High temperature nanoindentation of bcc HEA NbMoCrTiAl

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Project: Identification of the intrinsic deformation mechanisms of single phase body-centered cubic high entropy alloys
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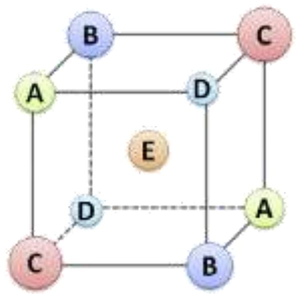
Background

- Representative composition of *bcc* refractory HEA with single phase

[*BCC structure*]

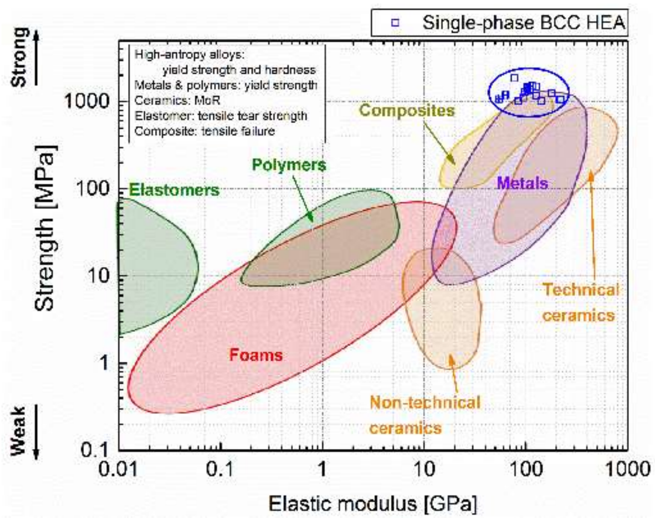


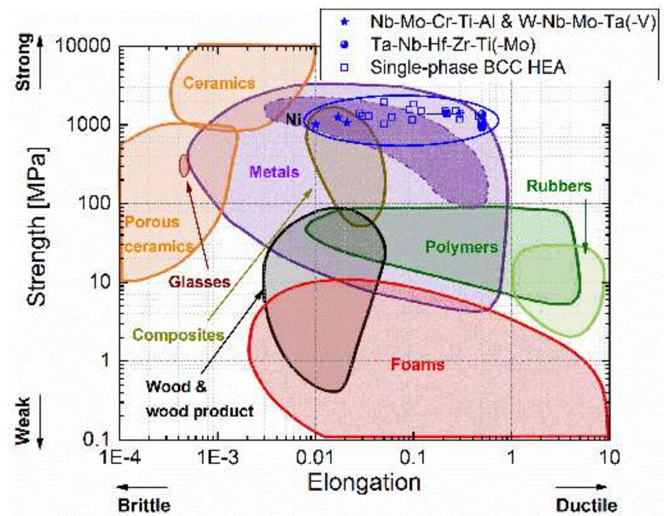
refractory elements with high melting point

Mo-Nb-Cr-Ti-Al,
Nb-Mo-Ta-W(-V),
Ta-Nb-Hf-Zr-Ti, …

✓ For high-temperature applications
- Superior strength
- Excellent thermal stability
- Oxidation resistance
- Other useful properties at room and high temperature

- Ashby maps showing properties of *bcc* refractory HEAs





Background

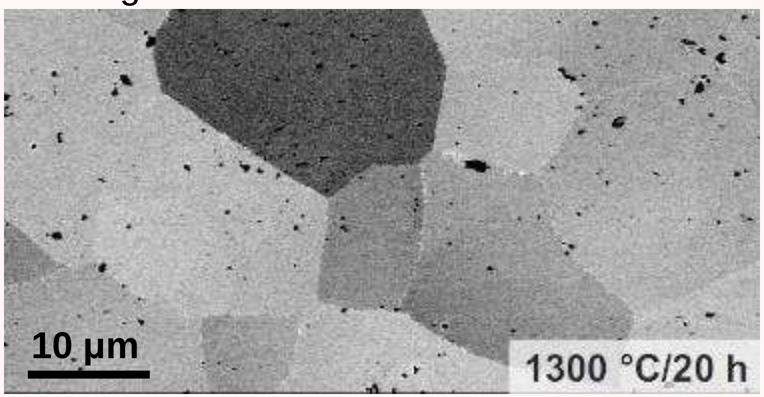
- Why NbMoCrTiAl?

passivating

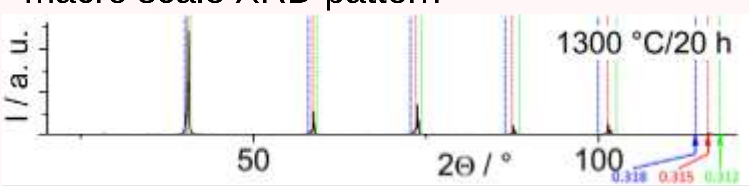
Nb-Mo-Cr-Ti-Al

high melting point   low density

* homogenized microstructure *

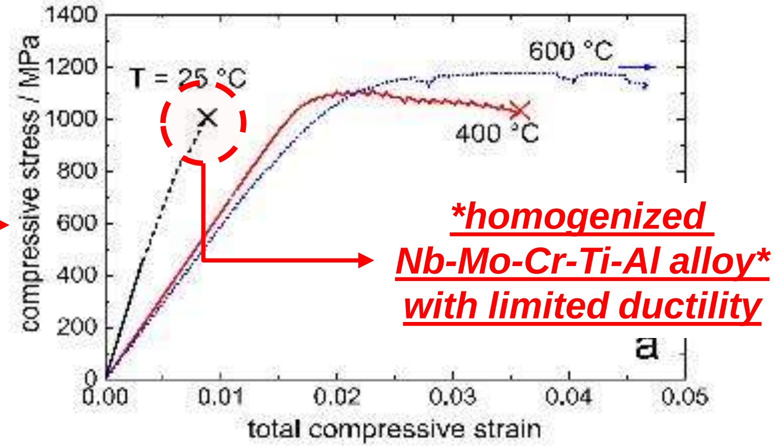


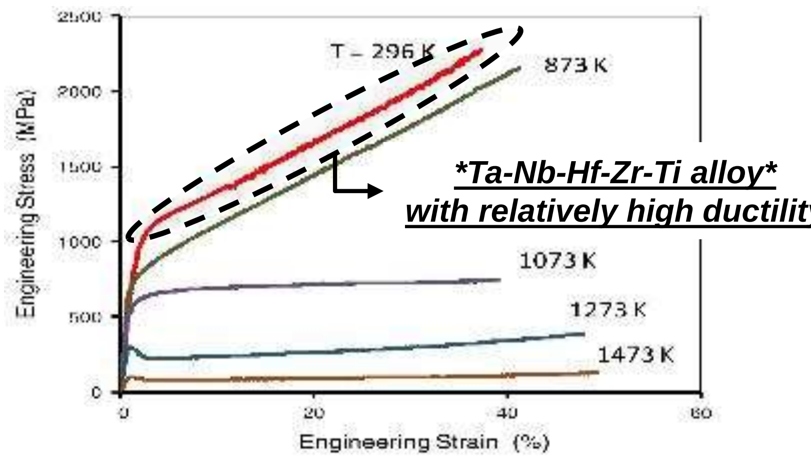
* macro scale XRD pattern *



Chen et al., J Alloys Comp., 661 (2016) 206

- Apparently different response on compressive stress





Senkov et al., J Mater. Sci., 47 (2012) 4062

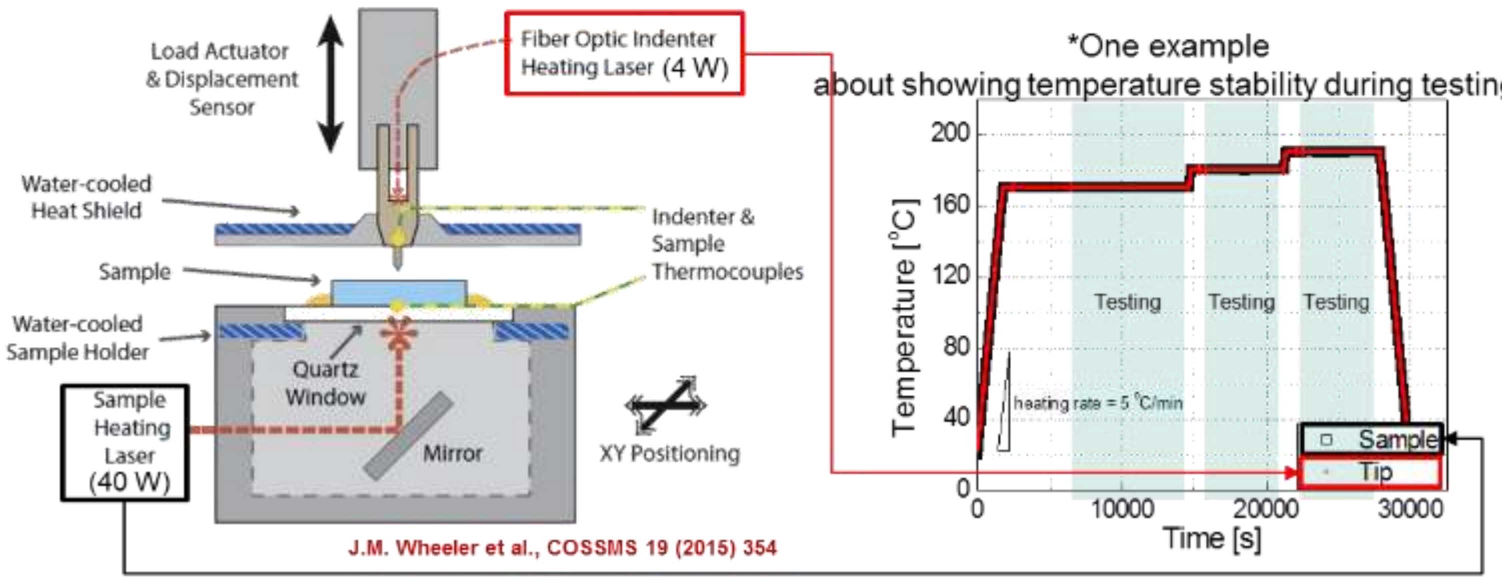
Background

- Proper characterizing system for both of brittle & ductile HEA systems

  = ***High-temperature (HT) nanoindentation***

  ✓ Equipment: Agilent G200 with laser-based heating system from SURFACE GmbH

  ✓ Measurable properties: *elastic modulus (E), hardness (H), strain-rate sensitivity (m), activation volume (V*)*

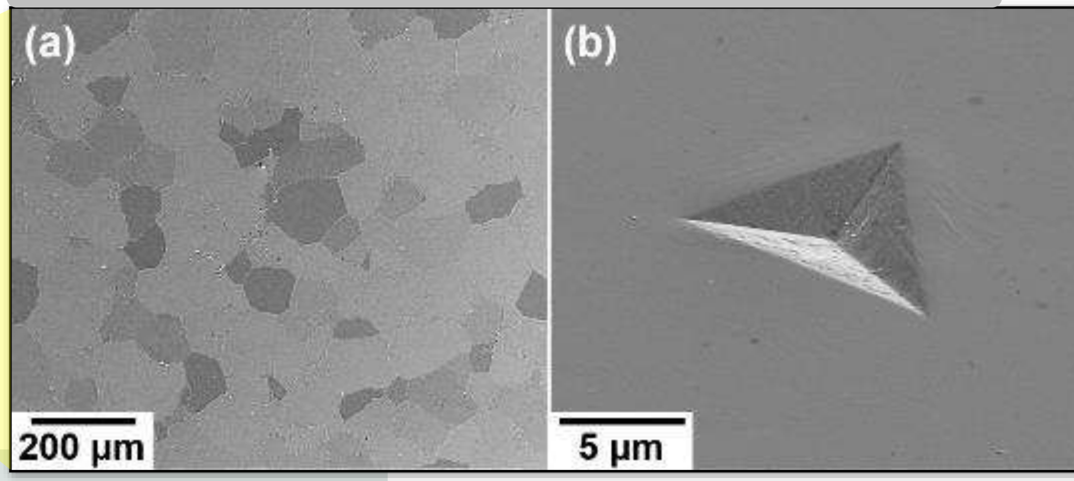


- Small heated volume (sample, tip, and mounting glue itself) for minimal influence on the measurement setup
- Minimized thermal drift and high stability by independent setting for tip and sample temperature

Objective

High-temperature nanoindentation system

Microstructure of brittle *bcc* refractory HEA & impression image



To build a database of the temperature- and rate-dependent plastic deformation characteristics and strength in bcc refractory HEAs

Experimental details

- The procedure to estimate mechanical properties and thermally activated parameters

Preparing and attaching specimens on stage (thickness < 3mm)

↓

Nanoindentation (Setting test parameters)

↓

Constant S^2/P with h — No → Correcting load frame stiffness (→ back to the loop)

Yes ↓

Constant E with h — No → Correcting tip area function (→ back to the loop)

Yes ↓

Measuring *E* and *H*

Yes ↓

End test for each condition (More than 4 measurements) → **Calculating *m* and *V** from SRJ tests**

* *T*-dependent elastic modulus of the sapphire indenter tip was taken into account in the analysis.

* How to obtain mechanical properties?

- By standard CSM method
- Averaging values of *E* and *H* in displacement regime of 1000-1700 nm

✓ Tip: Berkovich indenter (sx sapphire)

✓ Atmosphere: Forming gas (5% H_2 in Ar)

I. Constant strain-rate (CSR) test

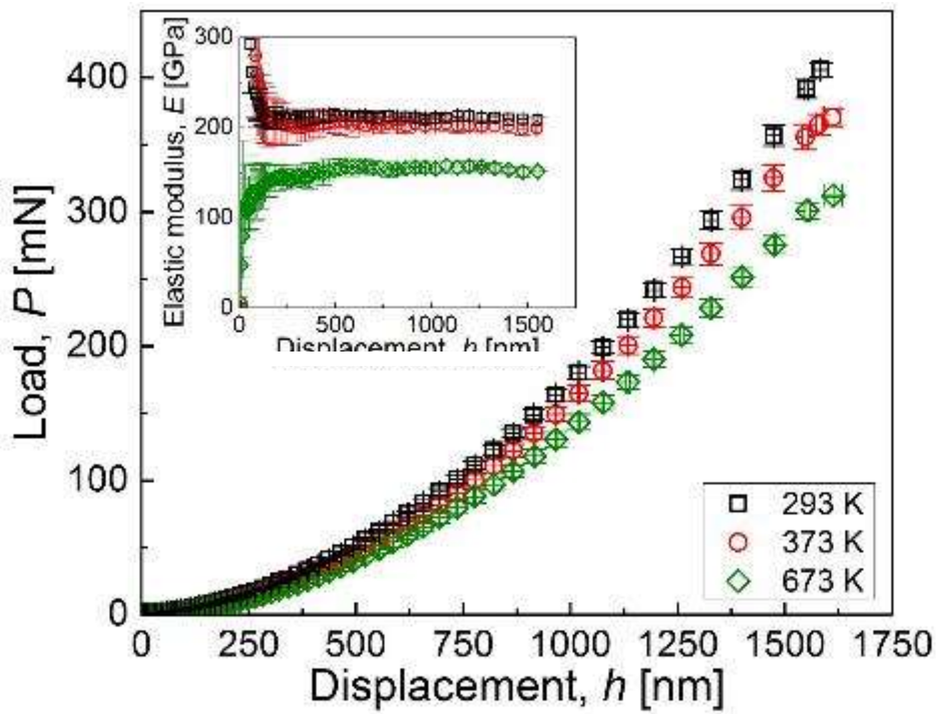
- $((dP/dt)/P) = 0.05\ s^{-1}$
- h_{max}=1.7 µm; Thermal drift < 0.2 nm/s
- Temperature: from 293 K to 673 K

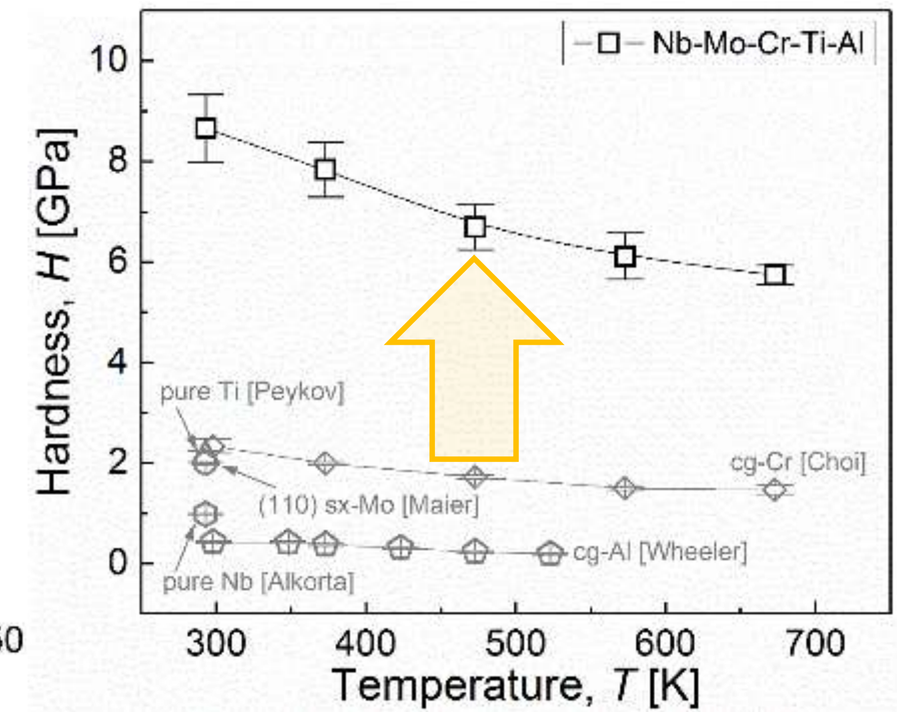
II. Strain-rate jump (SRJ) test

- $((dP/dt)/P)$=0.05, 0.016, 0.005 s^{-1}
- $h_{initial}$=500 nm, h for each strain rate = 300 nm ➔ h_{total}=1.7 µm
- Temperature: from 293 K to 673 K

Preliminary results

- *T*-dependent properties from CSR tests





<u>*To find reasons for variation in hardness and deformation morphology*</u>

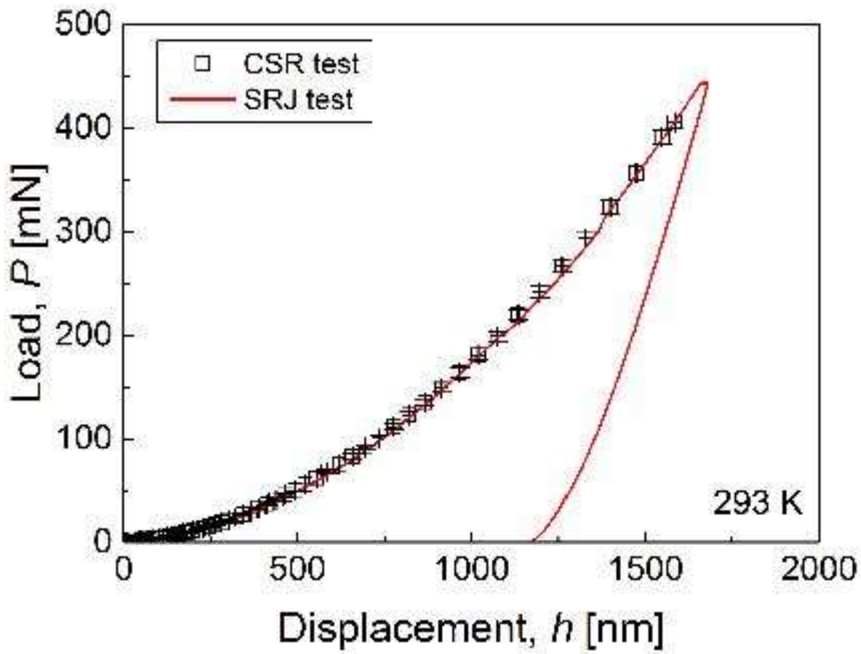
<u>➔ "Necessity of microstructural analysis"</u>

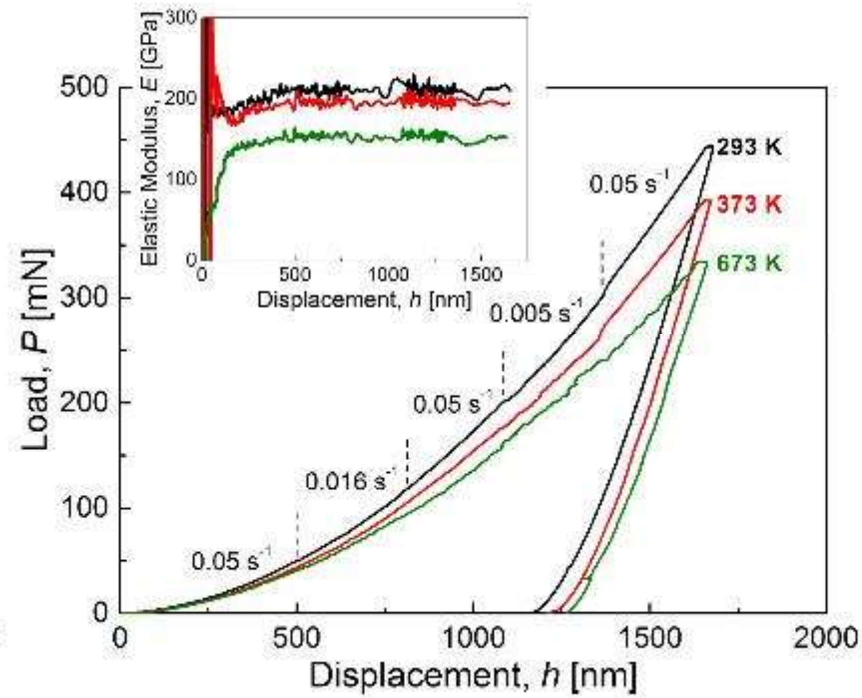
1. limited motion of dislocations by severe lattice distortion in HEA?
2. existence of other phases (i.e., ordered b2 structure)?
3. other possibilities, …?

Alkorta et al., Acta Mater., 56 (2008) 884
Maier et al., Philo. Mag., 95 (2014) 1766
Peykov et al., J. Mater. Sci., 47 (2012) 7189
Wheeler et al., MSEA, 585 (2015) 108
Choi et al., Acta Mater., 140 (2017) 107

Preliminary results

- *T*-/rate-dependent behavior from SRJ tests





Relatively lower rate-dependent behavior compared to m value of pure bcc elements*

➔ ***"Necessity of theoretical study combined with microstructural analysis"***

1. different kinetics for thermally activated dislocation motion in bcc HEAs?
2. influence of interaction between dislocation and other phases (e.g., with ordered b2 structure)?
3. other possibilities, …?

Summary

- The mechanical properties of fully annealed NbMoCrTiAl alloy was characterized by high-temperature nanoindentation.

- Constant strain-rate tests: Compared to hardness data on high-purity elements, NbMoCrTiAl HEA shows much higher value of hardness at room temperature.

- Strain-rate jump tests: Compared to rate-sensitive deformation behavior of pure bcc-structured elements, NbMoCrTiAl HEA shows relatively low strain-rate sensitivity.

- To identify the relevant deformation mechanisms and relationships between composition, microstructure, and properties, future research will be focused on:
  - Investigating deformation mechanisms through various microstructural analysis tools
  - Theoretical study correlated with simulation works
  - Characterizing mechanical properties on various composition of NbMoCrTiAl system
  - Comparison the results with Senkov's ductile bcc HEA system

* Acknowledgments: Samples were provided by H. Chen, Dr. A. Kauffmann, Prof. M. Heilmaier (KIT), which is greatly appreciated.