Micro Materials
Excellence in Nanomechanics

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.

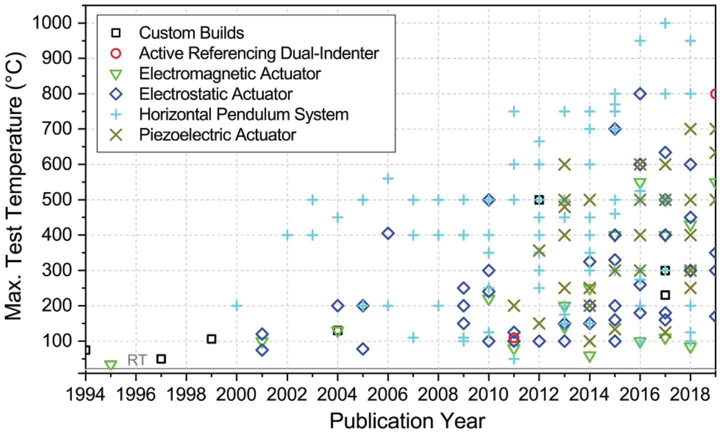
Nanomechanical tests have been performed with Micro Materials NanoTest systems at test temperatures up to 1000 °C. This has led to a number of publications on a wide range of materials.

In the tables on pages 2-3 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) fuel cell materials
(4) aerospace materials
(5) materials with other high temperature applications

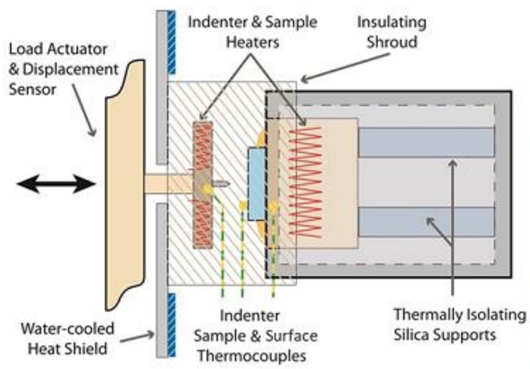
Within each category the published studies have been listed in order of the maximum test temperature. The choice of indenter material and test environment is influenced by the sample being tested and the test temperature range. These factors are discussed in detail in *Nanomechanics to 1000 °C for high 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 ±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.

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_2	500	2019	9
PM2000 ODS alloy	cBN	argon	600	2014	10
PH 13-8 Maraging steel	cBN	95%Ar/5%H_2	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

2. PVD hard coatings

Materials System	Indenter material	Test environment	Max. test temp. (°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, 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

3. Fuel cell materials

Materials System	Indenter material	Test environment	Max. test temp. (°C)	Year	Reference
$(Pr,Ce)O_{2-\delta}$ cathode material	cBN	argon/N_2	600	2016	30
G18 glass-ceramic	cBN	argon	750	2011	31

4. Aerospace materials

Materials System	Indenter material	Test environment	Max. test temp. (°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

5. Other materials

Materials System	Indenter material	Test environment	Max. test temp. (°C)	Year	Reference
δ-$Mg_{17}Al_{12}$ phase	sapphire	air	278	2016	36
Magnesium	diamond	air	300	2015	37
NiTiHf shape memory alloy	diamond	air	340	2017	38
$MgAl_2O_4$ 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_2O_3 coating	cBN	95%Ar/5%H_2	700	2015	44
WC-Co	cBN	vacuum	700	2020	45
Silicon	cBN	vacuum	770	2017	46
Cr_2AlC MAX-phase	sapphire	vacuum	980	2019	47

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