



Evidence of dislocation loop preferential nucleation in irradiated aluminum under stress

D. Da Fonseca^{a,e}, F. Momprou^{d,e}, T. Jourdan^a, J.-P. Crocombette^a, A. Chartier^c, F. Onimus^{b,*}

^a Université Paris-Saclay, CEA, Service de recherche en Corrosion et Comportement des Matériaux, SRMP, F-91191 Gif-sur-Yvette, France

^b Université Paris-Saclay, CEA, Service de Recherche en Matériaux et procédés Avancés, F-91191 Gif-sur-Yvette, France

^c Université Paris-Saclay, CEA, Service de recherche en Corrosion et Comportement des Matériaux, F-91191 Gif-sur-Yvette, France

^d Centre d'Elaboration de Matériaux et d'Etudes Structurales, CNRS UPR 8011, 29 rue J. Marvig, BP 94347, Toulouse cedex 4 31055, France

^e Université de Toulouse, UPS, F-31055 Toulouse, France

ARTICLE INFO

Keywords:

Irradiation creep
Dislocation loop
In-situ straining irradiation
Object kinetic Monte-Carlo
Frenkel pair accumulation

ABSTRACT

To explain irradiation creep, several mechanisms have been proposed. Some are based on the effect of stress on either nucleation or growth of dislocation loops. To investigate these mechanisms in aluminum we combine in-situ transmission electron microscope irradiation under stress and two simulation approaches (object kinetic Monte-Carlo, molecular dynamics Frenkel pair accumulation). We observe the selectivity of Frank loop variants under electron irradiation and applied stress. When the stress is turned on after loop formation, there is no loop variant selectivity, suggesting the absence of preferential absorption on already formed loops. Object kinetic Monte-Carlo simulations, including the effect of stress on the diffusion of point defects, show no growth rate difference between loop variants. Frenkel pair accumulation simulations exhibit variant selectivity of nucleated loops. This shows that loop selectivity is due to preferential nucleation of well oriented loops under stress and not to differential growth of loops.

Under irradiation and applied stress, a specific deformation process known as irradiation creep arises in many materials, such as steels, nickel-based and zirconium alloys [1–3]. Its phenomenology, well documented, is very different from thermal creep, which essentially operates at high temperature. Depending on irradiation flux, temperature and stress magnitude, irradiation creep can be responsible for deformation rates far higher than those related to thermal creep [4]. Understanding the mechanisms underlying irradiation creep is therefore of prime importance.

Several mechanisms have been proposed to explain irradiation creep [5,6,3]. Some of them are based on the effect of stress on dislocation loops and account for various observations. In some experiments, interstitial loops in planes where the normal stress is the largest have been shown to be larger [7,8], resulting in a net strain. Other experiments have evidenced an increase in interstitial loop density with the normal stress on the loop habit planes, also resulting in a net strain [9,7,10–13]. The mechanisms which can explain such results can be classified in two main categories: the stress-induced preferred absorption (SIPA) phenomenon [14,15] and the stress-induced preferred nucleation (SIPN) of interstitial dislocation loops. SIPA is

due to the anisotropic diffusion of self-interstitials and vacancies under stress [16,17]. It has been shown to be able to explain not only differential loop growth, but also preferential loop formation in some planes [18]. The first version of SIPN was based on the classical nucleation theory, which proved erroneous for interstitial dislocation loops due to the absence of activation barrier for nucleation [19]. Later, it was suggested that the reorientation of small SIA clusters under stress could explain the different loop densities on different habit planes [20]. However, the magnitude of this effect was found too low to match experimental results. Although this process is not a classical nucleation process, we consider it as SIPN, as opposed to the purely diffusive SIPA process.

Whether the contribution of loops to irradiation creep is due to a SIPN or a SIPA mechanism is still an open question [21]. Results are not all consistent, with some experiments leading to opposite trends to what would be expected due to SIPN [22] or SIPA [23]. In addition, internal stresses are difficult to determine and probably affect the results [24–26]. Finally, in high dose irradiations, loops interact with the dislocation network. This interaction alters the loop size distributions and makes the sole effect of stress on loops less easy to observe, espe-

* Corresponding author.

E-mail address: fabien.onimus@cea.fr (F. Onimus).

<https://doi.org/10.1016/j.scriptamat.2023.115510>

Received 20 January 2023; Received in revised form 27 March 2023; Accepted 21 April 2023

Available online 26 April 2023

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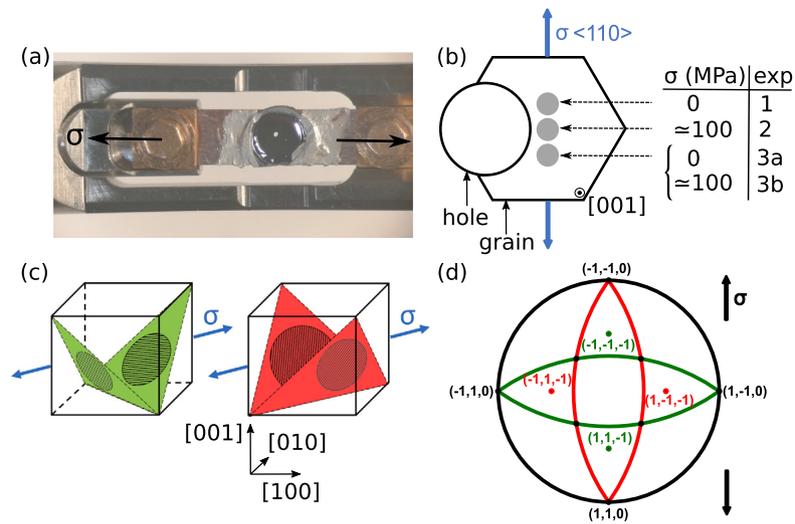


Fig. 1. (a) Straining TEM sample holder, (b) sketch of the experiments: three irradiations are conducted in the same grain close to the electropolished hole, with different stress conditions (without or with stress estimated to be around 100 MPa), the final microstructures are presented in Fig. 2, (c) orientation of the two different Frank loop types with respect to the tensile axis, and (d) the corresponding stereographic projection with the associated tensile axis. Well-oriented loops are presented in green (type B) and unfavorably oriented loops are presented in red (type A). The method used to evaluate the applied stress is described in the Supplementary Material.

cially if irradiations are performed ex-situ [27–29]. Therefore, in-situ, low dose irradiations should be preferred to investigate stress effects on loops.

In the following we combine both experimental in-situ straining transmission electron microscopy (TEM) irradiations of aluminum thin foils and two simulation methods, namely object kinetic Monte-Carlo (OKMC) and molecular dynamics using Frenkel pair accumulation (FPA) process, to determine whether stress has an impact on the nucleation and/or on the growth of loops.

In-situ observations were conducted in a JEOL 2010HC microscope operated either at 140 kV, *i.e.* well below displacement threshold for setting up experiments and at 180 kV for irradiation. A pure aluminum (99.999%) annealed 0.5 mm thick foil was used for the experiments. It ensures a large grain microstructure with a strong cube texture, *i.e.* foil normal close to [001] and rolling direction along the [100] direction. 3 mm disk specimens were extracted from the foil plane, mechanically grinded and eventually doubled-jet electropolished to electron transparency around a central hole using a solution of perchloric acid/ethanol (5/95) at -30°C and 30 V. Then the specimens were glued, with a cyanoacrylate glue, on a copper tensile grid and placed in a Gatan straining holder, as shown in Fig. 1.a. Samples were oriented on the copper grid so that the $\langle 110 \rangle$ direction (at 45° from the rolling direction) was aligned with the straining direction, with the normal of the foil close to $\langle 001 \rangle$ direction. The stress was imposed by a micrometer controlled motion of one of the sample grip while the other one stayed at rest. The method used to evaluate the applied stress is described in the Supplementary Material. Irradiations were conducted at room temperature without and with external stress. At this temperature (below $0.5 T_m$, where T_m is the melting temperature), emission of point defects from loops can be neglected and the contribution of the stress dependent emission rates to loop evolution is negligible. The beam was adjusted to obtain an electron flux of $1 \times 10^5 \text{ e}^-/\text{nm}^2/\text{s}$ at 180 kV which corresponds to $4.55 \times 10^{-5} \text{ dpa/s}$. The damage rate was computed as the electron flux times the adequate cross-section given in Oen's table [30]. In this condition, the irradiated zone covers a disk of $3 \mu\text{m}$ of diameter. Observations were made in the center of the irradiated area which was measured [31] to be around 170 nm thick (see Supplementary Material). Three experiments referenced as 1, 2 and 3 (Fig. 1.b) are detailed in the following. They were all carried out in the same grain in the area where the local stress is uniaxial and parallel to the tensile axis. Fig. 2 shows weak-beam dark field (WBDF) images taken after irradiation

with $g = 200$, using $g(3g)$ diffraction condition, in order to have all possible Frank loops visible.

After irradiation without applied stress, the microstructure was characterized in detail. The loop nature was determined using the inside/outside method [32,33]. Only interstitial loops were observed. Furthermore, most of these loops are Frank loops with Burgers vectors $b = 1/3\langle 111 \rangle$ and lying in $\{111\}$ habit planes [34]. In the tensile configuration used, Burgers vectors $1/3[1\bar{1}1]$ and $1/3[1\bar{1}\bar{1}]$ (denoted herein as type A loops in red in Fig. 1c-d) are both perpendicular to the straining axis, while Burgers vectors $1/3[11\bar{1}]$ and $1/3[1\bar{1}1]$ (denoted herein as type B loops in green in Fig. 1c-d) are tilted 35° away from the straining axis. Hence, the two types of loops are not equivalent with respect to the applied stress. For the type A loops, the component of the stress along the loop Burgers vector is equal to zero. Moreover, all the normals of the habit planes of the loops make a 55° angle with the foil normal which allows the observations of the loops with the same apparent projections, but with different orientation with respect to the tensile axis: the long axis of the ellipse of the A loops is vertical, parallel to the tensile axis while the long axis of the ellipse of the B loops is horizontal, perpendicular to the tensile axis.

Experiment 1 (Fig. 2.a) is a 38 minutes stress free irradiation. It yields a proportion of 60% of type A loops and 40% of type B loops. Although the proportions are not exactly equally balanced, it can be considered as a reference state. The authors want to point out that a stress-free state, with nearly balanced proportion of loops, is not systematically obtained. This is presumably due to residual stresses. Only samples with nearly stress-free state, with initially quasi-balanced proportion of loops, have thus been considered for this work.

In experiment 2, the specimen was first strained at lower voltage to avoid atomic displacement until the first dislocation motion was observed. Then, the displacement of the tensile grip was slightly reduced. Irradiation started with immobile dislocations under a stress slightly below the yield stress that can be roughly estimated from dislocation curvature, *i.e.* of the order of 100 MPa in a thin foil (see Supplementary Material). After irradiation, the tensile grip was again slightly moved (a few microns), inducing dislocation glide, in order to check that the stress did not relax during irradiation. Fig. 2.b shows the microstructure after 41 minutes of irradiation. Contrary to experiment 1, 10% of loops are of type A and 90% of type B. This result is consistent with most of the observations in literature where the populations with the largest projection of the tensile stress vector on the normal to their

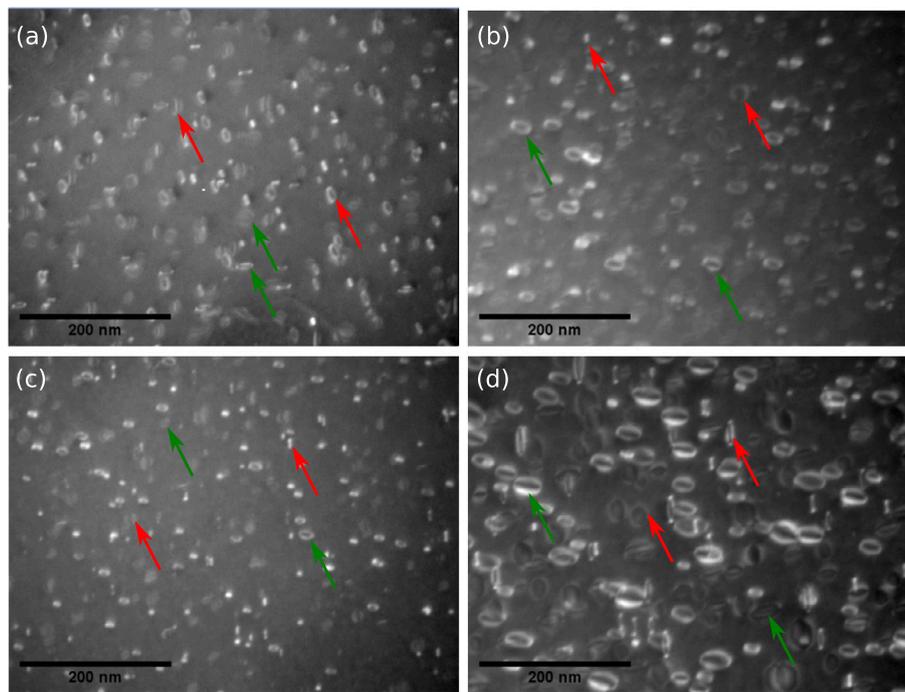


Fig. 2. Irradiations conducted in the same grain with a flux of $1 \times 10^5 \text{ e}^-/\text{nm}^2/\text{s}$ at room temperature: (a) experiment 1 without applied stress for 38 minutes, (b) experiment 2 under stress for 41 minutes, (c) experiment 3a without stress for 16 minutes, (d) experiment 3b with 48 additional minutes under stress. Red arrows point out some unfavorably oriented loops and green arrows some well-oriented loops.

habit plane are preferentially formed [9,7,10,24–26,11,13]. This strong anisotropy of loop population is a clear evidence of the influence of the applied stress, but this experiment is not self-sufficient to ascertain between preferential absorption and preferential nucleation mechanisms, as some studies explained the anisotropic population with SIPA mechanism [24,13] based on the work of Wolfer [20].

A third experiment was hence conducted, in two steps, to discriminate between SIPA and SIPN. In a first step (experiment 3a), a new area was irradiated without applying an external stress, until the loops were large enough to allow their type classification. The irradiation was stopped after 16 minutes (see Fig. 2.c). As expected for an irradiation without stress, proportions are balanced with 47% of type A loops and 53% of type B loops and a measured mean diameter of 13.3 nm and 12.4 nm, respectively. This corresponds to a mean growth rate in diameter of 0.013 nm/s. Using the same protocol as in experiment 2, the sample was then stressed and irradiated for 48 minutes in the same area (experiment 3b) (see Fig. 2.d). The proportion of the loops did not significantly evolve: 40% of loops are of type A and 60% of type B. The loops have a mean diameter of 27 nm for both types.

Comparing the results of experiments 2 and 3 shows that stress has a strong impact only in early stages of irradiation, suggesting that the contribution of SIPA should be very moderate. The shrinkage of type A loops and the growth of type B loops would be expected due to SIPA but instead both types of loop grow and only the shrinkage of new small loops is observed, becoming sacrificial loops for the growth of previously formed loops. Suzuki and Sato [11] did an equivalent experiment to our experiment 3 in Fe-18Cr-14Ni alloy under 1 MeV electron irradiation at temperature ranging between 340 °C and 456 °C, and they drew the same conclusion. To confirm this analysis, SIPA and SIPN were evaluated separately using two different simulation methods. The results are presented in the following.

In order to evaluate the role of SIPA on loop growth under experimental conditions, OKMC [35–37] simulations were used. Accordingly, the x , y and z directions of the simulation box are chosen along [1 10], [1 10] and [001] directions, respectively. The dimensions of the box along these directions are 100, 100 and 200 nm. The height of 200 nm in the z direction corresponds approximately to the foil thickness.

Free surfaces are hence introduced in the z direction while periodic boundary conditions are considered in x and y directions. Initially, the simulation box contains no point defect. Frenkel pairs are then generated at a dose rate of $5 \times 10^{-5} \text{ dpa/s}$ to mimic TEM irradiation. Point defects migrate in the simulation box and can recombine with defects of opposite type, agglomerate with each other (thus forming interstitial loops or cavities) or escape to the surfaces. In addition, immobile traps are randomly placed in the system before the irradiation starts, to mimic impurities present in the material. These impurities are assumed to strongly bind to migrating point defects. Without such traps, SIAs would all recombine with vacancies or escape to free surfaces, leaving the simulation box free of loops. A total of 10 impurities, corresponding to a concentration of 0.08 appm, is chosen to reproduce the observed loop density in experiment 3a, well below the estimated experimental concentration. This suggests that not all impurities are efficient traps for point defects. When SIAs agglomerate with an impurity to form a loop, the habit plane is chosen randomly and kept the same as the loop grows, so SIPN is purposefully discarded. More details about the parametrization are given in the Supplementary Material.

The migration of point defects is affected by the internal stress generated by dislocation loops in the thin foil and the externally applied stress [37]. The elastic interaction energy between point defects and the local stress field, which biases the migration of point defects and leads to SIPA, is given by an elastic model based on the elastic dipole tensors and diaelastic polarizabilities of point defects [38]. These properties have been obtained recently in aluminum from density functional theory calculations [17]. Image forces induced by the presence of loops near surfaces [39] are neglected, as it has been shown that they affect dislocation loop growth only when loops are a few nanometers away from the surface [37].

Simulations were run at 300 K without or with a 100 MPa uniaxial stress applied in the y direction. Simulations were stopped after 10 minutes of irradiation. Cluster distributions are obtained from 100 independent simulations carried out with different spatial distributions of impurities.

Simulations performed without stress show an almost equal proportion of type A (49.4%) and type B (50.6%) loops with mean diameters

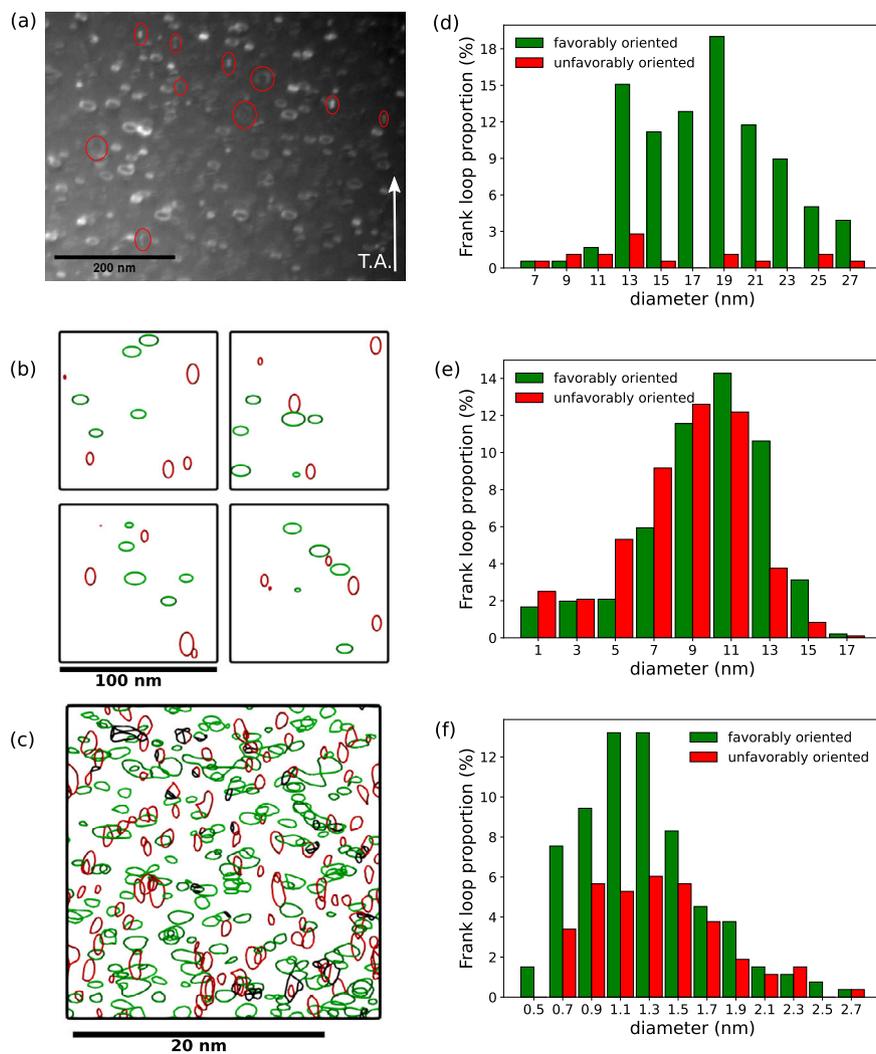


Fig. 3. Comparison of the three methods to evaluate the main mechanism involved. On the left (a), (b), (c) are the microstructures studied with the same orientation of the applied stress showed by a white arrow. (a) Experiment 2: in-situ irradiation under stress, unfavorably oriented loops are in minority, some of them are highlighted by red ellipses, (b) four out of 100 systems simulated by OKMC method, (c) system simulated with FPA method, black lines correspond to non-Frank dislocations. For the simulations in (b) and (c), type A dislocations are in red and type B are in green. On the right (d), (e), (f) are the corresponding normalized distributions of loop diameters in nanometer.

7.67 nm and 7.99 nm respectively, leading to an average growth rate of 0.013 nm/s. This growth rate is the same as in experiment 3a, which validates our simulation method. Typical microstructures and loop size distributions when a stress is applied are shown in Fig. 3.b and Fig. 3.e, respectively. Proportions of type A (48.5%) and type B (51.5%) loops under applied stress are still balanced with mean diameters of 8.45 nm and 9.89 nm respectively. Well-oriented loops are slightly larger, indicating only a moderate effect of the stress on differential growth. This effect is however below the experimental error and hence could not be ascertained by TEM. More importantly, the simulations fail to reproduce the strong selectivity on loop variants (Fig. 3.d and Fig. 3.e). These results thus suggest that the microstructure evolution under stress cannot be explained by a SIPA mechanism. They are in line with the small effect of stress on absorption efficiencies of dislocations derived in a previous work [17]. Absorption efficiencies of loops are found to be much more dependent on other factors, such as loop size and local loop environment.

To further highlight the importance of SIPN, molecular dynamics simulations of defect accumulation under stress were performed. Simulation boxes oriented along $[110]$, $[\bar{1}10]$ and $[001]$ directions were used. Their dimensions are of around $25 \times 25 \times 24$ nm³ and they contain 887520 atoms of aluminum whose interactions are described with

an EAM potential [40]. Periodic boundary conditions are applied in all three directions. Simulations are done at constant room temperature and constant stress using a Berendsen thermostat and a Parrinello-Rahman barostat, with fixed angles so that the box remains tetragonal during the simulations. TEM experiments, *i.e.* electron irradiations, are modeled by periodically introducing Frenkel pairs in the box, following the same FPA method as in previous works [41–46]. 200 Frenkel pairs are generated every 2 ps, which corresponds to a dose rate of 1.13×10^8 dpa/s, far from the experimental reality. The total simulated time is 0.5 ns, which makes long range diffusion negligible and therefore excludes any SIPA process. In addition, results cannot be directly compared with experiments at the same absolute value of dose. A snapshot of atomic configurations is taken every 2 ps, just before each insertion of point defects. The cell is then visualized with the OVITO software [47] and dislocations are identified with the DXA algorithm [48]. Note that only closed dislocations made up of segments with the same Burgers vector are counted in the total number of Frank loops. The evolution of loops is tracked until they are too big and interact with each other thus forming a dislocation network (around ~ 0.03 dpa).

A reference simulation was performed at zero stress. Type A loops come out at 48.6% to 51.4% for type B, thus the code reproduces correctly equiprobable loop nucleation between the two variants. In a

second simulation, we set a stress of 100 MPa in [1 10] direction and 0 MPa in [001] and $[1\bar{1}0]$ directions. Under stress, we obtain the microstructure shown in Fig. 3.c and the corresponding distribution in Fig. 3.f. Among all the dislocation segments detected by OVITO, 75.8% are of Frank type (265 Frank loops are counted in Fig. 3.c). What stands out from the snapshot and the bar plot is that well-oriented loops (B type loops) are in majority (65.7% of the total). This proportion is lower than what is shown in Fig. 3.d but the result still evidences preferential orientation of Frank loops under stress (even at this relatively low applied stress for MD simulations). We also found that the proportion of type B loop rises with the level of stress applied along [1 10] direction.

Our experiments show that the impact of stress is noticeable when it is applied from the start of the irradiation, suggesting that the anisotropic microstructure observed is due to preferential formation of well oriented loops under stress and not to the differential evolution of already existing loops. OKMC simulations confirm the small effect of SIPA and MD-FPA simulations yield an anisotropic population under stress. We thus prove that SIPN is stronger than SIPA under electron irradiation, suggesting that SIPA has only a minor contribution to irradiation creep deformation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors are grateful to Estelle Meslin, Benoit Arnal and Guilhem Sagnes for their help with sample preparation and TEM operation.

Appendix A. Supplementary material

Supplementary material related to this article can be found online at <https://doi.org/10.1016/j.scriptamat.2023.115510>.

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