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Temperature effect on radiation-induced dislocation loops in a FCC high purity CrFeMnNi multi-principal element alloy

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Abstract:

Temperature effect on dislocation loop density and radiation-induced segregation (RIS) phenomena are investigated on an ion irradiated 15Cr-46Fe-17Mn-22Ni (at.%) multi-principal element alloy (MPEA) over a wide range of temperatures. From 95K to 623K, loop densities are temperature independent and close to $\sim 5.0 \times 10^{22} \text{ m}^{-3}$. Above 623K, loop densities decrease until $\sim 1.05 \times 10^{21} \text{ m}^{-3}$ at 823K. This tendency is the same as in the conventional ternary Fe-Cr-Ni alloys. Besides, Ni enrichment and Cr, Fe, Mn depletion on loops are observed at 823K by scanning transmission electron microscopy coupled to energy dispersive X-ray spectroscopy and atom probe tomography with comparable RIS amplitude. This solute redistribution is explained by an inverse Kirkendall (IK) mechanism. In contrast, no obvious chemical segregation/depletion phenomena are detected at 298K, which reveals the temperature dependence of the IK effect. This comprehensive study of loop behavior as a function of temperature gives a better understanding of CrFeMnNi MPEAs under irradiation.

1 Keywords: Multi-principal element alloy; Crystal defects; Radiation-induced
2 segregation; Ion irradiation; Dislocation loop;
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5 Advanced irradiation-resistant materials, involving next-generation fission and
6 future fusion reactors, need to retain high mechanical performance under extreme
7 environments, such as high-dose radiation and/or elevated temperature [1-3]. These
8 harsh operating environments will potentially induce structural damage in alloys and
9 result in degradation issues, for example void swelling, radiation hardening and
10 irradiation creep [1]. For safety margin considerations and more efficient nuclear
11 energy systems, a novel generation of structural materials with superior radiation
12 damage resistance is mandatory to replace some internal parts or components of the
13 primary circuit composed by face-centered cubic (FCC) austenitic stainless steels.
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24 A new class of metallic materials with several principal elements named high-
25 entropy alloys (HEAs) have been drawing considerable attention due to their superior
26 properties, such as high strength, ductility, wear resistance, and corrosion resistance [4-
27 5]. The classical equiatomic CrFeCoNiMn HEA, named as Cantor's alloy [6], with FCC
28 structure, was shown to exhibit good irradiation resistance [7]. However, the presence
29 of Co leads to a high level production of the ^{60}Co radioisotope under neutron irradiation,
30 which makes it inappropriate for a use in nuclear reactors. Therefore, some CrFeMnNi
31 multi-principal element alloys (MPEAs) were designed, fabricated and characterized,
32 such as 18Cr-27Fe-27Mn-28Ni, 15Cr-35Fe-15Mn-35Ni, 20Cr-40Fe-20Mn-20Ni,
33 18Cr-29Fe-23Mn-30Ni (all the compositions in this study are given in at.%) [8-14].
34 Kumar *et al.* [8] reported that in the 18Cr-27Fe-27Mn-28Ni alloy irradiated with 5.8
35 MeV Ni ions up to 10 dpa, loop densities monotonically decrease at elevated
36 temperatures (from 673K to 973K, the blue solid circles in Fig.3) and the maximum
37 mean loop size of 5.45 nm is obtained at 973K. In addition, they also hypothesized that
38 the loop density is independent of dose and temperature between 298K to 673K with
39 values of $\sim 10^{23} \text{ m}^{-3}$. More recently, much larger loops, 27 nm for 18Cr-27Fe-27Mn-
40 28Ni and 10 nm for 15Cr-35Fe-15Mn-35Ni irradiated by 1 MeV Kr ions up to 2 dpa at
41 773K, were detected by Parkin *et al.* [9], and the loop density at 50K ($\sim 4.5 \times 10^{21} \text{ m}^{-3}$)
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1 was surprisingly lower than at 773K ($\sim 1.2 \times 10^{22} \text{ m}^{-3}$) in irradiated 15Cr-35Fe-15Mn-
2 35Ni samples (the green solid circles in Fig.3). What is also worth to notice that the
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4 loop densities in both 18Cr-27Fe-27Mn-28Ni ($\sim 6.7 \times 10^{21} \text{ m}^{-3}$) and 15Cr-35Fe-15Mn-
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6 35Ni ($\sim 1.0 \times 10^{22} \text{ m}^{-3}$) irradiated up to 0.3 dpa at 50K are nearly 10 times lower than in
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8 Kumar's study ($1.5 \times 10^{23} \text{ m}^{-3}$) at 298K at the same dose. Another aspect of interest is
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10 the solute redistribution in these alloys. A huge quantity of freely migrating point
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12 defects (PDs), self-interstitial atoms and vacancies, are created under irradiation. They
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14 migrate into the microstructure and may eliminate on PD sinks, giving rise to PD fluxes.
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16 Furthermore, these PDs may be coupled with solutes and, if the coupling is positive,
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18 drag solute atoms towards PD sinks [15]. This flux coupling will produce some
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20 radiation-induced segregation (RIS) phenomena at dislocation loops [14,16-17]. Up to
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22 date, only two studies reported RIS behavior near loops in CrFeMnNi MPEAs but with
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24 different results. Kamboj *et al.* [14], studied RIS phenomena in 18Cr-29Fe-23Mn-30Ni
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26 and found a significant Ni enrichment and Cr, Fe, Mn depletion on dislocation loops
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28 after irradiation with 6 MeV Fe ions up to 2 dpa at 773K using atom probe tomography
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30 (APT). However, RIS behavior on loops was not detected in neither 18Cr-27Fe-27Mn-
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32 28Ni nor 15Cr-35Fe-15Mn-35Ni irradiated with 3.7 MeV Ni ions to 75 dpa at 773K
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34 [18], with the same technique as Kamboj.

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38 It thus appears that loop density evolution as a function of temperature and RIS
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40 behavior around loops are still under debate for CrFeMnNi MPEAs. In this work, 15Cr-
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42 46Fe-17Mn-22Ni [19] was chosen as a model alloy and irradiated in-situ within the
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44 JANNuS-Orsay platform [20] up to 0.2 dpa with a dose rate of $1.6 \times 10^{-4} \text{ dpa/s}$. This low
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46 dose avoids the formation of radiation-induced damage microstructures other than
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48 dislocation loops [14], over a wide range of temperatures. The temperature effect from
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50 95K to 823K on loop density evolution is studied comprehensively, and its effect on
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52 RIS behavior near loops is also discussed.

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56 The high-purity 15Cr-46Fe-17Mn-22Ni (at.%) MPEA was elaborated by cold
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58 crucible induction melting at MINES Saint Etienne (EMSE, France) [19]. The alloy
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60 was firstly hot forged at 1273K, and then homogenized under argon flow at 1273K for
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1 two hours, followed by air-cooling. Prior to irradiation, transmission electron
2 microscopy (TEM) samples were electropolished with a solution composed of 30%
3 nitride acid in methanol (vol.%) at 243K under 10 V. Thin foils were irradiated in-situ
4 with 2 MeV Fe⁺ ions using a rastered beam at an ion flux of $\sim 1.8 \times 10^{11}$ ions·cm⁻²·s⁻¹ up
5 to a fluence of $\sim 2.5 \times 10^{14}$ ions·cm⁻² from 95K to 823K within the JANNuS-Orsay
6 platform [20]. We calculated the damage rate by using the Iradina code [21] in Kinchin-
7 Pease mode with a displacement energy of 40 eV. The depth profile of damage and Fe⁺
8 implantation profile are plotted in Fig.S1(a), the details are given in Table 1. After
9 irradiation, the radiation-induced dislocation loops were analyzed within a 200 kV FEI
10 TECNAI G² TEM. Loop densities were calculated as the surface densities of loops
11 divided by the local thickness of the analyzed zone. The latter was estimated by the
12 Convergent Electron Beam Diffraction (CBED) technique with $\mathbf{g}=\langle 202 \rangle$ reflection
13 [22-23]. Surface densities were calculated using the statistical method (details can be
14 found in [24]). Focused Ion Beam (FIB, FEI Helio 650 NanoLab dual beam scanning
15 electron microscope (SEM)) was applied to prepare transverse TEM thin foils, which
16 was followed by a flash polishing technique [25] and shape atom probe tomography
17 (APT) needles from irradiated thin foil, as shown in the schematic Fig.S1(b). Then,
18 compositional evolutions between dislocation loops and irradiated matrix were
19 followed by means of scanning transmission electron microscopy coupled to energy
20 dispersive X-ray spectroscopy (STEM-EDS) in the 200 kV JEM-ARM200F TEM and
21 APT technique. The APT needles were analyzed using a laser assisted APT (CAMECA
22 LEAP 4000X HR) with a pulse repetition rate of 200 kHz at 50K. Laser energy pulses
23 were set to an equivalent evaporation fraction of 20%. Reconstructions of the volumes
24 were performed with IVAS 3.6.8 (CAMECA software) using spatial distribution maps
25 (SDM) on low index crystallographic poles.
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52 Before irradiation, the microstructure and composition of the as-received samples
53 are checked with SEM-EDS (ZEISS 1525 equipped with a FEG source), as shown in
54 Fig.S2, and the measured chemical composition is shown in Table 2. After irradiation,
55 loop densities in the thin foils are analyzed from 95K to 823K. The damage evolution
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at 823K as a function of the dose in given in supplementary. The speed rate is accelerated 100 times. We report in Fig.1 a panel of the typical kinetic bright field (KBF) loops microstructure obtained at all the investigated temperatures at the end of irradiation. At temperatures lower than 623K, the microstructure is composed of a huge density of small-sized loops (<10 nm), which appear as black dots. With increasing temperature from 623K (Fig.1(c), loop size: 28 ± 12 nm) to 823K (Fig.1(e), loop size: 58 ± 16 nm), the loops grow up, while their densities decrease significantly. Both faulted Frank loops and perfect loops are observed at 823K. Due to the high density and small size of loops, a statistical analysis method [24] is used to get the total number density of loops for each irradiation temperature and account for the extinction condition. This method is illustrated in Fig.2 for a sample irradiated at 823K to the final dose. Firstly, the KBF TEM micrographs are taken on the same zone with five different \mathbf{g} vectors (at least two of them non-coplanar [24]), as shown in Fig.2(a)-(e). The visible loop density is calculated for each micrograph (see Fig.2f). Secondly, a system of coupled equations based on the invisibility criterion [26] is established for Frank and perfect loop families. The coupled equations are written as:

$$\begin{cases} N_{\mathbf{g}=\bar{1}\bar{1}1}^v = \frac{4}{4} N_{\mathbf{b}=\frac{1}{3}\langle 111 \rangle} + \frac{3}{6} N_{\mathbf{b}=\frac{1}{2}\langle 110 \rangle} \\ N_{\mathbf{g}=\bar{2}02}^v = \frac{2}{4} N_{\mathbf{b}=\frac{1}{3}\langle 111 \rangle} + \frac{5}{6} N_{\mathbf{b}=\frac{1}{2}\langle 110 \rangle} \\ N_{\mathbf{g}=0\bar{2}0}^v = \frac{4}{4} N_{\mathbf{b}=\frac{1}{3}\langle 111 \rangle} + \frac{4}{6} N_{\mathbf{b}=\frac{1}{2}\langle 110 \rangle} \\ N_{\mathbf{g}=022}^v = \frac{2}{4} N_{\mathbf{b}=\frac{1}{3}\langle 111 \rangle} + \frac{5}{6} N_{\mathbf{b}=\frac{1}{2}\langle 110 \rangle} \\ N_{\mathbf{g}=\bar{1}\bar{1}1}^v = \frac{4}{4} N_{\mathbf{b}=\frac{1}{3}\langle 111 \rangle} + \frac{3}{6} N_{\mathbf{b}=\frac{1}{2}\langle 110 \rangle} \end{cases}$$

in which $N_{\mathbf{g}}^v$ is the visible loop density for the applied \mathbf{g} vector, $N_{\mathbf{b}=\frac{1}{3}\langle 111 \rangle}$ is the total density of Frank loop, and $N_{\mathbf{b}=\frac{1}{2}\langle 110 \rangle}$ is the total density of perfect loop. Using the numerical values of loop densities, this system also can be expressed as (loops densities are in m^{-3}):

$$\begin{pmatrix} 6.12 \\ 8.19 \\ 7.60 \\ 8.21 \\ 6.15 \end{pmatrix} \times 10^{20} = \begin{pmatrix} 1 & 1/2 \\ 1/2 & 5/6 \\ 1 & 2/3 \\ 1/2 & 5/6 \\ 1 & 1/2 \end{pmatrix} \begin{pmatrix} N_{\mathbf{b}=\frac{1}{3}\langle 111 \rangle} \\ N_{\mathbf{b}=\frac{1}{2}\langle 110 \rangle} \end{pmatrix}$$

The least-squares method is employed to solve the system, and the error is given by standard deviation. The densities of Frank and perfect loops are respectively $N_{\mathbf{b}=\frac{1}{3}\langle 111 \rangle} = 1.7 \pm 0.1 \times 10^{20} \text{ m}^{-3}$ and $N_{\mathbf{b}=\frac{1}{2}\langle 110 \rangle} = 8.8 \pm 0.1 \times 10^{20} \text{ m}^{-3}$. Finally, the total loop density is $N_{\text{total}} = N_{\mathbf{b}=\frac{1}{3}\langle 111 \rangle} + N_{\mathbf{b}=\frac{1}{2}\langle 110 \rangle} = 10.5 \pm 0.1 \times 10^{20} \text{ m}^{-3}$. This method is then applied to the whole batch of irradiated samples (Fig.S3 to Fig.S6). The loop density distribution as a function of temperature is plotted in Fig.3. We also gather on the same graph the loop densities from literature about conventional austenitic steels irradiated to low dose until stationary state between 298K and 873K [27-33], as well as reported CrFeMnNi MPEAs [8-9]. This graph demonstrates the similar evolution of loop density between the studied MPEA in this work and the conventional stainless steels. From 95K to 623K, the loop density is nearly constant ($\sim 5.0 \times 10^{22} \text{ m}^{-3}$) and is temperature independent. Above 623K, it decreases drastically to reach $1.1 \pm 0.2 \times 10^{22} \text{ m}^{-3}$ at 723K and $10.5 \pm 0.1 \times 10^{20} \text{ m}^{-3}$ at 823K. It is worth noticing that the loop density at 95K ($1.7 \pm 0.2 \times 10^{22} \text{ m}^{-3}$) is a bit lower than at 298K ($5.0 \pm 0.1 \times 10^{22} \text{ m}^{-3}$), this could come from a poor contrast of small defects and/or overlapping effects arising from a rather thick working area ($\sim 220 \text{ nm}$) at 95K [8,34].

In order to study RIS phenomena near dislocation loops, STEM-EDS is firstly applied on the 823K sample with investigated loops edge-on. As a consequence, the FIB lamella is oriented on the [001] zone axis, in which only one third of the perfect $\langle 110 \rangle$ type loop planes are parallel to the incident electron beam. The STEM-BF image and corresponding EDS Ni mapping (Fig.S7) show that Ni enrichment occurs at each visible dislocation loop, and the strongest enrichment corresponds to edge-on loops. RIS amplitude has then been quantitatively estimated on one edge-on perfect loop. Fig.4(a) is a STEM-BF image taken from the FIB lamella on $z=[001]$ zone axis, Fig.4(b)

1 exhibits a perfect loop (loop circled in green circle in Fig.4(a)) in the edge-on
2 orientation. The composition profiles across the loop habit plane along the arrow (in
3 Fig.4(b)) are shown in Fig.4(c), and chemical segregation behavior with substantial Ni
4 enrichment and Cr, Fe, Mn depletion is observed. The corresponding STEM-EDS
5 mappings are shown in Fig.S8. To confirm this STEM-EDS measurement and get a
6 better spatial distribution of atoms along loop plane, APT analyses have been performed.
7 Dislocation loops are investigated indirectly through their possible chemical
8 redistribution in the vicinity of loops. They are decorated by enriched/depleted elements
9 and, as a result, can be easily identified. Fig.5(a) illustrates a 3D reconstructed APT
10 volume extracted 230 nm away from the surface after irradiation at 298K. No obvious
11 segregation/depletion is detected in the investigated volume. The composition profiles
12 in Fig.5(b) also show that the chemical composition of the sample is identical to the
13 composition of the as-received one measured by SEM-EDS (solid line is mean value,
14 dashed line is the error bar). However, disk-shaped segregations at 823K are obtained
15 in Fig.5(c) and are comparable to those detected in Fig.4(c), providing further
16 confidence that the object intercepted by APT is a dislocation loop. In order to highlight
17 chemical evolution between the adjacent matrix and the loop, a cylinder is inserted
18 perpendicular to this investigated loop (Fig.S9) and the concentration profiles confirm
19 the segregation in Ni and depletion in Cr, Fe and Mn around the loop (Fig.5(d)). Our
20 STEM-EDS and APT results reveal a good agreement on chemical
21 segregation/depletion amplitude (Fig.5(d)).
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44 In irradiated alloys, RIS phenomena is driven by a coupling with vacancies or self-
45 interstitial atoms (SIAs). In concentrated alloys, the vacancy mechanism is usually
46 considered and is related to the well-known inverse kirkendall (IK) effect, as in
47 austenitic stainless steels. When RIS occurs through an IK effect, slow diffusers
48 segregate while fast diffusers deplete at defect sinks [35]. It is linked to the preferential
49 exchange of a solute with vacancy flux. The flux of a particular atom is proportional to
50 the local atomic concentration, the vacancy diffusivity and the solute self-diffusion
51 coefficient [36]. Thanks to the investigation of radiotracer thermal diffusivity
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1 coefficients for all the elements in equiatomic CoCrFeNiMn HEA at elevated
2 temperatures [37], it is well known that the order of the self-diffusion coefficients is:
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4 $D_{Mn} > D_{Cr} > D_{Fe} > D_{Ni}$. Thus, we expect the fastest diffuser Mn to deplete and the slowest
5 one Ni to enrich on loops, as observed in our study. Depletion or enrichment of Cr and
6 Fe are dependent on the Cr self-diffusion coefficient (D_{Cr}) and Fe self-diffusion
7 coefficient (D_{Fe}) relative to the average self-diffusion coefficient (D_a) in the alloy: if
8 $D_{(Cr/Fe)}/D_a > 1$, Cr/Fe will deplete, otherwise Cr/Fe will enrich. In the present study, Cr
9 and Fe deplete in our study. This is consistent with the result obtained in the irradiated
10 FeNiMnCrCo HEA, in which slower diffusers (Ni, Co) enriched while faster diffusers
11 (Cr, Fe, Mn) depleted at sinks [16-17]. It is also worth to mention that the shape of the
12 Fe profiles obtained in Fig.4(c) and Fig.5(d) are not the typical “V-shape”, which is
13 usually obtained from solute depletion driven by IK mechanism, but a “W-shape”
14 [8,38], a transient state before the stationary state “V-shape” [39]. Finally, as already
15 mentioned, there are no obvious segregation phenomena detected by APT at 298K. At
16 this low temperature, vacancy diffusivity at 298K is probably not large enough to
17 permit any significant IK effect on dislocation loops. Therefore, no RIS phenomena are
18 detected at clusters in the 298K sample.

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36 In summary, temperature effect on loop density evolution in 15Cr-46Fe-17Mn-
37 22Ni has been carefully investigated between 95K and 823K for the first time: loop
38 densities are temperature independent and are close to $\sim 5.0 \times 10^{22} \text{ m}^{-3}$ from 95K to 623K.
39 Above 623K, loop densities decrease monotonically down to $\sim 1.05 \times 10^{21} \text{ m}^{-3}$ at 823K.
40 It shows the same trend within conventional ternary Fe-Cr-Ni alloys. Besides, RIS near
41 loops indicates a temperature dependent behavior: Ni enrichment and Cr, Fe, Mn
42 depletion are observed probably driven by IK mechanism at 823K by STEM-EDS and
43 APT with comparable RIS amplitude. In contrast, no obvious chemical
44 segregation/depletion phenomena are detected at 298K, where IK effect is probably not
45 activated. This comprehensive study of loop behavior as a function of temperature gives
46 a better understanding of CrFeMnNi MPEAs under irradiation.

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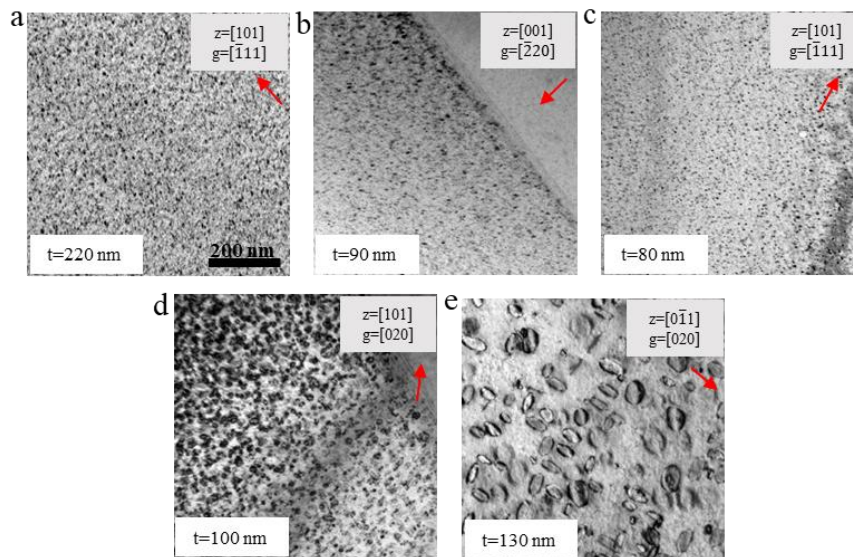


Fig.1 Kinetic BF TEM micrographs of irradiation-induced defects to the final dose at (a) 95K, (b) 298K, (c) 623K, (d) 723K, (e) 823K. “t” is the thickness of the zone observed.

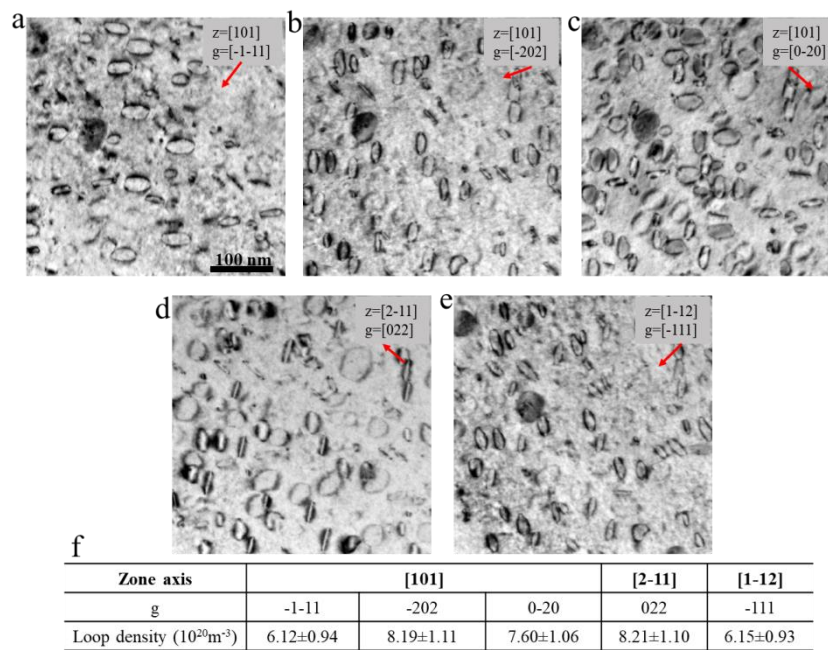


Fig.2 Dislocation loop density calculation by statistic method [24] of the sample irradiated to the final dose at 823K: (a-e) KBF TEM micrographs of the same zone showing visibility of loops under five g vectors; (f) calculated loop density with five different g used for statistic analysis.

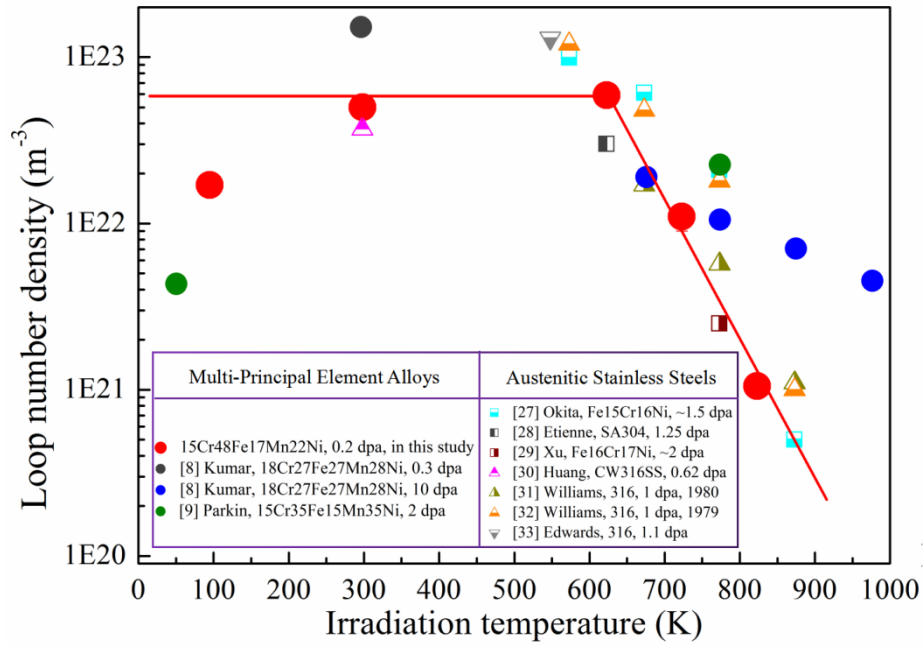


Fig.3 Evolution of dislocation loop density as a function of ion irradiation temperature.

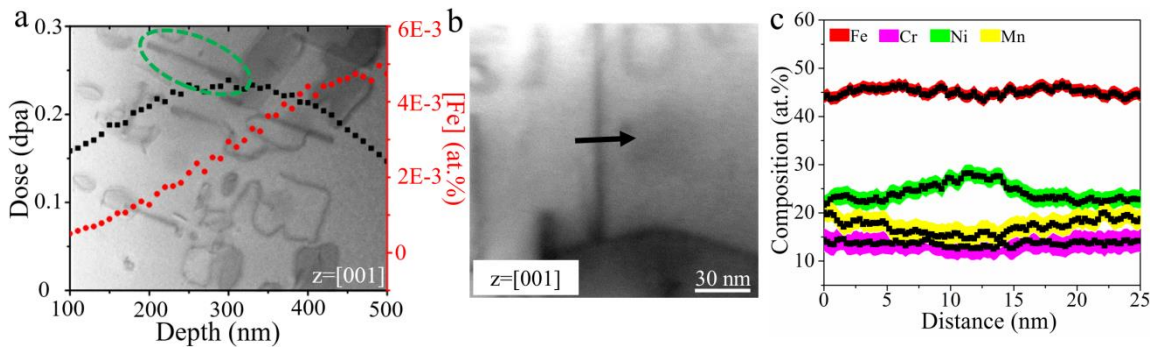


Fig.4 RIS on a dislocation loop at 823K by STEM: (a) STEM-BF image of FIB lamella on $z=[001]$ zone axis superimposed with damage (in black) and implanted (in red) ion depth profiles; (b) STEM-BF micrograph of the zoomed edge-on loop in (a); (c) composition profiles across the loop along arrow in (b).

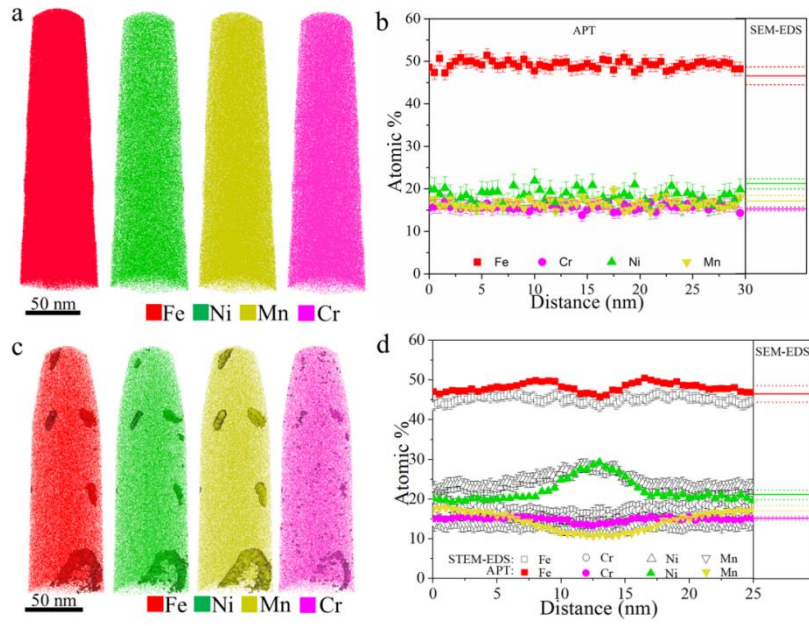


Fig.5 APT results of irradiated specimen: (a) APT reconstructed volume ($80 \times 80 \times 230 \text{ nm}^3$) and (b) corresponding concentration profiles at 298K; (c) 3D distribution of Fe, Ni, Mn, Cr ($60 \times 60 \times 200 \text{ nm}^3$) and (d) concentration profiles between matrix and dislocation loop at 823K.

Tables:

Table 1: Summary of ion irradiation conditions for the 15Cr-46Fe-17Mn-22Ni MPEA.

Ion	Dose (dpa) (at peak)	Dose rate (dpa/s) (at peak)	Temperature (K)
Fe ⁺ 2 MeV	0.2	1.6 10 ⁻⁴	95, 298, 623, 723, 823

Table 2: Chemical composition of the 15Cr-46Fe-17Mn-22Ni alloy, as-received from EMSE (nominal composition). Measurements have been performed by SEM/EDS.

at.%	Fe	Ni	Cr	Mn
15Cr-46Fe- 17Mn-22Ni	46.5±2.1	21.1±1.2	15.2±0.4	17.3±1.3