ARTHEMIS

Assessment of Radiation damage and Transmutation Helium Effects on structural reactor materials via MIcromechanical testing and advanced analysis methodS

This project has received funding from the European Union's NextGenerationEU through the Recovery and Resilience Plan for Slovakia under project No. 09I03-03-V04-00384.

Deliverable D2.3

Recommendation on micromechanical testing of ion irradiated materials

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1. Introduction

The assessment of radiation effects on structural materials is one of the main challenges in the development of advanced nuclear systems [1-3]. Neutron irradiation experiments provide the most representative conditions but are time-consuming, costly, and limited by induced radioactivity. As an accelerated approach, ion irradiation is widely used for investigating radiation-induced changes in structural materials under well-controlled conditions [1,4]. By introducing controlled levels of displacement damage and, where required, implanted gases such as helium, ion irradiation enables the systematic investigation of microstructural evolution under irradiation and its effect on material performance. Compared with neutron irradiation, ion irradiation offers advantages in terms of shorter irradiation times, controllability of the irradiation parameters, and the possibility of producing pre-defined damage and gas concentration profiles. However, the limited penetration depth of energetic ions results in a thin modified layer, which presents a significant challenge for the direct mechanical characterization of the irradiated material. Conventional mechanical testing techniques, such as tensile or fracture testing, generally require specimen dimensions substantially larger than the thickness of the ion-irradiated layer. Consequently, their measured response is dominated by the unirradiated substrate and cannot directly provide the mechanical properties of the irradiated region.

Micromechanical testing techniques, including nanoindentation and micropillar compression, offer an alternative approach because their characteristic sampling volumes can be adapted to the dimensions of the irradiated layer. These techniques can therefore provide information on irradiation-induced changes in hardness, strength and deformation behaviour using relatively small irradiated volumes [5-7].

The interpretation of micromechanical measurements on ion-irradiated materials nevertheless requires particular attention to the relationship between the irradiation profile, testing geometry and material microstructure. The damage and implanted-gas concentrations may vary with depth, while the mechanical response can be affected by indentation size effects, micropillar dimensions, crystallographic orientation, grain size, surface preparation and focused-ion-beam-induced modifications. In addition, the local strength measured by micromechanical testing cannot necessarily be directly equated with a macroscopic mechanical property without considering these effects. Appropriate experimental design and complementary microstructural characterization are therefore essential for obtaining physically meaningful results.

Within the ARTHEMIS project, micromechanical testing was employed to investigate the mechanical response of ion-irradiated candidate materials and to assess the influence of helium implantation on their mechanical performance. The work focused primarily on EUROFER97 and Alloy 800H. Nanoindentation was used to characterize the mechanical response of reference and He-implanted materials. Micropillar compression was implemented for Alloy 800H, where the coarse-grained microstructure enabled the fabrication and testing of individual pillars within selected grains. The experimental results were complemented by microstructural characterization to support the interpretation of the observed mechanical response.

The report focuses on the micromechanical testing approaches implemented within the project and on the methodological experience obtained from their application to ion-irradiated materials. It formulates recommendations for the design, execution and interpretation of micromechanical tests on ion-irradiated materials. Emphasis is placed on the relation between irradiation depth and testing dimensions, the influence of specimen size and material microstructure, and the requirements to reliable extract irradiation-induced hardening and local deformation behaviour. The recommendations are intended to support the future application of micromechanical testing as a tool for the characterization and development of radiation-tolerant structural materials.

2. Characteristics of ion-irradiated layers relevant to micromechanical testing

2.1. Limited penetration depth and depth-dependent irradiation effects

A fundamental characteristic of ion irradiation is the limited penetration depth of the incident ions in the target material. Depending on the ion species and irradiation energy, the resulting modified region typically extends from several hundred nanometres to 1-2 micrometres below the surface. Within this region, the concentration of irradiation-induced defects and implanted species generally varies with depth. Consequently, the mechanical response of an ion-irradiated specimen cannot be considered independently of the depth distribution of the irradiation damage.

For conventional mechanical testing, the thickness of the irradiated layer is usually insufficient to dominate the measured response. Tensile, bending or fracture specimens typically sample material volumes several orders of magnitude larger than the ion-irradiated region. The measured mechanical properties therefore represent a combination of the irradiated surface layer and the underlying unirradiated material. Micromechanical techniques overcome this limitation by reducing the characteristic testing volume and enabling measurements to be performed within or close to the irradiated region.

The compatibility between the irradiation depth and the characteristic dimensions of the mechanical test is therefore one of the primary considerations when designing an ion-irradiation and micromechanical-testing experiment. For nanoindentation, this compatibility is determined primarily by the indentation depth and the associated plastically deformed volume. For micropillar compression, it is determined by the pillar diameter, height and depth of the irradiated region. In both cases, the dimensions of the mechanically sampled volume must be considered together with the depth dependence of the irradiation-induced microstructure.

A further complication is that the irradiation-induced changes are not necessarily limited to displacement damage. When gas ions such as helium are implanted, the concentration of helium also varies with depth and can interact with irradiation-induced defects. Depending on the irradiation conditions, helium may form atomic-scale complexes, bubbles or other defect structures. The resulting mechanical response is therefore determined by the

combined effects of displacement damage, implanted-gas concentration and the evolution of the underlying microstructure.

2.2. ARTHEMIS irradiation approach

Within ARTHEMIS, a multi-energy helium implantation strategy was developed to produce an extended and quasi uniform helium-containing region suitable for subsequent materials characterization and micromechanical testing. EUROFER97 and Alloy 800H specimens were implanted using a sequence of helium-ion irradiation steps with energies decreasing from approximately 17 MeV to 500 keV. The use of multiple energies was intended to broaden the implantation profile and to obtain a substantially thicker modified layer than would be achieved using a single implantation energy.

For the representative irradiation campaign considered in ARTHEMIS, a total of 33 implantation steps were employed. The resulting helium-containing region extended over approximately 65 µm below the specimen surface with an average helium concentration of approximately 1000 appm. The calculated displacement damage was approximately 0.12 dpa. Figure 1 presents the calculated helium concentration and displacement-damage profiles for the multi-energy implantation. The individual implantation steps overlap to produce an extended region in which the helium concentration and displacement damage vary substantially less than for a single-energy implantation.

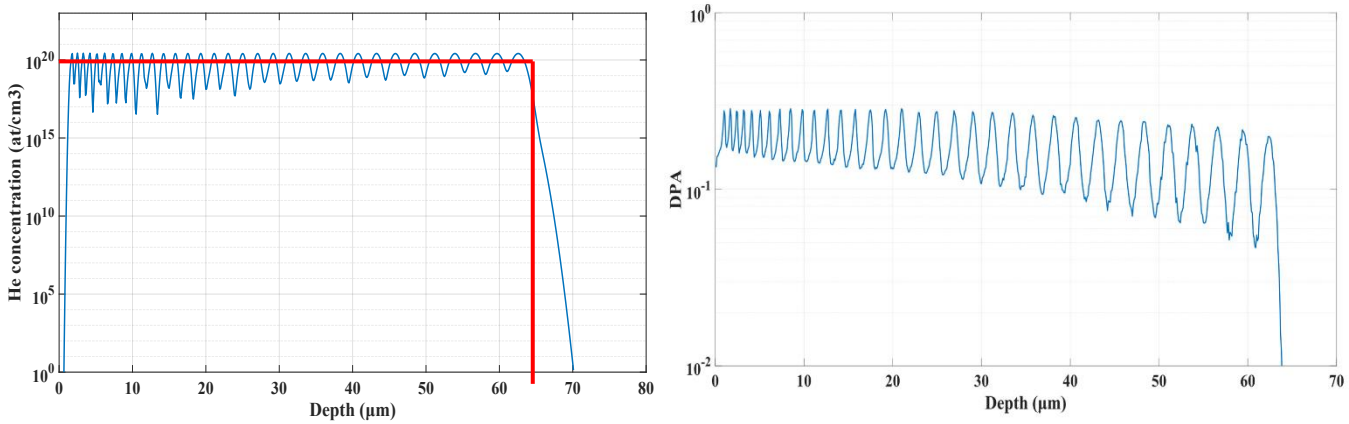


Figure 1. Calculated depth profiles of helium concentration and displacement damage for the multi-energy He implantation used in ARTHEMIS.

The irradiation profile is particularly relevant for micromechanical testing because it determines the maximum volume in which the mechanical response can be attributed predominantly to the modified material. In the present case, the approximately 65 µm thick implanted region provides substantially more flexibility in the selection of the indentation depth and micropillar dimensions than a conventional shallow implantation profile.

It should nevertheless be noted that the nominal thickness of the implanted region should not be interpreted as an exact boundary between irradiated and unirradiated material. Both the damage and helium concentration distributions are continuous functions of depth. In addition, the mechanically affected volume associated with a particular test geometry extends beyond the nominal contact or specimen dimensions. The transition from the irradiated to the unirradiated material must therefore be considered when selecting the testing parameters.

2.3. Relationship between irradiation profile, microstructure and testing volume

The irradiation depth represents only one of the length scales that need to be considered when selecting a micromechanical testing method. The characteristic microstructural dimensions of the material can impose equally important constraints. These include grain size, precipitate or second-phase dimensions, irradiation-induced defect spacing and, in the case of helium implantation, the characteristic size and distribution of He-containing defects.

This is particularly important when micropillar compression is considered. A pillar fabricated from a coarse-grained material may be contained entirely within a single grain, allowing the crystallographic orientation and slip behaviour to be controlled. In a fine-grained material, however, a pillar of the same diameter may contain several grains or may approach the dimensions of an individual grain. The measured response can then depend strongly on grain boundaries and the specific microstructure sampled by the pillar.

The ARTHEMIS materials illustrate this difference. Alloy 800H has a coarse-grained microstructure, with grains substantially larger than the approximately 2 µm diameter pillars used for micropillar compression. This enabled pillars to be fabricated within individual grains and tested under controlled crystallographic orientations. EUROFER97, in contrast, has a much finer ferritic/martensitic microstructure, with characteristic grain or prior-austenite-related dimensions of only a few micrometres. Consequently, the use of very

small pillars in EUROFER97 would introduce a strong coupling between pillar size and grain structure, while larger pillars would increasingly sample multiple grains.

For nanoindentation, the relevant length scale is different. The indentation depth determines the size of the plastically deformed zone beneath the indenter, while the lateral dimensions of the indentation determine the volume contributing to the measured response. If the indentation depth becomes too large relative to the thickness of the irradiated layer, an increasing fraction of the deformation occurs in the underlying unirradiated material. The measured hardness will then underestimate the hardness of the irradiated layer. Conversely, very shallow indentation can increase sensitivity to surface roughness, preparation artefacts and instrument-related uncertainties.

The selection of an appropriate test dimension therefore requires consideration of at least three length scales:

1. the irradiation depth and depth distribution of damage and implanted species,
2. the characteristic microstructural dimensions of the material,
3. the characteristic sampling volume of the selected micromechanical test.

The relationship between these length scales determines whether a measurement can be interpreted as a representative property of the irradiated material or whether the result is strongly affected by the substrate, grain structure or size effects.

2.4. Implications for micromechanical testing

The above considerations demonstrate that micromechanical testing cannot be treated as a separate step performed after irradiation. The irradiation conditions and the intended mechanical characterization should ideally be designed together. In particular, the irradiation profile should provide a sufficiently large and sufficiently well-characterized modified volume for the selected testing method.

For nanoindentation, the indentation depth should be selected such that the mechanically affected volume remains predominantly within the irradiated region over the range used for quantitative comparison. A series of indentation depths is generally preferable to relying on a single nominal depth because it allows the depth dependence of the measured hardness and modulus to be evaluated. Measurements on an identically prepared unirradiated

reference specimen are essential for distinguishing irradiation-induced changes from surface and indentation-size effects.

For micropillar compression, the pillar diameter and height must be selected by considering not only the irradiation depth but also the grain size and expected size effects. The pillar should be sufficiently small to remain within the irradiated region while being sufficiently large to limit excessive small-scale strengthening and fabrication-related effects. Where single-crystal pillars are used, crystallographic orientation should be characterized before fabrication and maintained consistently between reference and irradiated conditions.

The ARTHEMIS irradiation strategy illustrates the importance of this integrated approach. The production of an approximately 65 µm thick helium-containing layer enabled micromechanical testing over a substantially larger dimensional range than would be possible for a shallow single-energy implantation. At the same time, the different grain structures of EUROFER97 and Alloy 800H demonstrated that the suitability of a particular micromechanical test geometry is material/microstructure-dependent.

Therefore, an appropriate micromechanical testing strategy for ion-irradiated materials should be based on the combined consideration of irradiation depth, damage and gas concentration profiles, material microstructure, specimen geometry and the characteristic sampling volume of the mechanical test. These considerations form the basis for the recommendations developed in the subsequent sections of this deliverable.

3. Nanoindentation assessment of ion-irradiated materials

Nanoindentation was used within ARTHEMIS to assess the changes in local mechanical properties resulting from helium implantation. Measurements were performed on reference and He-implanted EUROFER97 and Alloy 800H. The technique was selected because its small characteristic sampling volume enables mechanical characterization of the near-surface irradiated.

The nanoindentation results demonstrated irradiation-induced hardening in both materials, although the magnitude of the response differed. EUROFER97 exhibited an increase in hardness of approximately 1.1 GPa following He implantation. This increase was accompanied by a reduction of approximately 17% in reduced modulus. Alloy 800H showed a smaller relative increase in hardness, approximately half that observed for EUROFER97 under the irradiation conditions investigated. The difference between the two materials illustrates that the mechanical response to ion irradiation depends not only on the irradiation conditions but also on the material's initial microstructure and its response to the formation and evolution of irradiation-induced defects. A representative comparison of the measured hardness changes in the investigated materials is shown in Figure 2.

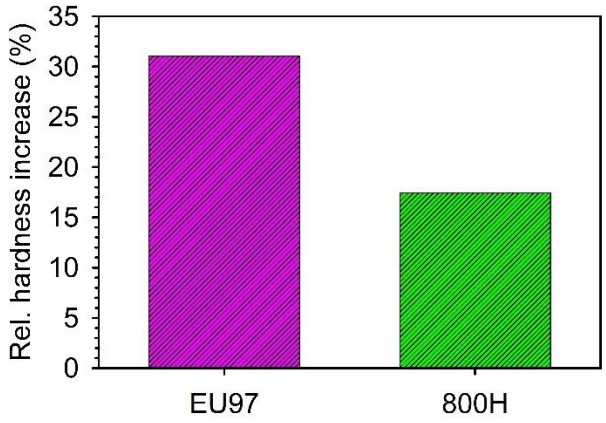


Figure 2. Comparison of the irradiation-induced hardness increase measured by nanoindentation in EUROFER97 and Alloy 800H.

3.1. Indentation depth and indentation size effects

The contact depth in the conventional single-indentation measurements was approximately 380 nm. To investigate the depth dependence of the mechanical response, cyclic indentation measurements were additionally performed over a contact-depth range of approximately 50–390 nm. The indentation size effect (ISE) was observed consistently in the unirradiated specimens, with the measured hardness increasing with decreasing contact depth. At larger indentation depths, the hardness approached the value obtained from the single-indentation measurements.

The observation of an ISE is important when comparing the reference and irradiated conditions. The measured hardness at shallow indentation depths can be affected by several factors, including surface preparation, surface roughness and the intrinsic size dependence of plastic deformation. Consequently, comparisons between irradiation conditions should preferably be performed at identical contact depths and using an appropriately characterized reference material.

In the present measurements, the irradiated material exhibited higher hardness than the corresponding unirradiated material over the investigated depth range. The persistence of this difference across the measured range supports the conclusion that the observed hardness increase is associated with irradiation-induced changes rather than being solely a consequence of the indentation size effect.

The maximum contact depth investigated in the cyclic measurements was approximately 390 nm and was limited by the applied force. At these relatively shallow depths, the indentation response remains within a small fraction of the approximately 65 µm thick He-implanted region described in Section 2. Consequently, the measured hardness can be considered representative of the near-surface irradiated material without requiring a correction for penetration into the unirradiated substrate. Nevertheless, the actual mechanically affected volume extends below and laterally beyond the nominal contact dimensions, and the irradiation profile should always be considered when selecting the indentation depth.

A direct validation of the depth dependence of the irradiated layer could be obtained by performing nanoindentation on a cross-section through the implanted region. Such measurements would allow the hardness to be mapped as a function of distance from the irradiated surface and could provide a direct comparison with the calculated irradiation profile. This measurement was not performed within ARTHEMIS because the irradiated

specimens were retained for complementary characterization. Cross-sectional nanoindentation can therefore be considered a useful follow-up approach for future work.

3.2. Lessons learned from nanoindentation

The ARTHEMIS results demonstrate that nanoindentation is a practical and sensitive method for detecting irradiation-induced hardening in ion-irradiated materials. Its principal advantage is the ability to characterize very shallow modified layers while requiring only a limited specimen volume. This makes it particularly suitable for comparative assessment of reference and irradiated materials.

Several methodological considerations should nevertheless be taken into account. First, the indentation depth must be selected in relation to the calculated irradiation profile and should remain sufficiently small to minimize the contribution of the underlying unirradiated material. Second, the indentation size effect should be characterized using a suitable range of depths rather than assuming that a single indentation depth is independent of depth. Third, measurements on the reference material should be performed under identical conditions to those used for the irradiated material. Finally, a sufficiently large number of indents should be used to account for local microstructural variations and experimental scatter.

For the ARTHEMIS conditions, the relatively large thickness of the multi-energy He-implanted layer provided a favourable geometry for nanoindentation. The approximately 380–390 nm maximum contact depth used in the measurements was small compared with the implanted-layer thickness, allowing the irradiation-induced hardness change to be assessed without a significant direct contribution from the unirradiated substrate. Cross-sectional nanoindentation represents a complementary approach for future studies where preservation of the irradiated specimen is not required.

Overall, nanoindentation provides a suitable first-line technique for identifying and quantifying irradiation-induced hardening in ion-irradiated materials. However, hardness should be interpreted as a local mechanical response rather than directly equated with macroscopic yield strength. Complementary micromechanical testing and microstructural characterization are required when the objective is to identify the mechanisms responsible for the observed hardening and to relate the local measurements to bulk mechanical behaviour.

4. Micropillar compression of Alloy 800H

Micropillar compression was employed in ARTHEMIS to complement nanoindentation by providing a direct measurement of the local mechanical response of the ion-irradiated material in the form of stress–strain curves. In contrast to nanoindentation, which provides hardness and reduced modulus from a localized indentation volume, micropillar compression enables the determination of a local flow stress and the investigation of deformation behaviour under uniaxial loading.

Alloy 800H was selected for micropillar testing because its coarse-grained microstructure is particularly well suited to this type of experiment. The grain size is typically larger than 200 µm, which is substantially larger than the dimensions of the micropillars investigated in this work. Individual pillars can therefore be fabricated entirely within a single grain, allowing the crystallographic orientation and active slip system to be controlled. This is an important advantage when comparing reference and ion-irradiated conditions because it minimizes the influence of grain boundaries and allows differences in the mechanical response to be related more directly to the irradiation-induced changes.

The relatively large thickness of the He-implanted layer was an additional prerequisite for micropillar testing. The approximately 65 µm thick modified region produced by the multi-energy implantation described in Section 2 is substantially larger than the dimensions of the micropillars. The pillars could therefore be fabricated and compressed entirely within the implanted layer, avoiding a direct mechanical contribution from the unirradiated substrate. The experimental approach followed the sequence:

Ion implantation → EBSD mapping → FIB milling → micropillar compression

This sequence was found to be important for obtaining reproducible and physically interpretable results.

4.1. Pillar fabrication and EBSD-guided experimental strategy

Micropillars were fabricated using focused ion beam (FIB) milling in a TESCAN Solaris FIB/SEM system. Pillars with top diameters below approximately 3 µm were investigated, with an aspect ratio maintained in the range of approximately 2:1–3:1 and a taper angle

below approximately 3°. Several pillar diameters were investigated to assess the influence of specimen dimensions on the measured mechanical response.

Electron backscatter diffraction (EBSD) was an integral part of the experimental workflow rather than an additional characterization step. The material surface was mapped prior to pillar fabrication, allowing grains with a suitable crystallographic orientation and sufficiently large area to be identified. Pillar locations were then selected from the EBSD map and subsequently fabricated by FIB milling. This approach provided control over both the grain in which the pillar was located and its crystallographic orientation with respect to the loading direction.

The large grain size of Alloy 800H provides an important advantage for this type of experiment. Since the grains are substantially larger than the pillar diameter, individual pillars can be positioned well within single grains and sufficiently far from neighbouring grain boundaries. This enables the deformation behaviour to be studied under conditions approaching those of a single crystal, while retaining the actual microstructural characteristics of the material.

For the experiments reported here, grains were selected such that the pillar loading direction corresponded approximately to the <123> crystallographic direction. This orientation was selected to provide a well-defined, single-slip deformation configuration under uniaxial compression. The same orientation-selection procedure was applied to both the reference and He-implanted conditions. This was important for minimizing variations in flow stress arising from crystallographic orientation and thereby improving the comparability of the two material conditions.

Figure 3 illustrates the resulting experimental workflow, comprising ion implantation, EBSD mapping and grain selection, FIB fabrication of the micropillars, and subsequent in-situ SEM compression testing. The combination of EBSD-guided site selection and controlled pillar fabrication enabled pillars with different diameters to be produced within suitably oriented individual grains.

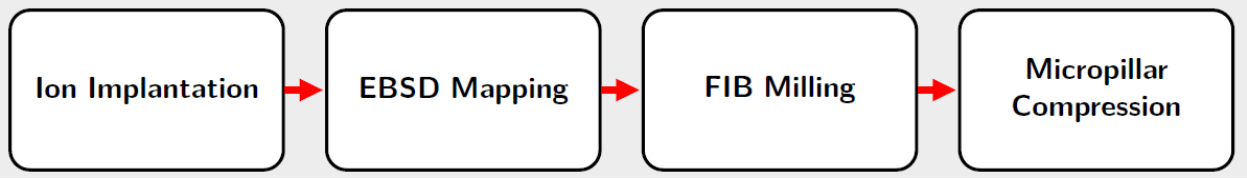
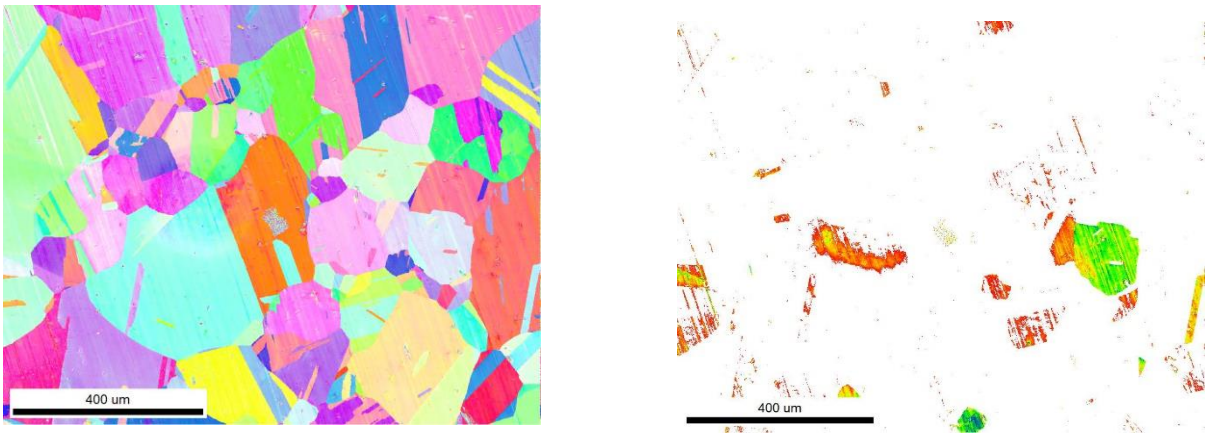


Figure 3. Experimental workflow for EBSD-guided micropillar compression of He-implanted Alloy 800H.

Figure 4 shows an example of the EBSD characterization used for pillar site selection. The orientation map was used to identify grains fulfilling the required crystallographic criterion for loading approximately along the <123> direction.

The use of EBSD-guided site selection therefore provided a controlled experimental configuration in which irradiation-induced changes in deformation behaviour could be investigated while reducing two important sources of variability in micropillar testing: grain-boundary interactions and crystallographic orientation.



a) b)

Figure 4. EBSD characterization of Alloy 800H used for micropillar site selection. (a) EBSD orientation map of the investigated area. (b) Selected grains fulfilling the orientation criterion for micropillar compression with loading approximately along the <123> direction.

4.2. In-situ micropillar compression

Micropillar compression was performed in an SEM using an Alemnis ASA micromechanical testing system. The in-situ configuration enabled the mechanical response to be recorded while simultaneously observing the deformation of the pillar.

The micropillars were compressed under displacement-controlled conditions using a flat-ended Ø10µm sized compression punch. The applied force and displacement were recorded during the test and converted into engineering stress and strain using the pillar geometry. The stress–strain response was evaluated primarily in terms of the flow stress, while the corresponding SEM observations were used to identify the deformation behaviour.

Representative engineering stress–strain curves for the reference and He-implanted Alloy 800H are shown in Figure 5. The He-implanted pillars exhibit a pronounced increase in the stress required for plastic flow compared with the reference condition. Under the investigated conditions, the flow stress of the He-implanted material was approximately a factor of two higher than that of the reference material.

The increase in flow stress is consistent with the irradiation-induced hardening observed by nanoindentation. The micropillar measurements provide complementary information because the mechanical response is expressed directly as a local stress–strain relationship rather than as an indentation hardness. The agreement in the direction of the mechanical change obtained using the two techniques supports the interpretation that He implantation produced a significant modification of the plastic deformation behaviour of Alloy 800H.

The magnitude of the measured flow stress should, however, not be interpreted directly as the macroscopic yield strength of Alloy 800H. At the micrometre scale, plastic deformation is affected by specimen dimensions, dislocation source availability, crystallographic orientation and free-surface effects. Consequently, the principal value of the micropillar results within the present study is the controlled comparison between reference and He-implanted conditions and the investigation of the associated deformation mechanisms.

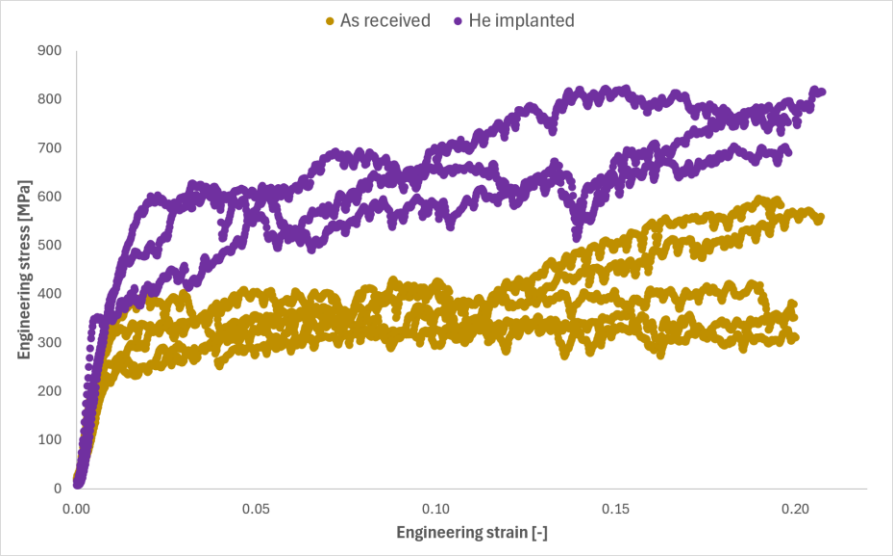


Figure 5. Representative engineering stress–strain curves obtained from micropillar compression of reference and He-implanted Alloy 800H.

SEM observation during and after compression provided additional information on the deformation mode of the micropillars. In the selected crystallographic orientation, plastic deformation occurred predominantly through slip, resulting in localized surface traces and/or shear offsets on the pillar surface. The deformation morphology provides an important complement to the stress–strain curves because it confirms that the measured plastic response is associated with crystallographic deformation rather than, for example, gross pillar instability or premature failure.

Representative SEM images before and after compression are shown in Figure 6. The comparison demonstrates the change in pillar geometry and the formation of deformation fbands associated with plastic slip.

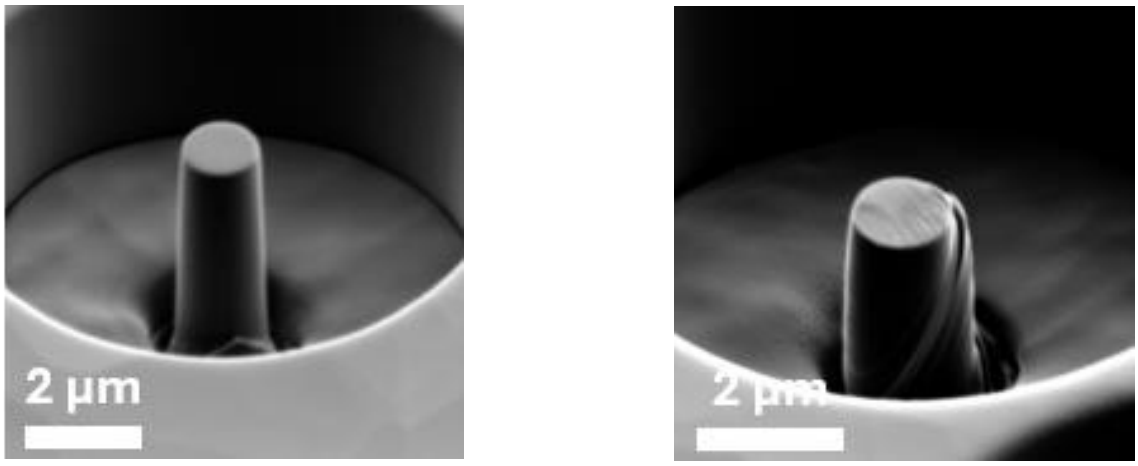


Figure 6. SEM images of representative Alloy 800H micropillars before and after compression.

The combination of controlled crystallographic orientation and in-situ SEM observation is particularly useful for distinguishing between different deformation responses. In the reference material, plastic flow occurs at lower applied stress, whereas the He-implanted material requires substantially higher stress to initiate and sustain plastic deformation. The increased flow stress can therefore be interpreted in the context of irradiation-induced obstacles to dislocation motion, provided that the corresponding microstructural information is taken into account.

The deformation observations also demonstrate the benefit of selecting pillars from grains with the same crystallographic orientation. Without such control, differences in the resolved

shear stress and available slip systems could produce substantial variations in the measured flow stress and could obscure the effect of irradiation.

4.3. Pillar size and microstructural considerations

Several pillar diameters were therefore investigated within the ARTHEMIS study. The use of a diameter series provides information on the extent to which the measured mechanical response is affected by specimen dimensions and is an important prerequisite for selecting an appropriate geometry for comparison between different irradiation conditions. A clear size effect, manifested by an increase in the measured flow stress with decreasing pillar diameter, was observed for the unirradiated micropillars, as shown in Figure 7.

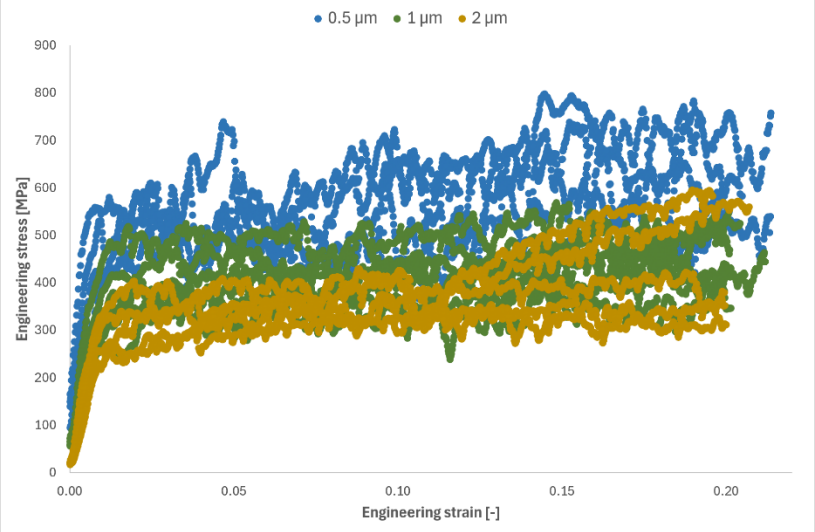


Figure 7. Flow stress of unirradiated Alloy 800H micropillars as a function of pillar diameter, showing the increase in measured flow stress with decreasing pillar size (size effect).

The pillar diameter must therefore be considered in relation to the characteristic microstructural length scales of the material, while the associated size effects must be taken into account when interpreting the measured mechanical response. This is particularly important when transferring a micropillar methodology between materials with substantially different microstructures. In Alloy 800H, the grain size exceeds 200 µm, which

is much larger than the investigated pillar dimensions. This allowed micropillars with diameters of approximately 2 μm to be fabricated entirely within individual grains. When positioned sufficiently far from a grain boundary, such pillars can be regarded as single-grain specimens, with a negligible contribution from grain-boundary interactions to the measured deformation behaviour.

The use of small pillars does not, however, eliminate the intrinsic size effect associated with micropillar deformation. Instead, the large grain size allows the effects of specimen dimensions to be investigated without introducing an additional contribution from grain boundaries. Consequently, pillar diameter, crystallographic orientation and grain size should be considered separately when interpreting micropillar strength.

The Alloy 800H results therefore provide an important methodological lesson for the micromechanical testing of ion-irradiated materials: pillar dimensions must be selected in relation to both the irradiation depth and the characteristic microstructural length scales of the material. The unusually large grains of Alloy 800H provide favourable conditions for controlled single-grain micropillar experiments, whereas application of the same approach to fine-grained materials requires additional consideration of grain-boundary effects and the relationship between pillar diameter and grain size.

4.4. Lessons learned from micropillar compression

The micropillar experiments on Alloy 800H provided several methodological insights relevant to the application of micropillar compression to ion-irradiated materials. The approximately 65 μm thick He-implanted region used in ARTHEMIS provided sufficient depth to accommodate the investigated micropillars entirely within the modified layer. This is an important consideration for ion-irradiated materials, as insufficient irradiation depth relative to the pillar dimensions can result in a significant contribution from the underlying unirradiated material to the measured mechanical response.

The experiments further demonstrated that specimen preparation and site selection are integral parts of the micromechanical testing methodology. EBSD-guided selection of grain position and crystallographic orientation enabled a controlled comparison between reference and He-implanted material. In particular, the use of the same crystallographic orientation for both conditions reduced the influence of orientation-dependent plasticity on the comparison. The experimental workflow can therefore be summarized as:

Ion implantation → EBSD characterization → grain and orientation selection → FIB pillar fabrication → in-situ SEM compression → stress–strain and deformation analysis.

Several additional considerations should be taken into account when applying this approach to other materials:

- Irradiation depth: The pillar dimensions should be selected such that the mechanical response is dominated by the irradiated region. The irradiation profile and pillar geometry should therefore be considered together during experimental design.
- Crystallographic orientation: EBSD-guided site selection is particularly beneficial when crystallographic orientation has a significant influence on plastic deformation. Using comparable orientations for reference and irradiated conditions improves the reliability of the comparison.
- Grain size: The pillar dimensions should be considered relative to the characteristic microstructural length scales. The large grains of Alloy 800H allow individual grains to be tested, whereas the application of similar pillar dimensions to fine-grained materials such as EUROFER97 would introduce substantially stronger grain-boundary effects.
- Pillar diameter: Micropillar strength exhibits a pronounced size dependence. A series of pillar diameters is therefore preferable when establishing a testing methodology and assessing the influence of specimen dimensions.
- Specimen preparation: FIB fabrication quality, pillar geometry and surface condition can affect the measured response. The fabrication procedure should consequently be validated before systematic comparison of irradiation conditions.
- In-situ observation: SEM observation during and after compression provides complementary information on the deformation behaviour and can help identify slip localization, deformation modes and possible preparation-related artefacts.
- Interpretation of strength: Micropillar flow stress represents a local micromechanical response and should not be directly equated with macroscopic yield strength. Size effects, crystallographic orientation, dislocation source availability and microstructural constraints must be considered when relating the results to bulk mechanical properties.

The combination of a sufficiently thick and well-characterized irradiated layer, EBSD-based site selection, controlled FIB fabrication and in-situ micropillar compression provided a suitable methodology for investigating the local mechanical response of He-implanted Alloy

800H. The approach is particularly advantageous for coarse-grained materials where individual grains can accommodate the complete pillar geometry. For fine-grained materials, the pillar dimensions and testing configuration require additional consideration of grain-boundary effects and the relationship between pillar size and grain size.

5. Recommendations for micromechanical testing of ion-irradiated materials

The results obtained within ARTHEMIS demonstrate that micromechanical testing can provide useful information on the mechanical response of ion-irradiated materials, provided that the characteristics of the irradiated layer and the limitations of the selected technique are considered during experimental design. In particular, irradiation depth and profile, material microstructure, surface condition and test dimensions should be considered together. Based on the experience gained from nanoindentation and micropillar compression, the following recommendations are formulated.

5.1. Design of irradiation and mechanical testing

The irradiation conditions and the intended mechanical characterization should ideally be designed together. Since ion irradiation produces a relatively shallow modified layer, its thickness and depth-dependent damage and implanted-species profiles directly influence the applicable micromechanical technique and test geometry.

For nanoindentation, the indentation depth and associated plastically affected volume should remain sufficiently small relative to the irradiated layer to minimize the contribution of the underlying unirradiated material. For micropillar testing, the pillar dimensions should likewise be selected with respect to the thickness and profile of the irradiated region. A sufficiently thick and well-characterized modified layer therefore provides greater flexibility in selecting the test geometry.

Where a relatively thick and approximately uniform modified region is required, multi-energy ion implantation can be considered. The irradiation profile should be calculated in advance and, where relevant, experimentally verified. The influence of the near-surface region, surface preparation and any depth-dependent variation in damage or implanted-species concentration should be considered when defining the mechanical test volume.

5.2. Nanoindentation

Nanoindentation is well suited for screening and quantifying irradiation-induced changes in hardness because it probes a small material volume and allows a relatively large number of measurements. The indentation depth should be selected based on the thickness and profile of the irradiated layer rather than on sample thickness alone.

A series of indentation depths is recommended to assess indentation size effects and to verify that the measured hardness is dominated by the irradiated region. At very shallow depths, surface roughness, preparation, tip calibration and other near-surface effects can influence the measurement, whereas excessive indentation depth can introduce a contribution from the underlying unirradiated material. Repeated measurements or arrays of indents with sufficient spacing should be used to distinguish systematic trends from local microstructural variability.

Where preservation of the original specimen is not required, cross-sectional nanoindentation can provide additional information by mapping hardness as a function of depth and directly comparing the mechanical response with the irradiation profile.

5.3. Micropillar compression

Micropillar compression provides complementary information when a local stress–strain response and direct observation of plastic deformation are required. It is particularly useful when the irradiated layer is sufficiently thick to accommodate the selected pillar geometry.

For materials with sufficiently large grains, EBSD-guided selection of pillar sites is recommended. Comparable crystallographic orientations should be used for reference and irradiated conditions where practical, thereby reducing the influence of orientation-dependent plasticity. Pillar dimensions should also be varied where possible because micropillar flow stress depends on specimen dimensions, crystallographic orientation, dislocation source availability and specimen preparation. A single pillar diameter should therefore not be interpreted as a representative material strength without consideration of these effects.

Pillar dimensions must additionally be selected relative to the material microstructure. In coarse-grained materials such as Alloy 800H, individual grains can accommodate the complete pillar geometry, allowing controlled single-grain measurements. In fine-grained materials such as EUROFER97, similar pillar dimensions may be comparable to the grain

size and introduce significant grain-boundary and orientation effects. Consequently, a micropillar geometry established for one material should not automatically be transferred to another material with a substantially different microstructure.

FIB fabrication and surface preparation should be controlled consistently for reference and irradiated conditions. Pillar geometry, taper, sidewall quality and surface damage should be monitored, and the fabrication procedure should be validated before systematic testing. A sufficient number of individual pillars should be tested to account for local microstructural and stochastic variability.

5.4. Complementary characterization and interpretation

Micromechanical measurements should preferably be combined with microstructural characterization. Mechanical testing can demonstrate a change in hardness or flow stress, but independent information on irradiation-induced defects, helium-related features, grain structure and composition is generally required to identify the underlying hardening mechanisms. Depending on the material and irradiation conditions, techniques such as TEM, SANS, EBSD and ion-beam analysis can therefore provide important complementary information.

In general, Nanoindentation and micropillar compression should be regarded as complementary rather than interchangeable techniques. Nanoindentation is particularly suitable for efficient assessment of local hardness changes over a relatively large number of measurements, whereas micropillar compression provides access to local plastic deformation and stress–strain behaviour under controlled crystallographic and microstructural conditions. In both cases, meaningful interpretation requires the test volume and geometry to be considered together with the irradiation profile and material microstructure. The resulting micromechanical properties should subsequently be related to macroscopic behaviour with appropriate consideration of scale, crystallographic and specimen-size effects.

Table 1. Summary of recommendations for micromechanical characterization of ion-irradiated materials

Technique / approach	Main purpose	Critical parameters	Main considerations / limitations
Nanoindentation	Screening and quantification of irradiation-induced hardness changes	Indentation depth relative to irradiated-layer thickness; indentation spacing and number of measurements	Indentation size and near-surface effects; excessive depth may include unirradiated material
Micropillar compression	Local stress–strain response and investigation of plastic deformation	Pillar diameter and aspect ratio; irradiation depth; crystallographic orientation; grain size	Size and orientation effects; FIB-induced effects and stochastic deformation; local flow stress is not directly equivalent to macroscopic yield strength
Cross-sectional nanoindentation	Depth-resolved assessment of irradiation-induced hardening	Cross-section quality; indentation depth and spacing	Destructive preparation; useful for comparison with the irradiation profile
Complementary microstructural characterization	Identification of irradiation-induced defects and interpretation of mechanical response	Appropriate spatial resolution and characterization depth	Required for linking mechanical changes to specific microstructural features or hardening mechanisms

6. Summary

Two The ARTHEMIS study demonstrated the applicability of micromechanical testing for assessing the mechanical response of ion-irradiated materials and provided practical recommendations for the selection and implementation of suitable testing approaches. The work focused on nanoindentation and micropillar compression, complemented by microstructural characterization, with particular attention to the specific constraints imposed by the limited penetration depth of ion irradiation.

Based on the experimental experience gained within ARTHEMIS, recommendations were formulated for the design and interpretation of nanoindentation and micropillar compression experiments on ion-irradiated materials. In particular, the irradiation depth and profile, material microstructure, crystallographic orientation, specimen dimensions and preparation conditions should be considered together when selecting the appropriate testing approach. The recommendations provide a practical framework for obtaining reliable and interpretable micromechanical data and for relating local mechanical response to the underlying irradiation-induced microstructural changes and, where appropriate, to macroscopic mechanical behaviour.

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