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Confinement of motion of interstitial clusters and dislocation loops in BCC Fe–Cr alloys

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ABSTRACT

This work is devoted to the study of the effect of Cr solutes on the mobility of self interstitial atom (SIA) clusters and small interstitial dislocation loops (of size up to a few nanometers) in concentrated Fe–Cr alloys. Atomistic simulations have been performed to characterize the variation of the free energy of interstitial loops in the Fe–15Cr alloy using the experimentally determined profile of Cr distribution along the path of a loop. It is shown that the presence of randomly distributed Cr in Fe leads to the creation of local trapping configurations for small SIA clusters. The strength (trapping energy) and density of these configurations depend on the Cr content. On the contrary, large SIA clusters (which can be described as 1/2⟨1 1 1⟩ dislocation loops) are strongly affected by the presence Cr–Cr pairs and larger Cr clusters, which act as barriers to their motion.

1. Introduction

High-Cr ferritic/martensitic steels are primary candidate structural materials for future nuclear power plants, due to their good void swelling resistance and superior mechanical properties [1]. These steels, of body-centred cubic crystallographic structure, contain 7–14 wt% Cr, about 1–2 wt% of other alloying elements (Mo, Nb, V, W, Ta, Mn, Ti ... depending on the type of steel) and about 0.1 wt% carbon [1]. Thus, the main alloying element is Cr and this is why Fe–Cr binary alloys can be used as model materials to study the behaviour of these steels. Irradiation, in general, leads to a degradation of the mechanical properties of materials, via creation of radiation defects and secondary phase particles, both of them hindering the motion and creation of dislocations in metals. The rationalization of radiation induced effects on the microstructure and their consequences on the material properties by developing predictive models is thus of great importance in view of a safe design of future nuclear reactor concepts. The typical defects observed under/after irradiation in Fe-based alloys, in particular in Fe–Cr alloys, are voids, dislocation loops having Burgers vectors ⟨1 0 0⟩ or 1/2⟨1 1 1⟩ and Cr-rich precipitates, whose number density and mean size mainly depend on dose, temperature, radiation source and Cr content. The formation of Cr-rich precipitates is usually observed in alloys containing from 8 up to 16%Cr; in presence of other alloying and substitutional elements, other type of precipitates (η, G, χ and other phases) can be formed as well [1]. Under neutron irradiation, dislocation loops are mainly of interstitial type according to transmission electron microscopy (TEM) studies. Atomistic studies (e.g. molecular dynamics, MD) also suggest the formation of small, TEM-invisible self interstitial atom (SIA) clusters, which can be produced directly in the displacement cascade induced by primary knock-on atoms receiving energy from neutrons (e.g. for Fe Ref. [2], for Fe–Cr Ref. [3]). Experimental studies indicate that Cr atoms in Fe exhibit attractive interaction with SIAs, which is stronger in concentrated alloys, but also exists in dilute alloys, whereas almost no interaction of Cr with vacancies is found [4]. Thus, it is expected that the Cr–SIA interaction plays a crucial role in the evolution of the microstructure in FeCr alloys and its variation, depending on Cr content. Early and recent experiments have indeed shown that the mobility of small dislocation loops (from 1 to 10 nm) is suppressed by the presence Cr [5,6] and enrichment of dislocation loops by Cr atoms was experimentally observed in Fe–9Cr [6,7] and Fe–10Cr [5] after electron irradiation.

In pure Fe, the motion of 1/2⟨1 1 1⟩ SIA clusters occurs via glide along the closed packed direction, with extremely low energy barrier (tens of meV) by independent motion of SIAs keeping ⟨1 1 1⟩ orientation, i.e. crowdions [8]. A strong binding between Cr and ⟨1 1 1⟩ SIA in Fe was found in recent studies using density functional theory (DFT) calculations [9]. By coupling DFT and MD techniques based on a DFT-fitted interatomic potential for Fe–Cr [10], it has been shown that the motion of small SIA clusters (tens of defects) is significantly reduced in the presence of Cr, due to the long range attractive interaction between ⟨1 1 1⟩ SIA and Cr atoms [11,12]. The relative mobility (ratio of the diffusion coefficients in Fe and FeCr) of small SIA clusters was found to depend in a non-monotonic way on the Cr content and on the cluster size [11,12]. These works, however, focused only on small clusters (up to 91

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SIAs), while well experimentally resolved interstitial dislocation loops have size of a few nanometers (i.e. contain hundreds of defects), or larger.

In this work we study the effect of Cr on the mobility of visible $1/2\langle 1\ 1\ 1\rangle$ interstitial dislocation loops of size 3.5 and 5 nm, using a method based on molecular static (MS) simulations developed in [11]. At the same time, an attempt at establishing a correlation between the variation of the local chemical composition near the loop and its formation energy is made. To do this, we have estimated the distribution of the loop formation energy in Fe–Cr along the direction of its Burgers vector and identified positions corresponding to low energy states (i.e. energetically favourable configurations, where the loop is expected to spend most time). By doing this we aimed at revealing whether the trapping of $1/2\langle 1\ 1\ 1\rangle$ loops occurs in regions significantly enriched by Cr or not, as this could explain some of the cited experimental observations [5–7]. The calculations of the distribution of the loop formation energy have been performed in the Fe–15%Cr alloy using the experimentally determined Cr profiles taken from [13], where a detailed study using transmission electron microscopy (TEM) of irradiated single Fe–15%Cr crystals was performed. The interstitial dislocation loops were identified and the distribution of Cr composition near the loops was estimated. In this way, the most probable (i.e. energetically favourable) locations can be compared directly with TEM observations.

2. Calculation details

The analytical model used for the assessment of self interstitial cluster mobility in Fe–Cr alloys, based on the result of MS calculations, was developed in [12]. The model is built on the idea that an attractive interaction between Cr atoms and crowdions forming an SIA cluster causes a change in the formation energy of the SIA cluster while it diffuses in the matrix [12]. Because of such interaction, the 1D migration of a cluster in an Fe–Cr alloy can be represented as the motion in a field of energy valleys and hills, attributed to different energy states of the cluster, corresponding to different numbers of Cr atoms interacting with it. The time a cluster spends in a particular configuration in the Fe–Cr matrix is assumed to be proportional to $\exp(\beta\Delta F_n)$, where $\beta = (k_B T)^{-1}$, k_B is the Boltzmann constant, T is the absolute temperature and ΔF_n is the change in the system free energy as compared to the lowest energy state. The diffusivity, D^{FeCr}, of a cluster in the alloy can thus be expressed as a function of that in pure Fe, D^{Fe}, as:

$$D^{FeCr} = D^{Fe} \exp(\Delta F / \mathrm{k_B} T), \tag{1}$$

The definition of the change in the cluster free energy was given in Ref. [12] as:

$$\Delta F = k_B T \langle \exp(E_b(x)/k_B T)\rangle^{-1}, \tag{2}$$

where $E^b(x)$ is the binding energy of the SIA cluster in a particular configuration x and $\langle\ldots\rangle$ denote averaging over different configurations. The binding energies entering Eq. (2) were defined as the differences between the maximum energy state (i.e. the maximum formation energy) encountered in the simulation box and the formation energy in each configuration. Here, we have performed static calculations to characterize the change in the free energy of small dislocation loops in Fe–Cr alloys using the same simulation techniques as in Ref. [11]. Calculations were carried out for SIA clusters of circular shape containing 167 and 331 defects in Fe–Cr with Cr concentration varying from 0 up to 20%. In the case of the Fe–15Cr alloy, the simulation box was subdivided into 100 sub-blocks, each of size $1 \times 1 \times 6\ \mathrm{nm}^3$. In each sub-block the total Cr concentration was varied according to the experimentally determined Cr profile (however, in each sub-block atoms were randomly distributed). Thus, the total Cr concentration in the 100 sub-blocks altogether was equal to 15%, but in each of them it could vary around this mean value. The atomic position relaxation of the SIA clusters was performed using a quenching procedure to zero temperature [11]. The EAM-type potential of Ackland et al. [14] was used for the Fe–Fe interaction, while the description of the Fe–Cr and Cr–Cr interaction was taken from Ref. [10].

3. Results

The variation of the cluster free energy versus Cr concentration is shown in Fig. 1, calculated putting MS data in Eq. (2). The data for small SIA clusters are taken from Ref. [10]. Applying Eq. (1), these results can be immediately converted into the diffusion coefficient of the cluster of the corresponding size. On the whole, ΔF appears to be a non-monotonic function of the Cr concentration. For clusters of 7, 19, 37, 61 and 91 defects there is a minimum, whose location depends on the cluster size. As was discussed in [12], for small SIA clusters (up 91 SIA) the minimum becomes shallower and its position shifts towards lower Cr concentrations with increase of the cluster size. This translates into a non-monotonic decrease of the diffusivity of these small SIA clusters in Fe–Cr versus Cr concentration, as compared to pure Fe, with a minimum at lower concentration: the larger the cluster size, the lower the concentration where the minimum is located. In addition, the influence of Cr on the cluster diffusivity gradually disappears for larger Cr contents. An explanation for this has been proposed in [11] using the following reasoning. For a given cluster size, adding Cr will initially increase the number of interacting Cr–crowdion pairs, thereby increasing the overall binding energy (i.e. decreasing the cluster self energy), until a free energy minimum is reached, when each crowdion in the cluster interacts with one Cr atom. Further increasing the Cr concentration will lead to a situation where each crowdion will interact with more than one Cr atom and the effective binding energy will therefore decrease, due to a saturation of the Cr–crowdion interaction. Since the local Cr distribution near a cluster of increasing size becomes more and more uniform, the effective binding energy decreases and the free energy minimum shifts to lower Cr concentrations.

For large SIA clusters containing 167 and 331 SIAs, ΔF exhibits only a very shallow minimum at 1%Cr and starts to decrease above 10% again, as can be seen from Fig. 1. The decrease is more pronounced for the larger cluster. 1%Cr was the lowest Cr content

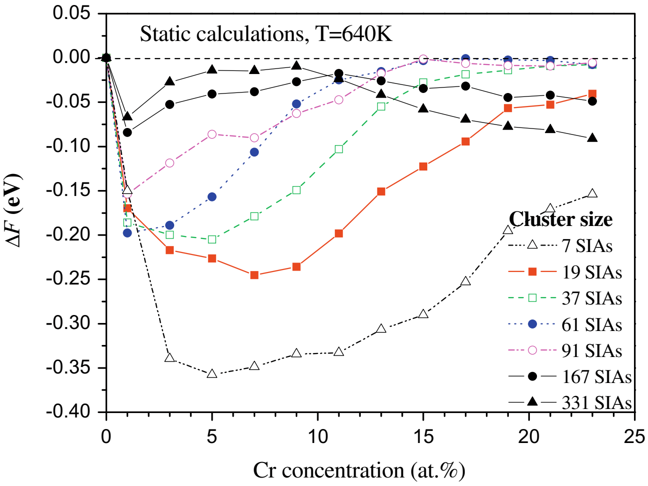

Fig. 1. Change in the free energy of SIA clusters of different sizes estimated for $T = 640$ K.

studied here, so the presence of a minimum there for different loops sizes suggests that the saturation of Cr–crowdion interaction probably has occurred already and the real minimum most likely is located below 1%Cr. The absolute value of ΔF for the 167 and 331-SIA clusters is much smaller than that for the 91-SIA cluster. Thus, either the amount of Cr–crowdion pairs is smaller, or the interaction strength of Cr–crowdion is weaker for the large SIA clusters, or both. Changes in the structure of large SIA clusters with respect to small clusters (up to 91 SIAs) can be the reason for a decrease in the interaction strength, as well as for a decrease in the number of actual crowdions interacting with Cr. The results obtained in earlier works [11,12] show that the highest binding energy between Cr and crowdion occurs in the region where the interatomic distance between two atoms along the $\langle 1\,1\,1\rangle$ direction ($dx_{\langle 111\rangle}$) is significantly smaller than b (Burgers vector length), i.e. the edge of the loop. The highest binding energy is found when the solute atom is located at the centre of the crowdion and the smaller the displacement between two atoms along the $\langle 1\,1\,1\rangle$ direction (moving away from its centre), the weaker the Cr–crowdion interaction energy. A study of the structure of SIA clusters [15], using the same Fe–Fe potential as here, has shown that, by increasing the cluster size from 7 to 331 SIAs, the distortion associated to the crowdions forming the cluster decreases and does so more rapidly in the central part of the cluster than at the edge [15]. In the case of a perfect dislocation loop, above a certain size the distortion field in the central part should become zero, while at the edge, in the limit of large sizes, it should correspond to the displacement field in the core of the $1/2\langle 1\,1\,1\rangle$ edge dislocation, which is not going to be coincident with the displacement field associated with a crowdion in a small cluster. Setting a cut-off crowdion displacement, at which the Cr–crowdion binding energy vanishes, one can define a critical size of the cluster above which it interacts with Cr only at its edge. Taking 0.05 eV as a cut-off energy, which is the average atomic kinetic energy at room temperature, a critical distance between two atoms along the $\langle 1\,1\,1\rangle$ direction of 0.8 lattice units is obtained [12]. The interatomic distances, $dx_{\langle 111\rangle}$, between two atoms in the habit plane of clusters of different sizes are shown in Fig. 2. It is clear that clusters containing more than 60 SIAs mainly interact with Cr via crowdions located at the edge. However, the ΔF curves given in Fig. 1 for 61- and 91-SIA clusters are quite similar to each other. Apparently the structure of the edge of SIA clusters containing less than 100 SIAs is still sufficiently close to a platelet of $\langle 1\,1\,1\rangle$ crowdions to provide the same type of interaction with Cr atoms, while above 100 SIAs it becomes closer to the structure of the edge dislocation and the actual number of properly called crowdions decreases, thereby decreasing the strength of the interaction with Cr atoms.

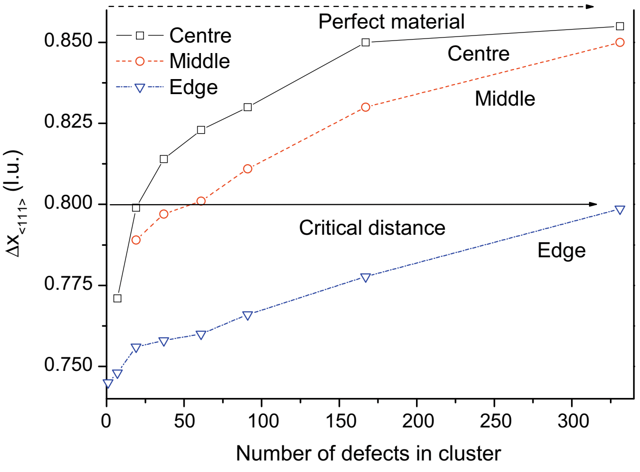

Fig. 2. Interatomic distance between two adjacent atoms for SIA clusters of different sizes along the line perpendicular to the cluster habit plane that crosses the cluster at its centre, edge and between edge and centre (so called middle). The critical distance indicated represents the interatomic distance along the $\langle 1\,1\,1\rangle$ direction below which the Cr–crowdion interaction is significant.

Thus, the change in the curvature of ΔF presented in Fig. 1 for 91-SIA cluster and for larger SIA clusters can be explained by the existence of differences between the Cr–crowdion interaction and Cr–edge dislocation interaction. If, as it appears, the interaction energy of the edge dislocation with Cr atoms is weaker than the Cr–crowdion interaction energy, then the low value of ΔF_n at 1%Cr can be explained. The decrease of ΔF_n at $C_{\mathrm{Cr}} > 10\%$ suggests, on the other hand, that the interaction of Cr–Cr pairs and larger Cr clusters with an edge dislocation is stronger than the interaction with isolated Cr atoms. To verify these assumptions we have estimated the interaction energy of the $1/2\langle 1\,1\,1\rangle$ $\{1\,1\,0\}$ edge dislocation with a single Cr atom and Cr–Cr pair depending on the location of Cr relative to the core of the dislocation. In the latter case one Cr was kept in the core of the dislocation, while the position of the second Cr atom was varied. The corresponding binding energies are presented in Fig. 3. As one can see from Fig. 3(a), a single Cr atom is attracted to the edge dislocation and the maximum binding energy occurs when the Cr atom is located in the core. Even in this case, though, the binding energy reaches 0.1 eV, which is about four times lower than the binding energy with a crowdion (0.42 eV) [12]. In all other positions the binding energy does not exceed 0.05 eV (see Fig. 3(a)). In contrast to it, the interaction between Cr and dislocation containing one Cr in the core is repulsive, as shown in Fig. 3(b). The maximum repulsion occurs when Cr atoms stay as first nearest neighbours, so that a line joining them is perpendicular to the dislocation line and both atoms are located in the slip plane of the dislocation. Note that the repulsive interaction energy, which is about 0.25 eV, is considerably higher than the binding energy of the Cr–edge dislocation (0.1 eV). This strong repulsion vanishes at a distance of about 1 nm (see Fig. 3(b)). In addition, the binding energy between two Cr atoms inserted in the core of the edge dislocation was found to be −0.18 eV (i.e. repulsive interaction), it decreases with increasing distance between Cr atoms and vanishes completely if the distance is larger than $3b$ (i.e. ∼1 nm). Clearly, therefore, the presence of Cr–Cr pairs standing as first nearest neighbours will increase the self energy of the edge dislocation and, therefore, will affect the self energy of large SIA clusters, whose structure is similar to that of a perfect $1/2\langle 1\,1\,1\rangle$ dislocation loop. Thus, in Cr-rich regions with relatively high concentration of Cr–Cr pairs, the presence of large SIA clusters is expected to be unfavourable.

The above presented analysis suggests that the binding energy of SIA clusters large enough to be defined as dislocation loops decreases in Cr-rich regions. While for small SIA clusters, which interact with them not just at the edge, but as a whole, and can be described as a bunch of 'independent' $\langle 1\,1\,1\rangle$ crowdions, it can grow (in particular, it has been shown in [12] that the interaction with Cr-rich precipitates is strongly repulsive). In order to verify this qualitatively different behaviour depending on size, the distribution of binding energy along the path of SIA clusters containing 37 and 331 defects in Fe–15Cr alloy are shown in Fig. 4, together with the Cr profiles. Dashed lines denote the average Cr content and average binding energy for the given cluster in the matrix. The Cr concentration profiles were taken directly from experimental work [13]. In the case of the 37-SIA cluster (see Fig. 4(a) and (b)) the peaks in the binding energy distribution coincide with the peaks of Cr concentration. Such correlation is not observed for the 331-SIA cluster, as can be seen from Fig. 4(c) and (d). In some cases, the binding energy of the cluster was clearly seen to decrease while the Cr concentration rises.

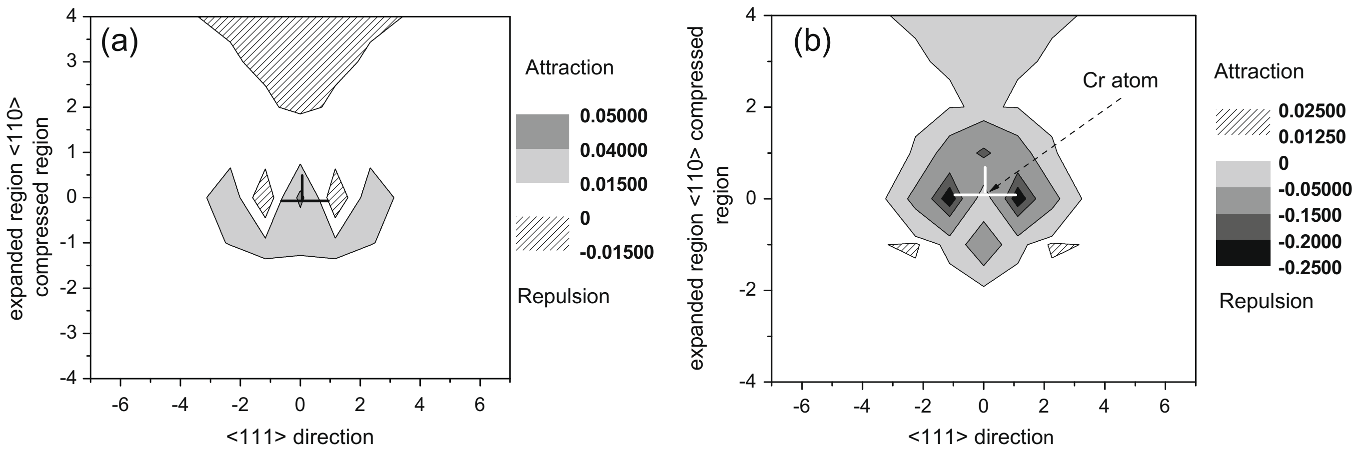

Fig. 3. Binding energy between the 1/2⟨1 1 1⟩ edge dislocation core and (a) isolated Cr atoms or (b) Cr–Cr pairs. The dislocation core is located at the centre and its line is perpendicular to the paper. The binding energies are displayed next to the corresponding figures.

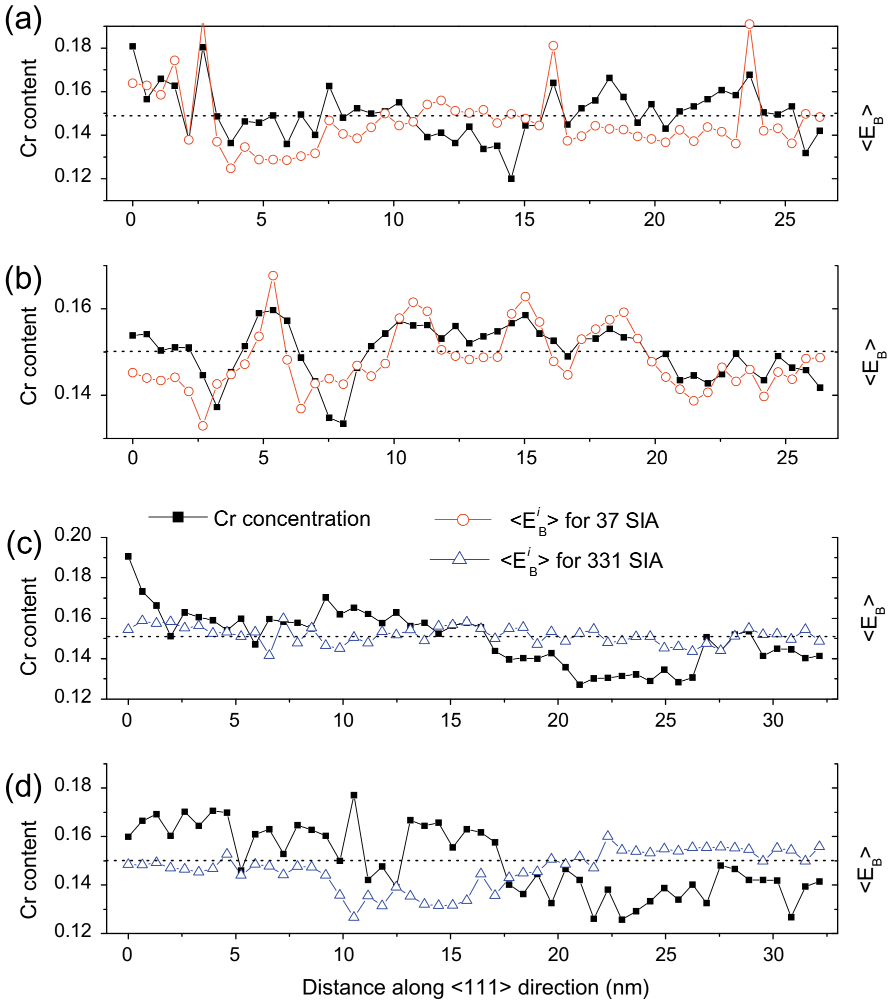

Fig. 4. Distribution of Cr profiles along ⟨1 1 1⟩ direction in a volume 1 × 1 × 6 nm^3 experimentally measured in [13] together with binding energy of 37-SIA cluster (a and b) and 331-SIA cluster (c and d) estimated using MS techniques.

4. Discussion

Summarizing the obtained results, we have shown that small SIA clusters (up to 37 SIAs) strongly interact with Cr atoms due to the attraction between ⟨1 1 1⟩ crowdions and Cr. However, with increasing cluster size, the distortion associated to the crowdions forming it changes. In a cluster containing more than 60 SIAs, the interaction with Cr occurs only or mainly near its edge, where the crowdions are still properly defined. Above this size, the structure (in terms of associated displacement field) of the crowdions at

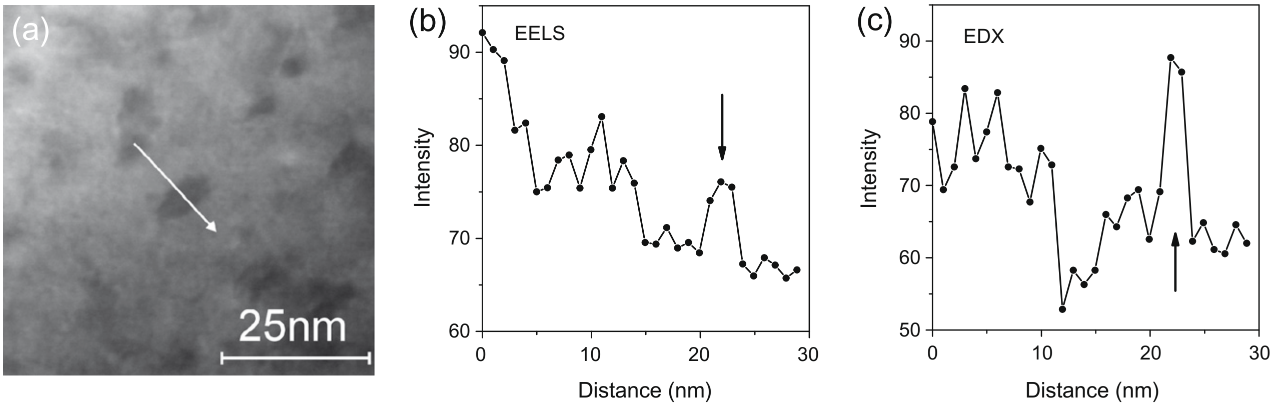

Fig. 5. The left side of the figure is the experimental TEM image of a Fe–15%Cr crystal irradiated with Au ions, the white arrow crosses a 1/2⟨1 1 1⟩ dislocation loop and shows the direction of the scanned area; the right side of the figure shows the signal intensity obtained using EELS (b) and EDX (c) techniques, used to build Cr concentration profiles. The TEM image and the data are from [13].

the edge of a cluster represents an intermediate case between the 1/2⟨1 1 1⟩ edge dislocation and an isolated ⟨1 1 1⟩ crowdion. The structure of the edge of the 331-SIA cluster is, on the other hand, very close to that of the 1/2⟨1 1 1⟩ edge dislocation. Thus, such cluster is better considered as a dislocation loop, rather than as a platelet of ⟨1 1 1⟩ crowdions. Although the presence of an isolated Cr in the core of the dislocation is slightly favourable (0.1 eV), adding another Cr atom next to it is not. These calculations have thus shown that the interaction between Cr and edge dislocation is attractive, but the energy involved is much weaker than the Cr–crowdion binding energy, while the interaction of the edge dislocation with Cr–Cr pairs is repulsive. Therefore, the formation energy of dislocation loops is much less affected by the presence of isolated Cr atoms than by Cr–Cr pairs and, probably, larger Cr clusters (contrary to SIA clusters, too small to be considered dislocation loops). In addition, static calculations have shown the presence of a strong repulsion (0.18 eV) between two Cr atoms being put at the core of the edge dislocation or just nearby it. Thus, only a certain amount of Cr can be 'accumulated' at the core and nearby the core of an edge dislocation. It follows that the 'optimal' content of Cr near the edge dislocation would be about 9–10%, which corresponds to a situation where the Cr atoms in the four ⟨1 1 1⟩ rows along the core are separated by a distance of 3***b***. Thus, if the Cr content is below 10%, segregation of Cr at the core of the edge dislocation as well as at the edge of the interstitial dislocation loops may occur, while in alloys with Cr content >10% it should not.

However, beside the above mentioned energetic considerations, the segregation of solutes at dislocation loops and in general at sinks for point defects may occur via a flux of defects transferring solutes. In this case, however, a relatively strong interaction between segregating solutes and point defects is necessary. In the case of the Fe–Cr system, the Cr–SIA (⟨1 1 0⟩ dumbbell) binding energy is about 0.15 eV (according to DFT [9], which is also reproduced with the used potential [16]). Thus, in Fe–Cr alloys under irradiation conditions single-interstitials will migrate in the ⟨1 1 0⟩ Fe–Cr mixed dumbbell configuration, thereby dragging Cr towards sinks [16]. In this case, the growth of dislocation loops may be accompanied by a local Cr enrichment near them. This logic, however, is applicable to dilute or low-concentration alloys, where the influence of surrounding Cr atoms on the migration of Fe–Cr mixed dumbbell can be neglected. In concentrated alloys, the drag of Cr atoms via dumbbell will be suppressed, since the Cr–Cr ⟨1 1 0⟩ dumbbell configuration, unavoidably forming during the long range migration, is unstable (according to DFT [9] and used potential [16]). The critical Cr concentration at which long range Cr drag will not occur any longer was proposed to be about 10% [16]. Thus, the enrichment of dislocation loops by Cr observed in Fe–9%Cr [6] and Fe–10%Cr [5] under electron irradiation is probably a consequence of non-equilibrium segregation of Cr via interstitials.

In recent experimental work by Klimenkov et al. [13], a systematic study of Cr concentration profiles near 1/2⟨1 1 1⟩ loops has been carried out in Fe–15%Cr single crystals after ion implantation (for irradiation conditions and experimental details see [13]). One of the goals of the study was to see whether Cr enrichment at dislocation loops occurs and, if so, whether this effect can be detected systematically. The TEM image of the 1/2⟨1 1 1⟩ dislocation loop is shown in Fig. 5(a). The intensity of Cr along the Burgers vector of the inspected loop was estimated using electron energy loss spectroscopy (EELS) and energy dispersive X-ray analysis (EDX) techniques and the results are presented in Fig. 5(b) and (c), respectively. Note that the results obtained by both techniques confirm the increase of Cr concentration near the location of the dislocation loop. However, the application of such analysis to other well resolvable 1/2⟨1 1 1⟩ loops has shown that a slight Cr enrichment (by 18–20%Cr) was detected only in three out of 30 inspected loops i.e. only 10%. In all other cases, no enrichment of Cr concentration near a position of the dislocation loop has been established, therefore making the existance of Cr enrichment doubtful, at least in a Fe–15Cr matrix. The cases in which enrichment was observed should be therefore attributed to local fluctuations of Cr content, since the deviation from the average Cr concentration was not spectacular, as can be seen from Fig. 5. This result is, however, not in contradiction with the enrichment observed at lower concentration, because of the above considerations: energetically, in an Fe–Cr alloy containing more than 10%Cr it is not enrichment, but slight depletion that should be observed. Kinetic processes may, however, hinder the establishment of a depleted region, thereby giving as result no clear tendency.

5. Conclusions

The results of this work suggest that:

1. The interaction between SIA cluster and Cr atoms depends on the interplay between size of the SIA cluster and (local) Cr concentration: it becomes weaker for large SIA clusters which can be described as 1/2⟨1 1 1⟩ dislocation loops.
2. Dislocation loops, contrary to small SIA clusters, exhibit weak attractive interaction with isolated Cr atoms, while they are repelled by Cr–Cr pairs.

3. As a consequence, the mobility of small SIA clusters is affected more significantly in the low Cr region (up to 10%), where single Cr atoms are generally encountered, while the mobility of large SIA clusters (small dislocation loops) is affected more strongly in alloys containing more than 10%Cr, where the probability to encounter a Cr–Cr pair increases strongly.
4. It follows that in Fe–15%Cr alloy the accumulation of Cr at the edge of the small dislocation loops should lead to an increase of their formation energy. Thus, the non-systematic experimentally observed Cr enrichment of $1/2\langle 1\,1\,1\rangle$ loops by Cr in Fe–15Cr alloy [13] should be ascribed to the local fluctuations of Cr. The enrichment in Fe–9,10Cr reported in [5,6] is believed to be due to the non-equilibrium segregation of Cr atoms, transported via Fe–Cr mixed dumbbells eventually absorbed by the loops.

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