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Dislocation loops in ultra-high purity Fe(Cr) alloys after 7.2 MeV proton irradiation

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ABSTRACT

Ultra-high purity Fe(Cr) alloys (from 0 wt% Cr to 14 wt% Cr) were 3D homogeneously irradiated by 0–7.2 MeV protons to 0.3 dpa at nominal temperatures from 270 °C to 500 °C. Microstructural changes were observed by transmission electron microscopy (TEM). The results showed that evolution of dislocation loops depends on the Cr content. Below 300 °C, large ½ a_0 <111> loops are dominating. Above 300 °C, a_0 <100> loops with a habit plane {100} appear. Loop sizes of both types are more or less the same. At temperatures from 310 °C to 400 °C, a_0 <100> loops form clusters with the same {100} habit plane as the one of the loops forming them. This indicates that <100> loops of the same variant start gliding under mutual elastic interaction. At 500 °C, dislocation loops form disc shaped clusters about 1000 nm in diameter and sitting on {111} and/or {100} planes in the pure Fe samples. Based on these observations a quantitative analysis of the dislocation loops configurations and their temperature dependence is made, leading to an understanding of the basic mechanisms of formation of these loops.

1. Introduction

Advanced nuclear technology, such as fusion and Generation IV fission reactors, is a technological solution for a sustainable production of clean energy [1]. However development of radiation resistant structural materials remains a key element not only for future but also for current nuclear technology [2]. Considering the extreme service conditions, with large heat loads and irradiation dose, assessment of the irradiation induced degradation of their mechanical properties is crucial for a safe and reliable performance of such nuclear plants. Fe(Cr) based steels and their oxide dispersion strengthened (ODS) versions are considered as promising candidates for such structural materials in advanced nuclear systems, for instance, as a cladding material in fast reactors [3]. Instead of complex Fe(Cr) steels, pure Fe(Cr) so-called model alloys are often chosen as representatives for fundamental understanding of radiation damage. Intensive studies on microstructural changes of Fe(Cr) alloys under irradiation have revealed that clusters of self-interstitial atoms and interstitial perfect dislocation loops are among the major microstructural damage features at intermediate temperatures (RT to 500 °C). They act as obstacles for dislocation movement causing irradiation hardening and low temperature embrittlement. Furthermore, dislocation loops could bias the flow of point defects in the materials that is responsible for swelling, irradiation creep and precipitation.

For several decades, a number of transmission electron microscopy (TEM) studies [4–16] investigated the formation of dislocation loops in bcc Fe and Fe(Cr) alloys. A summary is given in Table 1. The results show ambiguity, but nevertheless, some general consensus on the loop properties can be drawn, as follows. Loops formed after irradiation in Fe and Fe(Cr) alloys have a Burgers vector ***b*** of 1/2 a_0 <111> or a_0 <100>. In the following they will be called 1/2 <111> loops and <100> loops, respectively. For the <100> loops the habit plane has been unambiguously identified to be only {100}, i.e. they form <100>{100} loops. Conversely, for the 1/2 <111> loops, their elementary habit planes can be {110}, {211} and {111}, as determined by TEM investigations [9,10]. The 1/2 <111> loops can be composed of multiple habit planes, especially in the case of small loops (below around 20 nm) [10]. With increasing loop size, the preferred habit plane of 1/2 <111> loops is {111} [15].

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Table 1
Summary of experimental studies on dislocation loops in bcc Fe(Cr) alloys and steels. Symbols used: T: irradiation/annealing temperature; *b*: Burgers vector; I: interstitial; V: vacancy; p: proton; α: helium; n: neutron; e: electron.

	T (K)	*b*	habit plane	nature	specimen	irradiation	ref.
Fe	823	<001>	{001}	I	thin	p, Fe$^+$	[4]
	333	1/2 <111>	{111}		bulk	n	[5]
	353-593	1/2 <111>			bulk	n	[6]
		<001>					
	623-723	1/2 <111>	{111}	I	bulk	n	[7]
		<001>	{001}				
	703-763	1/2 <111>	{110}-{111}		thin	e	[8]
		<001>	{001}				
	333	1/2 <111>	{111}, {110}		bulk	n	[9]
	573	1/2 <111>	{110}, {211},{111}		quasi-bulk	α	[10]
		<001>	{001}				
pure Fe to Fe18%Cr	573–773	1/2 <111>		V	thin	Fe$^+$	[11]
		<001>					
Fe-10%Cr	653–733	<001>	{001}	I	bulk	n	[12]
	573-773	1/2 <111>	{001}		thin	e	[13]
		<001>					
Fe-15%Cr	473–873	1/2 <111>	{111}		thin	e	[14]
		<001>	{001}				
PM2000	573–773	1/2 <111>	{111}	I	quasi-bulk	α	[15]
(19%Cr)		<001>	{001}	I			
F82H	573	1/2 <111>	{111}	I	bulk	n	[16]
(8%Cr)		<001>	{001}	I			

TEM characterization demonstrates that both types of loops are mainly interstitial in nature in the bulk or in the quasi-bulk, i.e. foils around 100 μm in thickness or more. Note that near a free surface, within few nanometers, loops induced by low energy heavy ions irradiation can be vacancy in nature, as observed in experiments [11] and simulations [17]. However, their appearance is not relevant to bulk material under irradiation as they are created by an unbalance of interstitials and vacancies due to severe loss of interstitials and their clusters to the surface.

The observations of the damage microstructures in irradiated iron and Fe(Cr) alloys still differ significantly from the structures observed in other bcc metals such as molybdenum [18], tungsten [19], niobium [20] and vanadium [21]. Notwithstanding the multiple habit planes of 1/2 <111>loops, the most important anomalous features of the radiation-induced microstructures in iron and Fe(Cr) alloys is that besides of 1/2 <111> loops, a rather large portion of <100> loops form during irradiation, specially at elevated temperatures. The loops observed in other irradiated bcc metals are mainly 1/2<111>loops. It should be noted that among them tungsten may exhibit some proportion of <100> loops in low energy heavy ions irradiated thin samples [22,23]. There are a few studies on the temperature dependence of loop properties in Fe(Cr) alloys [24,25] based on heavy-ion irradiation of thin foil samples. The glide and loss of 1/2<111> loop to free surfaces, which is dependent on the foil orientation [24], has been reported to strongly impact the ratio of <100> to 1/2<111>loops [24,25]. Besides, some results exist on the impact of the Cr content, reporting that Cr content affects the ratio of <100> to ½ <111> loops in the Fe(Cr) alloys [26,27]. Very recently, it was reported that in irradiated Fe(Cr) at room temperature the Cr may promote the formation of <100> loops, regardless of its impact on the mobility of the ½ <111> loops [28]. With respect to the mechanism of the <100> loop formation, the authors in Ref. [24] indicate that the primary dislocation loops in Fe(Cr) are the glissile ½<111>. And further more they infer that at room temperature the <100> loops result mainly from the interaction between two ½ <111> loops, according to the reaction $\frac{1}{2}[1\bar{1}1] + \frac{1}{2}[11\bar{1}] \rightarrow [100]$, which is the so-called ‘111’ mechanism of Masters [4]. The ‘111’ mechanism is at variance with the experimental findings showing that when two ½<111> loops meet, the larger loop absorbs the smaller one [29]. The reaction between two 1/2<111> loops (both containing 37 interstitials) in Fe(Cr) at 10 at.% Cr studied by self-evolving atomistic kinetic Monte Carlo (SEAKMC) method shows the formation of either 1/2<111> or <100> loop, with an energy barrier of 0.85 eV [30]. It should be emphasized that according to simulations the ‘111’ mechanism occurs only when the two 1/2<111> loops are of the same size [30]. Note also that the ‘111’ mechanism was never observed directly by experiments. In Ref. [24] it is suggested that the presence of Cr favors the formation of <100>loops, indicating that they may stem from the transformation of undefined 3D clusters, such as the C15 cluster recently revealed in pure Fe by simulation [31]. However, no specific atomistic transformation mechanism is given. The formation mechanisms of interstitial loops in Fe(Cr) alloys, in particular the <100> loops are therefore still unclear. There is thus still a need for well controlled experiments and thorough investigations on the impact of temperature and Cr content on the ratio of <100> loops to ½<111> loops produced by well controlled irradiation in Fe(Cr). Bulk irradiation is desired so as to avoid the impact of the free surfaces on the induced damage. We have thus undertaken such an experimental study to investigate the dependence of dislocation loop properties on both temperature and Cr content in irradiated bulk specimens of ultra-high purity (UHP) Fe(Cr) alloys. Mechanisms of formation and transformation of various loops in Fe(Cr) alloys are discussed on the basis of experimental and modelling results.

2. Experimental

2.1. Materials

Ultra-high purity (UHP) iron and Fe(Cr) alloys having a nominal content of 5 wt.% Cr, 10 wt.% Cr and 14 wt.% Cr were designed and produced at the Ecole des Mines de Saint-Etienne, France, for the modeling validation oriented experiments under European Fusion Development Agreement [32]. The chemical compositions of the investigated materials are listed in Table 2. It is emphasized that the concentration of C and P is <4 and 10 wt.-ppm, respectively, and other impurities are in even lower amounts.

The material was received in the form of 12-mm diameter cylindrical rods. It was cut into 300 μm slices by spark erosion. The