WP6: NANOINDENTATION

WP leader: A. Ruiz Moreno, JRC

WP6 is organized in four tasks; three of them are devoted to numerical and analytical modeling of nanoindentation processes, and the last one is dedicated to experimental activities. Recent work has been focused on:

1. Exploring the use of step-wise MD simulations of nanoindentation to provide useful information for upper scale models;
2. Establishing approaches for strain gradient and crystal plasticity finite element models of nanoindentation processes, accounting for indentation size effects and able to incorporate the effects of irradiation damage;
3. Developing a methodology to derive information of radiation effects from nanoindentation curves, using mechanically deformed Fe as a proxy and supported by optimized nanoindentation testing protocols and TEM analyses of the indented materials.
4. Performing nanoindentation testing on pure Fe, Fe-9Cr model alloys and Eurofer97, irradiated by 5 MeV and 275 keV Fe^{2+} ions at 300°C and 450°C up to 0.1 and 1 dpa.
5. Optimising surface preparation of neutron irradiated Fe-9Cr for nanoindentation testing.

Task 6.1. Modelling at the grain scale (M1 – M48)

Task leader: A. Kuronen, UH; other partners: U. Aalto, SCK-CEN

Molecular dynamics (MD) simulations of indentation of pure Fe samples were performed. The defects mimicking irradiation effects were introduced in the same manner as previously done for the Fe-9%Cr alloy, i.e. by introducing random atom displacements that generate dislocations and vacancy clusters. The resulting force-depth curves did not show any effect unambiguously related with the presence of defects, similar to the case of the alloy.

The possibility of using the step-wise indentation in MD simulations was further studied. In this scenario, indentation was first performed with a constant speed, followed by a relaxation stage in which the indenter is not moved. In **Figure 6.1** we see the results of a single indent-relax cycle. Here the indenter with a diameter 43 Å and a pure, defect-free Fe-9%Cr alloy sample were used. First the indenter was lowered with a constant speed of 0.1 Å/ps, after which it was stopped and the system was allowed to relax. Simulations were performed in the temperature range between 300 and 1100 K. What we see here is that the relaxation time does not depend strongly on the temperature. Also, the indenter force and the total length of dislocations seem to settle to a constant value during the 1 ns relaxation period.

The incipient plasticity data in the form of force-depth curves obtained from MD simulations are being compared with those obtained from FEM simulation, based on the MD simulation of compression stress-strain data. The aim of this work is to determine whether it is possible to get similar size dependence of hardness in the nanometer scale.

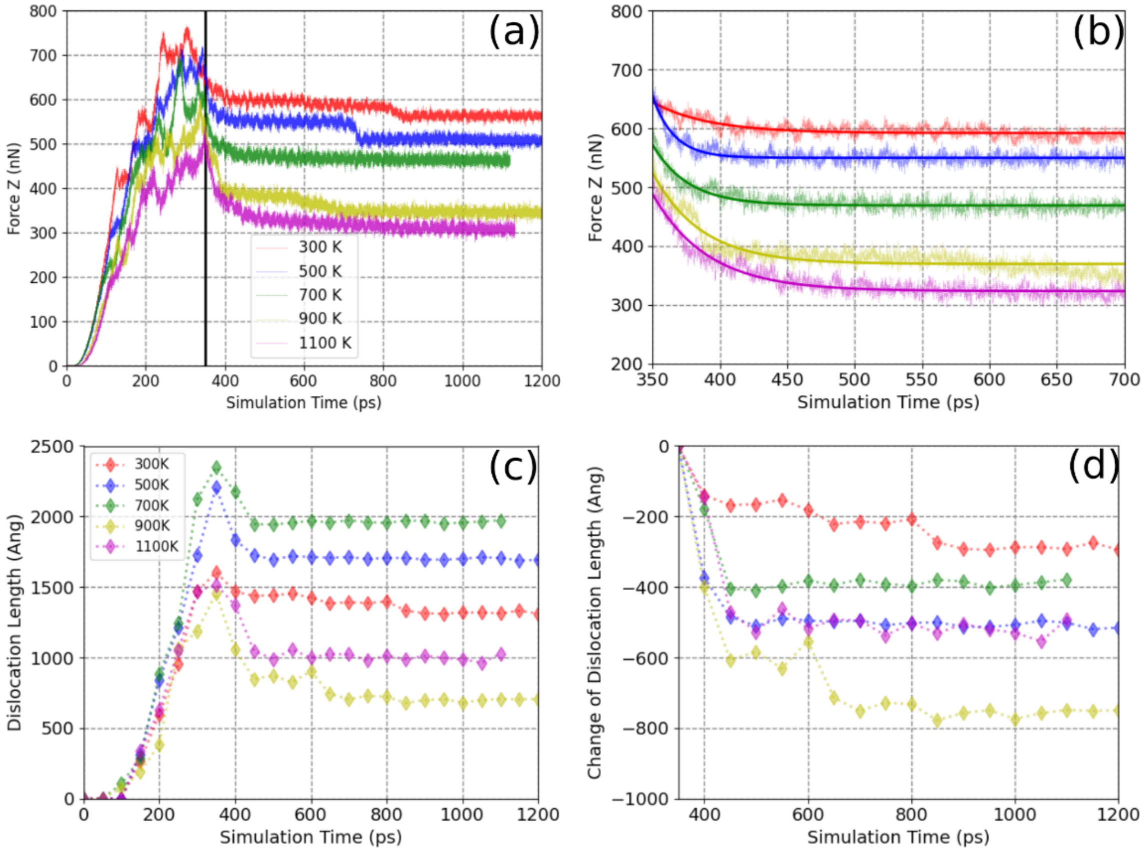

Figure 6.1. *(a) Force on the indenter as a function of the simulation time in different temperatures. The vertical line shows the point where the indenter was stopped. (b) Same as (a) but only showing the beginning of the relaxation. (c) Total dislocation length during the simulation. (d) Change of the total dislocation length in the relaxation phase.*

Task 6.2: Modelling local plastic deformation at the indentation scale (M1 – M48)
Task leader: D. Terentyev, SCK-CEN; other partners: U. Aalto

Work in ongoing to implementation a FEM crystal plasticity (CP) approach to model the nanoindentation load-displacement based on the true stress – true strain curves otherwise derived from the tensile tests. The model is currently being developed to introduce flexible CP laws such that the effect of neutron irradiation and interaction between the plastic deformation and neutron irradiation defects (under progressive plastic deformation) could be accounted for.

Berkovich and spherical nano-indentation experiments have shown that the Eurofer97 samples display indentation size effect. The spherical nanoindentation load-displacement curves derived following the Pathak-Weaver approach show a strong dependence on the tip curvature. The indentation size effect is taken into account in FEM simulations by using a strain gradient plasticity model with a proper scaling factor. An approach based on conventional theory of mechanism-based strain-gradient plasticity has been adopted and has been implemented in the Abaqus code. The calculations performed have shown that the scaling factor adopts a value within the range 0.1 µm to 1 µm. This value yields a decent compatibility of indentation stress-strain curves at different values of tip radius. The influence of the experimental uncertainties such as surface roughness, surface detection procedure and its influence on zero-point determination and coefficient of friction between the tip and the test material will be considered.

Task 6.3: Semi-empirical modelling to identify different types of irradiation damage by nanoindentation data (M1 – M44)
Task leader: N. Jennett, U. Coventry; other partners: SCK-CEN

This task backfills the work needed to underpin a final indentation protocol and analysis method to identify different types of irradiation damage on the nanoindentation curves. Key issues have been identified. It is extremely important to:

- Achieve sample surfaces prepared in a manner that does not change their mechanical properties. This means careful removal of the previous work hardened layer in each stage of polishing and a final polish that is either chemo-mechanical or electro-polishing to remove the final mechanical damage layer;
- Know or measure the existence or generation of residual stresses and/or pile-up;
- Select an indentation cycle that optimises the determination of the contact stiffness and minimizes the effect of creep on the force removal gradient;
- Understand the effect on hardness of total amount of indentation creep allowed.

Methods to quantify the offset in results caused by residual stress and/or pile up have been investigated using the measured stiffness and a reference elastic modulus value. The effect of calibration, creep and other sources of instrumental or methodological bias have also been investigated. (**Figure 6.2**) Fe tensile specimens strained to 15 % engineering strain at room temperature, 125 °C and 300 °C have been analysed by nanoindentation and TEM. Results are currently being extended using newer more optimized indentation cycles. Pre-strained specimens exhibited higher hardness as compared to the non-strained counterpart due to dislocation multiplications during straining, but creep rates vary and affect the hardness values obtained. Differences in pile-up and residual stress are also being investigated. The focus of the task is now to optimize the correction of hardness data using elastic modulus, and the procedures to fit data sets to semi-empirical models to obtain the relationship between the derived fitting parameters and the microstructure and damage state.

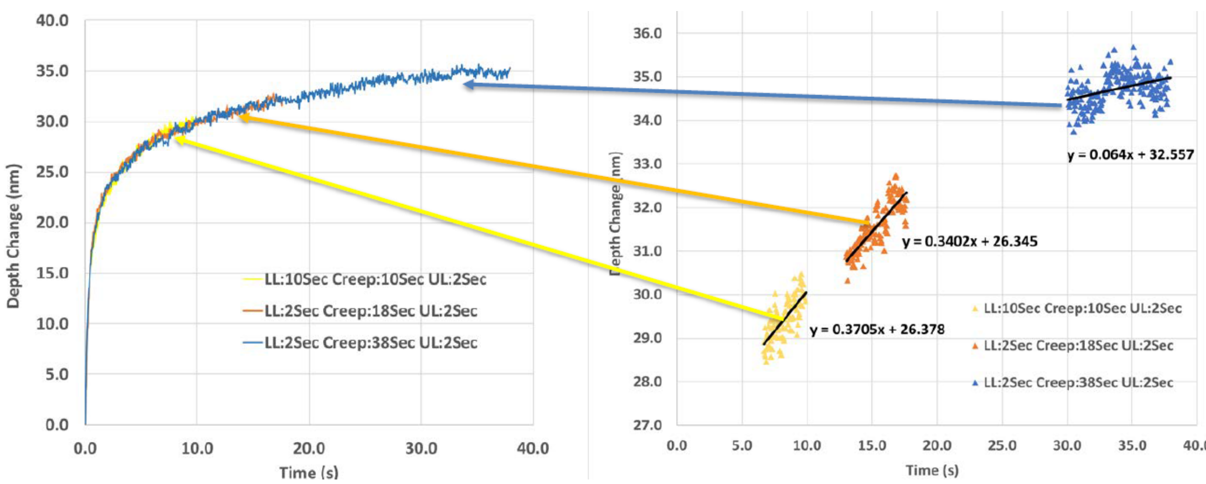

Figure 6.2. Selection of holding time to compensate for creep during NI experiments on pre-strained Fe.

Task 6.4: Nanoindentation testing of reference, ion- and neutron-irradiated materials (M1 – M48)

Task leader: F. Bergner, HZDR; other partners: U. AALTO, CIEMAT, CV REZ, JRC, NCBJ, PSI, UCov, SCK-CEN

Nanoindentation tests have been performed in four different ferritic/martensitic materials: the steel Eurofer97, the Fe-9Cr model alloy with ferritic microstructure, the Fe-9Cr model alloy with more complex martensitic-ferritic-bainitic microstructure and pure Fe. Berkovich and spherical nanoindentations have been carried out on the non-irradiated materials and in the materials irradiated with the following conditions:

- Fe^{2+} self-ions at 5 MeV and the following temperatures and doses:
  - RT, 1 dpa
  - 300 °C, 0.1 dpa
  - 300 °C, 1 dpa
  - 450 °C, 0.1 dpa
  - 450 °C, 1 dpa
- Fe^{2+} self-ions at 275 keV and the same T and doses as above (except 450 °C, 1 dpa).

Neutron-irradiated ferritic Fe-9Cr has been also characterized using a Berkovich indenter and applying continuous stiffness measurements. A protocol to optimize surface preparation of the neutron-irradiated samples has been optimized with the aim of obtaining equivalent surface finish in order to compare the nanoindentation response of unirradiated, and ion- and neutron-irradiated materials (**Figure 6.3**).

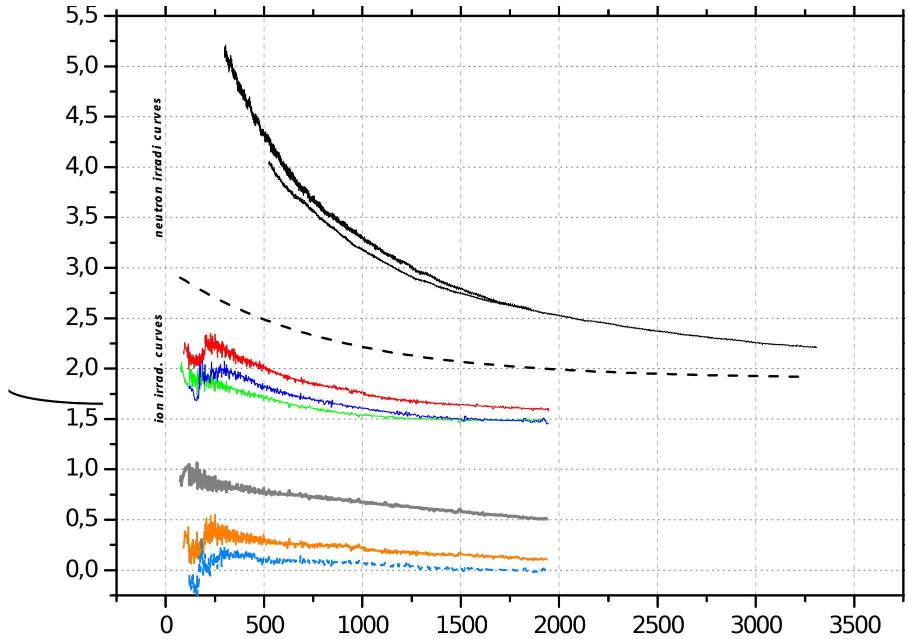

Figure 6.3. *Continuous stiffness measurement results for Fe9Cr samples. Corrected curve due to finished surface is shown as dotted line.*