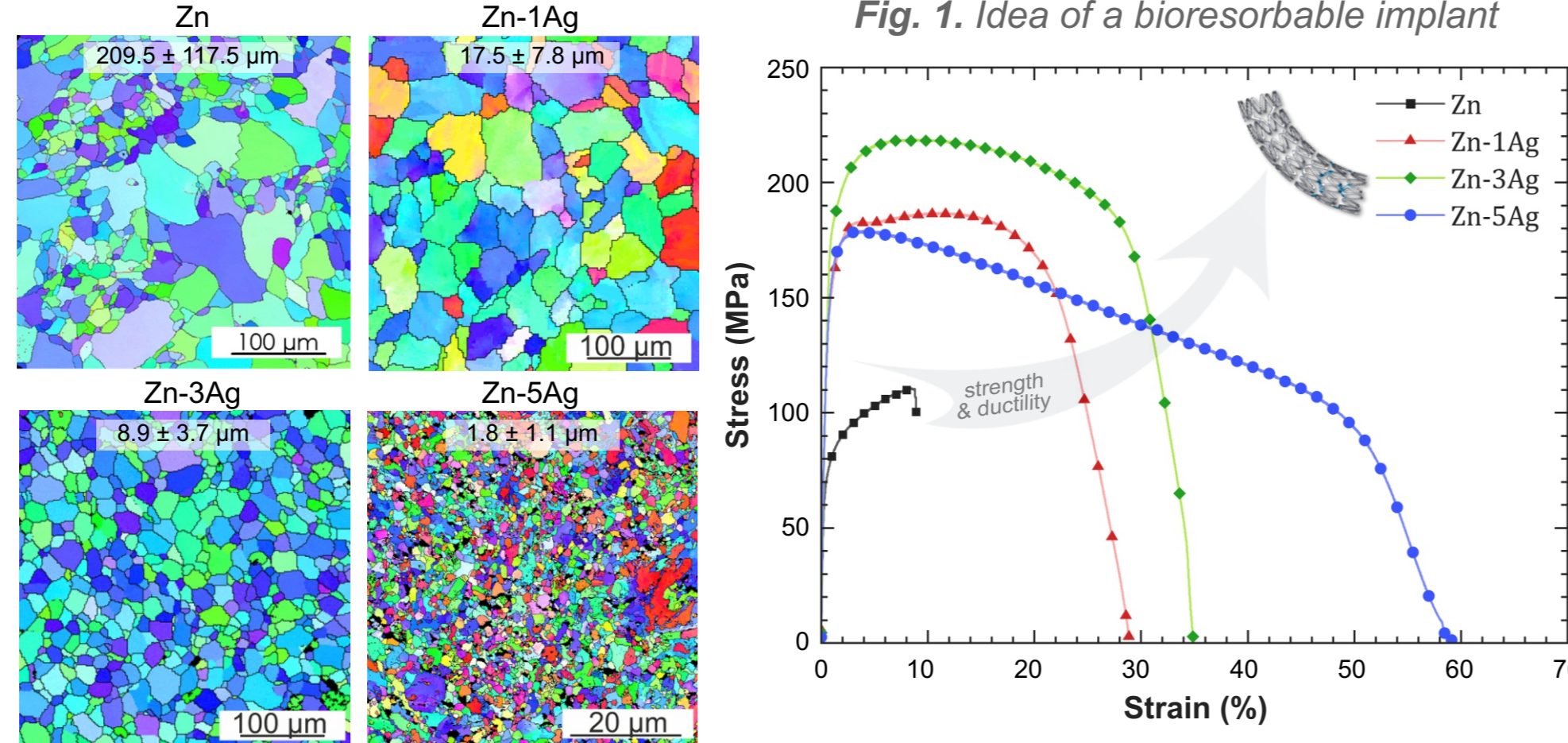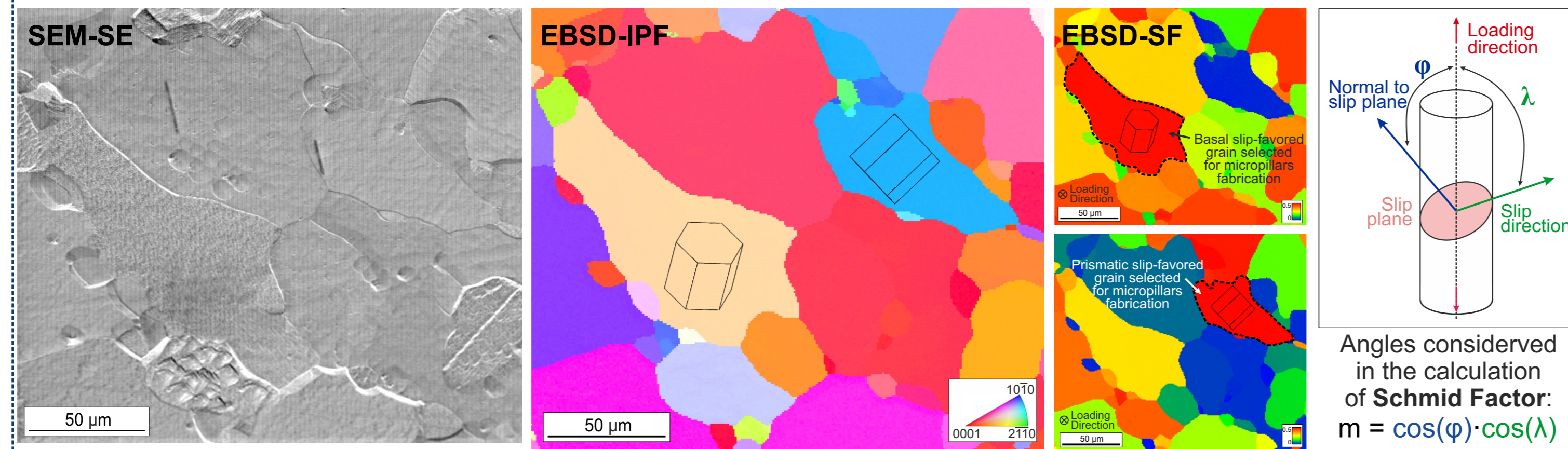
Fig. 1. Idea of a bioresorbable implant

Fig. 2. Effect of Ag on the microstructure and mechanical properties of hot-extruded 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 systems** were selected for the micropillars fabrication.

Angles considered in the calculation of **Schmid Factor**: $m = \cos(\varphi) \cdot \cos(\lambda)$

Fig. 3. Microstructure of the sample; EBSD-IPF orientation map; SF maps for basal and prismatic slip system; Schmid factor calculations.

* Micropillars were fabricated by multi-step Ga+ focused ion beam milling at 30kV and 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] since the transition from twinning to dislocation slip dominant deformation mechanism occurred within this grain size range.

* **According to the Schmid Law**, CRSS can be calculated 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.
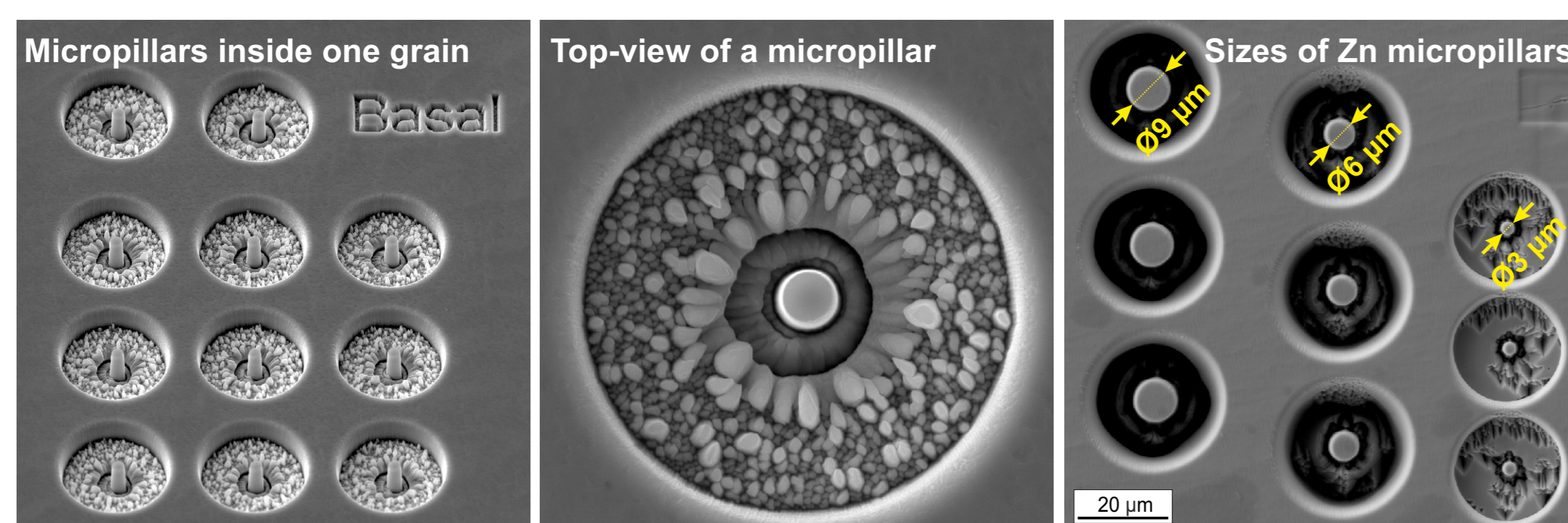
Fig. 4. Micropillars prepared for the compression tests withing single grain with basal- or prismatic- slip favoured orientation

IN SITU MICROPILLARS COMPRESSION

* Micropillars compression tests were performed using diamond flat punch installed in Alemnis Standard Assembly *in situ* nanoindentation system *(Fig. 5)* 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.**

HCP favorable orientation for: Basal slip / Prismatic slip

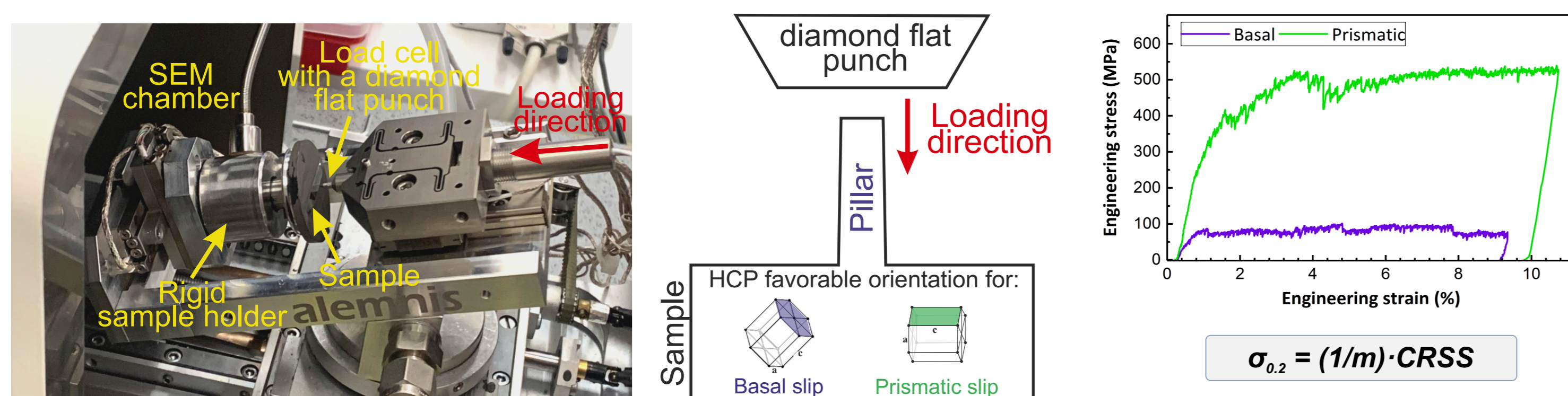$$\sigma_{0.2} = (1/m)\cdot CRSS$$

Fig. 5. Setup for the in situ micropillars compression and the example of stress-strain curves

FUTURE PERSPECTIVES

► Current studies provide fundamental knowledge about the possibilities of Zn solid solution strengthening.

► Calculated CRSS can be further implemented in crystal plasticity models, as input data, for designing fabrication processes of bioresorbable implants requiring specific mechanical properties.

► The Zn-Ag system can be used as a thin antibacterial coating on biomedical devices, such as orthopaedic 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 bioresorbable maxillofacial implant applications.
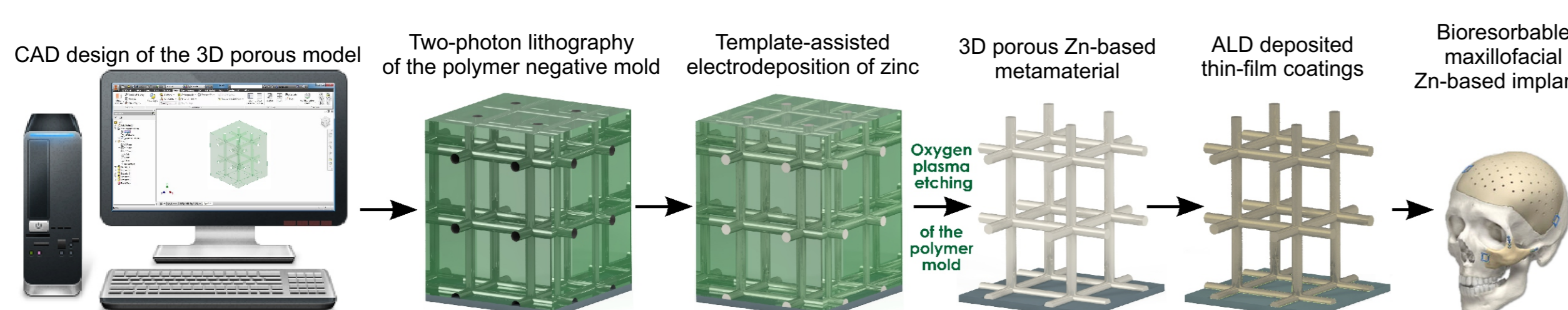
Fig. 6. Process diagram for fabrication of coated Zn-based metamaterials
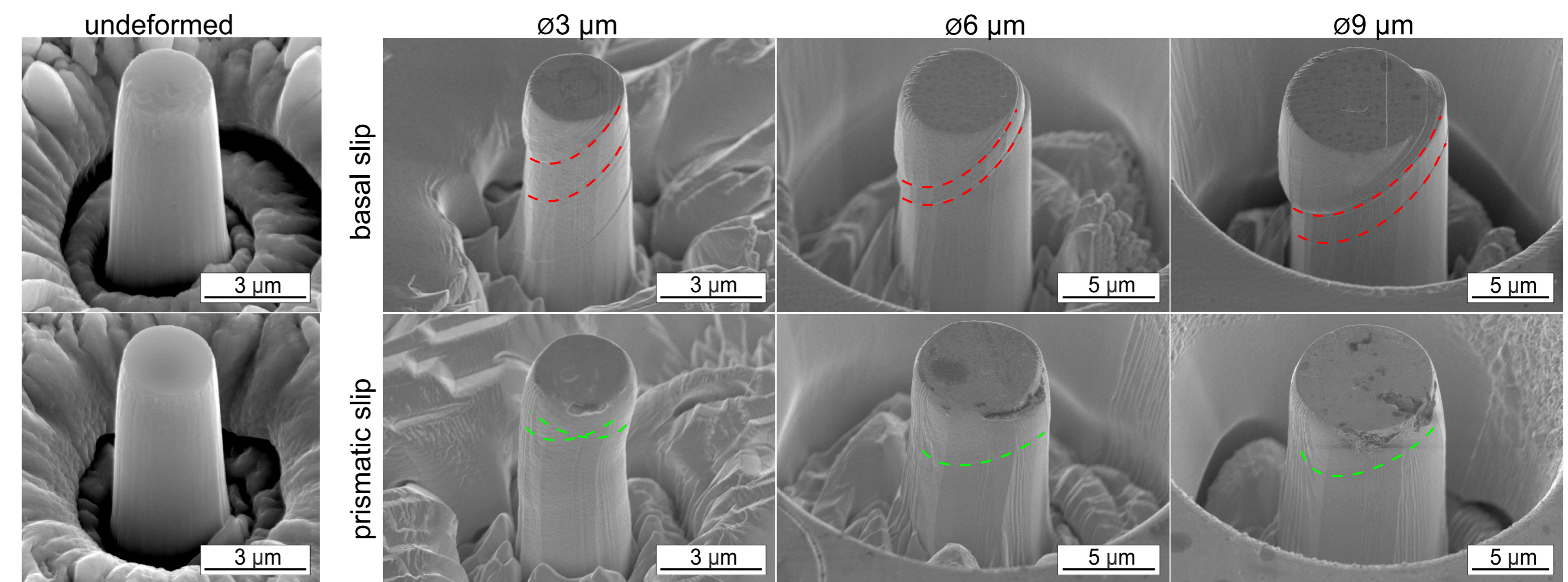
SIZE EFFECT IN PURE ZINC

Fig. 7. SEM images of undeformed and deformed Zn micropillars with different sizes showing basal and prismatic slip after compression
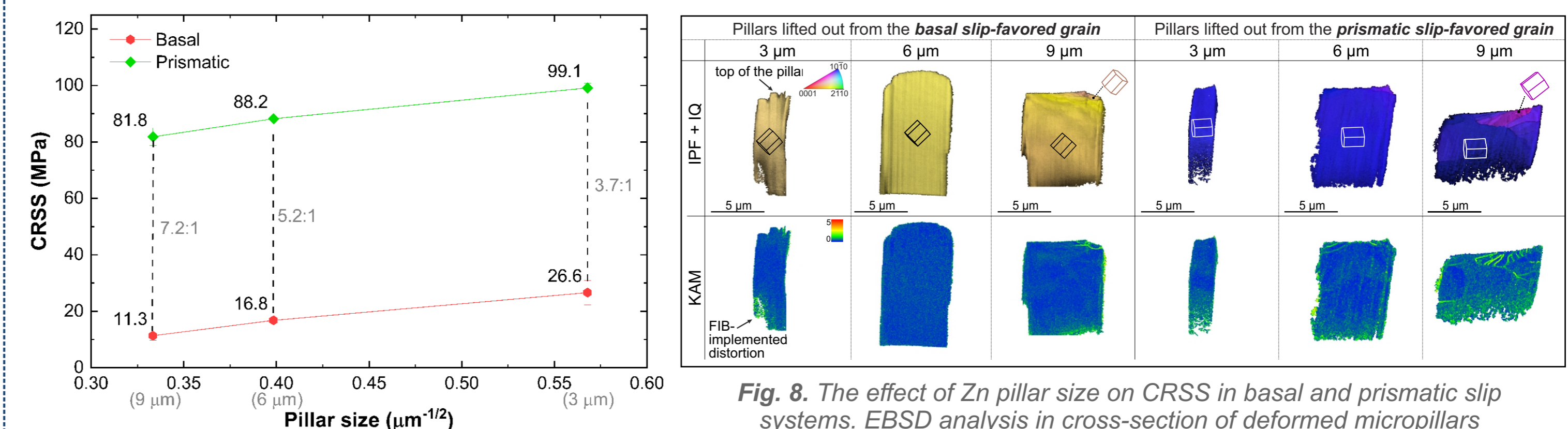
Fig. 8. The effect of Zn pillar size on CRSS in basal and prismatic slip systems. EBSD analysis in cross-section of deformed micropillars

* A significant **yield stress and CRSS increase** with the micropillar **diameter reduction** was observed.
* The deformation in the **basal slip** system produced **multiple slip traces** along the basal plane, while in the **prismatic slip** system, **single slip traces** were observed.
* A **uniform orientation within the deformed 3 μm pillars** was observed, while **in bigger pillars, a localized deformation** occurred on the top of the pillar, resulting in a lattice rotation. No twins were nucleated.

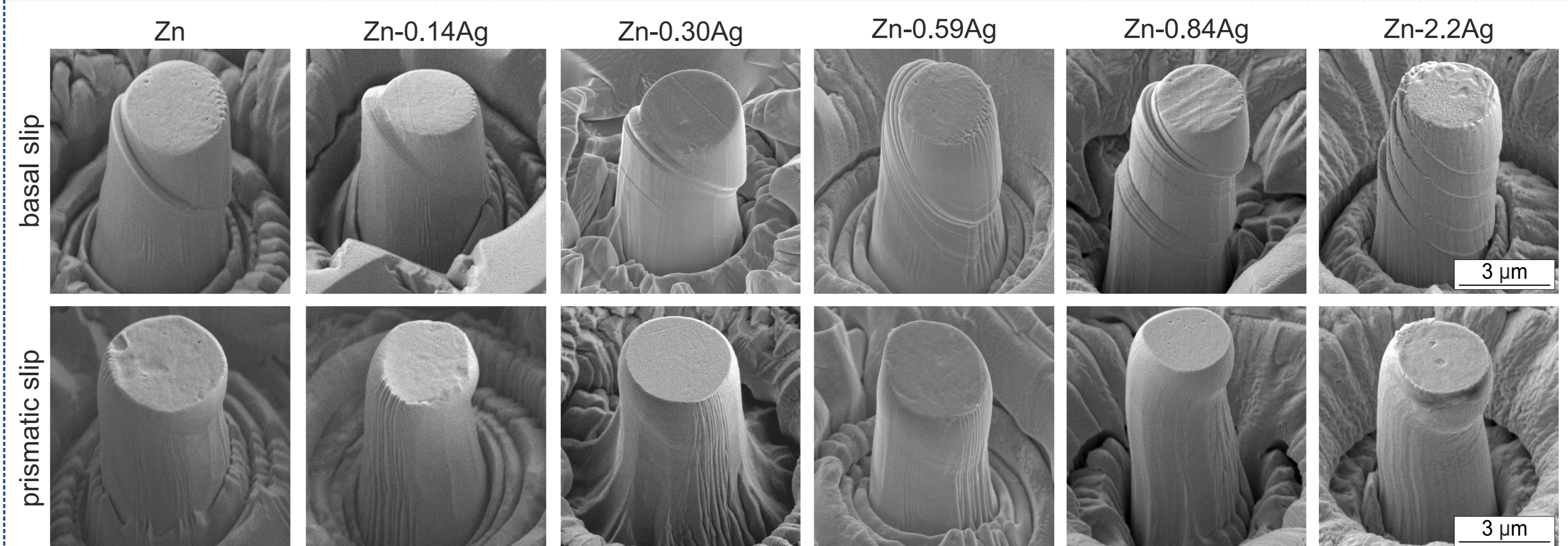## SOLID SOLUTION EFFECT IN Zn-xAg ALLOYS

Fig. 9. SEM images of deformed Zn-xAg micropillars showing basal and prismatic slip after compression

→ Ag content is increasing →
→ the recrystallization at RT is suppresing →
→ dislocation density is increasing →
→ the probability of finding dislocation source is increasing →

* Basal slip changes the character from localized deformation in pure Zn to uniform in the Zn-2.2Ag alloy.
* Prismatic slip occurs in two favourable planes resulting rather in buckling than clear localized shearing.
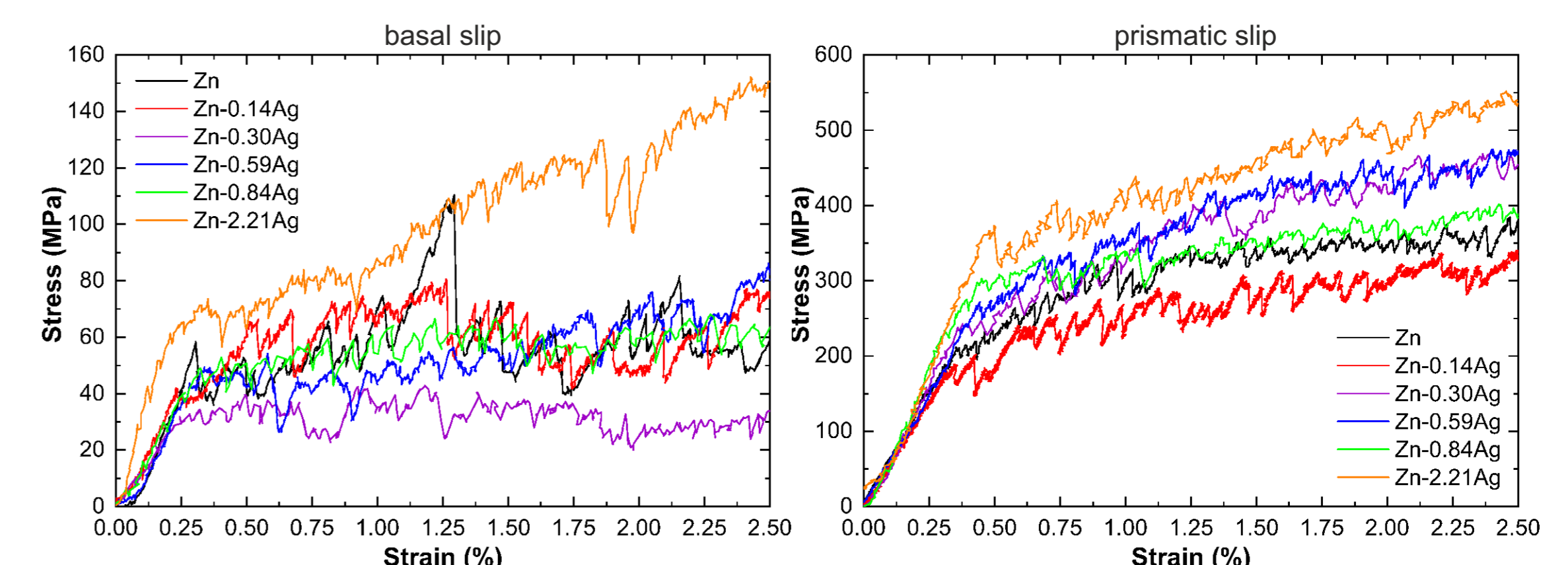
Fig.10. Engineering stress-strain curves of Zn and Zn-xAg micropillars with basal- and prismatic slip-favored orientation
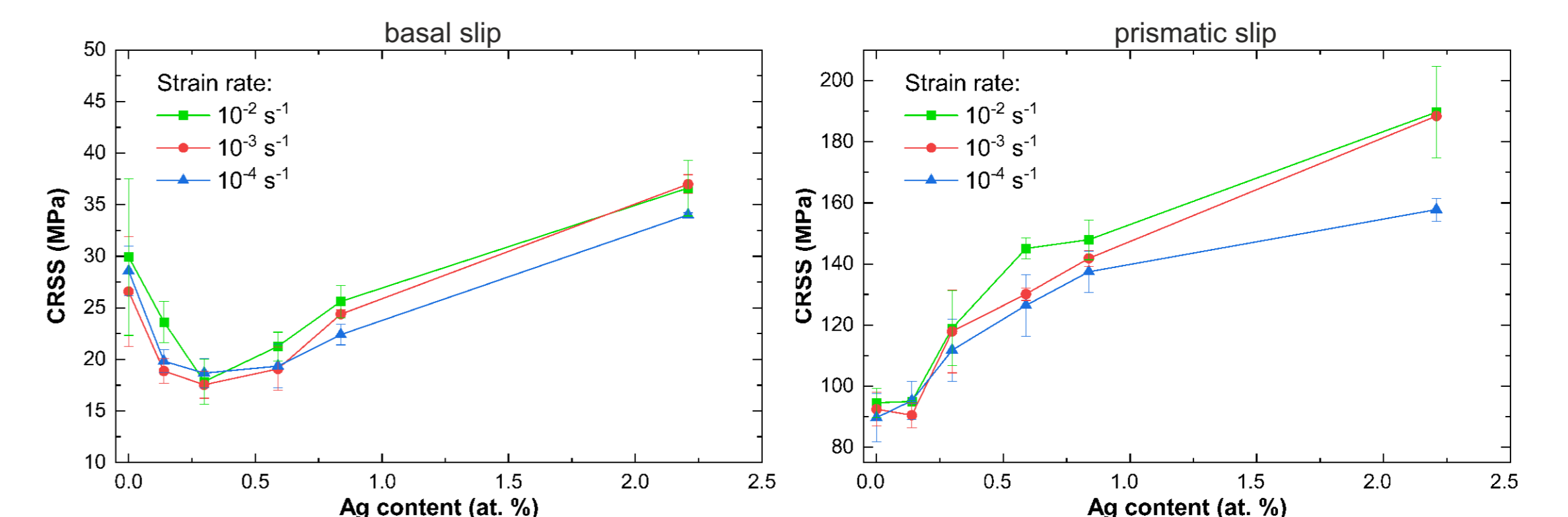
Fig. 11. The effect of Ag additions on CRSS for the basal and prismatic slip systems in Zn-xAg alloys at various strain rates

* **Strain rate sensitivity:** no significant effect in the basal slip system, while in the prismatic slip system, the pronounced effect was seen above 0.59 at.% Ag.

* **Strengthening effect:** small Ag additions result in a 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 increases the CRSS in both cases.

* According to [3], initial drop and further increase of CRSS with Ag additions result from increasing dislocation density in the micropillars. The transition in strengthening sources 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.