

*Unique microstructure and mechanical properties of ultra-finegrained
Al-Zn alloys processed by high-pressure torsion*



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Background, general observations

- **Bulk Ultrafine-grained (UFG) materials having high strength can be mostly achieved by using severe plastic deformation (SPD) techniques such as well-known equal-channel angular pressing (ECAP) or high- pressure torsion (HPT) methods**
- **The UFG materials have reasonably saturated microstructures with a steady-state (saturated) dislocation density**
- **The role of the grain boundaries is enhanced in the post-SPD deformation processes**

- **Unusual super-ductility at room temperature in an ultrafine-grained aluminum alloy**

HPT-processed Al-30Zn alloy:

- Ultrafine-grained
- Grain size of about 300-400 nm

- **Super-ductility at room temperature:**

- **Maximum elongation of about 150-160%**

- **Relatively high strain rate sensitivity**

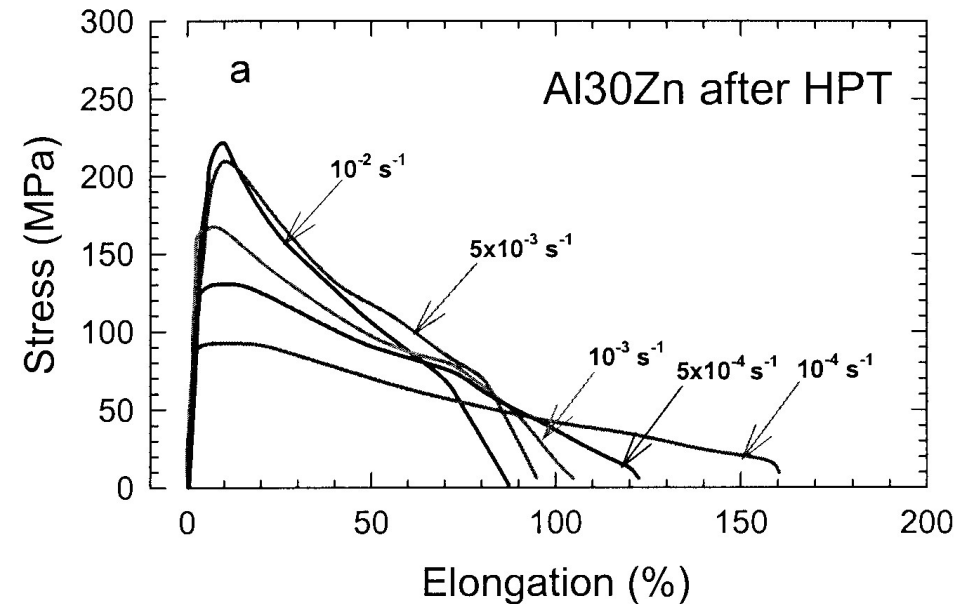
(*Ruslan Valiev et al., J Mater. Sci. 45 (2010) 4718.*)

(for Al alloys at RT, in general:

max. elongation < 30-40%,

for UFG alloys:

max. elongation < 10-20%)



Present talk:

-The influence of Zn concentration

-Unique mechanical and plastic behaviors of UFG Al-30%Zn alloy

Investigated by:

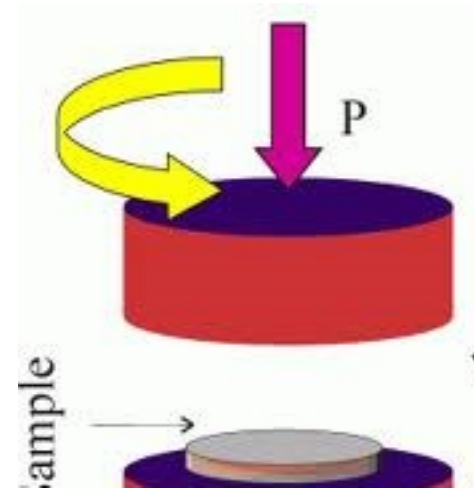
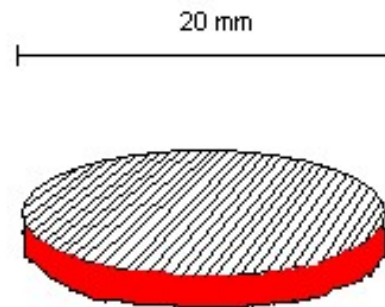
- **Transmission electron microscopy (TEM),**
- **Energy-dispersive X-ray spectroscopy (EDS)**
- **Depth-sensing indentation (DSI)**
- **Focused ion beam and scanning electron microscopy (FIB/SEM)**
- **Differential scanning calorimetric (DSC)**

Investigated Materials:

-High purity (4N) **Al-2Zn, Al-5Zn, Al-10Zn and Al-30Zn** (wt%)

Processing by High Pressure Torsion (HPT) (in Ufa, Russia)

P = 6 GPa, N = 5 turns

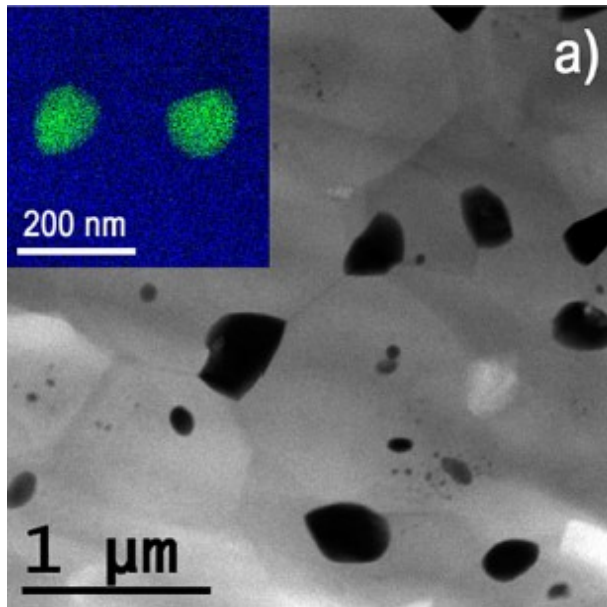


Microstructure of HPT-processed samples:

Al-2Zn and Al-5Zn: Ultrafine-grained (UFG), $d \sim 1 \mu\text{m}$

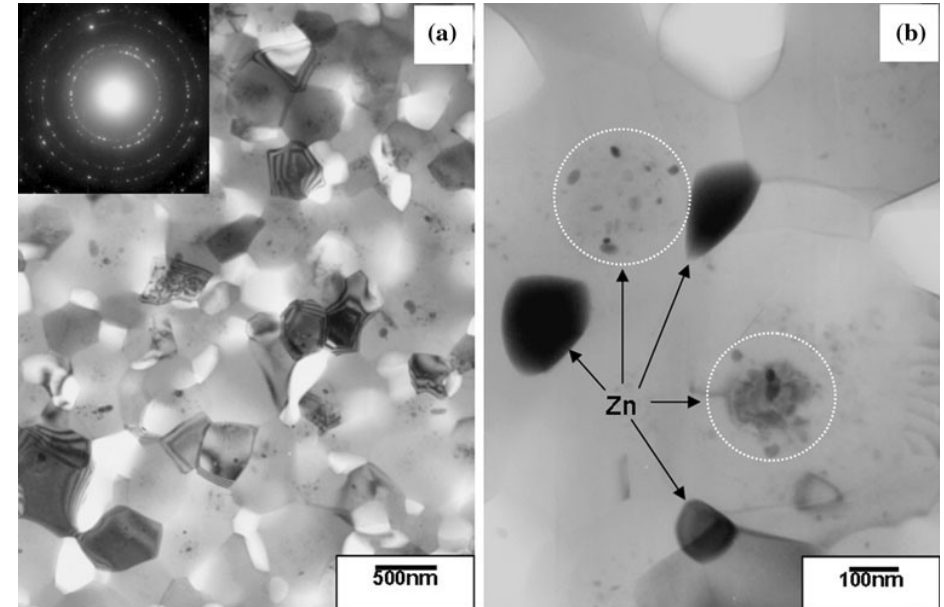
Al-10Zn: UFG, $d \sim 500\text{-}700 \text{ nm}$, decomposition

Al-30Zn: UFG, $d \sim 300\text{-}400 \text{ nm}$, decomposition



X. Sauvage et al., Adv. Eng. Mater (2015),

E.V. Bobruk et al., Rev. Adv. Mater. Sci. (2015)



R. Valiev et al., J Mater. Sci. (2010)

Nguyen Q. Chinh et al., Adv. Eng. Mater. (2014)

Decomposition in microstructure of Al-10Zn and Al-30Zn samples.

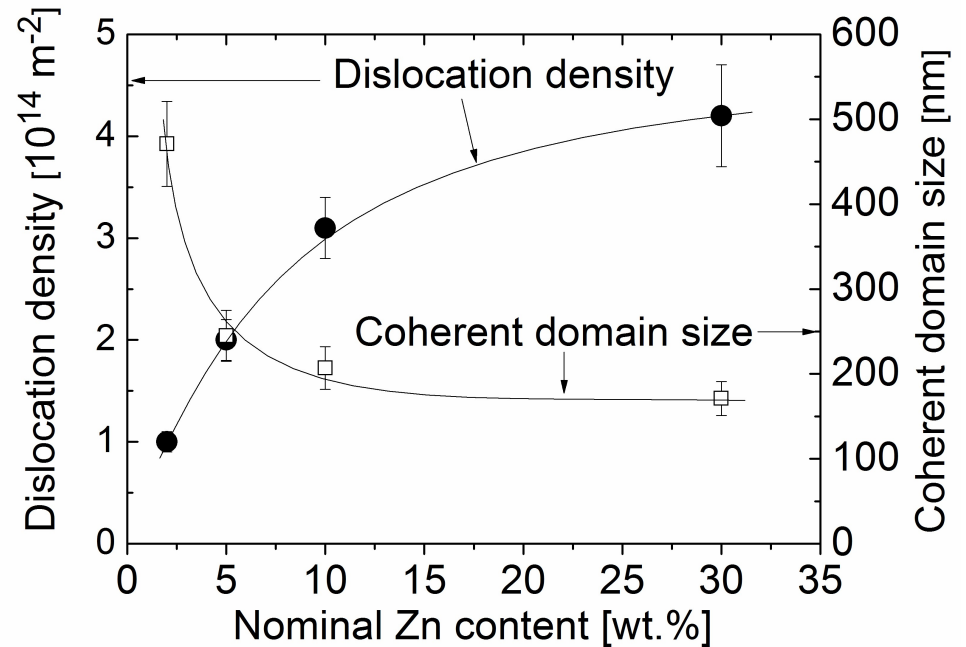
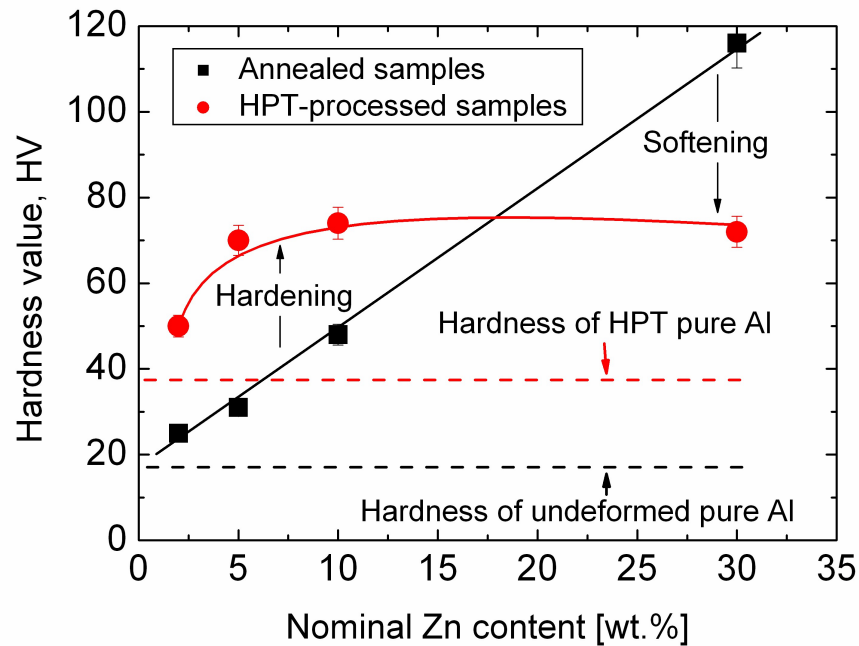
Y.H. Zhu, Mater. Transactions (2004),

A.A. Mazilkin et al., Acta Materialia (2006), Mater. Letters (2012)

Ali Alhamidi et al., MSEA (2014),

M. Borodachenkova et al., Int. J. Plasticity. (2014),

Mechanical Properties:



● For Zn concentrations ≤ 10 wt%, the hardness of the HPT-processed samples is higher than that of the annealed samples: *Normal strengthening effect of SPD.*

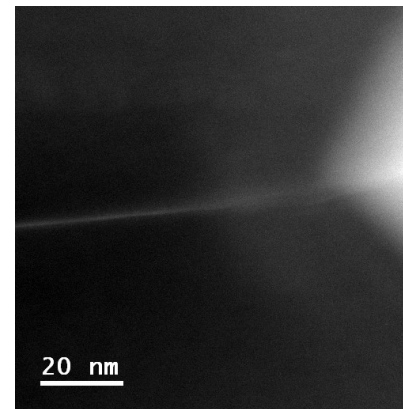
● In the case of the Al-30Zn alloy there is a strong reduction in hardness due to HPT:

Abnormal softening effect of SPD

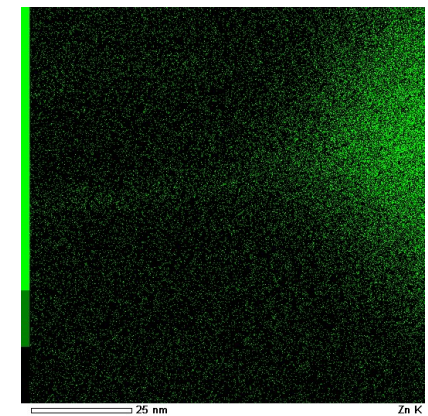
• **Decomposition in microstructure of Al-10Zn and Al-30Zn alloy processed by HPT**

Zn-rich layers in Al/Al boundaries

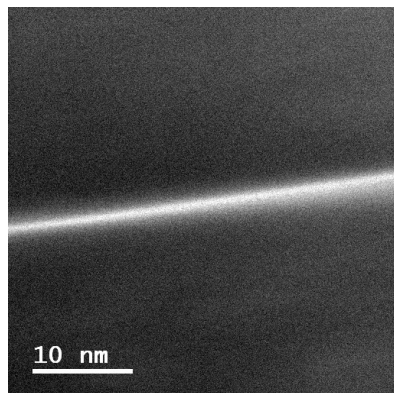
High magnification STEM as a) HAADF image (Z contrast) showing a Zn particle and a brightly imaged grain boundary and b) Corresponding EDS map, confirming the high Zn concentration in the particle and the Zn grain boundary segregation.



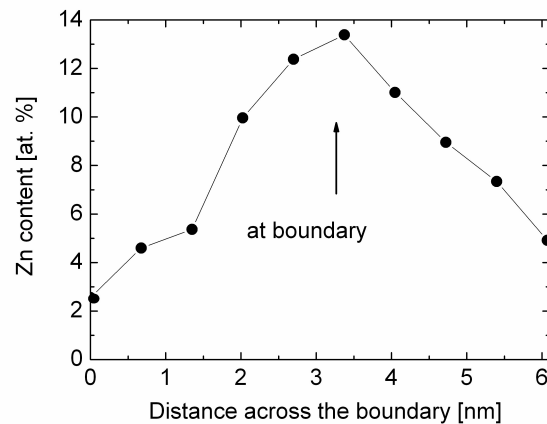
a)



b)



a)



b)

High magnification STEM as a) HAADF image (Z contrast) showing Zn segregation along a grain boundary and b) EDS line profile analysis showing the local Zn concentration across the boundary

Nguyen Q. Chinh et al., Adv. Eng. Mater. (2014)

B. B. Straumal et al., Scripta Mater. (2014)

X. Sauvage et al., Adv. Eng. Mater (2015)

Unique microstructure and mechanical properties of Al-Zn alloys processed by HPT

The effect of the Zn-rich boundary layers in UFG Al-Zn alloys:

- Enhancing the strain rate sensitivity (SRS)

The strain rate sensitivity of the HPT-processed Al-Zn alloys.

Sample	Al-2Zn	Al-5Zn	Al-10Zn	Al-30Zn
SRS value (± 0.02)	0.08	0.14	0.17	0.25

- The process of plastic deformation is controlled by the Zn diffusion along Al/Al grain boundaries (Nguyen Q. Chinh et al., Mater. Sci. Eng. A (2012).)

-Enhancing the role of grain boundary sliding at room temperature

-Leading to super-ductility at room temperature in HPT-processed Al-30Zn:

- Maximum elongation of about 150-160%

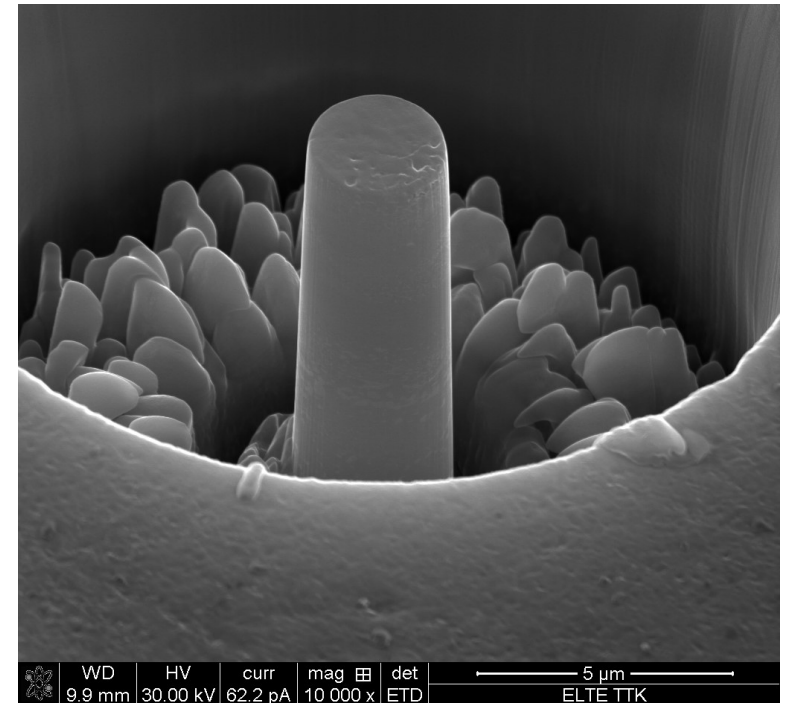
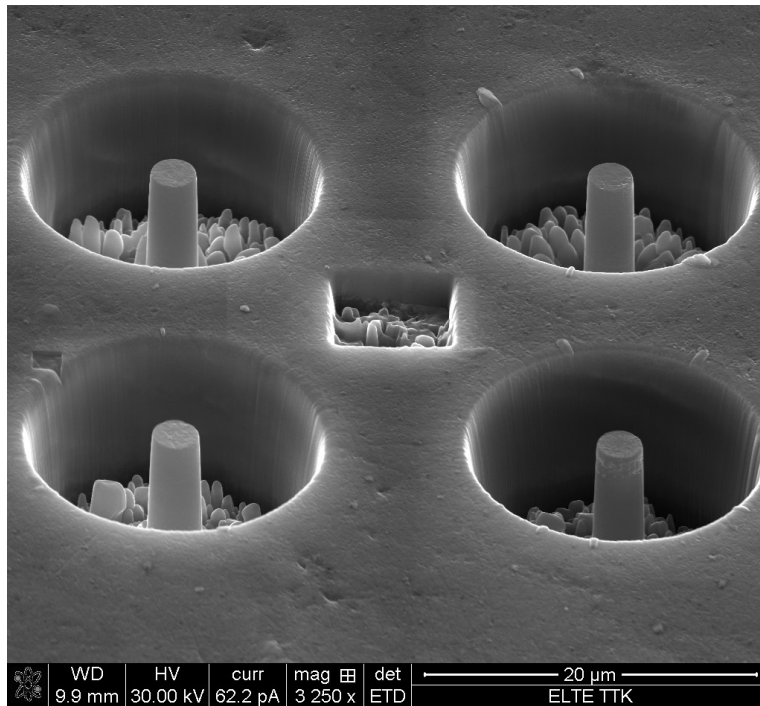
(Ruslan Valiev et al., J Mater. Sci. (2010)

(For Al-10Zn alloy: max. elongation was lower than 50%, E.V. Bobruk et al., Rev. Adv. Mater. Sci. (2015)

Deformation mechanism of the Ultrafine-grained Al-30Zn alloy:

Unique plastic behavior of micro-pillars of UFG Al-30Zn alloy:

Micro-pillars having diameters of $\sim 3 \mu\text{m}$ and heights of $\sim 10 \mu\text{m}$ were prepared on the surfaces of **both conventional and UFG alloy disks** using SEM and FIB (with Ga^+ ions)

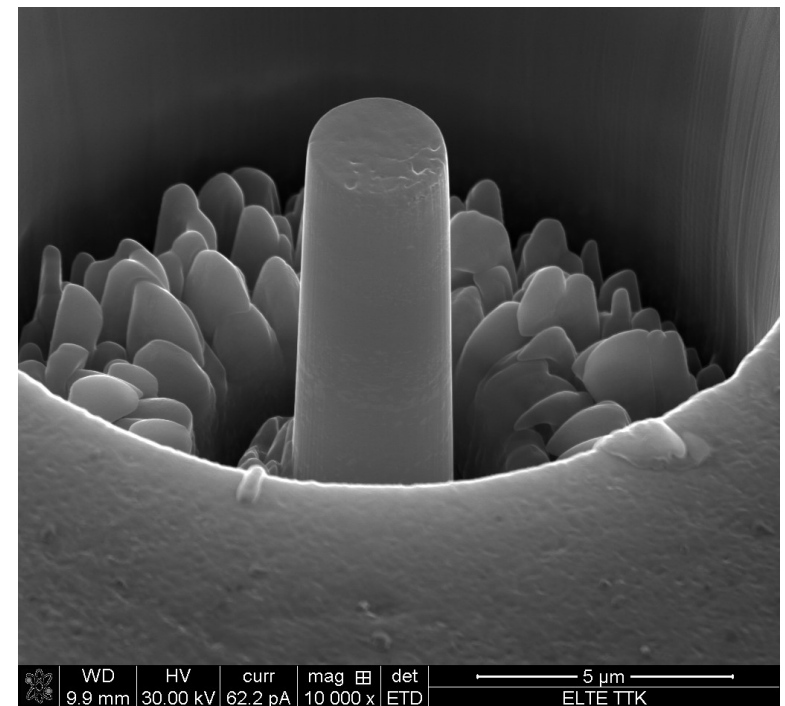
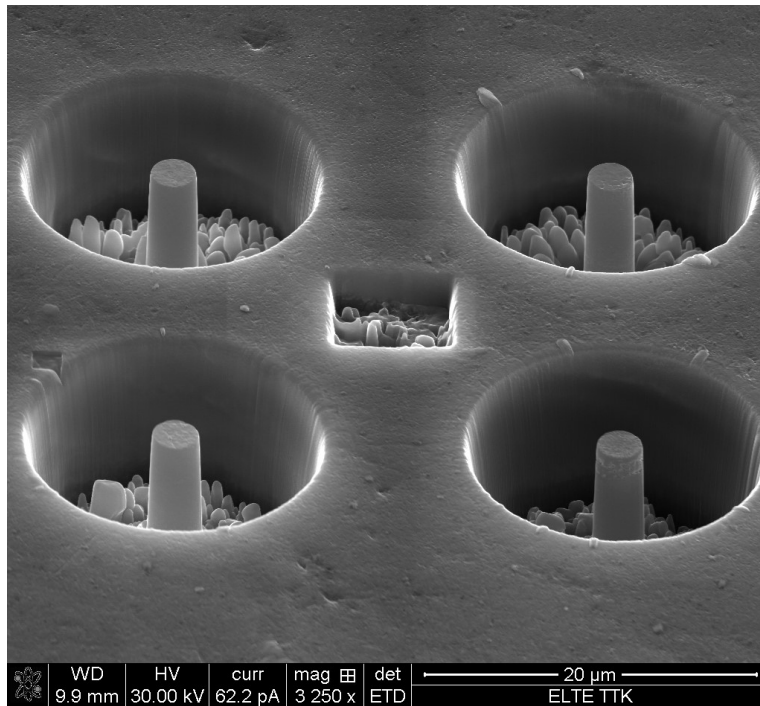


Nguyen Q. Chinh et al., Adv. Eng. Mater. (2014), DOI: 10.1002/adem.201300450

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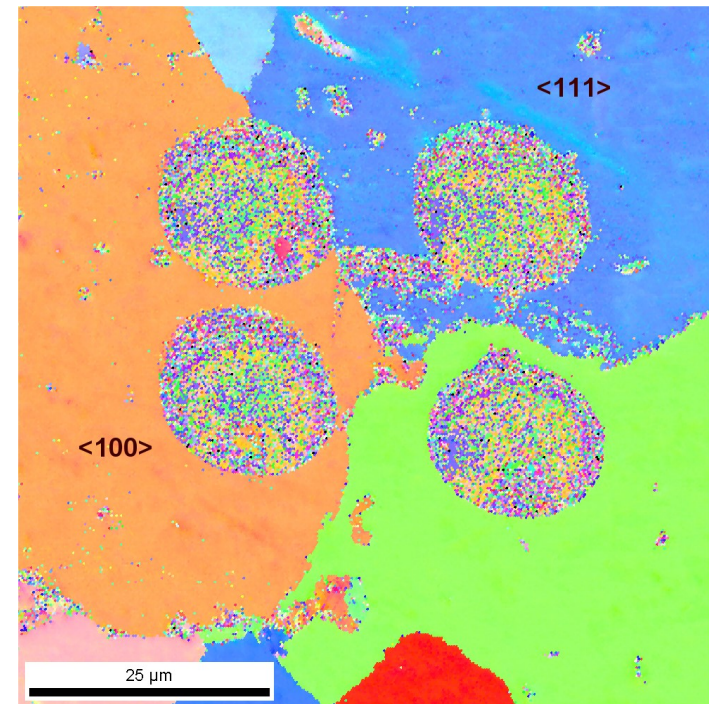
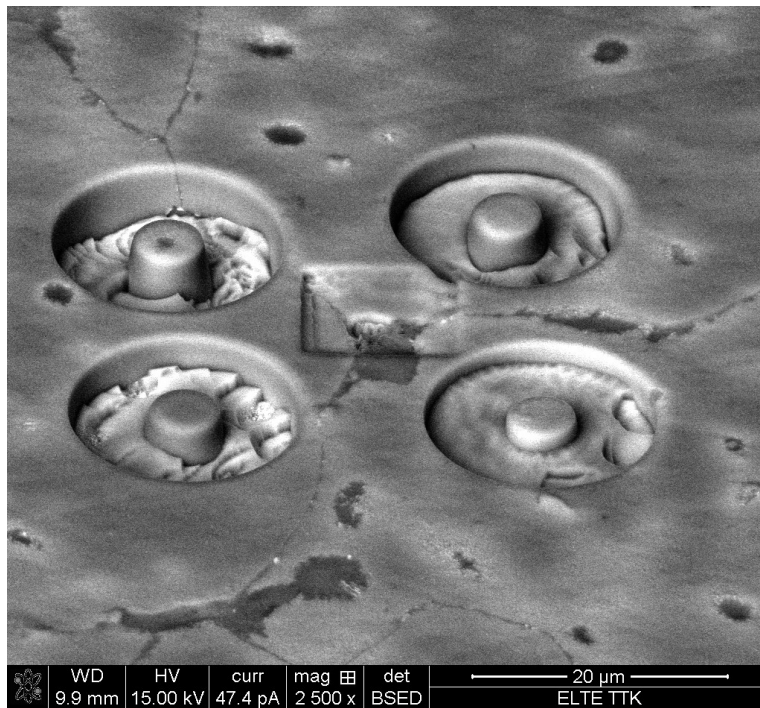
Ultrafine-grained Al-30Zn alloy: grain size of about 300-350 nm

The micro-pillars are polycrystals

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Back-scattering electron diffraction (EBSD)

Conventional Al-30Zn alloy: grain size of about $60 \mu\text{m}$

The micro-pillars are single-crystals (Adv. Eng. Mater. (2014), DOI: 10.1002/adem.201300450)

Deformation mechanism of the Ultrafine-grained Al-30Zn alloy:

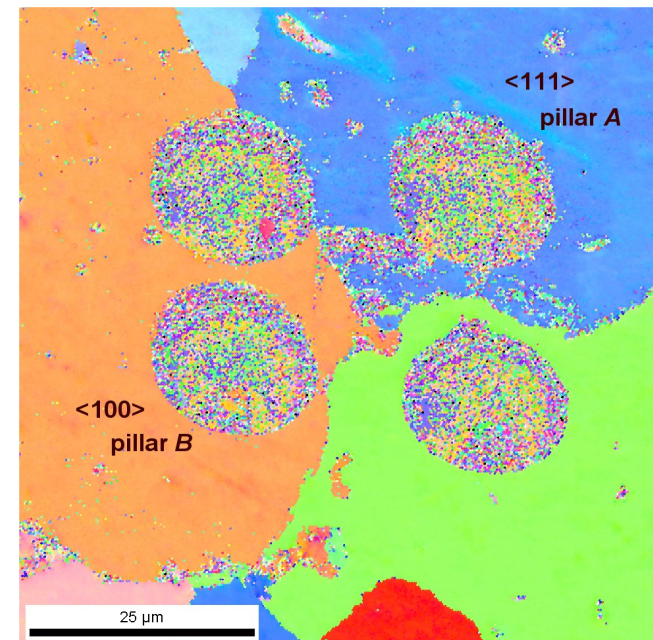
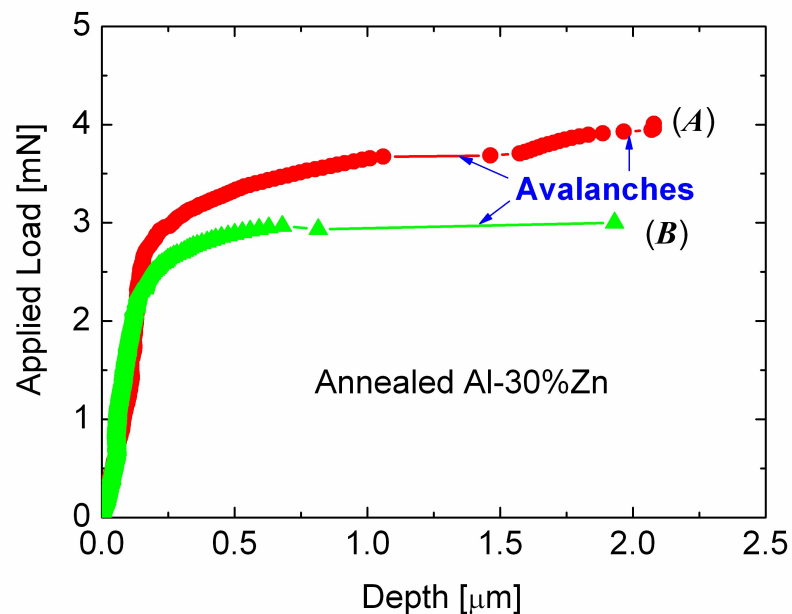
Unique plastic behavior of micro-pillars of UFG Al-30Zn alloy:

• Nanocompression:

Nanocompressions were made at room temperature using a flat-ended conical diamond indenter within a depth-sensing ultra-microhardness testing machine operating under a force increasing up to 4 mN with loading rate of 10^{-2} mN s⁻¹, for a maximum compression depth of 2 μ m.

(Nguyen Q. Chinh et al., *Adv. Eng. Mater.* (2014), DOI: 10.1002/adem.201300450)

Micro-pillars on the surface of conventional (annealed) sample:



Deformation mechanism of the Ultrafine-grained Al-30Zn alloy:

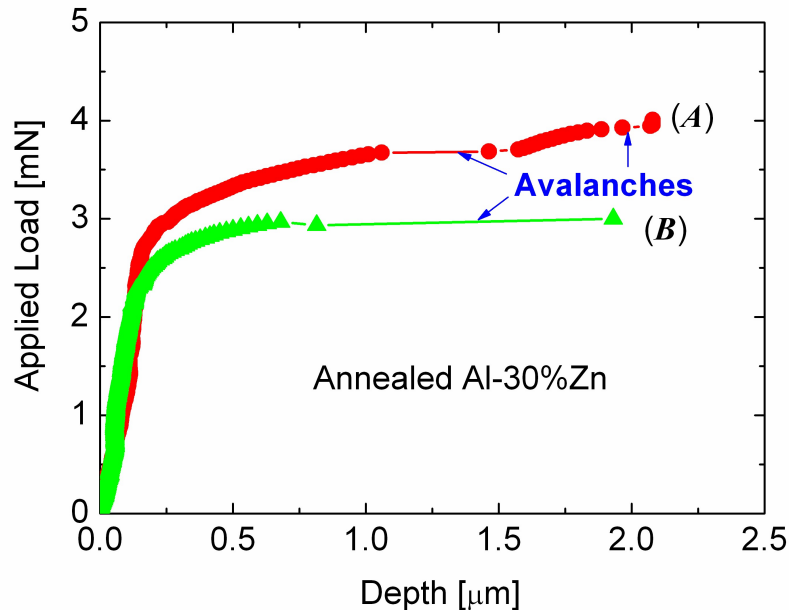
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Micro-pillars on the surface of conventional (annealed) sample:



- Visible strain avalanches
- Well-known phenomenon for single-crystals
- Catastrophic failure, difficulties in plastically forming
- Micrometer-sized samples of coarse-grained metals are not suitable for use in the fabrication of micro-devices

Deformation mechanism of the Ultrafine-grained Al-30Zn alloy:

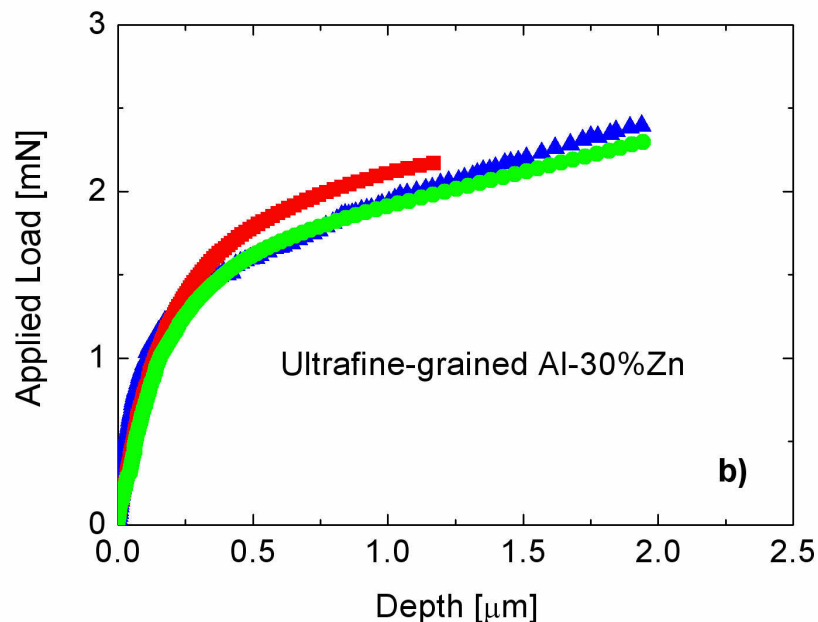
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Micro-pillars on the surface of Ultrafine-grained sample:

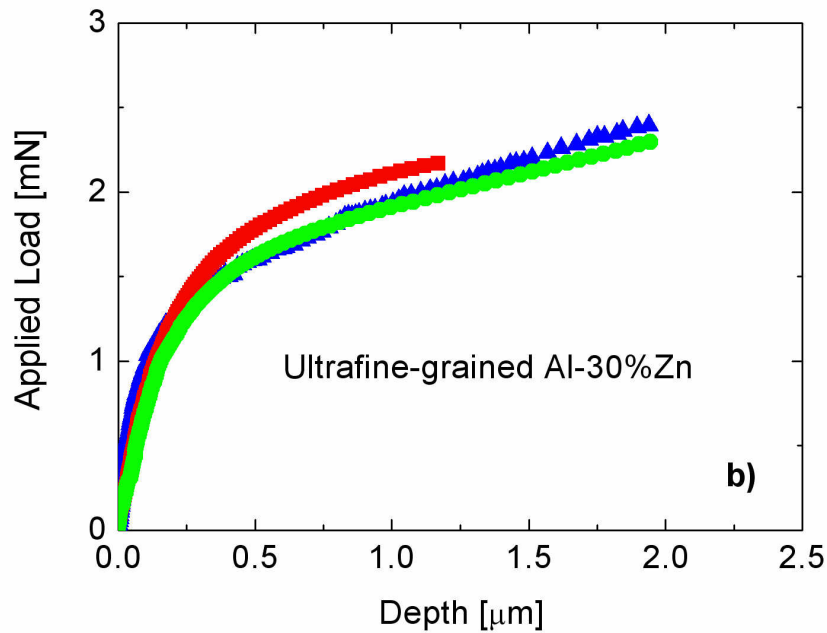


- Smooth, absence of any strain avalanches
- Polycrystalline micro-pillars
- The role of grain boundaries

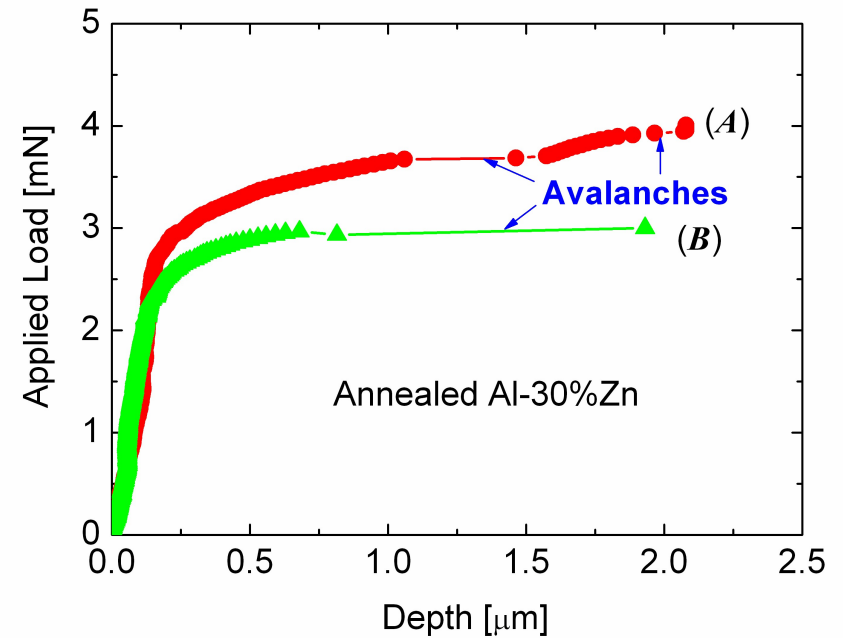
Deformation mechanism of the Ultrafine-grained Al-30Zn alloy:

Unique plastic behavior of micro-pillars of UFG Al-30Zn alloy:

- Nanocompression:



Reproducibility with a scatter of <math><10\%</math>



Deviations of more than 30%.

Deformation mechanism of the Ultrafine-grained Al-30Zn alloy:

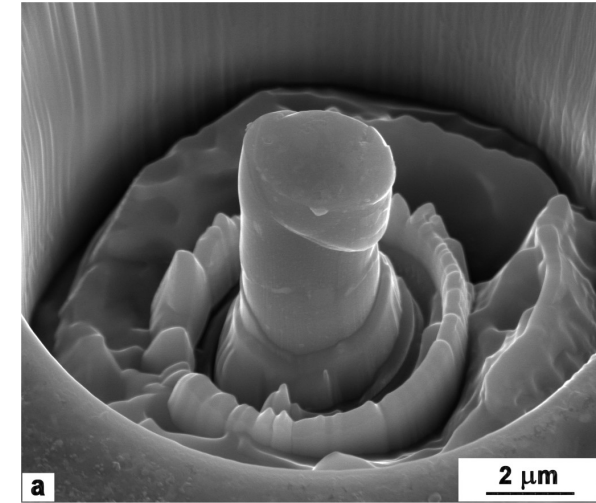
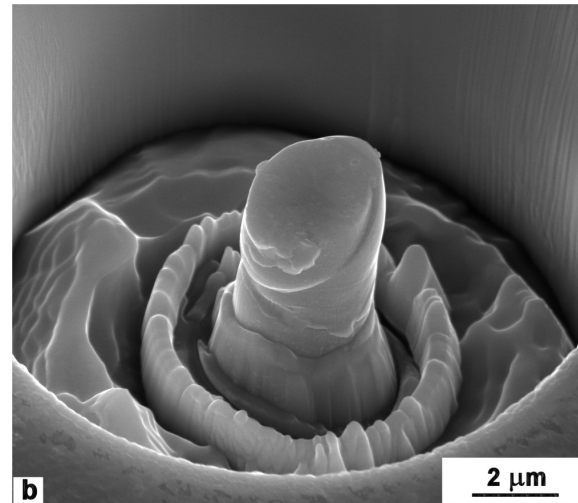
Unique plastic behavior of micro-pillars of UFG Al-30Zn alloy:

Pillar *A*, near to $\langle 111 \rangle$

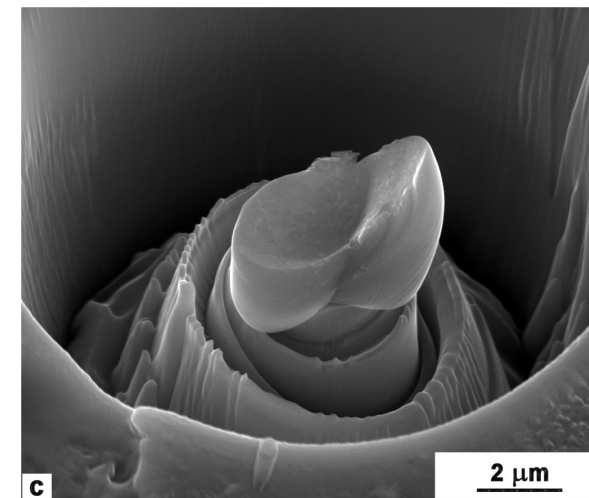
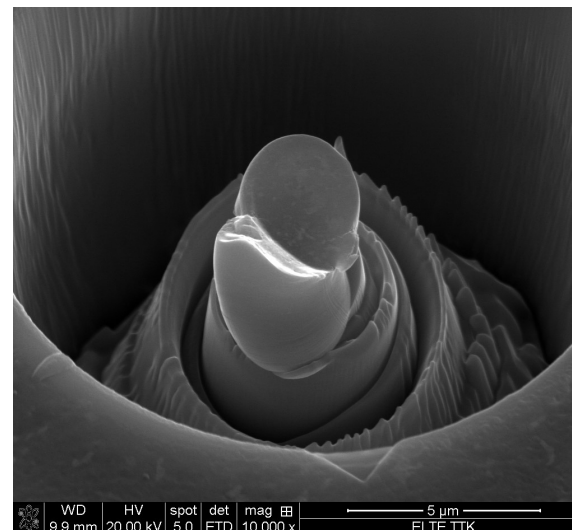
Examination of the compressed
micro-pillars by SEM:

a) Single-crystal pillars:

- **Strain localizations and extreme slip bands**
- **Maximum shear stress at 45° orientation**



Pillar *B*, near to $\langle 100 \rangle$



(Nguyen Q. Chinh et al.,
MRS Communications 2 (2012) 75-78,
Adv. Eng. Mater. (2014),
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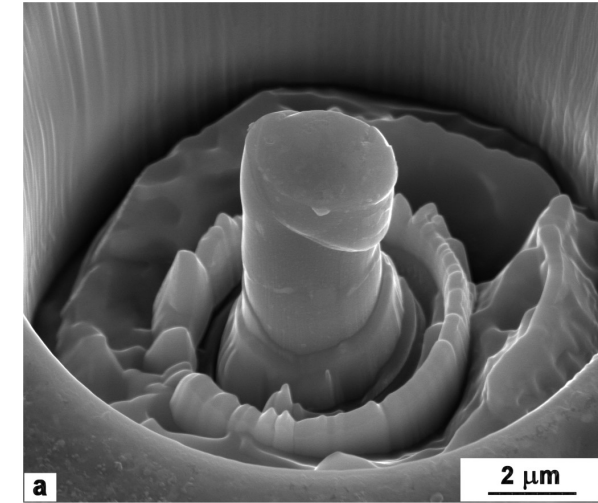
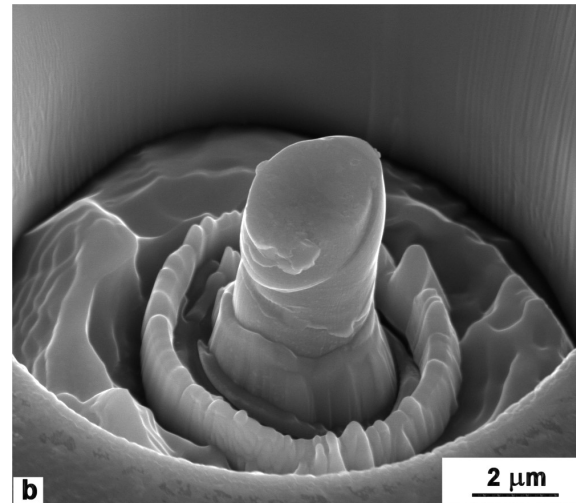
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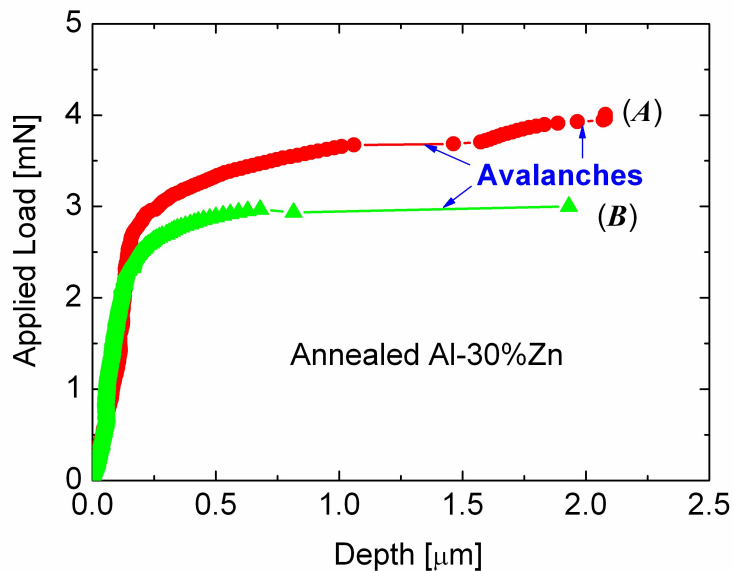
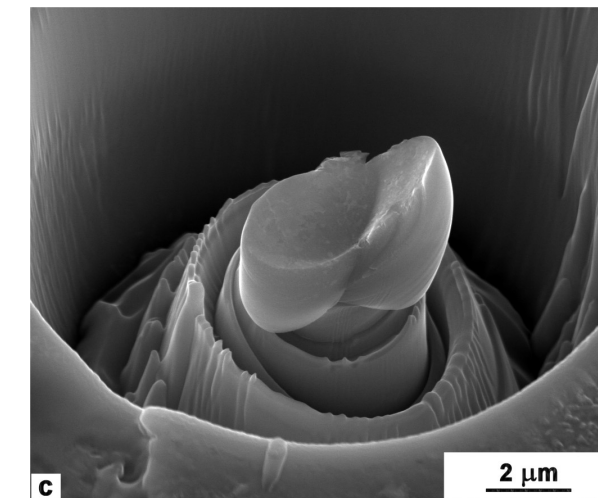
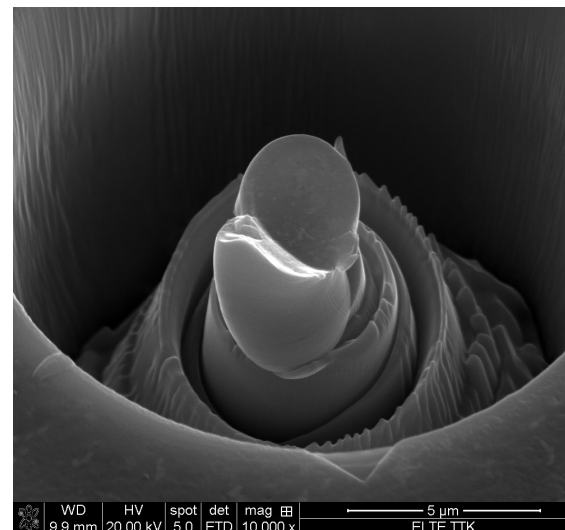
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Pillar *B*, near to $\langle 100 \rangle$



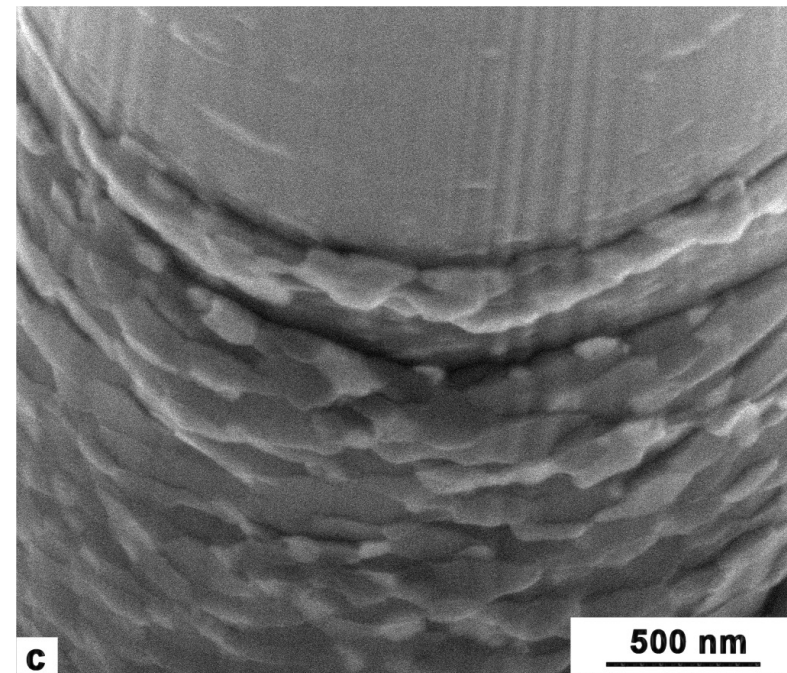
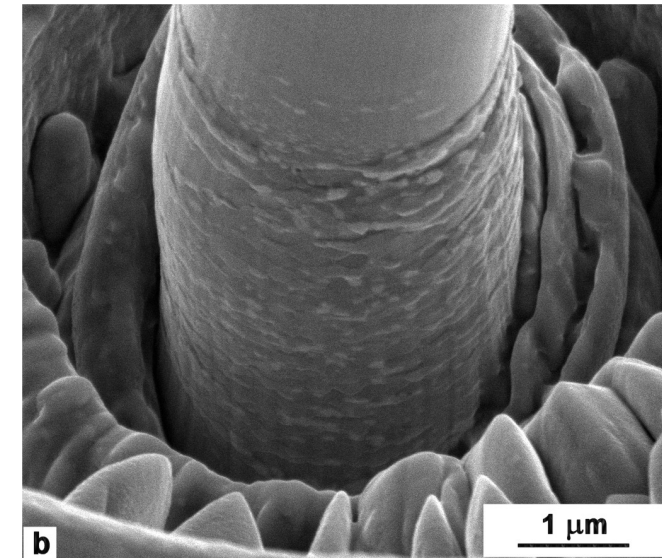
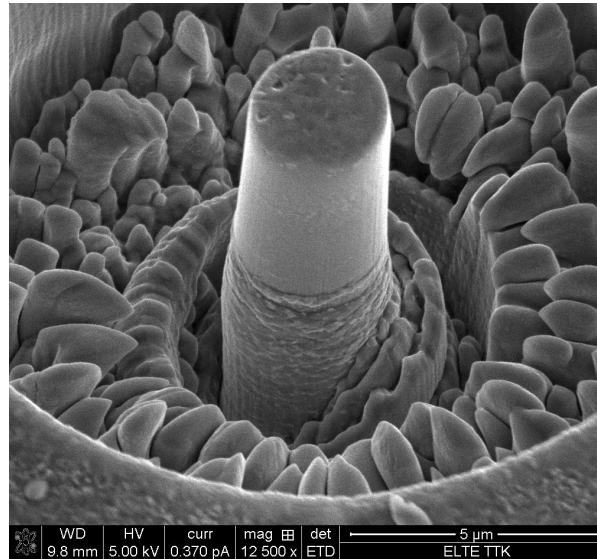
Deformation mechanism of the Ultrafine-grained Al-30Zn alloy:

Unique plastic behavior of micro-pillars of UFG Al-30Zn alloy:

Examination of the compressed
micro-pillars by SEM:

b) UFG (polycrystal) pillars:

- Occurrence of intensive GBS without localization and extreme slip
- Deformation having cylindrical symmetry



(Nguyen Q. Chinh et al.,

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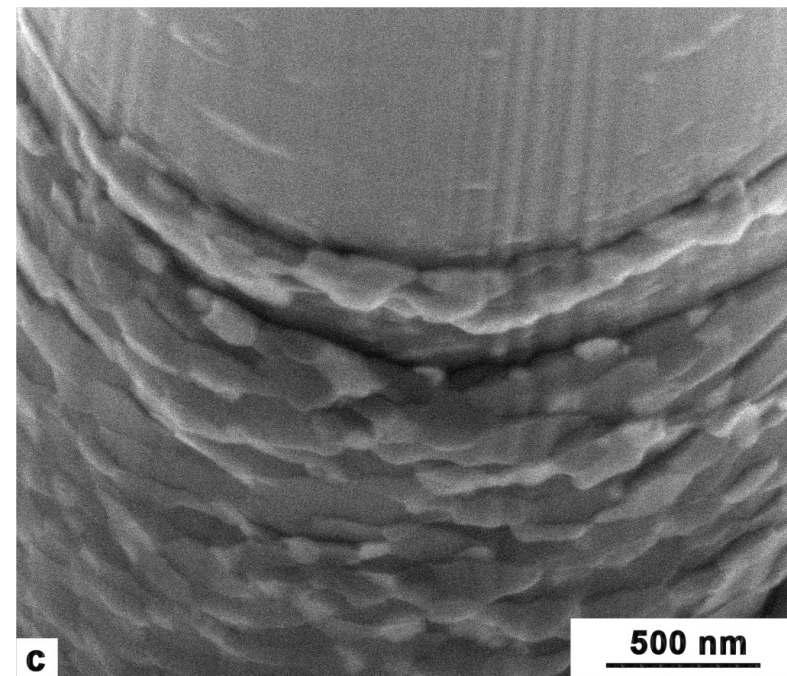
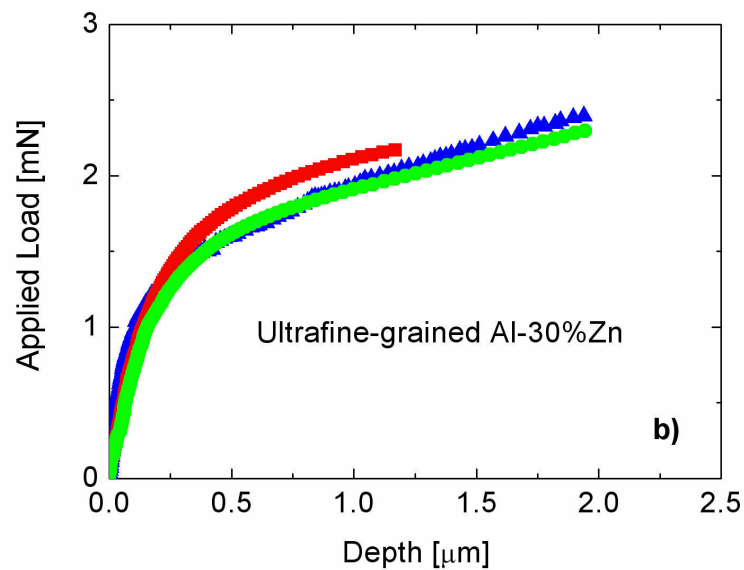
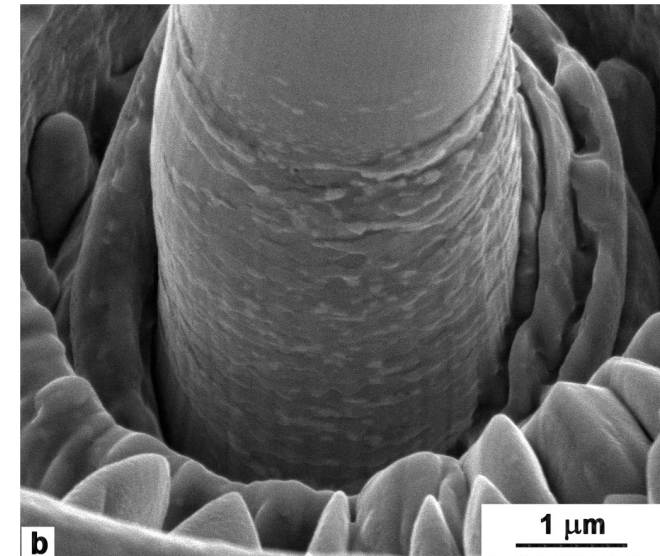
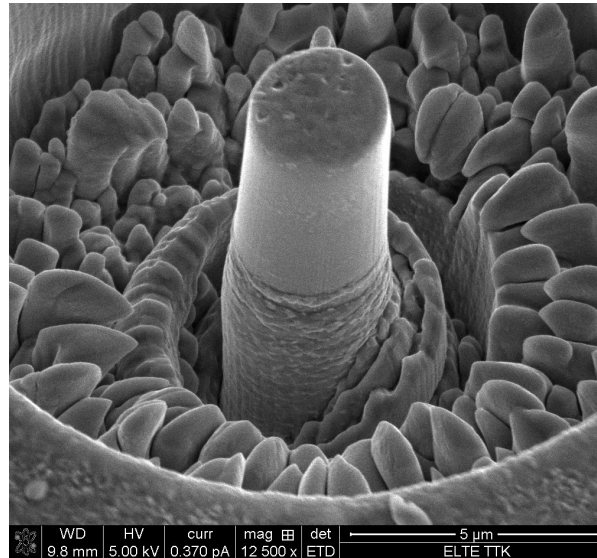
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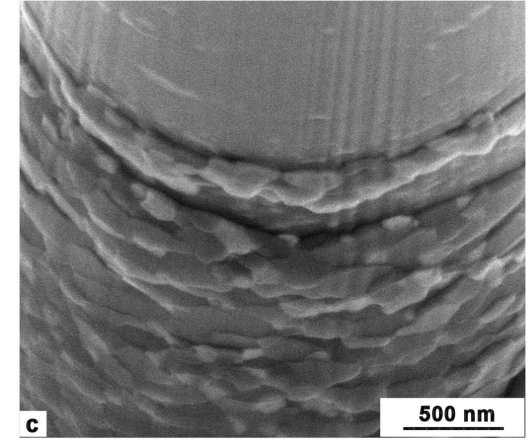
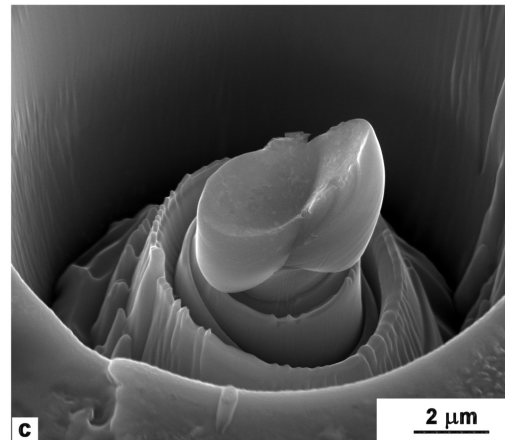
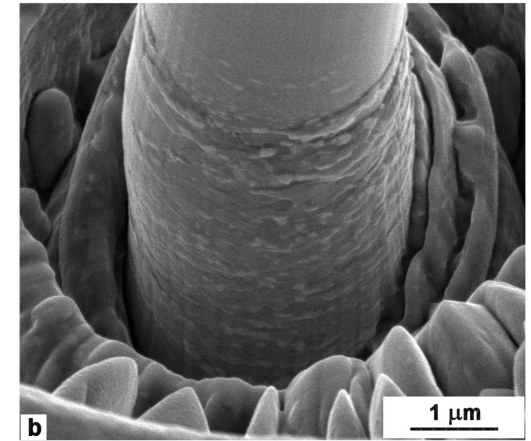
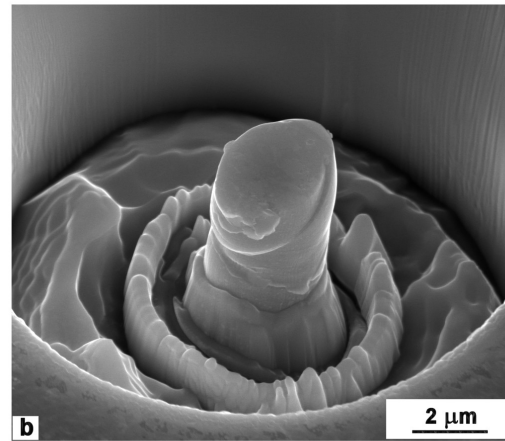
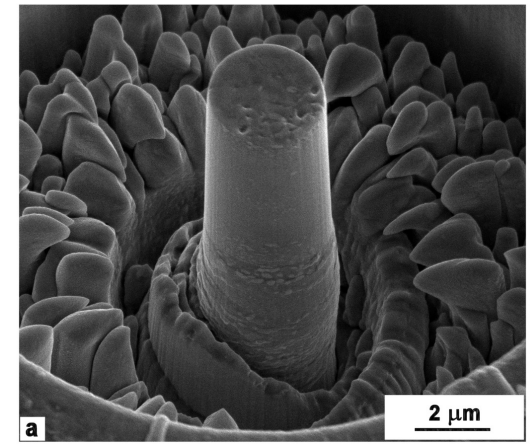
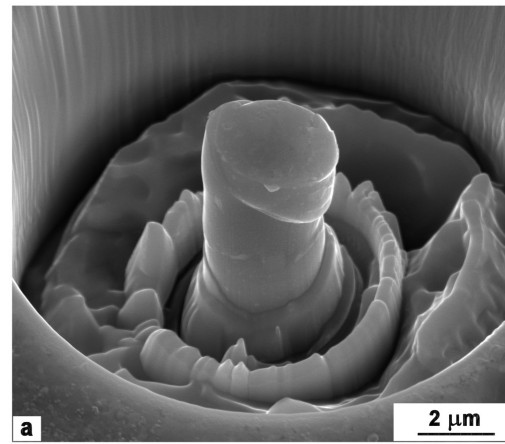
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**Deformation mechanism of the
Ultrafine-grained Al-30Zn alloy:**

***Unique plastic behavior of
micro-pillars of UFG Al-30Zn alloy***

The stable deformation by grain boundary sliding (GBS) of the ultrafine-grained (UFG) micro-pillars, without the occurrence of catastrophic avalanches, emphasizes the advantage of the GBS mechanism and suggests a potential for using the UFG materials in the fabrication of micro-devices.



Summary

●Microstructures and mechanical properties of HPT-processed UFG Al-Zn alloys were studied by using transmission electron microscopy (TEM) depth-sensing indentation (DSI), scanning electron microscope and focused ion beam (SEM/FIB), as well as by differential scanning calorimeter (DSC).

●For low Zn contents normal strengthening was dominant whereas for the highest Zn concentration an abnormal softening was observed due to microstructural decomposition.

●The strong microstructure decomposition in high Zn-concentrated alloy leads also to the formation of Zn-rich grain boundary layers which wet the Al/Al grain boundaries and enhance the role of grain boundary sliding in plasticity with an unusually high strain rate sensitivity.

●Occurrence of intensive grain boundary sliding at room temperature in the UFG Al-30wt%Zn alloy, leading to its super-ductility at room temperature.

●As a consequence of the role of the grain boundaries, the deformation process of UFG materials is relatively homogenous and this may have important practical implications for the electronics industry and especially for using these materials in micro-devices.

Acknowledgements

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-The Hungarian-Russian bilateral Research program (TÉT) No. 2017-2.3.4-TÉT-RU-2017-00005

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