Crystal plasticity finite element analysis of nanoindentation process in pure iron and ferritic/martensitic steels at elevated temperatures

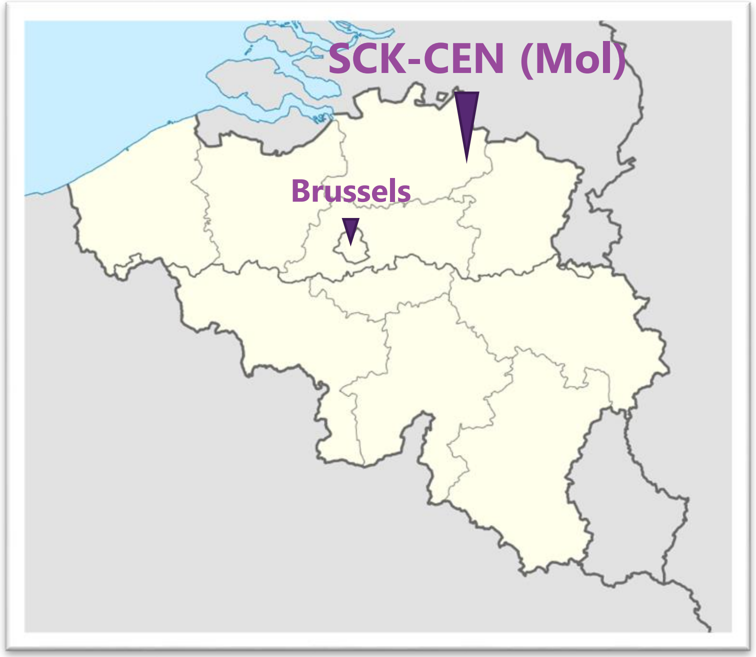
Tymofii Khvan*, Dr. Dmitry Terentyev, Prof. Ludovic Noels

*tymofii.khvan@sckcen.be

Indentation 2021
13.10.2021-15.10.2021

sck cen

- Belgian Nuclear Research Centre
- Located in Mol, Antwerp province, Belgium
- Carries the research in different fields of nuclear sciences
- **Belgian reactor 2 (BR2)** of SCK-CEN is a high thermal neutron flux reactor used for nuclear materials research as well as nuclear medicine and electronics



SCK-CEN on the map of Belgium

Belgian Reactor 2

Structural MAterials (SMA):

1. The selection, specification and validation of materials for applications in nuclear installations (fusion and fission reactors):
   a) Reduced activation Ferritic/Martensitic (RAFM) steels
   b) Austenitic steels
   c) Tungsten, copper, chromium, etc.
2. The evolution of mechanical properties of selected materials by using thermomechanical treatment or changes in chemical compositions:
   a) Ageing, hot-rolling, quenching, etc.
   b) Composition variations of W, Ta, C, Cr, etc.
3. The assessment of mechanical properties degradation due to neutron irradiation:
   a) Hardening
   b) Loss of ductility
   c) Decrease of creep resistance

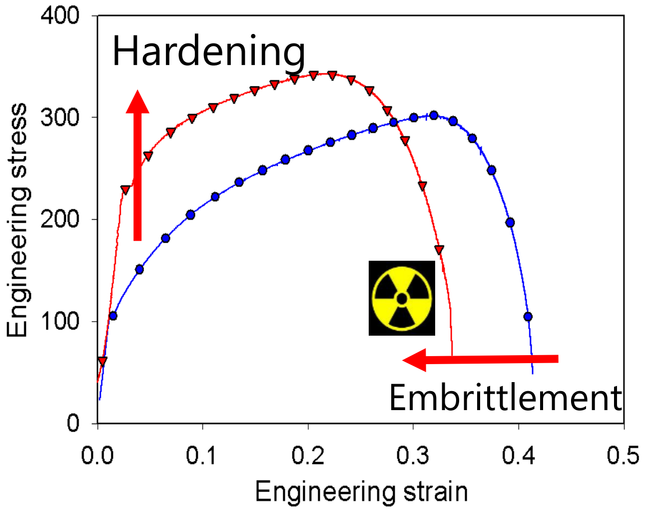
Harmful conditions in nuclear reactors

Structural materials endure challenging operational conditions:

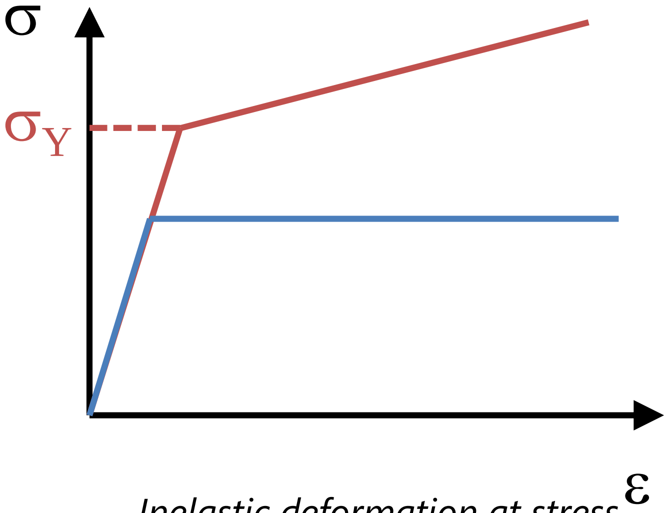
- Neutron irradiation with high doses (over 50 dpa over lifetime of in-vessel components in commercial Fusion reactors)
- High temperatures (up to 650 °C)
- High mechanical loads due to thermal expansions

Mechanical properties of RAFM steels (material of the study) degrade under mentioned conditions:

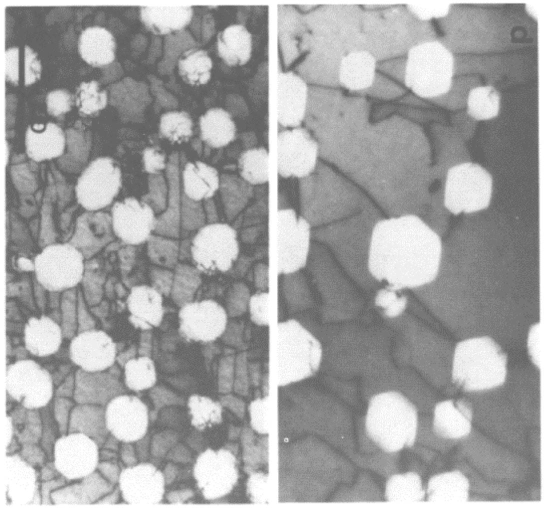
- Irradiation at low temperatures: hardening & embrittlement
- Irradiation at high temperatures: swelling & creep



Increase of yield stress (hardening) and loss of ductility (embrittlement)



Inelastic deformation at stress level below yield stress (creep)



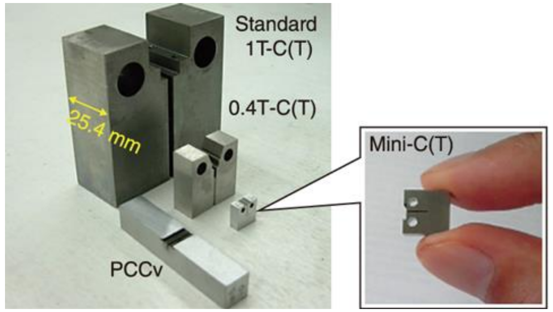
Nucleation and growth of voids (swelling)

Nanoindentation in nuclear materials sciences

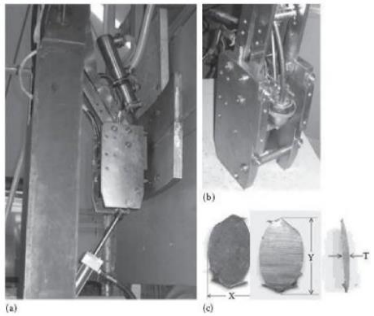
Miniaturization concept

The concept aimed on reduction of specimens dimensions

- Decrease of activity after irradiation
- Reduction of irradiation time and costs
- Requires less irradiation space



Compact tension specimens of different dimensions



A specimen taken from reactor core shroud in BARC, India

Variety of specimens for tensile testing of different dimensions

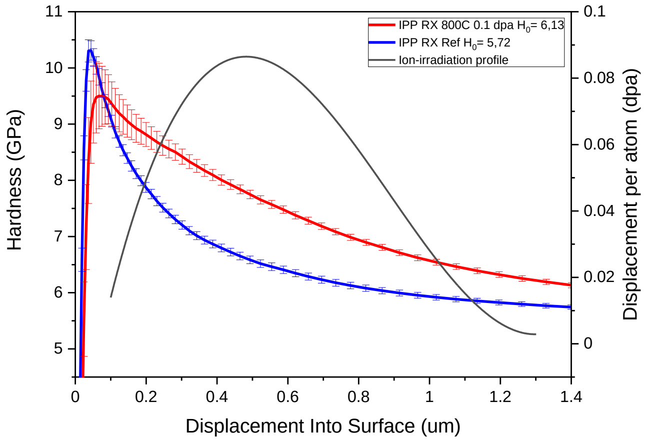
Assessment of ion irradiation damage

A tool to surrogate the neutron irradiation damage with ions

- Ion irradiation is cheaper and faster
- A specimen can be used for post-irradiation testing immediately and without safety protocols

BUT

- Ion irradiation has non-uniform damage profile
- Indentation size effect complicates the hardness evaluation
- There are no direct connection of neutron and ion irradiation damage and it differs a lot depending on irradiation parameters



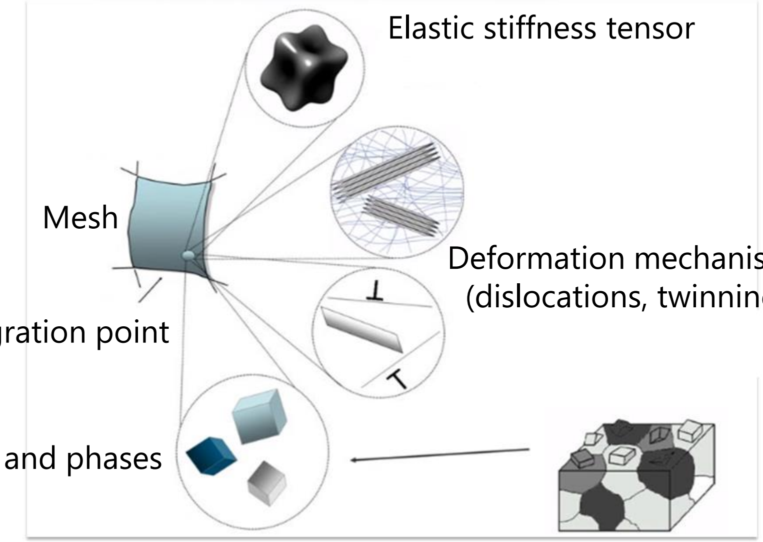
Hardness vs depth curves for ion-irradiated (red) and reference (blue) tungsten. Black line is roughly estimated ion damage profile

Global challenge

To combine the accessible research tools (mechanical testing, computational analysis, microstructure examinations) for development and validation of a computational model of nanoindentation process on ferritic/martensitic steels before and after irradiation in a relevant (operational) temperature range.

Features:

- **Finite element method** – a great computational tool to simulate behavior of complex structures under different boundary conditions
- **Crystal plasticity theory** – a well-defined theory of plasticity based on defect interactions in crystals
- **Ion irradiation damage layer** – to estimate the effect of irradiation on constitutive laws

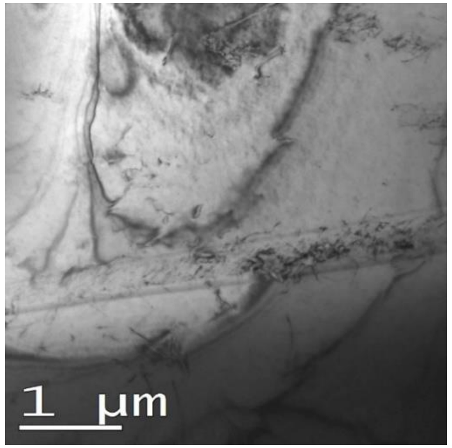


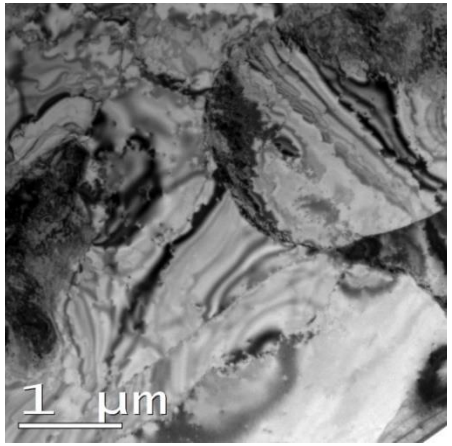
Crystal plasticity aspects in FEM

Reference F. Roters, P. Eisenlohr, L. Hantcherli, D.D. Tjahjanto, T.R. Bieler, D. Raabe. Acta Materialia 58 (2010) 1152–1211

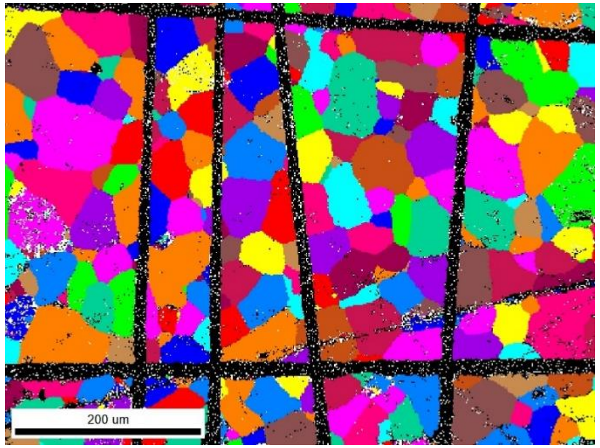
Development steps and microstructure

Step 1: **Pure Fe**





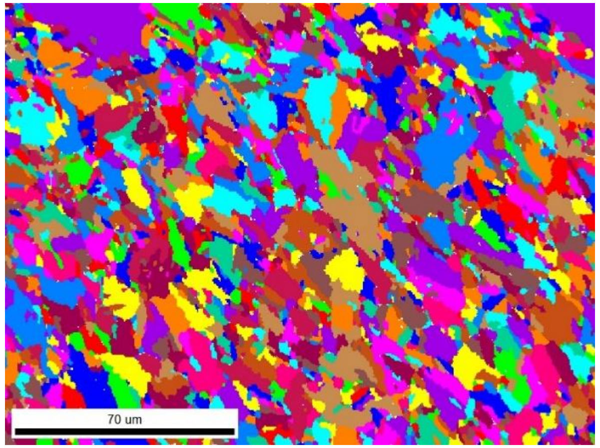
TEM pictures of Pure Fe



Grain map of pure Fe with 15° tolerance angle

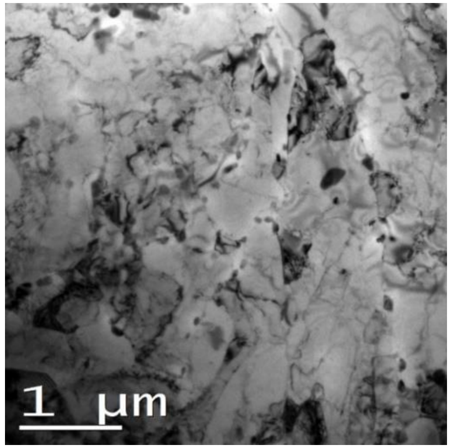
- Average grain size: 93.4 µm
- Average dislocation density: 1.0×10^{12} m^{-2}
- Basis for Eurofer97

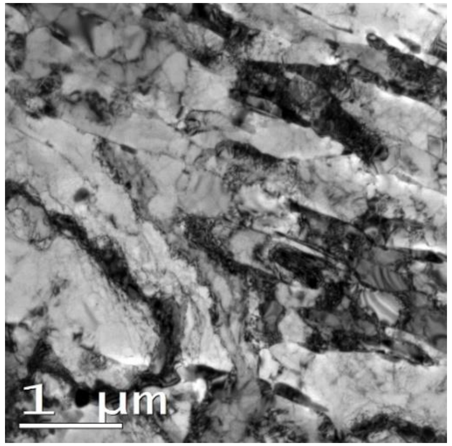
Step 2: **Eurofer97**



Grain map of Eurofer97 with 15° tolerance angle

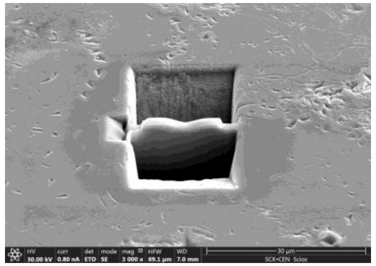
- Reduced Activation Ferritic Martensitic (RAFM) steel to be applied in Fusion reactor
- Average blocks size: 16.9 µm
- Average dislocation density: 1.5×10^{14} m^{-2}



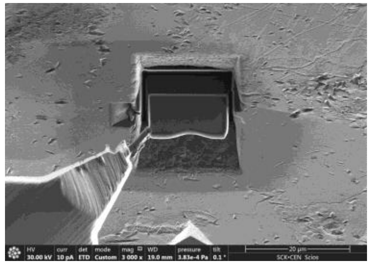


TEM pictures of E97

Microstructure inspection using FIB and TEM



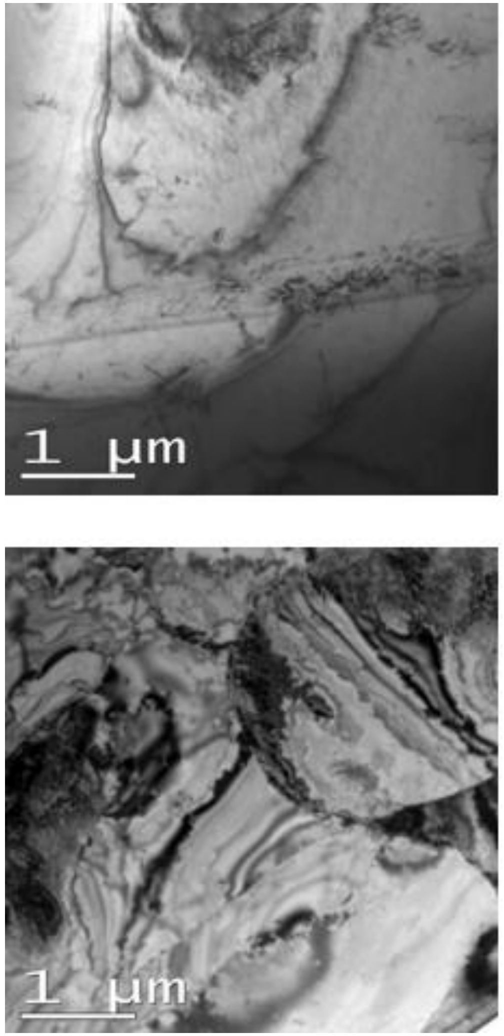
Cut-out of the lamella



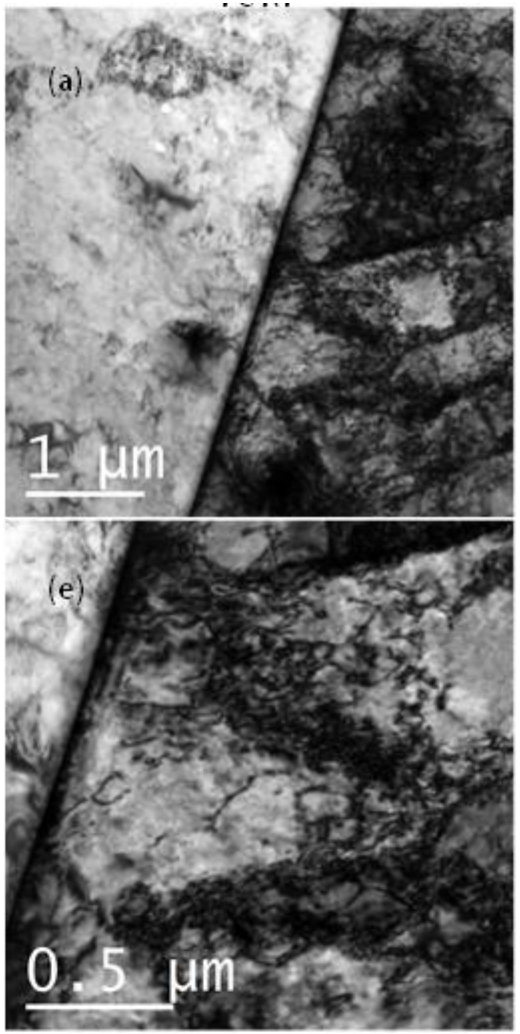
Lift-out of the lamella

Lamella inspected by TEM in the reference part of the specimen

ρ_0 = 10^{14} m^{-2}

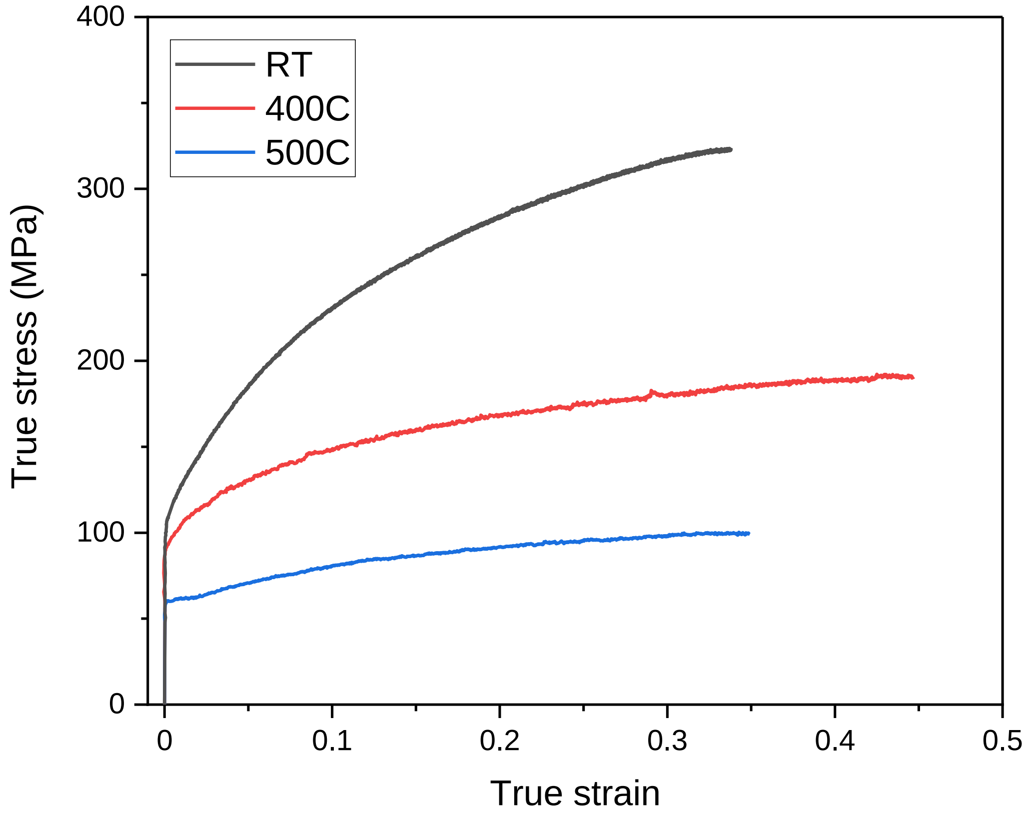


TEM patterns of iron before straining
ρ = 10^{12}

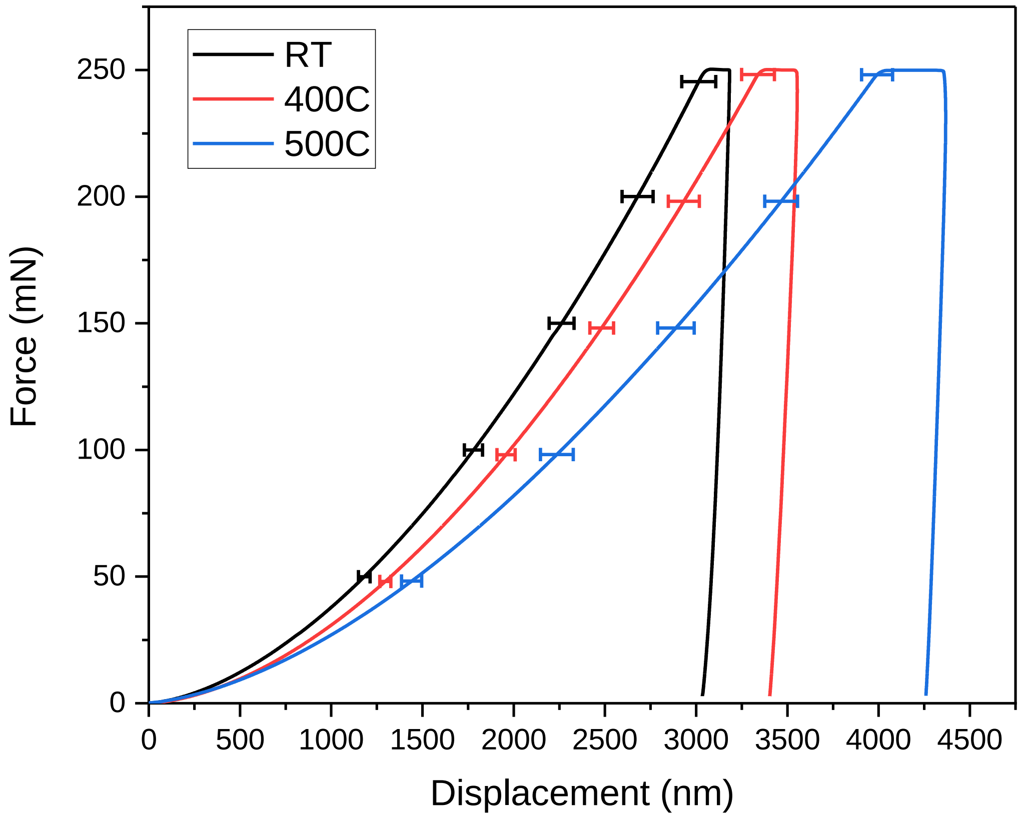


TEM patterns of prestrained to 15% iron
ρ = $2{\cdot}10^{14}$

Testing of pure iron



True stress – strain curves of pure iron



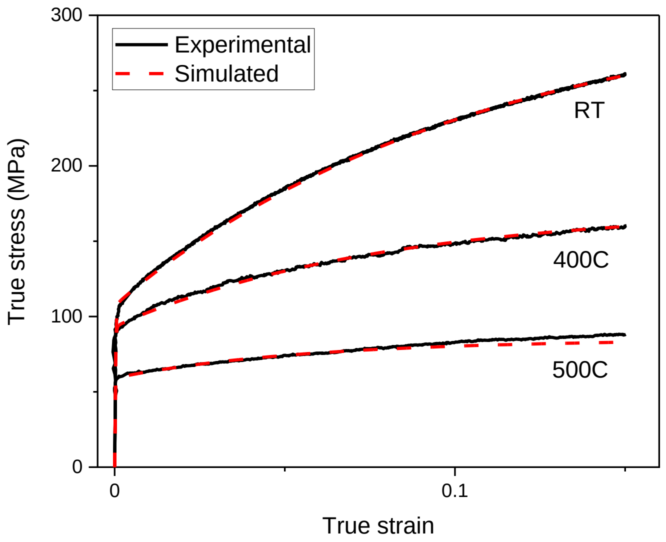
Testing conditions:

- Strain rate: 6.6·10^{-5}
- Miniaturized dogbone: 16x4.2x1.5 mm
- RT, 400 °C, 500 °C
- Recalculated to true stress-strain

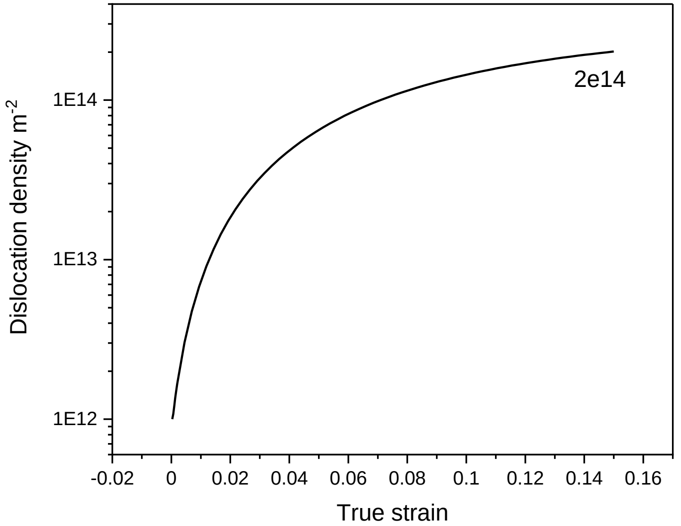
Testing parameters:

- Tests done in Bruker GmbH, Aachen, Germany
- Hysitron TI980 nanoindenter and xSOL heating stage
- SiO_2 40 nm layer was applied to the iron specimen to avoid oxidation
- Polishing up to 1 µm diamond paste + OP-U solution

Description of constitutive laws of pure iron using crystal plasticity



Simulated (red) and experimental (black) true stress – strain curves of pure iron



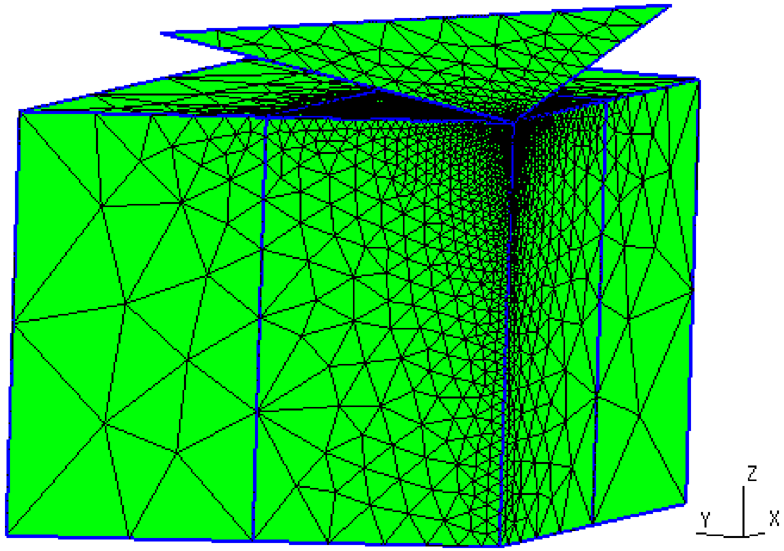
Dislocation density versus true strain at RT

Parameter	Value	Source
Elastic coefficient, *C11*	235 *GPa*	Literature
Elastic coefficient, *C12*	135 *GPa*	Literature
Elastic coefficient, *C44*	115 *GPa*	Literature
Reference slip rate, $\dot{\gamma}_0$	0.1 s^{-1}	Fitted
Activation energy, $2H_k$	2.365 *eV*	Literature/ Artificially increased
Hall-Petch contribution, $S_0(T)$	32.0 – 18.0 *MPa*	Fitted
Burger's vector, *b*	0.2482 *nm*	Literature
Initial dislocation density, ρ_0	10^{12} m^{-2}	Measured
Dislocations interaction coefficient, $\alpha(T)$	0.2 – 0.04	Fitted
Saturated-initial dislocation density ratio, $\rho_{sat}(T)/\rho_0$	$6\text{-}5\cdot10^2$	Measured
Kocks-Mecking parameter, k_1	$1.3\cdot10^8$ m^{-1}	Fitted

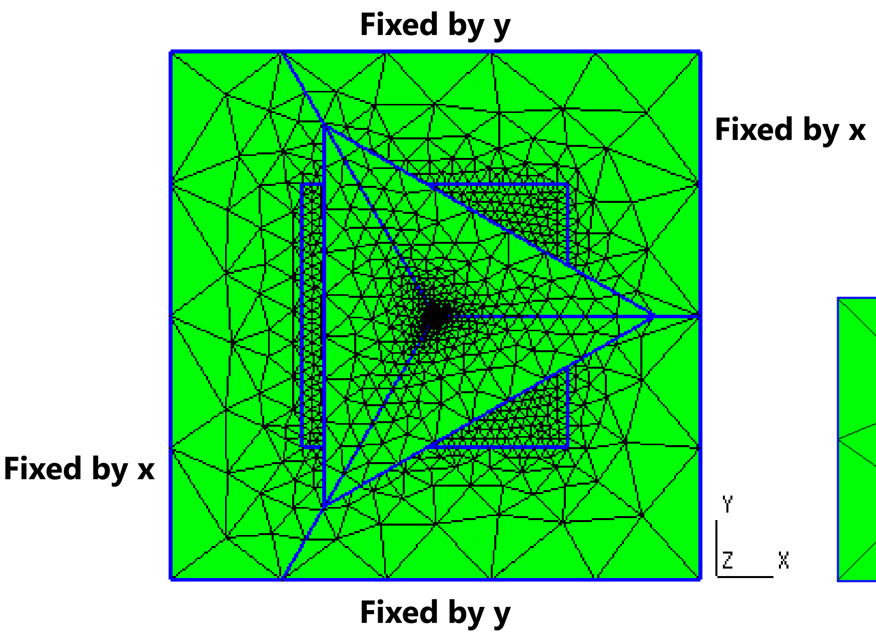
Table of parameters used for tensile curves replication

FEM setup

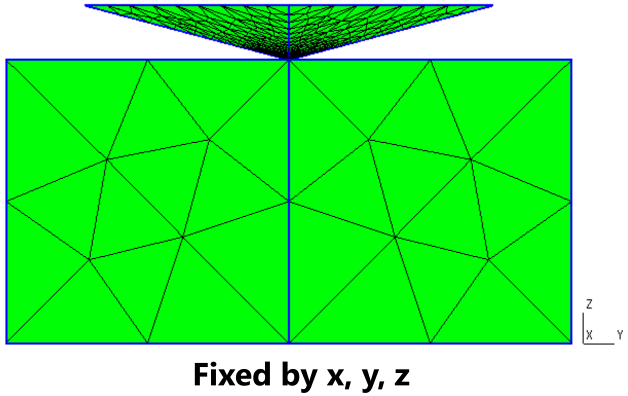
- Open-source FEM solver called **Cm3Libraries for gmsh** coupled with crystal plasticity model using UMAT interface
- Berkovich indenter with 12.95° attack angle and perfect tip
- Specimen box is 60x60x30 μm^3
- [100], [101], [111] directions of indenter immersion into a **single grain**
- Mesh refinement below the indenter tip. 7284 nodes and 34968 hexahedral 1st order elements in total.



Geometry of FEM setup: inner view

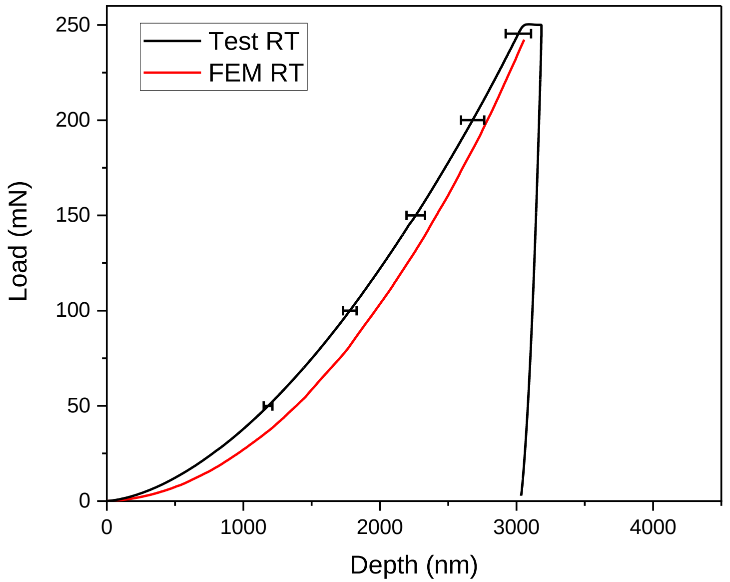


Geometry of FEM setup: top view

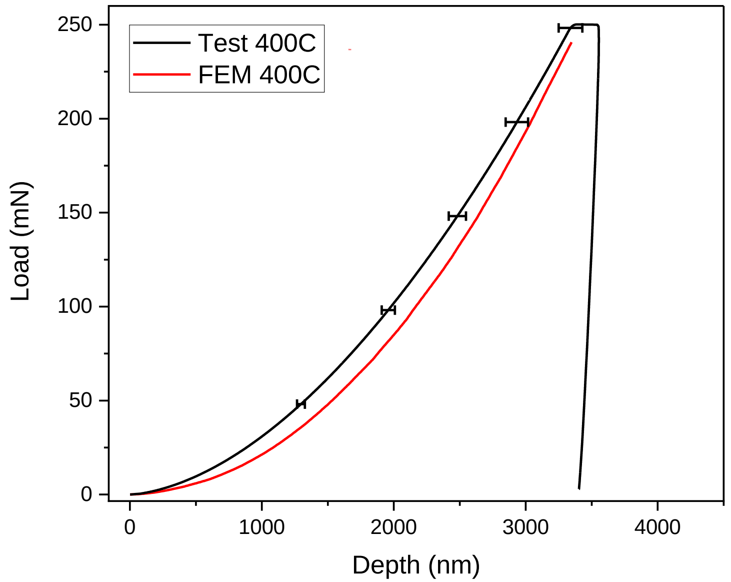


Geometry of FEM setup: side view

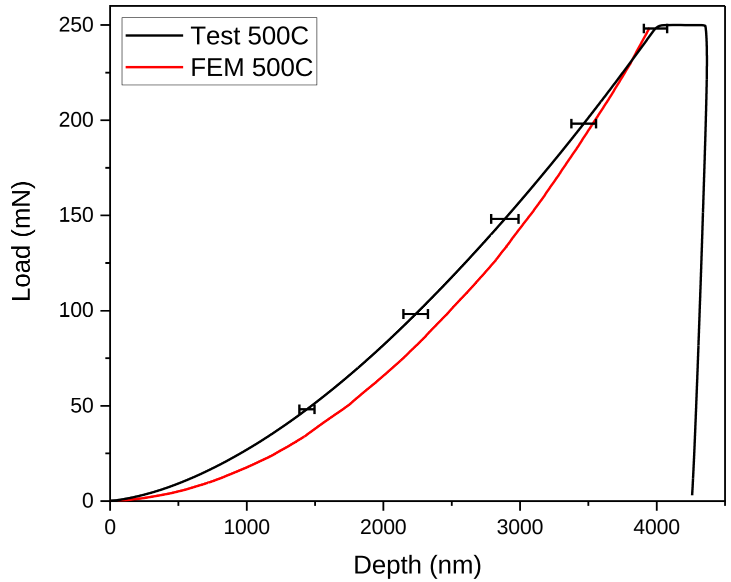
CPFEM nanoindentation simulations of pure iron



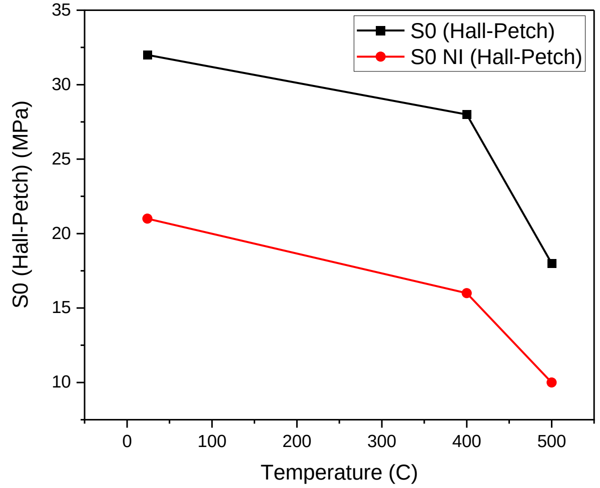
FEM and experimental force-displacement curves of pure iron at RT



FEM and experimental force-displacement curves of pure iron at 400C

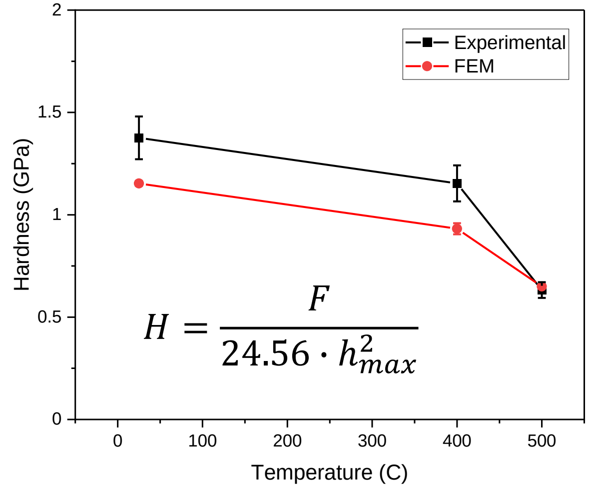


FEM and experimental force-displacement curves of pure iron at 500C



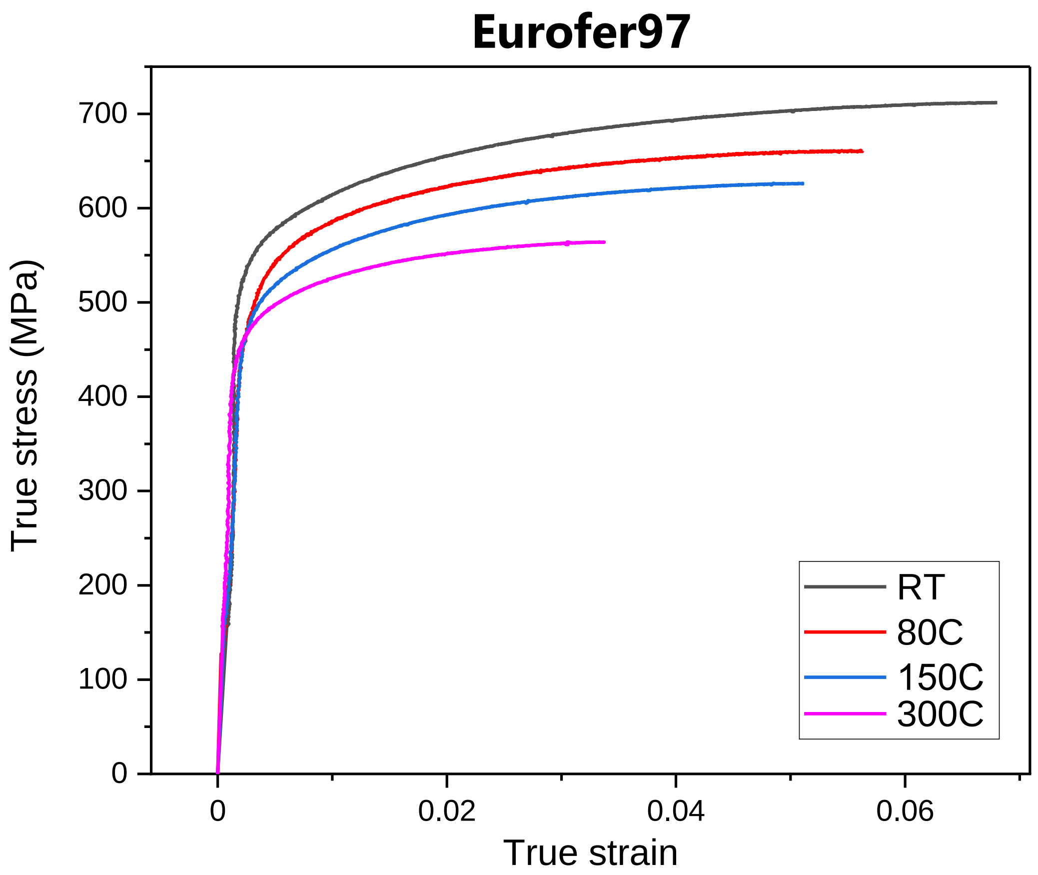
Hall-Petch contribution for tensile (black) and nanoindentation (red) simulations

ρ_0 = $10^{12} \rightarrow 10^{14}$ m^{-2} for nanoindentation

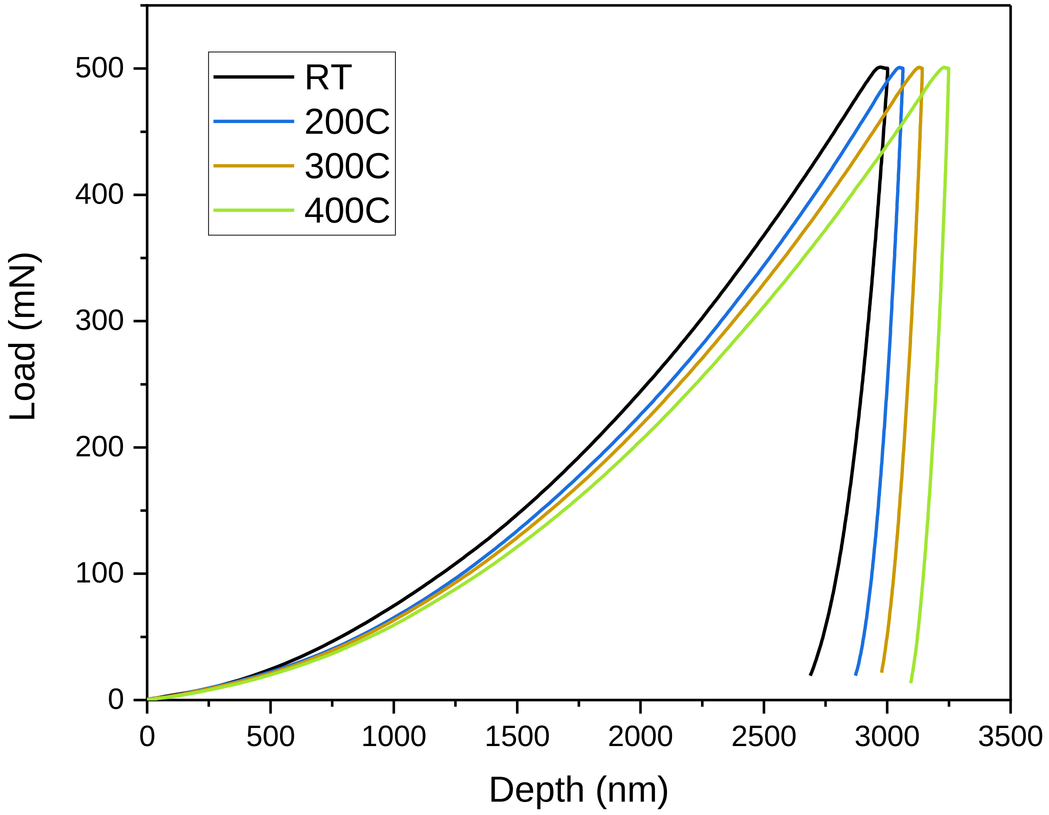


$$H = \frac{F}{24.56 \cdot h_{max}^2}$$

Hardness values measured experimentally (black) and estimated from FEM (red)



True stress – strain curves of Eurofer97



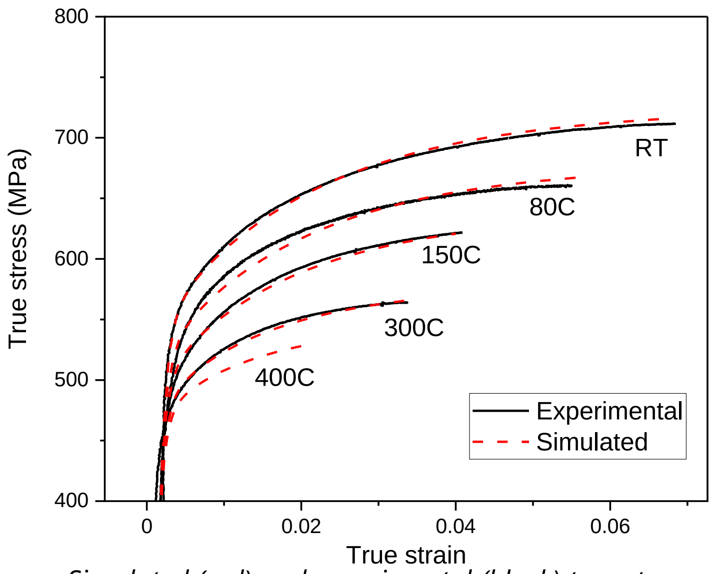
Testing conditions:

- Strain rate: $1.0 \cdot 10^{-5}$
- Cylindrical specimen: L = 24 mm, d = 2.4 mm
- RT, 80 °C, 150 °C, 300 °C
- Recalculated to true stress-strain

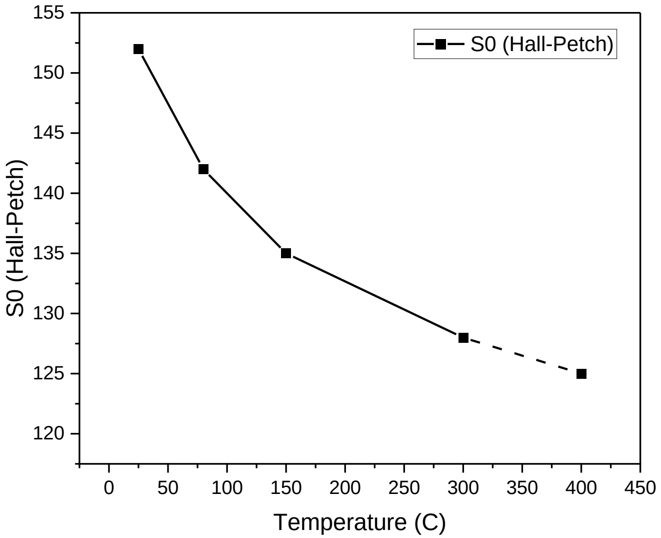
Testing parameters:

- Tests done in Bruker GmbH, Aachen, Germany
- Hysitron TI980 nanoindenter and xSOL heating stage
- Polishing up to 1 μm diamond paste + OP-U solution

Description of constitutive laws of Eurofer97 using crystal plasticity



Simulated (red) and experimental (black) true stress – strain curves of Eurofer97

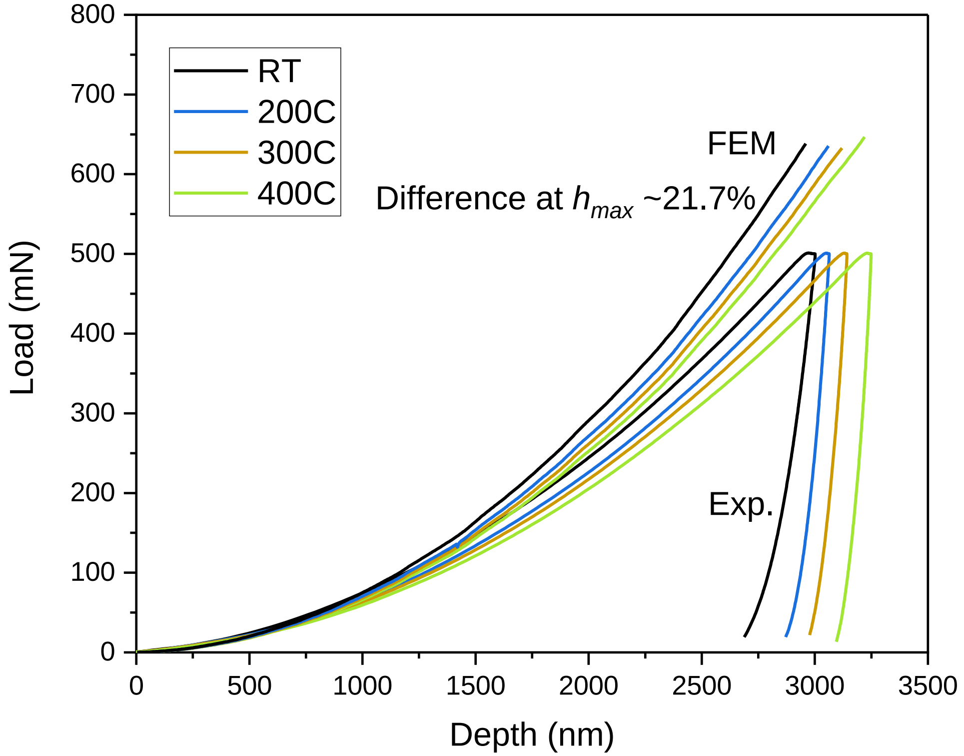


Hall-Petch contribution as function of temperature with predicted value for 400C

Parameter	Value	Source
Elastic coefficient, *C11*	235 *GPa*	Literature
Elastic coefficient, *C12*	135 *GPa*	Literature
Elastic coefficient, *C44*	115 *GPa*	Literature
Reference slip rate, $\dot{\gamma}_0$	1 s^{-1}	Fitted
Activation energy, $2H_k$	2.365 *eV*	Literature/ Artificially increased
Hall-Petch contribution, $S_0(T)$	154.0 – 125.0 *MPa*	Fitted
Burger's vector, *b*	0.2482 *nm*	Literature
Initial dislocation density, ρ_0	$1.5 \cdot 10^{14}$ m^{-2}	Measured
Dislocations interaction coefficient, $\alpha(T)$	0.18	Fitted
Saturated-initial dislocation density ratio, $\rho_{sat}(T)/\rho_0$	12.0	Fitted
Kocks-Mecking parameter, k_1	$9.8 \cdot 10^8$ m^{-1}	Fitted

Table of parameters used for tensile curves replication

CPFEM nanoindentation simulations of Eurofer97



FEM and experimental force-displacement curves of Eurofer97 at 400C

Potential reasons:

- Crystal plasticity model only accounts plastic slip due to dislocations and grain boundaries effect, but no solid solution hardening and complex grain structure which are present in Eurofer97
- In case of pure iron, the same material was used, however Eurofer97 tensile and nanoindentation specimens were taken from different sources where different thermomechanical treatment applied was possible, what can change the yield stress of the material on ~15%

Summary

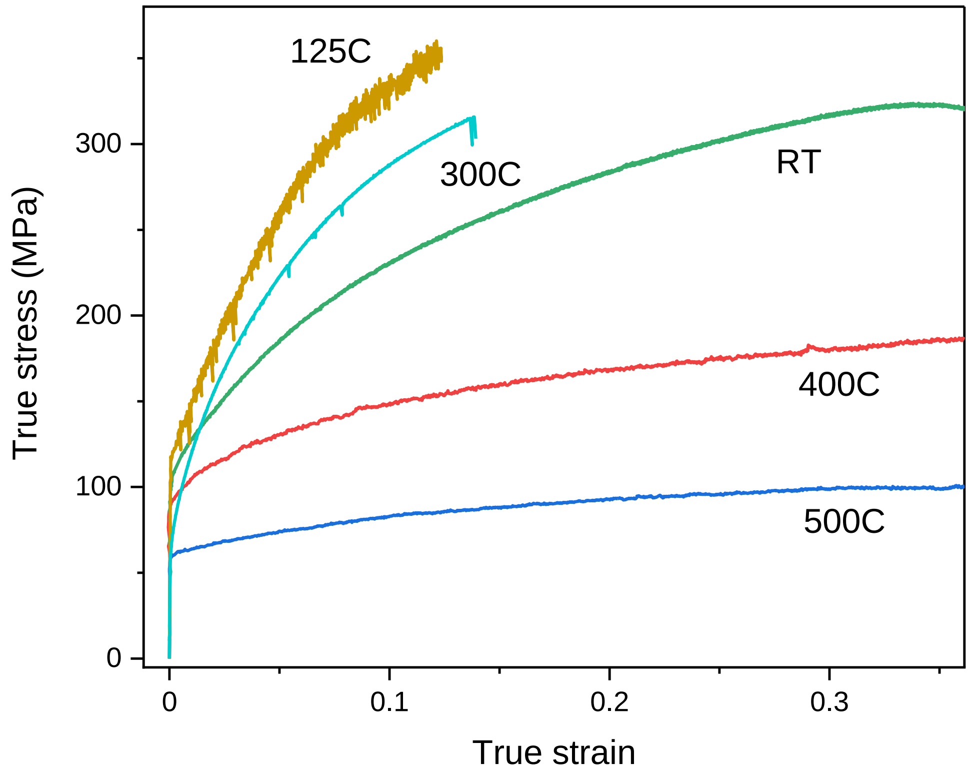
Within the presented work, the mechanical tests together with microstructure analysis were applied in order to obtain a correct input for CPFEM model of nanoindentation process to simulate the material response of pure BCC iron and Eurofer97 products on indenter immersion.

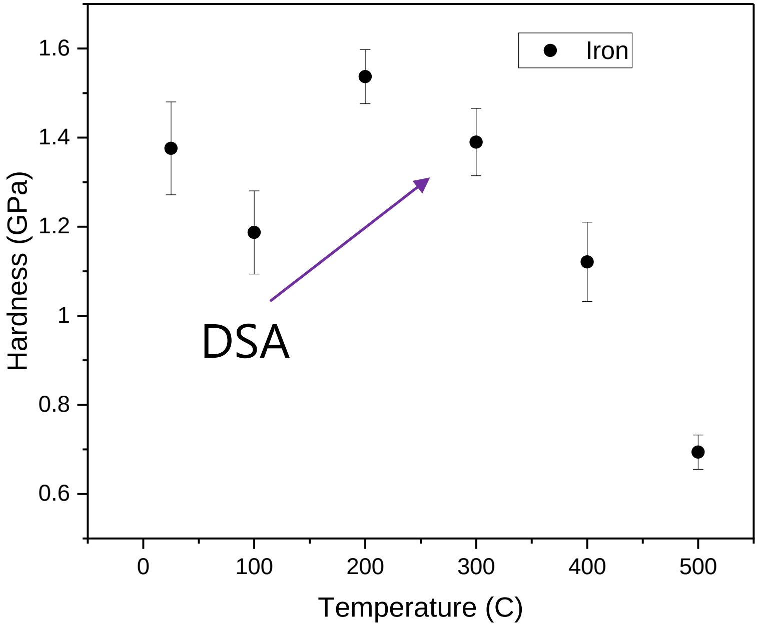
Conclusions

- The set of constitutive parameters used to describe the constitutive laws of pure iron and Eurofer97 is precise enough to reproduce the true stress-strain curves of these materials, and has physically correct trends in case of temperature dependence present for a particular parameter
- The obtained constitutive laws used as an input of established CPFEM setup can correctly reproduce a nanoindentation force-displacement curve of pure iron in a given set of temperatures
- Hardness calculations for pure iron are also in a good agreement with experimental values if obtained just by using the calculations for perfect indenter which we apply in the present simulations

Thank you for your attention!

tymofii.khvan@sckcen.be


125C
300C
RT
400C
500C
True stress (MPa)
300
200
100
0
0
0.1
0.2
0.3
True strain


Iron
DSA
Hardness (GPa)
1.6
1.4
1.2
1
0.8
0.6
0
100
200
300
400
500
Temperature (C)

Slip rate in slip system α

Activation energy

Stress in slip system

Pierels potential barrier
p = 0.5, q = 1.5

$$\dot{\gamma}^{\alpha} = \dot{\gamma}_0 \exp\left(-\frac{G_0 \mu b^3}{k_b T}\right) 2 \sinh\left(\frac{G_0 \mu b^3}{k_b T}\left(1 - \left(1 - \left(\frac{\tau_\alpha}{\hat{\tau}}\right)^p\right)^q\right)\right)$$

Reference slip rate

Boltzman & temperature

Dislocations interaction coeff.

Dislocation density

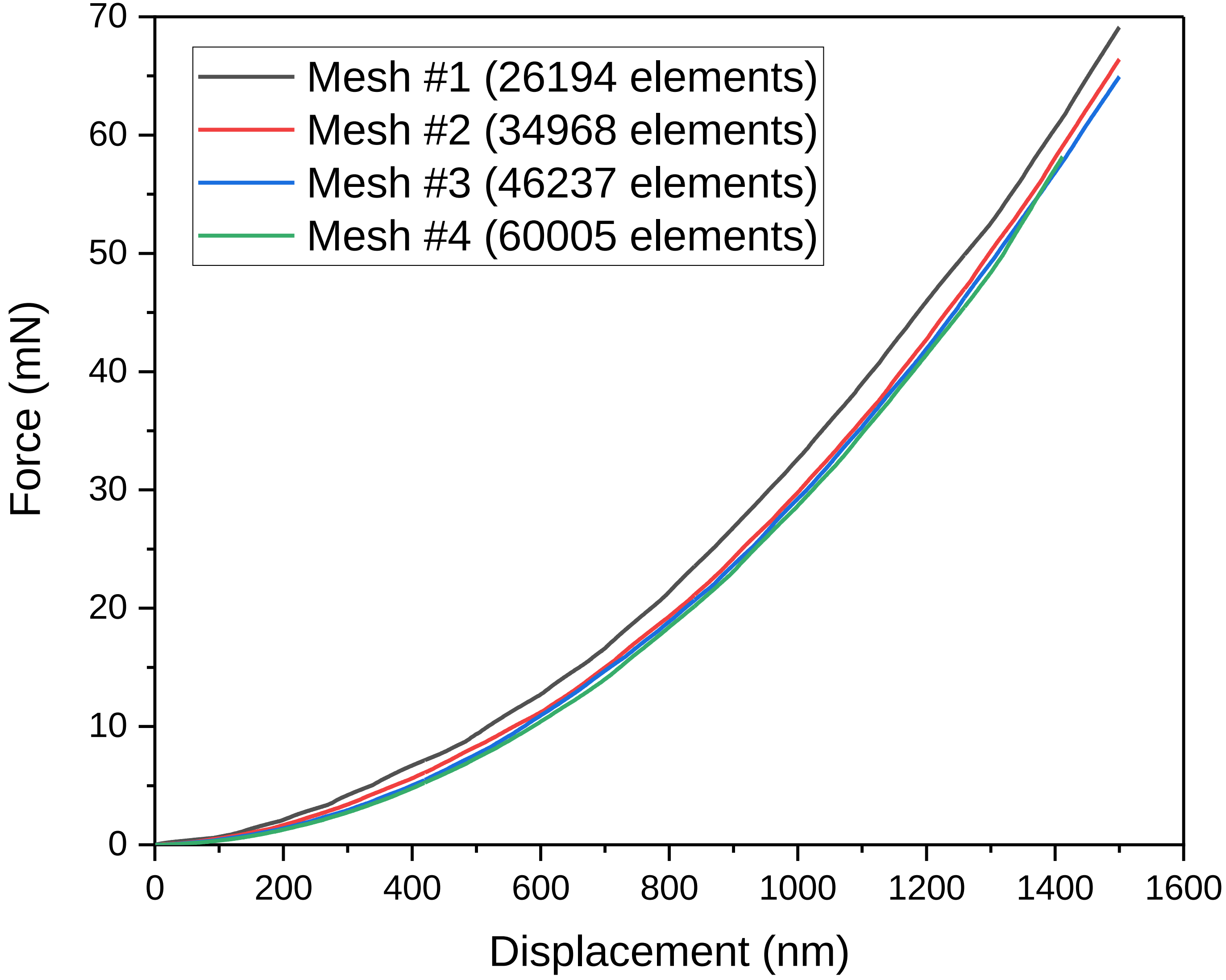
$$\hat{\tau} = S_0 + \alpha \mu b \sqrt{\rho}$$

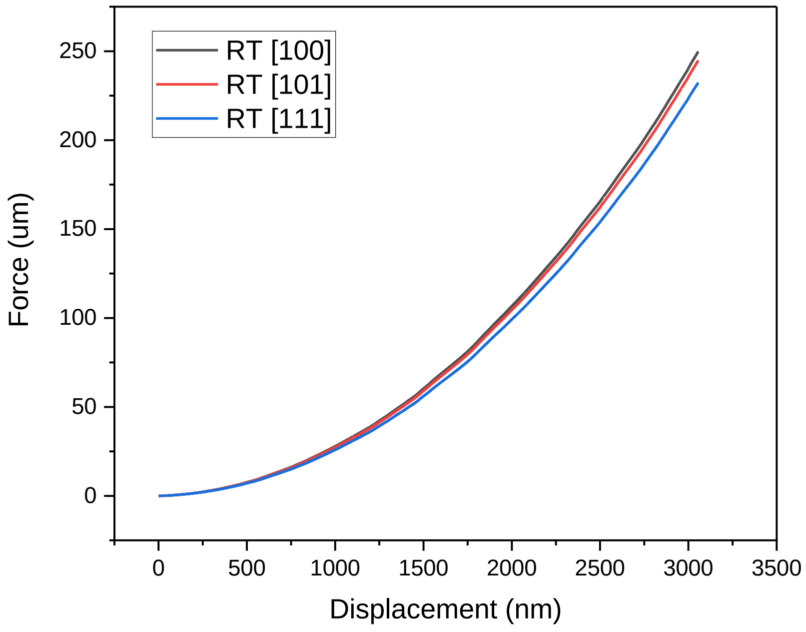
Hall-Petch

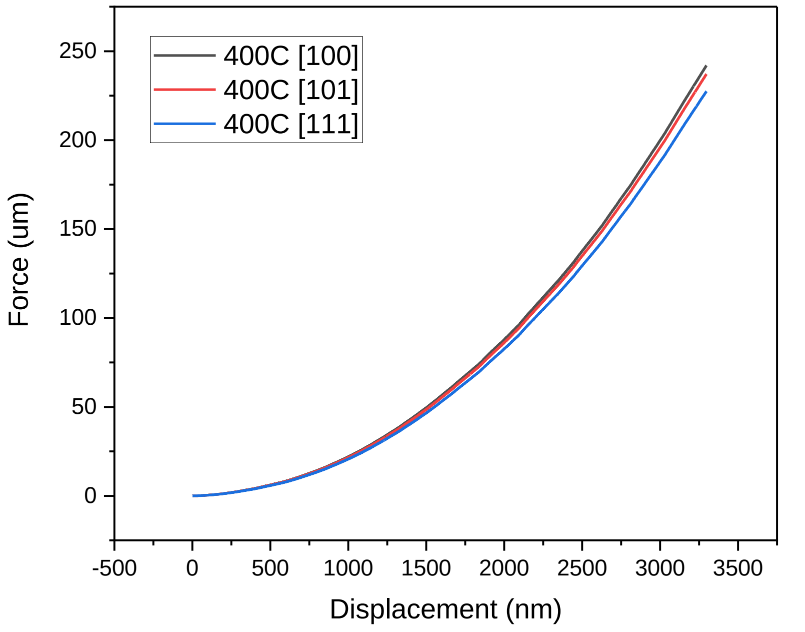
$$\dot{\rho} = (k_1 \sqrt{\rho} - k_2 \rho)\dot{\Gamma}$$

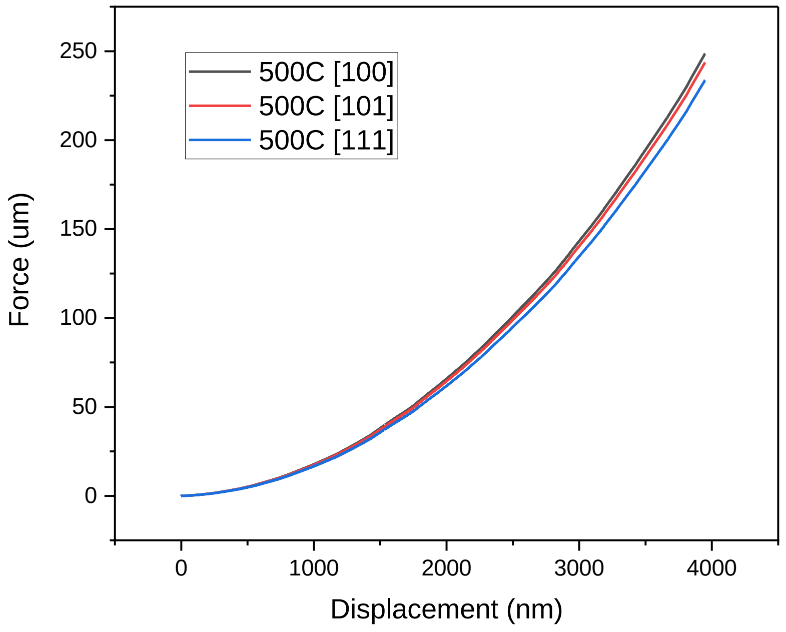
Kocks-Mecking parameters

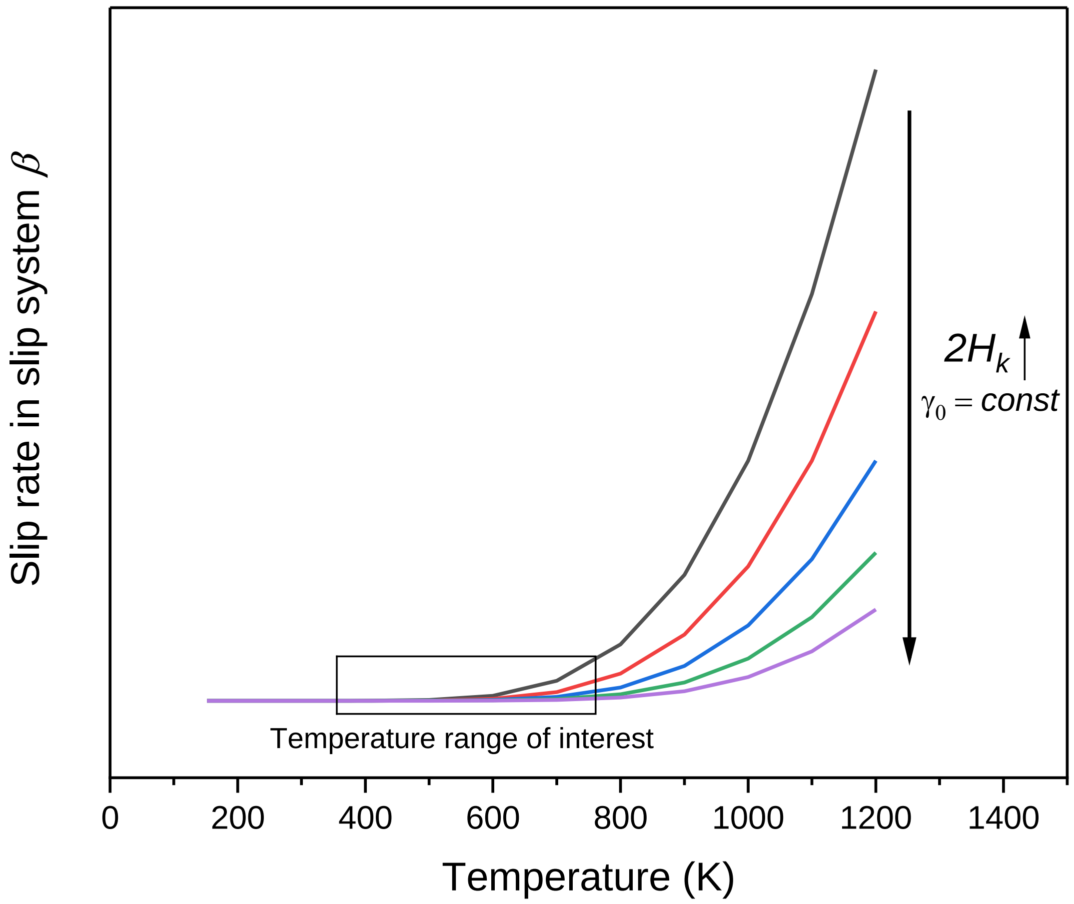
$$\dot{\Gamma} \triangleq \sum_{\alpha} |\dot{\gamma}_\alpha|$$

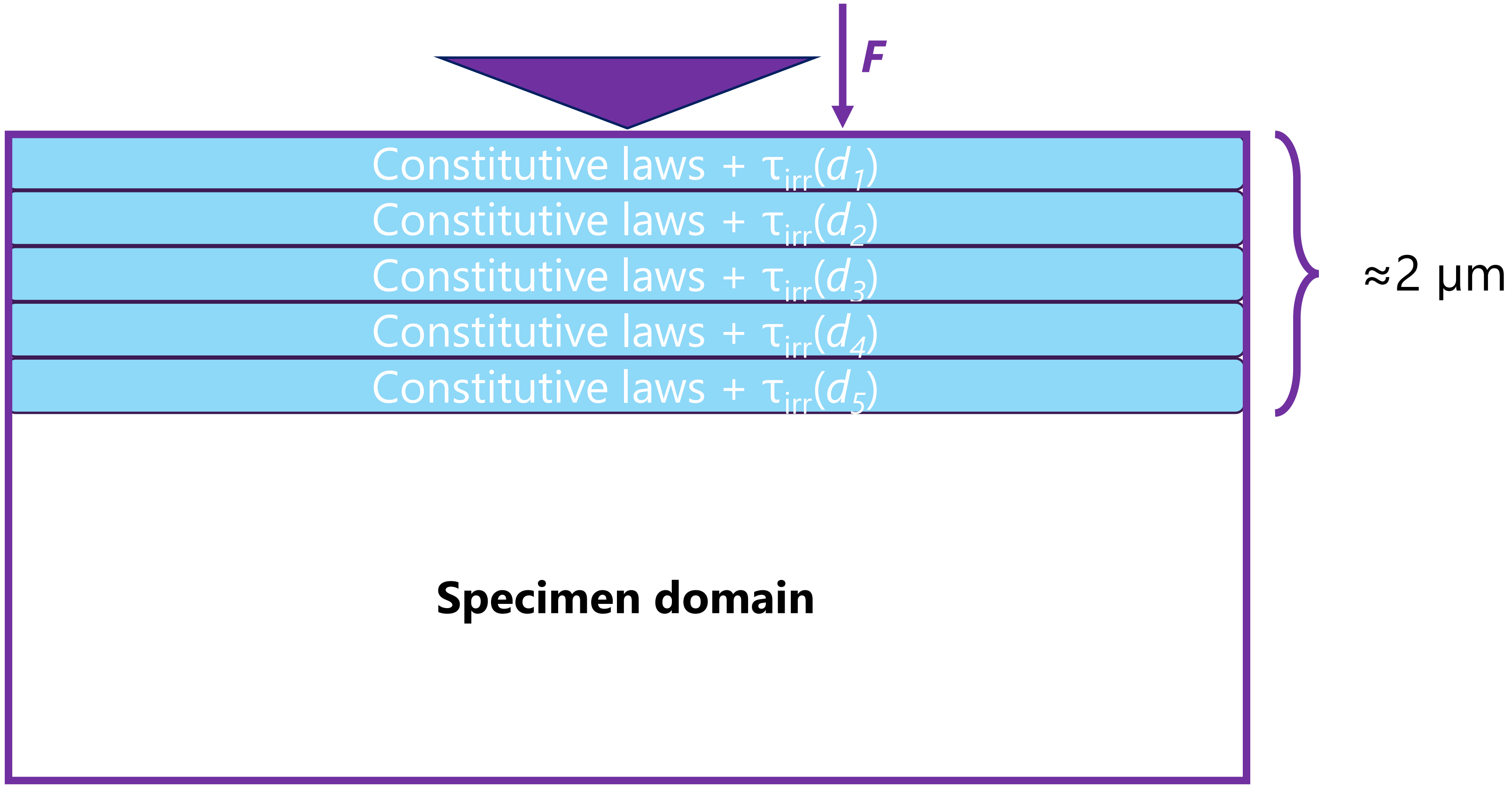

Mesh #1 (26194 elements)
Mesh #2 (34968 elements)
Mesh #3 (46237 elements)
Mesh #4 (60005 elements)
Force (mN)
Displacement (nm)
0
10
20
30
40
50
60
70
0
200
400
600
800
1000
1200
1400
1600


RT [100]
RT [101]
RT [111]
Force (um)
Displacement (nm)


400C [100]
400C [101]
400C [111]
Force (um)
Displacement (nm)


500C [100]
500C [101]
500C [111]
Force (um)
Displacement (nm)


Slip rate in slip system β
2H_k
γ_0 = const
Temperature range of interest
0
200
400
600
800
1000
1200
1400
Temperature (K)


F
Constitutive laws + $\tau_{irr}(d_1)$
Constitutive laws + $\tau_{irr}(d_2)$
Constitutive laws + $\tau_{irr}(d_3)$
Constitutive laws + $\tau_{irr}(d_4)$
Constitutive laws + $\tau_{irr}(d_5)$
≈2 μm
Specimen domain