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Nanoindentation, micropillar compression and nanoscratch testing of ZrB2 grains

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Introduction

Zirconium diboride (ZrB_2), owing to its high hardness, electrical and thermal conductivity, is an ideal candidate for ultra-high temperature ceramics. It can withstand extreme chemical and thermal environments and it finds applications in the field of cutting tools, plasma-arc electrodes and thermal protection system for hypersonic vehicles.

It is well known that the mechanical properties of bulk polycrystalline ceramics are strongly influenced by the texture. Thus, the investigation of the anisotropy on strength, hardness and wear resistance is important in order to understand its macroscopic mechanical properties.

Experimental

Material:
- spark plasma sintered (SPS) ZrB_2 by two steps SPS process

Method:
- grain orientation by SEM, EBSD
- pillar fabrication by FIB (8 pillars)
- micro-compression (flat-punch tip)
- nanoindentation (Berkovich tip, CSM mode, 900 indents, h_{max}=200 nm)
- nanoscratch testing (Berkovich tip, F=50, 100, 150 mN, l=200 μm)
- surface morphology by confocal microscope, SEM, AFM

Fig.1 Agilent G200 Nano Indenter

Plasticity and anisotropic deformation

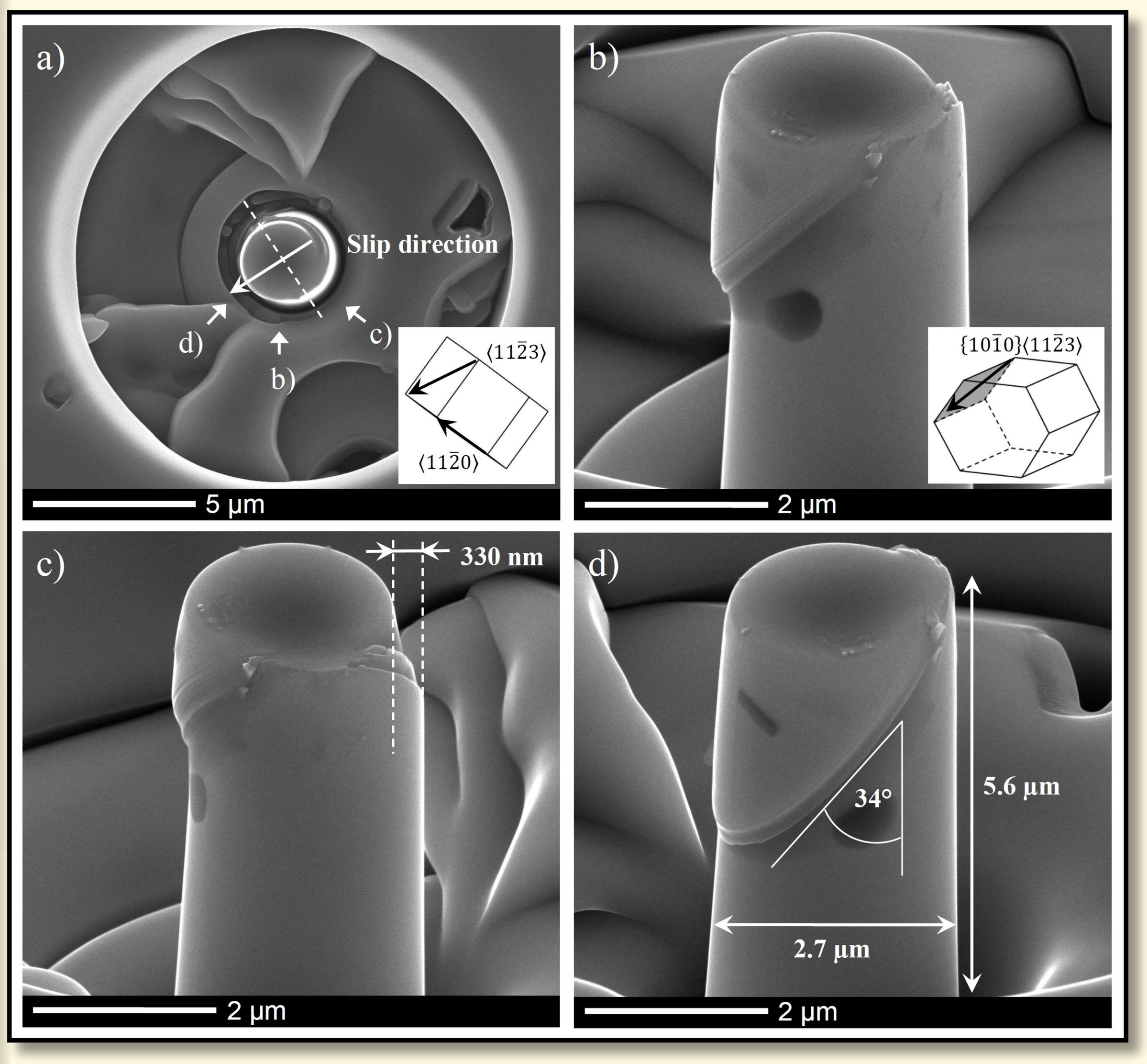


Fig.2 Single-slip activation of the {10-10}<11-23> system during micro-compression of a prismatic oriented pillar from a) top and b) side and side views after a rotation of c) -45° and d) 45° [1].

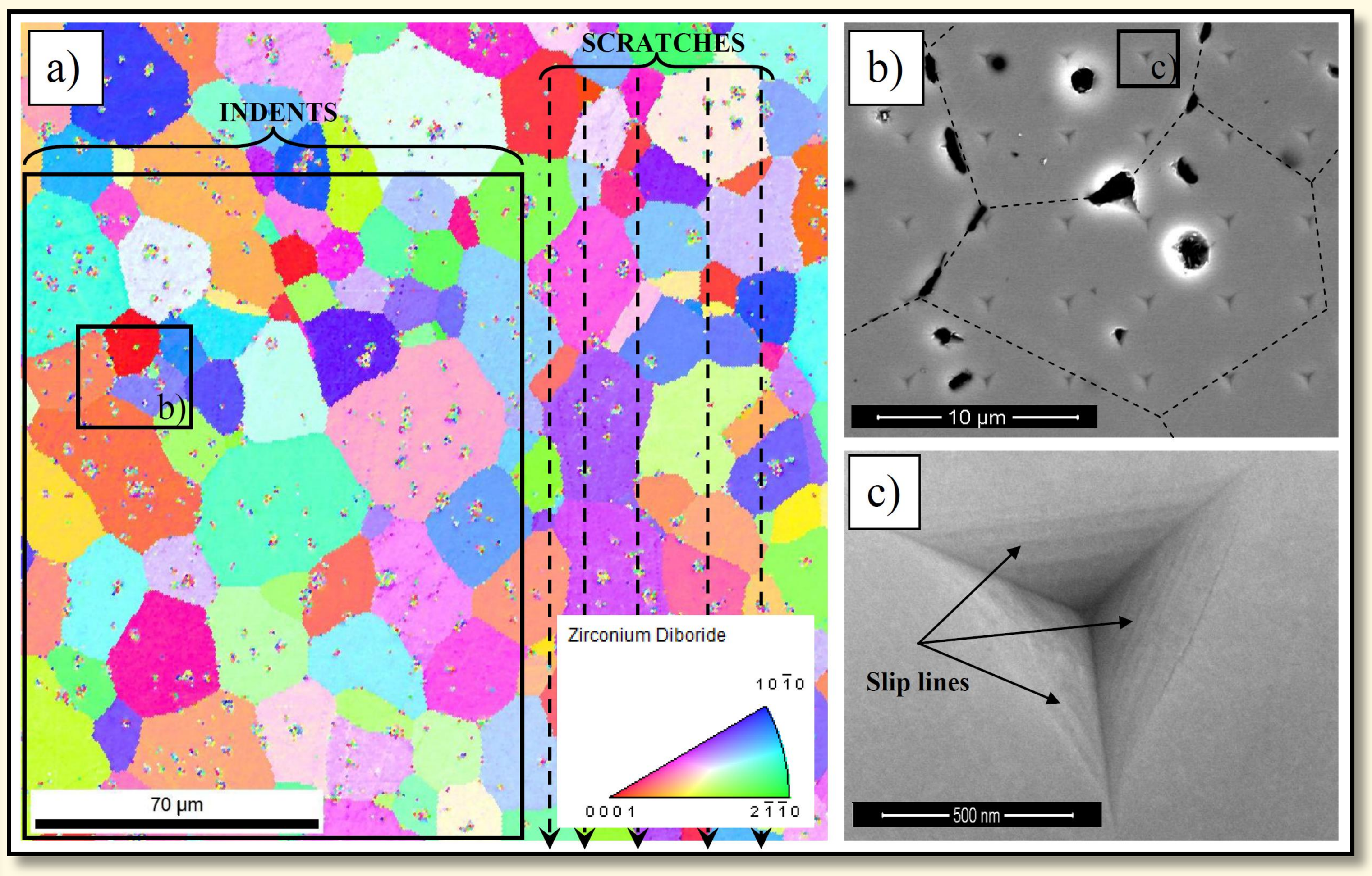


Fig.3 Characteristic maps of polycrystalline ZrB_2 made by a) EBSD prior to indentation and scratch testing; b) SEM on a particular part of the indented surface (grain boundaries are marked) together with; c) a selected indent located in grain close to basal orientation [2].

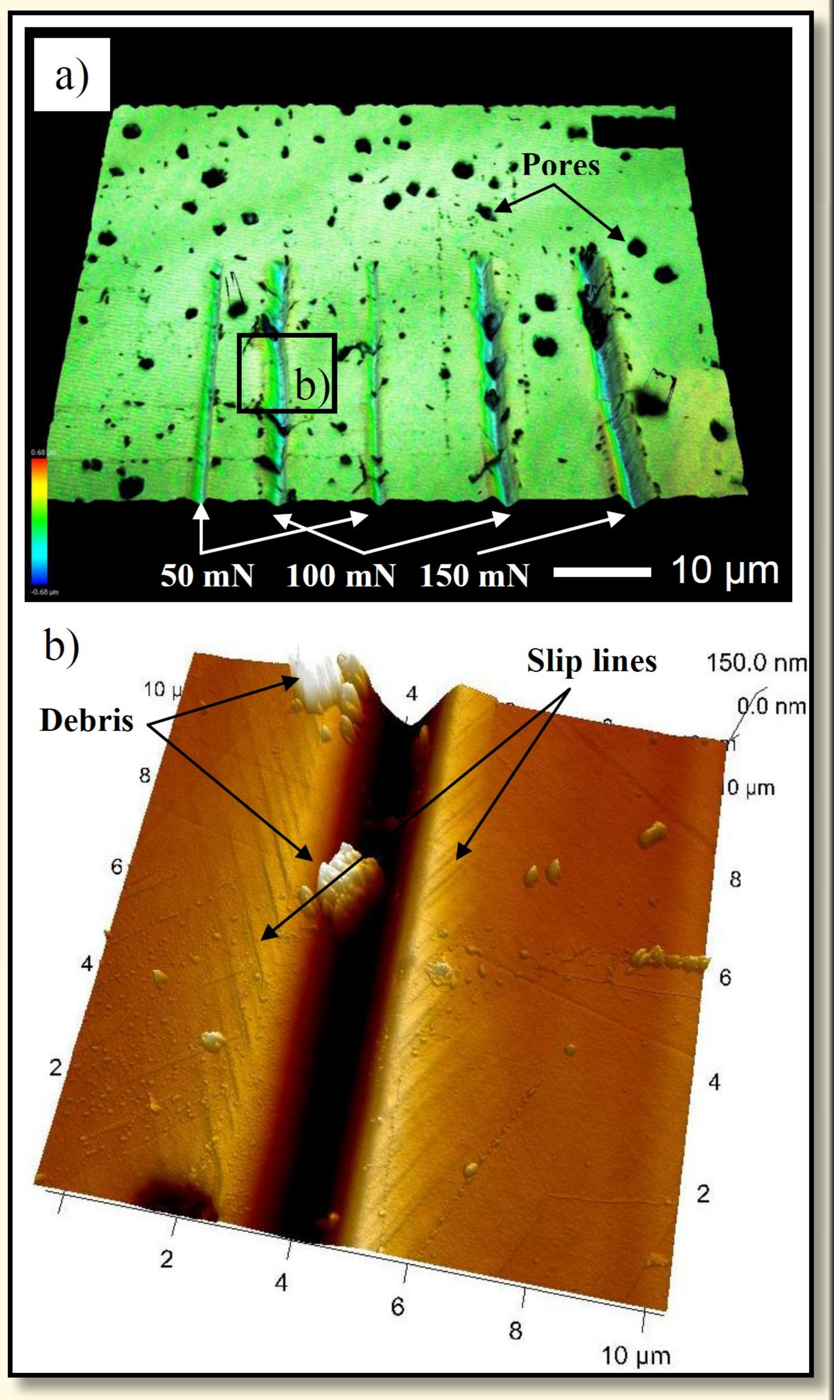


Fig.4 a) Confocal micrograph on scratch lines crossing differently oriented ZrB_2 grains together with b) the details of surface morphology around a particular part of scratch line corresponding to 100 mN load.

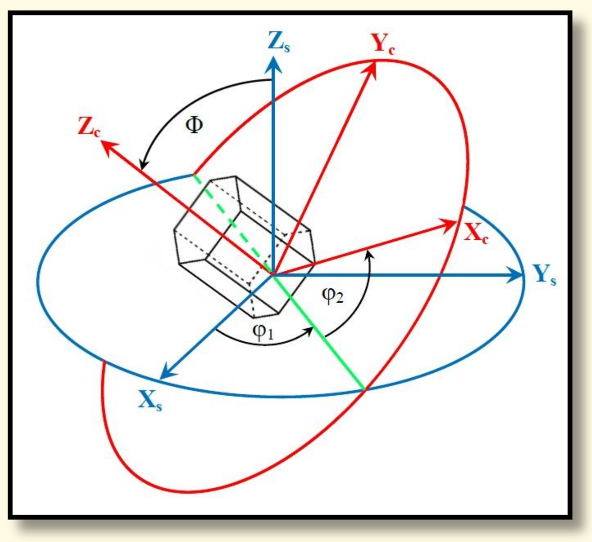
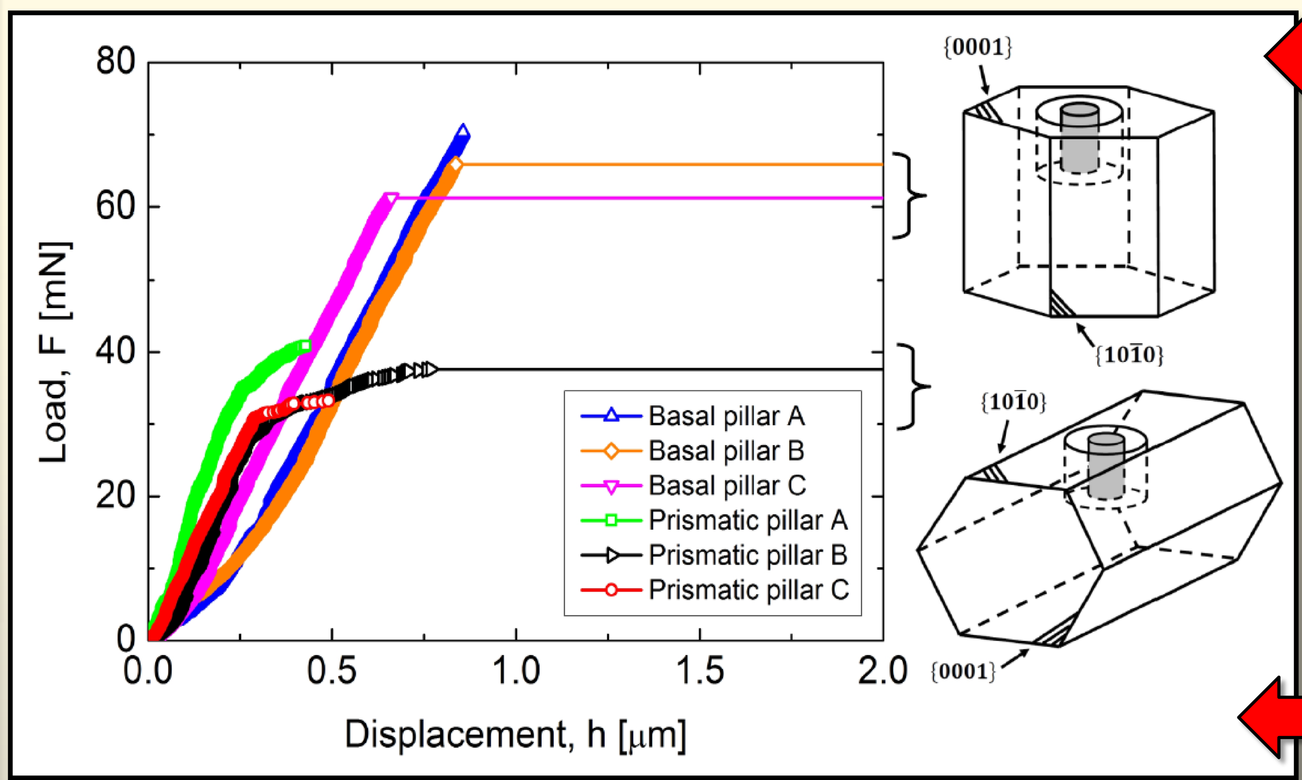


Fig.5 Schematic representation of the orientation of crystal relative to the sample coordinate system [3].

Fig.6 Characteristic load-displacement curves for basal and prismatic oriented pillars under micro-compression [1].



Φ-dependence is the most significant!!

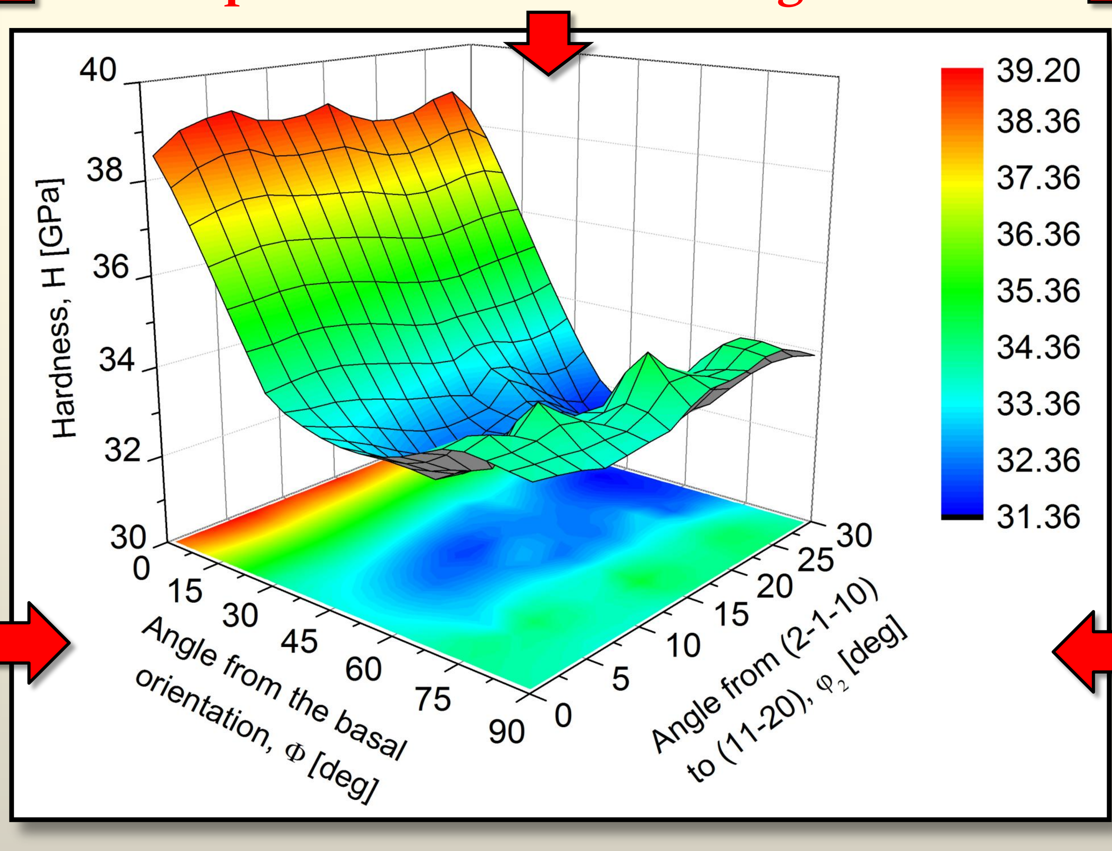


Fig.7 The orientation dependence of hardness of ZrB_2 grains in three-dimensional representation [2].

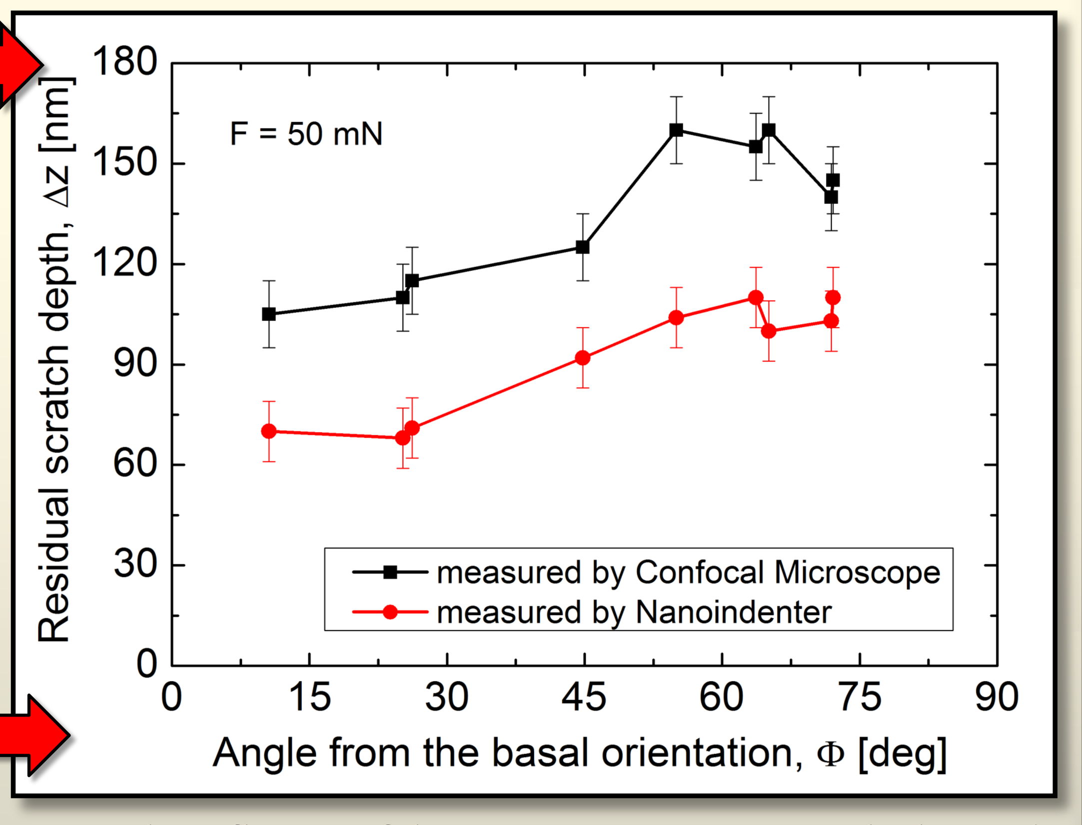


Fig.8 The influence of the grain orientation on residual scratch depth measured by confocal microscope and nanoindenter.

CORRELATION BETWEEN STRENGHT, HARDNESS AND WEAR RESISTANCE!!

Summary and conclusions

- Anisotropic deformation behavior with dislocation based plasticity is observed during micropillar compression, nanoindentation and nanoscratch testing [1,2] revealing the activation of the {10-10}<11-23> type slip system in prismatic oriented pillars [1].
- In all cases, the most significant factor on the orientation dependence is the rotation angle (Φ) between the axis of the hexagonal crystals and the surface normal [1,2,3].
- The anisotropic ratio of rupture stresses is around 1.8 with values of $\sigma_{r,basal}$=13.4 GPa and $\sigma_{r,prism}$=7.6 GPa, respectively [1].
- The nanohardness decreases from the basal towards prismatic orientations with a minimum value corresponding to a rotation angle of ~50°-60° [2].
- The residual scratch depth of ZrB2 crystals shows an increase from the basal orientation towards the prismatic and it is in agreement with recent studies on strength of micropillars and nanohardness anisotropy [1,2].

References

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