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Deformation Mechanisms of Zirconium Alloys after Irradiation Studied by Dislocation Dynamics Simulations and In Situ Straining Experiments in TEM

Citation

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ABSTRACT

Zirconium alloys, used in the nuclear industry, are subjected to neutron irradiation that affects their mechanical properties. At the microscopic scale, neutron irradiation creates small dislocation loops that act as obstacles against dislocation glide, explaining the irradiation hardening. Transmission electron microscopy observations performed after post-irradiation mechanical tests have shown that loops are swept out by gliding dislocations, creating thin zones free of defects. Observations have proven that slip occurs preferably in the basal plane, a puzzling fact as dislocations mainly glide in the prismatic plane in unirradiated conditions. In order to understand this phenomenon, discrete dislocation dynamics (DD) simulations, on complex configurations, have been performed. The input parameters of this code have been adjusted on molecular dynamics simulations. Then interactions between loops and mixed dislocations

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gliding either in the prismatic or basal plane have been simulated. These simulations show that prismatic glide is always impeded in the mixed-screw direction, whereas for basal slip, clearing or a weak interaction occurs in the mixed-screw direction, allowing an easy glide of basal dislocations. Furthermore, all three basal systems can contribute to clearing in the basal plane contrary to prismatic slip. These two reasons explain the easy basal glide and clearing of loops after irradiation. Moreover, in situ straining experiments inside a transmission electron microscopy have been conducted on ion-irradiated recrystallized Zircaloy-4. Several interactions between dislocations and loops have been observed in situ. The DD code has been used to simulate these interactions. A fair agreement is obtained between simulations and experiments, showing the relevance of the DD numerical tool.

Keywords

dislocation, deformation, simulation, loop, irradiation

Introduction

Zirconium (Zr) alloys are widely used as cladding tubes for the nuclear fuel of light water reactors. During in-reactor operation, these materials undergo radiation damage due to fast neutrons. Moreover, they are subjected to various mechanical loadings leading to irreversible visco-plastic deformation. In order to ensure the integrity of the cladding, various empirical models are used. To improve the predictive capability of the numerical simulations, it is of interest to develop physically based models that take into account the microscopic mechanisms.

At the microscopic scale, fast neutron irradiation leads to the formation of point defects that diffuse and cluster together in the form of small dislocation loops. In zirconium alloys, the most stable point defect clusters are the perfect $\langle a \rangle$ loops, with a Burgers vector $\underline{b} = 1/3\langle 11\bar{2}0 \rangle$, located close to the prismatic habit planes.^{1,2} These loops can be either interstitial or vacancy loops. They appear rapidly after the start of irradiation. They are small, typically 10 nm in diameter, and very numerous, with a typical number density of 10^{22} m⁻³.

The formation of these irradiation defects has a strong influence on the mechanical behavior of the material. It is well established that the hardening measured after irradiation is directly related to formation of $\langle a \rangle$ loops. Indeed, these loops act as obstacles against dislocation glide. The hardening is associated with a decrease of the uniform elongation and an early necking, with the failure remaining ductile. This is due to a loss of work-hardening capability. These phenomena are related to the formation of $\langle a \rangle$ loops and to their interaction with gliding dislocations.

In order to understand the underlying mechanisms at the origin of the plastic deformation of irradiated zirconium alloys, several authors^{3–13} have analyzed, by transmission electron microscopy (TEM), thin foils taken out of samples tested in hot cell after neutron irradiation. Thin, white, cleared bands were observed after testing. In these bands, loops have been swept away by gliding dislocations, creating zones where further dislocations can glide freely and thus remain confined in these

channels. It was shown in Onimus et al.^{10,11} and in Onimus, Béchade, and Gilbon¹³ that, for transverse tensile and internal pressure tests conducted at 350°C, the channels observed after testing were only basal channels, whereas in the same testing conditions, easy prismatic slip was observed in the nonirradiated material.

To obtain a better understanding of the processes leading to the channeling phenomenon, some authors have conducted molecular dynamics (MD) simulations of elementary interactions between individual dislocations and loops.^{14–16} Serra and Bacon¹⁵ studied the interaction between a small square loop and a dislocation, either edge or screw, gliding in a prismatic plane. Ghavam and Gracie¹⁶ studied the interaction between the two slip systems is provided by the authors. Following the work done by Serra and Bacon,¹⁵ Drouet et al.¹⁷ have studied small square loops interacting with a dislocation, either screw or edge only. For this study, the authors used dislocation dynamics (DD) simulations. By applying this method to dislocations gliding either in the prismatic plane or in the basal plane, the authors have been able to understand the reason for the easier glide in the basal plane. However, because the study was limited to pure screw and pure edge dislocations using a small simulation box with periodic boundary conditions, some artifacts remained and the overall picture was not fully clarified.

On the experimental side, Onimus et al.¹⁸ and Drouet et al.¹⁹ have conducted in situ TEM straining experiments to observe, at the nanometer scale and in real time, the interactions between dislocations and loops. They managed to observe interactions between loops and dislocations gliding in the prismatic and pyramidal planes. In particular, the incorporation of a vacancy loop into the gliding dislocation was clearly observed in situ and simulated using DD.¹⁹

In order to overcome previous limitations and provide a better understanding for the reason of the easier basal channeling, a new study, based on multiscale numerical simulations and experiments at the dislocation scale, has been undertaken and is presented in this article.

Tools and Methods

EXPERIMENTS

A thin sheet made of recrystallized Zircaloy-4 characterized by equiaxed grains 6 μ m in diameter has been used. Small samples have been machined out in the form of dog-bone specimens. The middle of the gauge length of the specimen was electropolished on one side only. Then these specimens have been irradiated at a temperature of 450°C using 600 keV Zr ions. Two doses were reached: 8×10^{17} ions/m² and 2.4×10^{18} ions/m², with an ion flux equal to 1.6×10^{14} ions/m²/s. After ion irradiation, the irradiated side was protected by a varnish and the other side was electropolished to create a small hole with a thin area on its side that can be observed by TEM. The mean loop diameter was 30 nm, the loop number density was 6×10^{20} m⁻³, and they were mainly interstitial loops.²⁰

These specimens were then installed on a specific TEM sample holder that was able to apply a strain to the sample while heating. In situ straining experiments were then done at various temperatures. After recording an interesting sequence, the orientation of the grain was analyzed using electronic diffraction. Two interactions are reported in the last part of the article.

NUMERICAL SIMULATIONS

In order to assess, starting from the smallest length scale, the details of the interactions between dislocation and loops, MD simulations have been conducted using the code LAMMPS.²¹ Following the work done by Serra and Bacon,¹⁵ interactions between dislocation and loops have been computed.

Because MD simulations are time consuming and limited to a small-length scale, other types of numerical simulation tools are required in order to investigate more complex configurations and to go up to a larger length scale. This is why a discrete DD code, called NUMODIS,¹⁹ has been used. In this model, the dislocations are represented by a set of nodes connected by segments. The nodal forces arise from the core energy of the dislocation and the Peach-Koehler forces, due to the local stress tensor acting on the segments. The local stress tensor is the result of the superposition of the external applied stress and of the stress fields created by all dislocation segments, including the considered segment, present in the simulation box. The velocity of each node is then deduced from a viscous mobility law of the dislocation segment connecting the nodes, which is given by equation (1).

$$v = \frac{\tau b}{B} \tag{1}$$

In equation (1), *B* is the friction coefficient, which depends on the glide system of the segment; τ is the resolved shear stress applied on the considered segment; *b* is the Burgers vector; and *v* is the segment velocity.

Discrete DD simulation requires the introduction of various materialdependent parameters. The elastic constants, the shear modulus (μ) and the Poisson's ratio (ν), assuming isotropic elasticity, the friction coefficients *B* for the various slip systems, and also two parameters, a_0 and E_{core}^0 , are needed to describe the energy per unit length of a dislocation. The elastic energy of a dislocation segment, which is related to the stress field created by the dislocation, is described by the nonsingular continuum isotropic elasticity theory of dislocations developed by Cai et al.²² In this theory, a parameter a_0 , related to the spread of the dislocation core, is introduced. In addition to the elastic energy, the energy of the core of the dislocation is taken into account in the modeling. It is orientation dependent, edge or screw, as for the elastic energy and can then be expressed as equation (2).

$$E_{core}(\varphi) = \frac{1 - \nu cos(\varphi)}{1 - \nu} E_{core}^{0}$$
⁽²⁾

In equation (2), φ is the angle between the Burgers vector and the dislocation segment, and E_{core}^0 is the core energy of a screw dislocation.

A method has been developed to adjust these parameters on MD simulations. From the anisotropic elastic moduli (C_{ii}) of Zr computed at 300K, the isotropic elastic parameters are deduced using the procedure described by Scattergood and Bacon.²³ The shear modulus is found equal to 36.3 GPa, and Poisson's ratio is found for the prismatic plane equal to 0.223 and 0.365, respectively, for the basal plane. The parameters obtained, after adjustments on MD simulations, for the parameters describing the energy per unit length of a dislocation are $a_0 = 3.3$ Å and $E_{core}^{0} = 0.137 \text{ eV/Å}$. The values obtained by MD simulation for the friction coefficients for the prismatic B_P and basal B_B slip are $B_P = 2.96 \times 10^{-5}$ Pa.s and $B_B =$ 4.56×10^{-5} Pa.s. Furthermore, it has been found that there is an additional dragging force on the constricted nodes on a jogged dislocation, at the intersection of the prismatic and basal planes. This phenomenon was already observed by Rodney et al.²⁴ for nickel. In order to account for this additional friction, a simple model was proposed where a specific friction is attributed to these constricted nodes (p expressed in Pa.s.m) and is found to be equal to $p = 1.72 \times 10^{-13}$ Pa.s.m. This value has also been introduced into the DD simulations.

Then, as a second step, the MD simulations of interactions between one dislocation and one loop done by Serra and Bacon¹⁵ have been reproduced using precisely the same geometrical configurations and loading conditions. Qualitatively and quantitatively, the mechanisms observed are the same as the one described in Serra and Bacon.¹⁵

It must be pointed out that, due to the computational limitations of MD simulations, the box used in Serra and Bacon¹⁵ is very small. Furthermore periodic boundary conditions and the choice to simulate only pure screw and pure edge dislocations induce very symmetrical configurations. This is especially true when the interaction of a screw dislocation with a loop is simulated. Indeed, in that case, a helical turn is formed, expanding along the line of the screw dislocation. Because of the too-small simulation box and the periodic boundary conditions, the segments of the dislocation, gliding originally in the prismatic plane, disappear. Only a segment in the basal plane remains. To overcome the helical turn, a new segment, gliding in the prismatic plane, nucleates in the middle of the remaining basal segment. For less symmetrical configurations, the segments gliding in the prismatic plane do not disappear, and this mechanism is thus not needed. From this analysis, it appears that it is very important to simulate lesssymmetrical configurations.

Taking advantage of the adjustment and validation of the DD code on MD simulations, more complex configurations than pure screw or pure edge dislocations have been studied. A dislocation with a mixed character, resulting from the activation of a Frank-Read source, has been introduced into a simulation box. The simulation box consists of a thin 40-nm box in the *z* direction, parallel to the glide plane normal, either ($10\overline{1}0$) or (0001). The length in the two other directions (*x* and *y*) is equal to 6 µm. Impenetrable surfaces are used. These surfaces represent grain boundaries. The initial Frank-Read source, screw or edge, of a length equal to 150 nm, is located in the middle of the box (fig. 1). The Burgers vector of the dislocation $\underline{b}_G = \underline{b}_2 = 1/3[1\overline{2}10]$ is always parallel to the *y* axis. A constant shear stress equal to $\sigma_{yz} = 120$ MPa is applied to activate the source. After the closing of the source, the resulting dislocation is circular in the prismatic plane and slightly elliptical in the basal plane. A dislocation segment can be tracked by the polar angle, θ , as shown in figure 1.

The interaction of the dislocation and one loop is then systematically studied starting from the screw-type interaction location ($\theta = 0^{\circ}$) to the edge-type interaction location ($\theta = 90^{\circ}$), with the loop being respectively located close to the screw or to the edge part of the dislocation, through all the possible mixed type interactions. Furthermore, the Burgers vector of the loop is also systematically changed and can be $\underline{b}_L = \underline{b}_2$, $\underline{b}_L = \underline{b}_1$, or $\underline{b}_L = \underline{b}_3$. The loop introduced is an interstitial loop in the shape of a square with sides 20 nm long. In these simulations, the nature of the loop can be easily changed by taking the opposite Burgers vector. This essentially modifies the geometry of helical turn (right-handed instead of left-handed, for instance) or modifies the location of the super-jog (above or below) with respect to the gliding plane. In all the following simulations, the dislocation glide plane cuts the loop to pin the dislocation or to be swept away by the dislocation is then analyzed.

Finally, several other DD simulations have been performed on geometry representative of the configurations observed during in situ TEM straining experiments. In that case, the two impenetrable upper and lower surfaces of the box account for the TEM thin foil surfaces that are covered by a thin oxide layer. The orientation of the grain analyzed by TEM is introduced into the simulation. A uniaxial tensile

FIG. 1 (*A*) Four-indices Miller-Bravais convention for hexagonal close-packed (HCP) structure. (*B*) Schematic describing the systematic study of interactions between mixed dislocations and loops. (*C*) Snapshot of the simulation box showing the interaction between a loop and a dislocation with the same Burgers vector ($\theta = 10^\circ$).



loading is applied with a constant applied stress. The other details concerning these simulations will be described further in the following sections of the paper.

Study of Interactions between Loops and Mixed Dislocations Gliding either in the Prismatic or Basal Plane

Following the work described in Drouet et al.,¹⁷ systematic DD simulations have been performed for dislocations gliding either in the basal or in the prismatic plane and interacting with loops. However, in this previous study, small simulation boxes with periodic boundary conditions along the dislocation line were used. As described previously, this induced some artifacts on the resulting interaction. This is why in this new study, DD simulations have not been restricted to pure screw or pure edge dislocations, and larger simulation boxes without periodic boundary conditions have been used.

Starting from the Frank-Read source (fig. 1), various locations for the loop along the dislocation line (every 10°) were tested, with each location for the loop being a single computation. It appears that the resulting interaction depends strongly on the location of the loop with respect to the local character of the dislocation, defined by the angle, φ , between the local dislocation segment and the Burgers vector. For the sake of simplicity, the polar angle θ between the *x*-axis and the loop location, starting from the center of the elliptical dislocation, has been used. In the case of a circular dislocation, which is the case for the dislocation source in the prismatic plane, the angle φ and the polar angle θ are equal. The dislocation source in the basal plane is slightly elliptical. This is because of the difference in the Poisson's ratio computed by the Bacon-Scattergood procedure.

The ratio between the half-length of the ellipse along the *x*-axis (l_x) and the half-length of the ellipse along the *y*-axis (l_y) is equal to 1.29 for basal dislocations; whereas it is equal to 1.03 for prismatic ones. There is a relationship, given by equation (3), between the angle φ characterizing the local character of the dislocation and the polar angle θ .

$$\tan(\varphi) = \left(\frac{l_x}{l_y}\right)^2 \tan(\theta) \tag{3}$$

First, interactions between a loop and a dislocation gliding in the prismatic plane are simulated. The gliding dislocation has a Burgers vector $\underline{b}_G = \underline{b}_2$, parallel to the *y*-axis. The results of the interaction between the dislocation and interstitial loops with Burgers vector $\underline{b}_L = \underline{b}_2$ are described in the following.

0° ≤ θ ≤ 10°: Strong pinning, no clearing. There is formation of a helical turn and then closing of the helical turn. The loop is left behind unchanged (fig. 2A-D).

FIG. 2 Sequence showing an interaction between a mixed dislocation of Burgers vector $\underline{b}_G = \underline{b}_2$ gliding in the prismatic plane and a loop with Burgers vector $\underline{b}_L = \underline{b}_2$. (A) to (D) θ = 10°: strong pinning, no clearing. (E) to (H) θ = 72°: partial clearing, no pinning.



- 15° ≤ θ ≤ 40°: Strong pinning, no clearing. The loop is incorporated into the dislocation line as a helical turn. Then, half of the loop is left behind. Later, the other half is also left behind.
- 45° ≤ θ ≤ 75°: No pinning and partial clearing. The loop is incorporated into the dislocation as a helical turn. A reaction then occurs leading to the formation of a double super-jog dragged by the dislocation. Half of the loop is left behind; partial clearing (fig. 2E-H).
- 80° ≤ θ ≤ 90°: Weak pinning and partial or no clearing. The loop is incorporated into the dislocation, slightly dragged by the dislocation and eventually left behind unchanged. Depending on the height of the loop with respect to the gliding plane, the loop can also be incorporated as a double super-jog.

The results of the interaction between the dislocation $(\underline{b}_G = \underline{b}_2)$ and loop $(\underline{b}_L = \underline{b}_1)$ are described in the following. Due to the symmetry of the system, the interactions with a loop with Burgers vector $\underline{b}_L = \underline{b}_3$ yield to exactly the same results.

- 0° ≤ θ ≤ 5°: Strong pinning, no clearing. There is formation of a junction. The dislocation fully reacts with the loop and leaves the loop behind with a Burgers vector modified and equal to <u>b_L</u> = <u>b₂</u>.
- $10^{\circ} \le \theta \le 30^{\circ}$: Strong pinning, no clearing. There is formation of a junction that reacts with the loop creating a helix turn. The helix turn is then closed and pulled on both sides by the gliding dislocation. Then the loop is left behind with a modified Burgers vector $\underline{b}_L = \underline{b}_2$ (fig. 3*A*-*D*).

FIG. 3 Sequence showing an interaction between a mixed dislocation of Burgers vector $\underline{b}_G = \underline{b}_2$ gliding in the prismatic plane and a loop with Burgers vector $\underline{b}_L = \underline{b}_1$. (A) to (D) $\theta = 21^\circ$: strong pinning, no clearing. (E) to (H) $\theta = 72^\circ$: full clearing, no pinning.



- 35° ≤ θ ≤ 45°: Medium pinning and limited clearing. There is formation of a junction that reacts with the loop, creating a double super-jog. The double super-jog is slightly dragged on its cylinder. The double super-jog is then closed and pulled on both sides by the gliding dislocation. Then the loop is left behind with a modified Burgers vector <u>*b*</u>_{*L*} = <u>*b*</u>₂.
- 50° ≤ θ ≤ 90°: Weak pinning and full clearing. There is formation of a junction. The junction reacts with the loop, leading to the full incorporation of the loop as a double super-jog. The double super-jog is dragged by the dislocation, leading to a full clearing (fig. 3*E*-*H*).

The results of the interactions are summarized schematically in figure 4A to C using a polar plot. The angular domain (shown in black) is where the interaction leads to strong pinning and no clearing because of the incorporation of the loop into the dislocation as a helical turn. The angular domain (shown in light gray) is where the interaction leads to partial or full clearing and no pinning because of the incorporation of the loop into the dislocation as a double super-jog that is dragged on its cylinder. The white domain corresponds to the nearly pure edge configuration where the interaction is relatively weak and can lead either to no or partial clearing depending on the height of the loop with respect to the glide plane.

These new results appear to be rather consistent compared with previous simulations involving only pure edge and pure screw dislocations.¹⁷ Indeed, for pure edge dislocations, the previous results yield to partial or full clearing for $\underline{b}_L = \underline{b}_2$ or $\underline{b}_L = \underline{b}_1$, respectively. The white interaction type was not observed in these early

FIG. 4 Results of interactions between loops and a mixed dislocation with Burgers vector $\underline{b}_G = \underline{b}_2$ gliding in (*A*) to (*C*) the prismatic plane or in (*C*) to (*E*) the basal plane. (*A*) and (D) Loop with Burgers vector $\underline{b}_L = \underline{b}_2$. (B) and (*E*) Loop with Burgers vector $\underline{b}_L = \underline{b}_2$. (B) and (*E*) Loop with gray: clearing and no pinning. Black: strong pinning and no clearing. White: special configuration with weak pinning and partial or no clearing. Dark gray: no clearing but also no pinning.



simulations. Concerning pure screw dislocations, the agreement is also good because all interactions yielded to the incorporation of the loop into the dislocation as a helical turn. However, in the small simulation box with periodic boundary conditions used previously, the helical turn expanded, leading to the disappearance of the dislocation segments gliding in the prismatic plane. This is not the case for these new simulations.

Because, in the material, $\langle a \rangle$ loops with the three different Burgers vectors can be found in the same proportion, when one prismatic slip system, with one Burgers vector ($\underline{b}_G = \underline{b}_2$), is activated, all these interactions can be found to occur at the same time. One-third of the interactions will be of the type $\underline{b}_L = \underline{b}_G = \underline{b}_2$ (fig. 4A), and two-thirds of the interactions will be of the type $\underline{b}_L = \underline{b}_1$ (fig. 4B). As illustrated on figure 4, partial or full clearing occurs only close to the edge direction, up to 45° to the edge direction. Along the screw direction, a strong pinning occurs in every case, and no clearing occurs. This leads to a very difficult expansion of dislocation source in the prismatic planes. This explains the significant hardening of the prismatic slip after irradiation. Furthermore, the clearing process can only proceed along the edge direction of the dislocation source, again resulting in a limited and difficult channeling process in the prismatic plane.

In a second step, interactions between a loop and a dislocation gliding in the basal plane are simulated. For dislocations gliding in the basal plane, the same parameters for the dislocation energy per unit length as the one deduced for prismatic slip are used.

The results of the interaction between the dislocation and loops with Burgers vector $\underline{b}_L = \underline{b}_2$ are described in the following.

- 0° ≤ θ ≤ 15°: Strong pinning, no clearing. There is formation of a helical turn, then closing of the helical turn. The loop is left behind unchanged (fig. 5A-D).
- 20° ≤ θ ≤ 40°: Strong pinning, no clearing. The loop is incorporated into the dislocation line as a helical turn. The helical turn is slightly dragged on its cylinder. Then half of the loop is left behind. Later, the other half is also left behind. For θ = 40°, the full loop is left behind.
- 45° ≤ θ ≤ 90°: No pinning and partial clearing. The loop is incorporated into the dislocation as a helical turn. A reaction then occurs leading to the formation of a double super-jog dragged by the dislocation. Half of the loop is left behind; partial clearing (fig. 5*E*-*H*).

The results of the interaction between the dislocation ($\underline{b}_G = \underline{b}_2$) and loops with Burgers vector $\underline{b}_L = \underline{b}_1$ are described in the following.

- 0° ≤ θ ≤ 20°: No pinning, no clearing. There is formation of a crossed state, with very weak interaction. The loop is slightly pushed aside, gliding on its cylinder. Then the loop is left unchanged.
- 25° ≤ θ ≤ 65°: No pinning, no clearing. There is formation of a junction, glissile in the basal plane. The loop is slightly pushed aside on its cylinder. Then the dislocation easily overcomes the loop, leaving the loop unchanged (fig. 6*A*-*D*).
- 70° ≤ θ ≤ 90°: Strong pinning, no clearing. There is formation of a junction. The loop does not move. The dislocation overcomes the loop after significant pinning, leaving the loop unchanged (fig. 6*E*-*H*).
- **FIG. 5** Sequence showing an interaction between a mixed dislocation of Burgers vector $\underline{b}_G = \underline{b}_2$ gliding in the basal plane and a loop with Burgers vector $\underline{b}_L = \underline{b}_2$. (*A*) to (*D*) θ = 10°: strong pinning, no clearing. (*E*) to (*H*) θ = 84°: partial clearing, no pinning.



FIG. 6 Sequence showing an interaction between a mixed dislocation of Burgers vector <u>b_G</u> = <u>b</u>₂ gliding in the basal plane and a loop with Burgers vector <u>b_L</u> = <u>b</u>₁.
(A) to (D) θ = 32°: no pinning, no clearing. (E) to (H) θ = 84°: strong pinning, no clearing.



In the case of basal slip, contrary to prismatic slip, the system is no longer symmetric. Simulations with $\underline{b}_L = \underline{b}_3$ are then also described in the following.

- 0° ≤ θ ≤ 10°: No pinning, no clearing. There is formation of a crossed state, with very weak interaction. The loop is slightly pushed aside, gliding on its cylinder. Then the loop is left unchanged.
- 15° ≤ θ ≤ 65°: No pinning, significant clearing. There is formation of a crossed state or a junction, glissile in the basal plane. The loop is pushed aside on its cylinder over a long distance. Eventually, the loop is left unchanged after significant clearing. For θ = 54°, the loop is dragged during the full simulation duration (fig. 7A-D).
- 70° ≤ θ ≤ 90°: Strong pinning, limited clearing. There is formation of a junction. The loop is slightly pushed on its cylinder. The dislocation overcomes the loop after a strong pinning, leaving the loop unchanged (fig. 7*E*-*H*).

The results of the interactions are also summarized in figure 4D to F. In this plot, the dark gray domain corresponds to the case when the interaction between the dislocation and the loop is very weak (no pinning) but the loop is not cleared by the dislocation.

Again, these results are consistent with previous simulations involving only pure edge and pure screw dislocations.¹⁷ For pure screw dislocation with a different Burgers vector than that of the loop, in early simulations, the formation of a crossed

FIG. 7 Sequence showing an interaction between a mixed dislocation of Burgers vector $\underline{b}_G = \underline{b}_2$ gliding in the basal plane and a loop with Burgers vector $\underline{b}_L = \underline{b}_3$. (*A*) to (*D*) θ = 54°: no pinning, significant clearing. (*E*) to (*H*) θ = 84°: strong pinning, no clearing.



state was also obtained, which was assumed to allow the clearing of loops. These new simulations bring a renewed light on the role of the orientation of the loop glide cylinder with respect to the character of the dislocation. Indeed, clearing only occurs, for dislocations and loops with different Burgers vector ($\underline{b}_G = \underline{b}_2$ and $\underline{b}_L = \underline{b}_3$), for the polar angle (θ) from 15° up to 65° (fig. 4*F*). This angular domain is around the glide cylinder of the loop, orientated at 30° with respect to the x-direction. Because the dislocation is not circular in the basal plane, the maximum clearing does not occur for a polar angle of 30° but for a higher polar angle. The local normal direction of the dislocation (velocity vector) makes an angle of $\phi = 30^{\circ}$, for a polar angle, this equals $\theta = 43^{\circ}$. This position should then be the best orientation for the glide of the loop along its cylinder. However, the best orientation for the glide of the loop on its cylinder is $\theta = 54^{\circ}$. This may be explained by the fact that, as the ellipse expands, the loop is dragged on its cylinder along its Burgers vector $(\underline{b}_{L} = \underline{b}_{3})$, leading to the progressive decrease of the angle θ . This, in turn, results in a decrease of the φ angle that can eventually lead to the release of the loop. This is probably why starting with a higher θ can lead to a delayed release of the loop.

Furthermore, it is worth noticing that, for the other configurations involving dislocation and loop with different Burgers vector ($\underline{b}_G = \underline{b}_2$ and $\underline{b}_L = \underline{b}_1$), the interaction between the dislocation and the loop is very weak for a polar angle between 0° and 65° (fig. 4*E*). Although there is no clearing for this configuration, there is also no pinning. This allows the dislocation to glide easily through loops for this entire angular domain.

Of course, as for prismatic slip, the dislocation gliding in the basal plane encounters the three types of loops. The resulting interaction is therefore a mix of all these interactions. One type of loop (one-third, with Burgers vector \underline{b}_2) will be partially cleared close to the edge direction. The loops with Burgers vector \underline{b}_3 (onethird) will be either fully or partially cleared in the angular domain between 15° and 65°. Finally, the loops with Burgers vector \underline{b}_1 (one-third) will be fully or partially cleared in the angular domain between 115° and 165°. In addition, the dislocation will be able to glide in this angular domain rather easily, being only pinned by one type of loops (\underline{b}_2). Therefore, in the presence of loops, the glide of dislocations in the basal plane is easier than the glide of the prismatic plane because of less pinning. It also explains that the clearing of loops in the basal plane is easier than in the prismatic plane.

Another major difference between prismatic glide and basal glide that can contribute to the formation of defect-free channels in irradiated zirconium alloys is the fact that, if the three different basal slip systems (different Burgers vectors) are activated at the same time in close parallel planes, then all the loops can be easily cleared away.

Experimental Results and Comparison with Numerical Simulations

In order to bring experimental proof concerning dislocation and loop interactions, in situ straining experiments have been conducted in a TEM on recrystallized Zircaloy-4 irradiated at 450°C with Zr ions. After irradiation, large interstitial $\langle a \rangle$ loops were observed. Furthermore, because of alloying elements present in Zircaloy-4, the effect of ion irradiation may not be only to create dislocation loops. This could play some role in the deformation mechanisms as compared to pure zirconium.

Several interactions between dislocations and loops have been observed. Two interactions, representative of the two main phenomena observed, are reported here. The first interaction (fig. 8) shows the strong pinning of a dislocation by a loop. The dislocation glides in the first-order pyramidal plane at 400°C. Then the dislocation overcomes the loop. The second interaction obtained at 350°C (fig. 9) shows the incorporation of a loop into the dislocation, also gliding in the first-order pyramidal plane. The loop is first incorporated on the edge side of the dislocation, creating a double super-jog; then it moves to the screw side of the dislocation and becomes a helical turn.

Following the approach described in Drouet et al.,¹⁹ these two configurations have been simulated by DD owing to the introduction of experimental inputs. First, the orientation of the grain has been determined by electron diffraction. The analysis of the slip traces observed on the two foil surfaces indicate that, in the two cases, the dislocations glide in a first-order pyramidal plane. The size of the loop was evaluated directly from the image. The loop nature was taken as interstitial because,

FIG. 8 Pinning case. In situ observation in a TEM of a dislocation interacting with a loop (left) and DD simulation of the dislocation-loop interaction (right): (*A*) t = 0 s, initial time; (*B*) Experimental time t = 2.64 s, the loop is incorporated into the dislocation; (*C*) Experimental time t = 23.56 s, the dislocation bends to overcome the pinning point; and (*D*) Experimental time t = 26.44 s, the dislocation has overcome the loop.



FIG. 9 Incorporation case. In situ observation in a TEM of a dislocation interacting with a loop (left) and DD simulation of the dislocation-loop interaction (right). (*A*) t = 0 s, initial time; (*B*) Experimental time t = 16 s, the dislocation interacts with the first loop; (*C*) Experimental time t = 35.72 s, the double super-jog is dragged by the dislocation; and (*D*) Experimental time t = 104.72 s, the dislocation interacts with the second loop.



after Zr ion irradiation, mostly interstitial loops have been observed.²⁰ Because the position of the loop in the foil thickness, with respect to the glide plane of the dislocation, is unknown, various positions are then tested to obtain the best agreement between simulation and experiment. Furthermore, the shear stress moving the dislocation was estimated from its curvature, knowing its Burgers vector and its glide plane.^{25,26}

As explained earlier, several material-dependent parameters must be chosen to simulate the behavior of $\langle a \rangle$ dislocations gliding in a pyramidal plane in Zircaloy-4 at these temperatures. The anisotropic elastic coefficients for temperatures of 350°C and 400°C can be found in Fisher and Renken.²⁷ Isotropic elastic coefficients have been deduced using Voigt's procedure, described in Li and Thompson,²⁸ yielding $\mu = 29.2$ GPa and $\nu = 0.36$. These coefficients lead to a Young's modulus of 79 GPa, which is in correct agreement with experimental values measured at these temperatures.^{29,30} Concerning the dislocation energy parameters, they have been chosen equal to the ones used in a previous work,¹⁹ which are $a_0 = 3.23$ Å and $E_{orre}^0 = 0.052$ eV/Å.

EXPERIMENTAL CASE #1: PINNING OF THE DISLOCATION BY THE LOOP

In a previous study, the friction coefficient was evaluated as being equal to 3×10^5 Pa.s for $\langle a \rangle$ pyramidal slip.¹⁹ This value has also been used to start with here. However, when conducting the simulations, it appeared that the loop was a much stronger obstacle than expected based on the previous simulations. The friction coefficients for the other slip systems (basal and prismatic) were increased $(B_P = 3 \times 10^6 \text{ Pa.s}; B_{\pi a} = 3 \times 10^5 \text{ Pa.s}; B_B = 3 \times 10^7 \text{ Pa.s})$, leading to the locking of the loop. The additional friction due to the constricted nodes is not taken into account here. For this simulation, a uniaxial tensile stress is applied along the *y*-direction. The magnitude of the tensile stress is $\sigma_{yy}=158$ MPa, accordingly to the evaluation based on the curvature of the dislocation.

It can be noticed on figure 8 that, with these parameters, both the kinetics and the geometry of the interaction are satisfactorily reproduced. From these simulations, it can be deduced that this loop acts as a stronger pinning point than expected probably because of solute atoms, such as oxygen, have diffused to the loop creating a solute atmosphere that can lock the loop motion. It can also be deduced that the loop has probably the same Burgers vector as the dislocation because this configuration was the one that led to the best agreement, compared to other trials.

EXPERIMENTAL CASE #2: INCORPORATION OF THE LOOP AS HELICAL TURN

Starting with the same set parameters, simulating a second interaction was attempted. From the curvature of the dislocation, the applied stress was estimated as $\sigma_{yy} = 124$ MPa, considering a uniaxial applied stress along the *y*-direction. However, the dislocation velocity was very low in this experiment. The reason for this very low velocity was not clearly understood. This may be explained by an overestimated applied stress because of a curvature that is not due to the stress but to a

motion mechanism involving a kink pair to overcome strong Peierls valleys, as was observed previously for $\langle a \rangle$ -pyramidal slip.³¹ In order to account for this very low dislocation velocity, the friction for the pyramidal plane was artificially increased. The other friction coefficients were also modified in order to obtain the best possible agreement $B_P = 1 \times 10^6$ Pa.s; $B_{\pi a} = 3 \times 10^6$ Pa.s; $B_B = 1 \times 10^7$ Pa.s.

It can be seen in **figure 9** that, with this parameter set, the agreement between the simulation and the experiment is correct; although from the timescale point of view, there are still significant discrepancies. From these simulations, it can be deduced that the loop probably does not have the same Burgers vector as the dislocation. The loop is incorporated as a double super-jog into the dislocation on the edge side. Then the loop is pushed aside, on the screw part leading to the formation of a helical turn. In the simulation, a second loop has also been introduced, which is difficult to see clearly on the film. This second loop also explains the wavy shape of the dislocation.

Discussion

From the systematic study of interactions between dislocations and loops using DD simulations adjusted on MD simulations, a new light has been brought on the deformation mechanisms occurring in neutron-irradiated zirconium alloys. Indeed, the origin of the easier basal glide and basal loop clearing observed by TEM after irradiation,^{8,11-13} is now better understood. It is clearly due to the weak interactions between dislocation and loops in the screw-mixed direction (for loops with different Burgers vectors than the dislocations) and to the easy clearing of loops along their glide cylinder, which is along the mixed direction. On the contrary, dislocations gliding in the prismatic planes are always strongly pinned in the screw-mixed direction. The dislocation source is thus very strongly constrained for prismatic slip, resulting in a high flow stress. On the other hand, the dislocation source in the basal plane is able to expand along the screw-mixed direction, leading to a lower flow stress needed to activate the dislocation source. Another reason for the easier clearing of loops by basal glide is the fact that the three $\langle a \rangle$ Burgers vectors all belong to the basal plane. If three dislocation sources, with different $\langle a \rangle$ Burgers vectors, are activated in close parallel basal planes, they should be able to clear out all the loops, leading to the formation of a dislocation channel.

To be able to reproduce in situ straining experiments done on Zircaloy-4 using DD simulations, the friction coefficient (here, for pyramidal $\langle a \rangle$ slip) must be increased by a factor of 10^{10} compared to the friction coefficient measured for prismatic slip by MD simulations. This is very surprising and can be partly attributed to the fact that a first-order pyramidal slip with $\langle a \rangle$ Burgers vector is more difficult to activate than the prismatic slip and has therefore a higher friction coefficient compared to the prismatic slip.

This discrepancy may also be attributed to the nonlinearity of the viscoplastic flow law (equation [1]). Indeed, the linearization of the nonlinear flow low at a very

high shear strain rate, as is the case in MD simulations, yields a very low friction coefficient (B); whereas for the very low shear strain rate used in the experiments, the linearization of the flow law leads to a very high friction coefficient.

Lastly, this discrepancy has been attributed¹⁹ to the effect of solute atoms such as oxygen that may strongly affect the dislocation motion. Indeed, it has been recently established, using ab initio calculations,³² that oxygen atoms induce a local cross-slip of a screw dislocation, initially gliding in the prismatic plane, leading to the formation of jogs that have to be dragged by the dislocation during its glide in the prismatic plane. This explains the hardening effect of oxygen atoms. Furthermore, at specific temperatures, a dynamic strain-aging phenomenon also occurs with oxygen atoms that may increase the hardening effect. This last issue should thus be investigated in more detail to extend the conclusions, obtained on pure zirconium, to zirconium alloys.

Conclusion

A multiscale numerical study has been conducted to investigate interactions between dislocations and radiation-induced loops in zirconium. Complex configurations involving mixed curved dislocations interacting with loops have been simulated using dislocation dynamics. The input parameters were adjusted on MD simulations. The result is a clear picture of the reason why dislocation glide and clearing of loops in the basal plane is easier than in the prismatic plane after irradiation, as shown by the experiments. Indeed, in the mixed-screw directions, dislocations in the prismatic plane are always strongly pinned, whereas in the basal plane, for some configurations, clearing of loops or weak interactions occur, allowing clearing and easy glide in this plane. Furthermore, all three basal systems can contribute to clearing in the basal plane contrary to prismatic slip. These two reasons explain the easy basal glide and clearing of loops after irradiation.

Moreover, in situ straining experiments inside a TEM, conducted on ionirradiated zirconium alloy samples, show interactions between dislocations and loops. The dislocation dynamics simulations are able to reproduce the TEM observations, after adjustment of some parameters, showing the relevance of this numerical tool. Some experiments also suggest that solute atoms, such as oxygen, could play a role in the pinning of some loops. More studies are probably needed to assess this last point.

In a future prospect, massive DD simulations, involving many loops and many dislocations, could now be used with confidence to simulate the formation of defect-free channels in irradiated zirconium. This will eventually give access to a fully predictive constitutive law, at the grain scale, allowing the prediction of the mechanical behavior of zirconium alloys after neutron irradiation.

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Discussion

Question from Anand Garde, Zira Shri, LLC:—For unirradiated HCP alloys whether basal slip or prism slip dominates appears to depend on (c/a) ratio. Did you measure (c/a) ratio before and after irradiation, and can it explain your observations to some extent?

Authors' Response:—Based on literature data, the c/a ratio does not change with irradiation. The change of an easy slip system with irradiation is the result of the differences in interaction between loops and dislocations depending on their gliding plane.

Questions from Arthur Motta, Penn State University:

- 1. The comparison of experimental in situ dislocation interactions with computer calculation did not produce good agreement, as per your presentation, with the loop seen in experiments being apparently pinned. You hypothesized that the loop was pinned by alloying elements. Do you have any evidence that this was the actual pinning agent? Could the pinning have been caused by small defect clusters, invisible in the TEM?
- 2. How do you believe your DD/MD approach would change if solutes segregate to dislocations?

Authors' Response:

- To explain the disagreement between simulation and experiment we have hypothesized that the significant pinning of the dislocation by the loop is the result of solute atoms that lock the loop. It could indeed be the result of other small point defect clusters invisible by TEM or even other dislocations that were invisible with this diffraction vector.
- 2. The effect of solute elements, such as oxygen (O), can be studied by molecular dynamics simulations, provided that a reliable Zr-O empirical potential is available. Taking this into account, an oxygen atmosphere is possible in the dislocation dynamics simulation through different possible procedures— ad hoc or more physically based. This could be a future prospect for this study.

Question from J. Fonseca, University of Manchester:—Thomas et al.,¹ clearly shows prismatic channeling; however, this seems to be inconsistent with the conclusion you draw from your MD simulations. Why does prismatic channeling also occur?

Authors' Response:—This is a complicated issue. After irradiation at 350°C, basal slip seems to be favored compared to prismatic slip. At room temperature, both slip systems seem to be competing, with a significant activation of prismatic

slip. This may be due to the different evolution of the various critical resolved shear stresses (CRSS) with temperature. Based on the dislocation dynamics simulations, we understand why basal channeling is easy. However, prismatic channeling is possible. Because of the lower CRSS of prismatic slip of the unirradiated material, there is a competition between basal and prismatic after irradiation.

 R. Thomas, D. Lunt, M. D. Atkinson, J. Q. da Fonseca, M. Preuss, F. Barton, J. O'Hanlon, and P. Frankel, "Characterisation of Irradiation Enhanced Strain Localisation in a Zirconium Alloy," *Materialia* 5 (2019): 100248.

Question from Philipp Frankel, University of Manchester:—The presentation showed that, to reproduce the observed pinning of dislocations by loops, a large friction coefficient is required. It was mentioned that solute atoms may contribute. Does this imply chemical segregation to the loops?

Authors' Response:—Yes, indeed. There is much evidence in the literature concerning the effect of oxygen on dislocations or dislocation loops, from a macroscopic point of view. However, to our knowledge, there is no microscopic observation of segregation of oxygen atoms on loops yet. Other elements such as iron or niobium could also segregate on loops, as suggested by some recent experimental results.

Question from Rick Holt, Queen's University, Kingston, Canada:—Very nice work. Do you have plans to extend this work to pyramidal c+a slip?

Authors' Response:—Thank you very much. From a crystallographic point of view, it is easy to extend the dislocation dynamics approach to c+a dislocations. However, the detailed motion process of c+a dislocation must be studied first to have the correct inputs for the dislocation dynamics simulation. This can be done by conducting in situ straining experiments in a TEM and molecular dynamics simulations. Some progress is currently being made in this direction.

Question from Michael Preuss, University of Manchester:—Don't you think that while irradiation might have a greater effect on hardening prismatic slip than basal slip, what is in fact activated in a polycrystalline material is greatly affected by the texture, as it is hard to see how one can activate basal slip for common textures when for example loading along RD?

Authors' Response:—At 350°C, it seems to be clear that basal slip becomes easier than prismatic slip. Nevertheless, prismatic slip is still possible, and both slip systems are competing. The Schmid factor, as for the unirradiated material, still plays an important role. This is why, for an axial tensile test (along the rolling direction), basal slip is not activated and prismatic slip is activated. Question from Ron Adamson, ZIRCOLOGY PLUS:—Your testing and simulations were for temperatures near 350°C. For the simulations, in order to produce the desired basal plane channeling, the "loop strength" of friction needed to be increased by some sort of hardening phenomenon. Since 350°C is near the peak of the irradiation anneal hardening curve whereby strength is increased by segregation of solutes to dislocations, could it be that at lower irradiation and testing temperatures (like 280°C) the propensity for prism plane channeling (as opposed to basal plane channeling) would increase?

Authors' Response:—Thank you for this suggestion. It is indeed possible that the segregation of oxygen atoms, associated with the radiation anneal hardening occurring at 350° C, increases the hardening of prismatic slip at this temperature, thus explaining that at this specific temperature, basal slip is favored.