Micro Materials
Excellence in Nanomechanics

High temperature nanoindentation of Refractory High Entropy Alloys
and
High strain rate experiments on fibre reinforced composites
IMDEA MATERIALS (Spain) – user case studies

Overview

At IMDEA, the nanoindentation technique is used to measure materials' hardness and Young's modulus as a function temperature for materials intended for use in severe environments, such as the new generation of High-Entropy Alloys (HEAs). A high-temperature chamber allows for measurements from RT up to 750 °C under a controlled atmosphere.

Refractory HEAs (i.e. MoNbTaW) are very attractive materials for high-temperature applications, such as higher service temperature combustion engines for the aerospace sector, which can result in superior yield of the combustion itself. In this work, the MoNbTaW system was optimized via directed energy deposition (DED) in-situ alloying with V additions and characterised for high-throughput composition screening [1].

The high strain rate characterization of polymers is of particular importance because these materials are highly rate-sensitive. New developments in this area will open the door to the calibration of micromechanical models of the impact behaviour of fibre-reinforced polymer composites, incorporating strain rate-dependent behaviour. A novel test set-up for high strain rate micropillar compression testing was proposed and applied to the study of the mechanical behaviour of an epoxy resin over a wide range of strain rates [2].

IMDEA NanoTest platform configuration

- Berkovich, Vickers, Cube Corner, Conical and Flat Punch indenters with nano-load head.
- HT measurements with diamond (up to 450°C) and cBN (up to 750°C) indenter tips for measurements under a controlled argon atmosphere.
- Optical microscope (up to 40x optical mag. > 3000x on-screen mag.)
- Load or depth-controlled modes.
- Nano-impact with an independent force sensor in direct contact with the sample.

High-temperature (HT) nanoindentation of RHEAs

Nanoindentation was performed at room temperature (RT), 350°C and 700°C. Each measurement is repeated 9 times with 50 microns spacing between the indents. All the indents are made by using a loading/unloading rate of 0.5 mN/sec and a dwell period for drift data correction of 60 s. Results revealed that V alloying of 22% promotes a 93% increase in hardness at RT and a 150% increase at 700 °C. After HT measurements, hardness increases considerably when compared to RT measurements. This phenomenon is related to a homogenisation of the structure during the HT measurement, resembling a heat treatment.

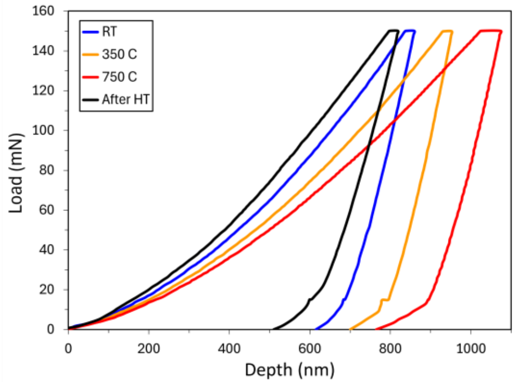

Figure. 1. Representative nanoindentation load-displacement curves for MoNbTaWV at the four temperatures tested.

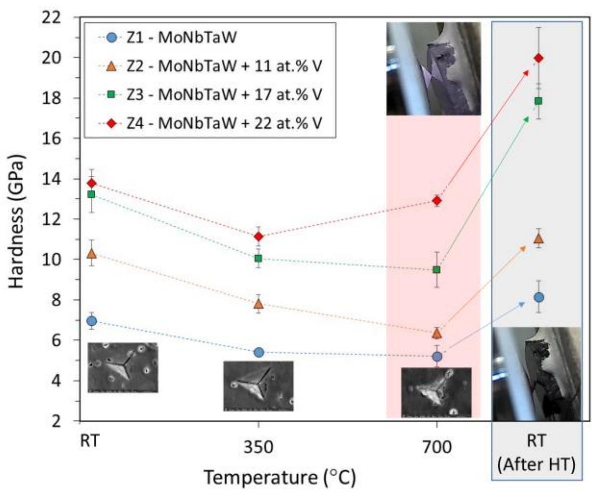

Figure 2. Hardness as a function of test temperatures for MoNbTaW with different V contents.

High strain rate experiments on polymers

A test set-up for high strain rate micropillar compression testing was applied to the study of the mechanical behaviour of an epoxy resin over a wide range of strain rates. The high strain rate test is based on impact loading, which is performed on the pendulum-based Nanotest platform modified to measure the actual force imposed on the sample via a piezoelectric force sensor. The micropillar compression tests on the epoxy resin revealed a significant strain rate sensitivity of the maximum compressive strength that correlated well with the values extracted from macroscopic compression tests in a wide range of strain rates.

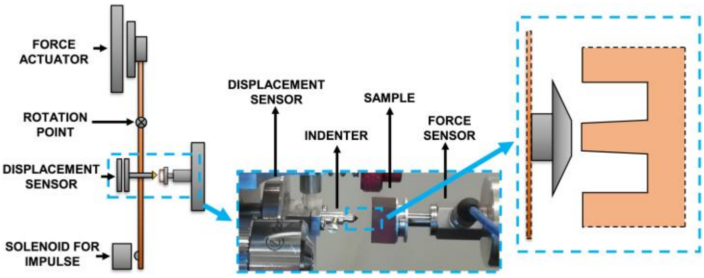

Fig. 3. Schematics of the pendulum impactor and the force sensor added to the setup.

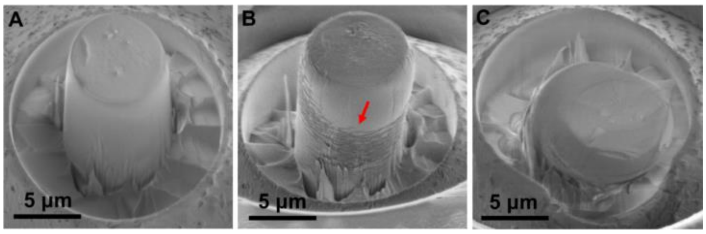

Fig. 4. SEM images of a pillar before testing (a), the same pillar after the low-speed test (b) and a pillar after the impact test (c).

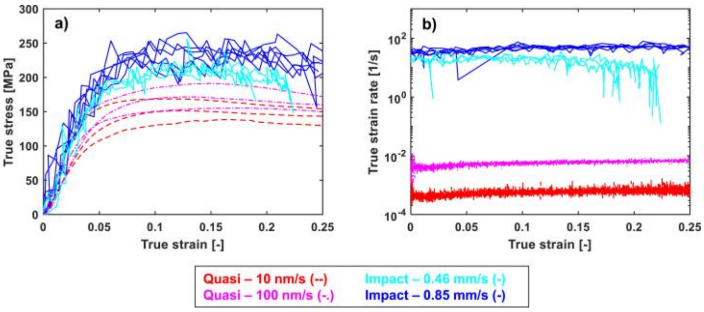

Fig. 5. Stress-strain curves from micropillar compression tests over a wide range of strain rates (10^{-3} to 10^{2} s^{-1}) and (b) true strain rate registered throughout the test.

User Profile

IMDEA Materials Institute is a leading research organisation in Spain. Recognized as a Maria de Maeztu excellence unit since 2019. Its objective is to perform research at the forefront of Materials Science and Engineering, attract talent from around the world and transfer technology to the industry. IMDEA Materials has an international reputation in the design, processing, characterization, modelling and simulation of advanced materials for applications in transportation, industry, energy and health. IMDEA Materials is made up of around 100 researchers from 15 nationalities organized into 16 research groups, whose activities revolve around 4 research programs: New materials; Advanced manufacturing; Multiscale characterization of materials and processes; Integrated computational materials engineering.

Highlights from the user

- Simple indenter mounting and stages that can accommodate large samples for high-temperature testing.
- Low thermal drift during high-temperature measurements.
- Excellent sensitivity in low load regime.
- Unique tool for the study of the high strain rate mechanical behaviour of all material types at the microscale.
- Important mechanical properties (elastic modulus, compressive strength, strain rate sensitivity) can be extracted from the stress–strain curves obtained from micropillar compression tests.

Quote from the user

"The thermal stability and large load range of the instrument, which can accommodate large samples, enables reliable high-temperature nanoindentation and micropillar compression tests"

References

1. *B.L. Ribeiro, M.A. Monclus, J.M. Torralba, M. Barbosa, R. F. Santos, E.W. Sequeiros, High-throughput screening of MoNbTaW-based Refractory High-Entropy Alloys through Direct Energy Deposition in-situ alloying of V, submitted to Materials Characterization.*

2. *M. Rueda-Ruiz, M. A. Monclus, B.D. Beake, F. Gálvez, J.M. Molina-Aldareguia, High strain rate compression of epoxy micropillars, Extreme Mechanics Letters, 40 (2020), 100905.*

Micro Materials Ltd

At the forefront of nanomechanics since 1988:

- First commercial high-temperature nanoindentation stage
- First commercial nano-impact stage
- First commercial liquid cell
- First commercial instrument for high-vacuum, high-temperature nanomechanics

Contact us:-

Micro Materials Ltd
Willow House, Yale Business Village,
Ellice Way, Wrexham, LL13 7YL, UK

Tel: +44(0) 1978 261615
E-mail: info@micromaterials.co.uk
www.micromaterials.co.uk