



## NanoTest High Temperature Publications

### Introduction

Material properties can vary greatly with changes in temperature. Thus, when developing or characterising the mechanical properties of coatings and bulk materials for high temperature applications, test conditions should mimic in-service conditions as closely as possible.

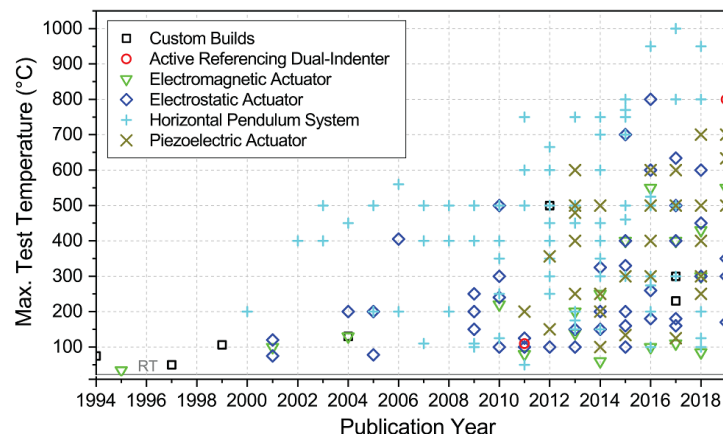
Nanomechanical tests have been performed with Micro Materials NanoTest systems at test temperatures up to 1000 °C. This has led to a number of publications on a wide range of materials.

In the tables on pages 2-5 of this note, published NanoTest studies are grouped into several categories based on the material tested:

- (1) Nuclear materials
- (2) PVD coatings for cutting tools
- (3) Aerospace materials
- (4) Materials with other high temperature applications

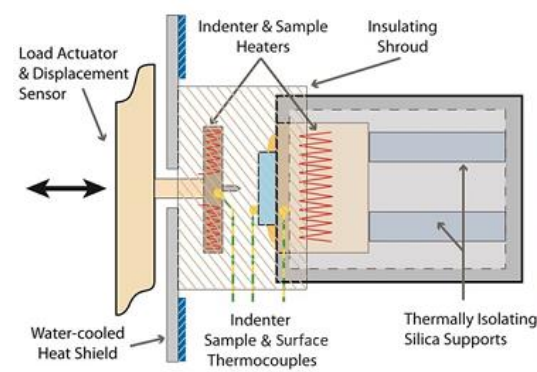
The maximum test temperature and year of publication is shown for each reference. The choice of indenter material and test environment is influenced by the sample being tested and the test temperature range. These factors are discussed in detail in *Nanomechanics to 1000 °C for high temperature mechanical properties of bulk materials and hard coatings* [ref 48].

The figure below shows the maximum published nanomechanical testing temperatures for various nanoindenter system configurations up to 2019. The dominance of the NanoTest (+) is demonstrated, in particular for temperatures > 500 °C.



### Important factors for High Temperature Testing

When measuring at elevated temperatures, it is essential that the sample and indenter are the same temperature when the indentation takes place. Any temperature mismatch will result in higher thermal drift, i.e. measurement error, caused by an expansion or contraction of the sample, indenter or instrument.



*The horizontal high temperature configuration of the NanoTest (left) and a close-up view of an indenter and sample at 950 °C in vacuum (right). (Figures above left and below left courtesy of Dr Jeff Wheeler, ETH Zurich.)*

NanoTest systems have design advantages which result in ultra-low thermal drift up to the maximum temperatures of 850 °C for the Vantage system, and 1000 °C for the NanoTest Xtreme:

**Active tip heating** – the indenter and the sample are both actively and independently heated, resulting in an isothermal contact *before* the experiment begins.

**Horizontal loading** – the unique load head configuration of the NanoTest systems means that there is no heat flow onto the loading head or depth measurement sensor.

**Highly localised heating** – a heat shield and insulating shroud around the heated zone ensures instrument stability during high temperature experiments.

**Patented control protocol** – software routines are used to precisely match the indenter and stage temperatures to  $\pm 0.1$  °C.

**Time-dependent measurements** – As no significant thermal drift occurs during high temperature measurements it becomes possible to perform long duration tests such as indentation creep tests and SPM imaging.

## 1. Nuclear materials

| Materials System               | Indenter material | Test environment       | Max. test temp. (°C) | Year             | Reference |
|--------------------------------|-------------------|------------------------|----------------------|------------------|-----------|
| 304 austenitic stainless steel | cBN               | argon                  | 300                  | 2015             | 1         |
| Zr-2.5%Nb                      | sapphire          | air                    | 400                  | 2011             | 2         |
| Nanoscale metallic multilayers | diamond           | argon                  | 400                  | 2013, 2014, 2018 | 3-6       |
| SiC in TRISO fuel particles    | diamond           | air                    | 500                  | 2015, 2016       | 7, 8      |
| SiC-SiC composite              | cBN               | 95%Ar/5%H <sub>2</sub> | 500                  | 2019             | 9         |
| PM2000 ODS alloy               | cBN               | argon                  | 600                  | 2014             | 10        |
| PH 13-8 Maraging steel         | cBN               | 95%Ar/5%H <sub>2</sub> | 625                  | 2016             | 11        |
| W-1%Ta alloy                   | cBN               | vacuum                 | 700                  | 2020             | 12        |
| Tungsten                       | cBN               | vacuum                 | 750                  | 2015             | 13        |
| Inconel 617                    | cBN               | air                    | 800                  | 2017             | 14        |
| Tungsten                       | cBN               | vacuum                 | 950                  | 2015, 2017, 2018 | 15-17     |
| Pyrolytic graphite             | diamond           | vacuum                 | 600                  | 2022             | 50        |
| δ-phase zircaloy hydride       | diamond           | nitrogen               | 300                  | 2020             | 51        |
| Inconel 718                    | cBN               | nitrogen               | 650                  | 2019             | 54        |
| Uranium dioxide                | cBN               | argon                  | 500                  | 2021             | 55        |
| Uranium dioxide                | cBN               | argon                  | 600                  | 2019             | 56        |
| ODS steels                     | cBN               | argon                  | 600                  | 2019             | 61        |
| Inconel 625/BNi-2              | diamond           | air                    | 490                  | 2020             | 69        |
| Inconel 617                    | diamond           | air                    | 200                  | 2016, 2018       | 72, 73    |
| Tungsten carbide               | diamond           | vacuum                 | 600                  | 2023             | 75        |

## 2. PVD hard coatings

| Materials System              | Indenter material | Test environment | Max. test temp.<br>(°C) | Year       | Reference |
|-------------------------------|-------------------|------------------|-------------------------|------------|-----------|
| TiAlN and TiN                 | diamond           | air              | 300                     | 2019       | 18        |
| TiAlN                         | diamond           | air              | 350                     | 2012       | 19        |
| TiAlN, AlCrN                  | diamond           | air              | 500                     | 2006       | 20        |
| AlTiN                         | diamond           | air              | 500                     | 2006, 2008 | 21-23     |
| TiAlN                         | diamond           | air              | 500                     | 2007       | 24        |
| TiAlCrSiYN/TiAlCrN            | cBN               | argon            | 600                     | 2012       | 25, 26    |
| TiAlSiN                       | cBN               | argon            | 600                     | 2019       | 27        |
| SiC, SiCN                     | cBN               | argon            | 650                     | 2015       | 28        |
| TiAlN, TiCN                   | cBN               | argon            | 750                     | 2014       | 29        |
| Ti <sub>2</sub> AlN MAX phase | sapphire          | vacuum           | 800                     | 2022       | 62        |
| DLC coatings                  | diamond           | air              | 400                     | 2020       | 66        |
| Al/SiC nanolaminates          | diamond           | air              | 150                     | 2020       | 67        |
| TiN/ZrN nanolaminates         | cBN               | argon            | 450                     | 2023       | 70        |
| Nanolayered CrAlTiN           | cBN               | argon            | 700                     | 2014       | 71        |

## 3. Aerospace materials

| Materials System                     | Indenter material | Test environment | Max. test temp.<br>(°C) | Year | Reference |
|--------------------------------------|-------------------|------------------|-------------------------|------|-----------|
| Ni-base superalloys                  | sapphire          | argon            | 400                     | 2008 | 32, 33    |
| Ni-base superalloy                   | sapphire          | vacuum           | 665                     | 2012 | 34        |
| Ni-base superalloy, MCrAlY bond coat | sapphire          | vacuum           | 1000                    | 2017 | 35        |
| SiC ceramic matrix composites        | sapphire          | argon            | 800                     | 2021 | 49        |

#### 4. Other materials

| Materials System                           | Indenter material | Test environment       | Max. test temp. (°C) | Year | Reference |
|--------------------------------------------|-------------------|------------------------|----------------------|------|-----------|
| (Pr,Ce)O <sub>2-δ</sub> cathode material   | cBN               | argon/N <sub>2</sub>   | 600                  | 2016 | 30        |
| G18 glass-ceramic                          | cBN               | argon                  | 750                  | 2011 | 31        |
| δ-Mg <sub>17</sub> Al <sub>12</sub> phase  | sapphire          | air                    | 278                  | 2016 | 36        |
| Magnesium                                  | diamond           | air                    | 300                  | 2015 | 37        |
| NiTiHf shape memory alloy                  | diamond           | air                    | 340                  | 2017 | 38        |
| MgAl <sub>2</sub> O <sub>4</sub> spinel    | diamond           | air                    | 400                  | 2009 | 39        |
| Silicon (100)                              | diamond           | air                    | 400                  | 2009 | 40        |
| AlCu alloy                                 | cBN               | argon                  | 460                  | 2016 | 41        |
| CuNb composite                             | cBN               | argon                  | 500                  | 2015 | 42        |
| Fused silica                               | cBN               | argon                  | 600                  | 2011 | 43        |
| Gold                                       | sapphire          | vacuum                 | 665                  | 2012 | 34        |
| CVD Al <sub>2</sub> O <sub>3</sub> coating | cBN               | 95%Ar/5%H <sub>2</sub> | 700                  | 2015 | 44        |
| WC-Co                                      | cBN               | vacuum                 | 700                  | 2020 | 45        |
| Silicon                                    | cBN               | vacuum                 | 770                  | 2017 | 46        |
| Cr <sub>2</sub> AlC MAX-phase              | sapphire          | vacuum                 | 980                  | 2019 | 47        |
| Ti-based bulk metallic glasses             | diamond           | argon                  | 400                  | 2020 | 52        |
| Bulk metallic glasses                      | diamond           | air                    | 200                  | 2020 | 53        |
| Sintered nano-copper                       | diamond           | air                    | 200                  | 2022 | 57        |
| magnesium/carbon nanotube nanocomposites   | cBN               | argon                  | 300                  | 2021 | 58        |

| Materials System                              | Indenter material | Test environment | Max. test temp. (°C) | Year | Reference |
|-----------------------------------------------|-------------------|------------------|----------------------|------|-----------|
| SiC/Al–Cu nanocomposites                      | diamond           | air              | 300                  | 2022 | 59        |
| MgCoNiCuZnO Entropy stabilized oxide          | cBN               | air              | 950                  | 2022 | 60        |
| Al/SiC Composites                             | diamond           | air              | 500                  | 2002 | 63        |
| Fe-based metallic glass                       | diamond           | air              | 450                  | 2021 | 64        |
| SmNiO <sub>3</sub> films                      | cBN               | air              | 200                  | 2019 | 65        |
| KH <sub>2</sub> PO <sub>4</sub> (KDP) crystal | diamond           | air              | 160                  | 2021 | 68        |
| WC-Co (3D printed)                            | cBN               | air              | 600                  | 2022 | 74        |
| NiCoCr medium entropy alloy                   | cBN               | air              | 750                  | 2022 | 76        |

## References

- H. Vo et al. Small-Scale Mechanical Testing on Proton Beam-Irradiated 304 SS from Room Temperature to Reactor Operation Temperature, *JOM* 67 (2015) 2959.
- B. Bose et al. Temperature dependence of the anisotropic deformation of Zr-2.5%Nb pressure tube material during micro-indentation, *J. Nucl. Mater.* 419 (2011) 235.
- M.A. Monclús et al. Optimum high temperature strength of two-dimensional nanocomposites, *APL Materials* 1 (2013) 052103.
- M.A. Monclús et al. Microstructure and mechanical properties of physical vapor deposited Cu/W nanoscale multilayers: Influence of layer thickness and temperature, *Thin Solid Films* 571 (2014) 275.
- M.A. Monclús et al. Effect of layer thickness on the mechanical behaviour of oxidation-strengthened Zr/Nb nanoscale multilayers, *J. Mater. Sci.* 53 (2018) 5860.
- L.W. Yang et al. Mechanical properties of metal-ceramic nanolaminates: effect of constraint and temperature, *Acta Mater.* 142 (2018) 37.
- N. Rohbeck et al. In-situ nanoindentation of irradiated silicon carbide in TSIRO particle fuel up to 500 °C, *J. Nucl. Mater.* 465 (2015) 692.
- N. Rohbeck et al. Evaluation of the mechanical performance of silicon carbide in TRISO fuel at high temperatures, *Nuclear Engineering and Design* 306 (2016) 52.
- D. Frazer et al. High-Temperature Nanoindentation of SiC/SiC Composites, *JOM* 2019; doi:10.1007/s11837-019-03860-7.
- Z. Huang et al. Nanoindentation creep study on an ion beam irradiated ODS alloy, *J. Nucl. Mater.* 451 (2014) 162.
- Z. Huang et al. A high temperature mechanical study on PH 13-8 maraging steel, *Mater. Sci. Eng. A* 651 (2016) 574.
- B.-S. Li et al. Measuring the brittle-to-ductile transition temperature of tungsten–tantalum alloy using chevron-notched micro-cantilevers, *Scr. Mater.* 180 (2020) 77.
- J.S.K.-L. Gibson et al. High-temperature indentation of helium-implanted tungsten, *Mater. Sci. Eng. A* 625 (2015) 38.
- Y. Zhang et al. High temperature indentation-based property measurements of inconel IN-617, *Int. J. Plasticity*, 96 (2017) 264.
- A.J. Harris et al. Extreme nanomechanics: vacuum nanoindentation and nanotribology to 950 °C, *Tribology* 9 (2015) 174.
- A.J. Harris et al. Development of high temperature nanoindentation methodology and its application in the nanoindentation of polycrystalline tungsten in vacuum to 950°C, *ExpMech* 57 (2017) 1115.
- B.D. Beake et al. Temperature dependence of strain rate sensitivity, indentation size effects and pile-up in polycrystalline tungsten from 25-950 °C, *Mater. Design* 156 (2018) 278.
- F. Giuliani et al. Deformation behaviour of TiN and Ti-Al-N coatings at 295 to 573 K, *Thin Solid Films* 688 (2019) 137363.
- V. Bhakhri et al. Instrumented nanoindentation investigation into the mechanical behavior of ceramics at moderately elevated temperatures, *J. Mater. Res.* 27 (2012) 65.
- G.S. Fox-Rabinovich et al. Impact of mechanical properties measured at room and elevated temperatures on wear resistance of cutting tools with TiAlN and AlCrN coatings, *SCT* 200 (2006) 5738.
- G.S. Fox-Rabinovich et al. Impact of annealing on the microstructure, properties, and cutting performance of AlTiN coating, *Surf. Coat. Technol.* 201 (2006) 3524.
- G.S. Fox-Rabinovich et al. Effect of annealing below 900°C on structure, properties and tool life of an AlTiN coating under various cutting conditions, *Surf. Coat. Technol.* 202 (2008) 2985.
- B.D. Beake et al. Coating optimisation for high-speed machining with advanced nanomechanical test methods, *SCT* 203 (2009) 1919.
- B.D. Beake et al. Investigating the correlation between nano-impact fracture resistance and hardness/modulus ratio from nanoindentation at 25-500°C and the fracture resistance and lifetime of cutting tools..., *Surf. Coat. Technol.* 201 (2007) 4585.

25. B.D. Beake et al. Why can TiAlCrSiYN-based adaptive coatings deliver exceptional performance under extreme frictional conditions? *Faraday Discussions* 156 (2012) 267.
26. G.S. Fox-Rabinovich et al. Mechanism of adaptability for the nanostructured TiAlCrSiYN-based hard physical vapor deposition coatings under extreme frictional conditions, *J. Appl. Phys.* 111 (2012) 064306.
27. B.D. Beake et al. Elevated temperature micro-impact testing of TiAlSiN coatings produced by physical vapour deposition, *Thin Solid Films* 688 (2019) 137358.
28. R. Ctvrtlik et al. Mechanical properties of amorphous silicon carbonitride thin films at elevated temperatures, *J. Mater. Sci.* 50 (2015) 1553.
29. B.D. Beake et al. Progress in high temperature nanomechanical testing of coatings for optimising their performance in high speed machining, *Surf. Coat. Technol.* 255 (2014) 102.
30. J.G. Swallow et al. Operando reduction of elastic modulus in (Pr, Ce)O<sub>2</sub>- $\gamma$  thin films, *Acta Mater.* 105 (2016) 16.
31. J. Milhans et al. Mechanical properties of solid oxide fuel cell glass-ceramic seal at high Temperatures, *J Power Sources* 196 (2011) 5599.
32. A. Sawant et al. High temperature nanoindentation of a Re-bearing single crystal Ni-base superalloy, *Scr. Mater.* 58 (2008) 275.
33. A. Sawant et al. High temperature nanoindentation of Ni-base superalloys, *Superalloys2008*, Eds. RC Reed et al, TMS (2008) 863.
34. S.K. Korte et al. High temperature microcompression and nanoindentation in vacuum, *J. Mater. Res.* 27 (2012) 167.
35. J.S.K.-L. Gibson et al. On extracting mechanical properties from nanoindentation at temperatures up to 1000 °C, *Extreme Mechanics Letters* 17 (2017) 43.
36. H.N. Mathur et al. Deformation in the  $\gamma$ -Mg<sub>17</sub>Al<sub>12</sub> phase at 25-278 °C, *Acta Mater.* 113 (2016) 221.
37. M. Haghshenas et al. Effect of temperature and strain rate on the mechanisms of indentation deformation of magnesium, *MRS Comm.* 2015. doi:10.1557/mrc.2015.57
38. P. Li et al. Rapid characterization of local shape memory properties through indentation, *Sci. Rep* 7 (2017) 14827.
39. S. Korte et al. Micropillar compression of ceramics at elevated temperatures, *Scr. Mater.* 60 (2009) 807.
40. S. Korte et al. Deformation of silicon – insights from micro compression testing at 25–500 °C, *Int J Plasticity* 27 (2011) 1853.
41. S. Koch et al. A high temperature nanoindentation study of Al-Cu wrought alloy, *Mater. Sci. Eng. A* 6 (2015) 218.
42. M.-M. Primorac et al. Elevated temperature mechanical properties of novel ultra-fine grained Cu-Nb composites, *Mater. Sci. Eng. A* 625 (2015) 296.
43. N.M. Everitt et al. High temperature nanoindentation - the importance of isothermal contact, *Philos. Mag.* 91 (2011) 1221.
44. M. Rebelo de Figueiredo et al. Nanoindentation of chemical-vapor deposited Al<sub>2</sub>O<sub>3</sub> hard coatings at elevated temperatures, *Thin Solid Films* 578 (2015) 20.
45. F. De Luca et al. Nanomechanical Behaviour of Individual Phases in WC-Co Cemented Carbides, from Ambient to High Temperature, *Materialia* 12 (2020) 100713.
46. D.E.J. Armstrong et al. Bending testing of Silicon Cantilevers from 21°C to 770°C, *JOM* 67 (2015) 2914.
47. J.S.K.-L. Gibson et al. Mechanical characterisation of the protective Al<sub>2</sub>O<sub>3</sub> scale in Cr<sub>2</sub>AlC MAX Phases, *J.Eur.Ceram.Soc.* 39 (2019) 5149.
48. B.D. Beake et al. Nanomechanics to 1000 °C for high temperature mechanical properties of bulk materials and hard coatings, *Vacuum* 159 (2019) 17.
49. C.H. Bumgardner et al. Probing the local creep mechanisms of SiC/SiC ceramic matrix composites with high-temperature nanoindentation, *Journals of Materials Research* 36 (2021) 2420-2433.
50. T.J. Marrow et al. High temperature spherical nano-indentation of graphite crystals, *Carbon* 191 (2022) 236-242.
51. H. Wang et al. Constitutive modelling of a  $\delta$ -phase zircaloy hydride based on strain rate dependent nanoindentation and nano-scale impact dataset, *International Journal of Plasticity* 133 (2020) 102787
52. Q. Zhou et al. A nanoindentation study of Ti-based high entropy bulk metallic glasses at elevated temperatures, *Journal of Non-Crystalline Solids* 532 (2020) 119878
53. Q. Zhou et al. Identifying the high entropy effect on plastic dynamics in bulk metallic glasses: A nanoindentation study at room and elevated temperature, *Materials & Design* 189 (2020) 108500.
54. H. Wang et al. Nanoindentation based properties of Inconel 718 at elevated temperatures: A comparison of conventional versus additively manufactured samples, *International Journal of Plasticity*, 120 (2019) 380-394.
55. D. Frazer et al. Elevated temperature nanoindentation creep study of plastically deformed and spark plasma sintered UO<sub>2</sub>, *Journal of Nuclear Materials* 545 (2021) 152605.
56. B. Gong et al. Nano- and-micro-indentation testing of sintered UO<sub>2</sub> fuel pellets with controlled microstructure and stoichiometry, *Journal of Nuclear Materials* 516 (2019) 169-177.
57. J. Fan et al. High-temperature nanoindentation characterization of sintered nano-copper particles used in high power electronics packaging, *Results in Physics* 33 (2022) 105168.
58. J. Thornby et al. Micromechanics and indentation creep of magnesium carbon nanotube nanocomposites: 298K-573K, *Materials Science and Engineering: A* 801 (2021) 140418.
59. X.M. Mei et al. Achieving enhanced mechanical properties of SiC/Al-Cu nanocomposites via simultaneous solid-state alloying of Cu and dispersing of SiC nanoparticles, *Materials Science and Engineering: A* 860 (2022) 144338.
60. C.M. Rost et al. On the thermal and mechanical properties of Mg<sub>0.2</sub>Co<sub>0.2</sub>Ni<sub>0.2</sub>Cu<sub>0.2</sub>Zn<sub>0.2</sub>O across the high-entropy to entropy-stabilized transition, *APL Mater* 10 (2022) 121108.
61. E. Wyszowska et al. Investigation of the mechanical properties of ODS steels at high temperatures using nanoindentation technique, *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms* 444 (2019) 107-111.
62. C. Tromas et al. Nanoindentation-induced deformation twinning in MAX phase Ti<sub>2</sub>AlN, *Acta Materialia* 227 (2022) 117665.
63. R.N. Sarafet al. Investigation and the thermal creep properties of Al/SiC composites made by the powder metallurgy technique, *Fourth Canada-Japan Workshop on Composites – CRC Press* (2020) 105-114.
64. C.C. Yuan et al. Anelastic and viscoplastic deformation in a Fe-based metallic glass, *Journal of Alloys and Compounds* 853 (2021) 157233.
65. J. Chen et al. Mechanical and correlated electronic transport properties of preferentially oriented SmNiO<sub>3</sub> films, *Ceramics International* 46 (2020) 6693-6697.
66. E. Bouzakis, Fatigue endurance assessment of DLC coatings on high-speed steels at ambient and elevated temperatures, *Coatings* 10 (2020) 547.
67. Y. Chen et al. High temperature indentation creep mechanism of metal-ceramic nanolaminates, *Mater. Sci. Eng. A* 802 (2021).
68. Q. Liu et al. On the improvement of the ductile removal ability of brittle KDP crystals via temperature-effect, *Ceram. Int.* (2021).
69. Y.-C. Zhang et al. Elastic modulus and hardness characterisation for microregion of Inconel 625/BNi-2 vacuum brazed joint by high temperature indentation, *Vacuum* 181 (2020) 109582.
70. S. Shen et al. Deformation mechanism and mechanical properties of TiN/ZrN nanolaminates by nanoindentation: effect of layer thickness and temperature, *Surf. Coat. Technol.* 455 (2022) 129230.
71. T. Polcar et al. High temperature behavior of nanolayered CrAlTiN coating: thermal stability, oxidation and tribological properties, *Surf. Coat. Technol.* 257 (2014) 70-77.
72. Y. Zhang et al. Visualizing in situ microstructure dependent crack tip stress distribution in IN-617 using nano-mechanical Raman spectroscopy, *JOM* 68 (2016) 2742-2747.
73. Y. Zhang et al. Visualizing stress and temperature distribution during elevated temperature deformation of IN-617 using nano-mechanical Raman spectroscopy, *JOM* 70 (2018) 464-468.
74. G. Rui et al. Small-scale mechanical response at intermediate/high temperature of 3D printed WC-Co, *Procedia CIRP* 108 (2022) 507-512.
75. F. De Luca et al. Nanomechanical response of tungsten carbide single crystals in extreme conditions: Temperature and strain rate dependence, *Materialia* 27 (2023) 101706.
76. B.B. Zhang et al. Inhibiting creep in nanograined alloys with stable grain boundary networks, *Science* 378 (2022) 659-663.b

