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In-situ TEM Studies of Microstructure Evolution under Ion Irradiation for Nuclear Engineering Applications

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One of the difficulties of studying processes occurring under irradiation (in a reactor environment) is the lack of kinetics information since usually samples are examined ex situ (i.e. after irradiation) so that only snapshots of the process are available. Given the dynamic nature of the phenomena, direct in situ observation is invaluable for better understanding the mechanisms, kinetics and driving forces of the processes involved. This can be done using in situ ion irradiation in a TEM at the IVEM facility at Argonne National Laboratory which, in the USA, is a unique facility. To predict the in reactor behavior of alloys, it is essential to understand the basic mechanisms of radiation damage formation (loop density, defect interactions) and accumulation (loop evolution, precipitation or dissolution of second phases…). In-situ Ion-irradiation in a TEM has proven a very good tool for that purpose as it allows for the direct determination of the formation and evolution of irradiation-induced damage and the spatial correlation of the defect structures with the pre-existing microstructure (including lath boundaries, network dislocations and carbides) as a function of dose, dose rate, temperature and ion type. Using this technique, different aspects of microstructure evolution under irradiation were studied, such as defect cluster formation and evolution as a function of dose in advanced Ferritic/Martensitic (F/M) steels, the irradiation stability of precipitates in Oxide Dispersion Strengthened (ODS) steels, and irradiation-induced grain-growth. Such studies will be reported in this presentation.

Microstructure Evolution in F/M Steels: Model F/M steels (Fe12Cr0.1C and Fe-9Cr-0.1C) were irradiated with 1 MeV Kr ions at 25°C, 200°C, 300°C, and 400°C to doses up to 10 dpa in-situ in a TEM. The microstructure evolution under irradiation was followed and characterized at successive doses in terms of defect formation and evolution, black dot density, and stability of as-fabricated microstructure using weak-beam dark-field imaging and g.b analysis for comparison with computations made using a spatially dependent rate theory model of cluster evolution in both compositional and geometric spaces under conditions of high energy ion irradiation. More specifically, in the model the concentrations of interstitial loops and voids are calculated as a function of time, number of interstitials/vacancies, spatial position, dislocation densities, temperature, dose and dose rate, impurities and so on. The modelling approaches solve spatially-dependent, coupled reaction-diffusion equations that fully incorporate interstitial and vacancy cluster sizes into the tens of thousands, with complete flexibility in terms of the number of mobile species and the transformations amongst cluster sizes. This modelling approach provides the ability to rigorously simulate the effect of surface sinks in thin-film in-situ irradiation studies, in addition to bulk irradiation conditions in which the extended defect microstructure provides a spatially-dependent sink microstructure. As an example of this modelling capability, the bottom right pane of Figure 1 presents the modelling predictions of visible interstitial cluster type density predicted for in-situ ion irradiation studies in the TEM; the left pane shows the experimental cluster density evolution obtained from in-situ experiments in relative agreement.

Precipitate Stability in ODS Steels under Irradiation was investigated in three ODS F/M steels (18CrODS, MA957 and 9CrODS). Initial synchrotron XRD characterization showed that, depending on the alloy, the initial precipitate population includes oxide particles, intermetallics and/or carbides which form during the alloy processing. TEM revealed two populations of nano-particles size-wise: nano-clusters smaller than 10 nm, and larger nano-particles up to few hundred nms. The alloys were ion-irradiated at 25°C and 500°C to doses up to 100 dpa. The stability of the initial microstructure (grain morphology, dislocation networks) and the bigger particles could be followed in-situ. The becoming of the smaller particles (2-5 nm) was determined ex-situ using EFTEM elemental mapping prior and after irradiation [2].

Grain Growth under Irradiation: In-situ grain growth experiments in nanocrystalline metallic foils under ion irradiation (done in a wide range of doses, temperature conditions for different pure metals) showed the existence of a temperature-independent regime (below about 0.15 to 0.22 T_m), and a thermally assisted regime [3]. A model was proposed to describe the kinetics in the temperature-independent regime, based on the direct impact of the thermal spikes on grain-boundaries. In the model, grain-boundary migration occurs by atomic jumps, within the thermal spikes, biased by the local grain-boundary curvature driving force. The jumps in the spike are calculated based on Vineyard's analysis of thermal spikes and activated processes. The model incorporates cascade structure features such as subcascade formation, and the probability of subcascades occurring at grain-boundaries. This results in a power law expression relating the average grain-size with the ion dose with an exponent of 3, in agreement with experimental observations.

References
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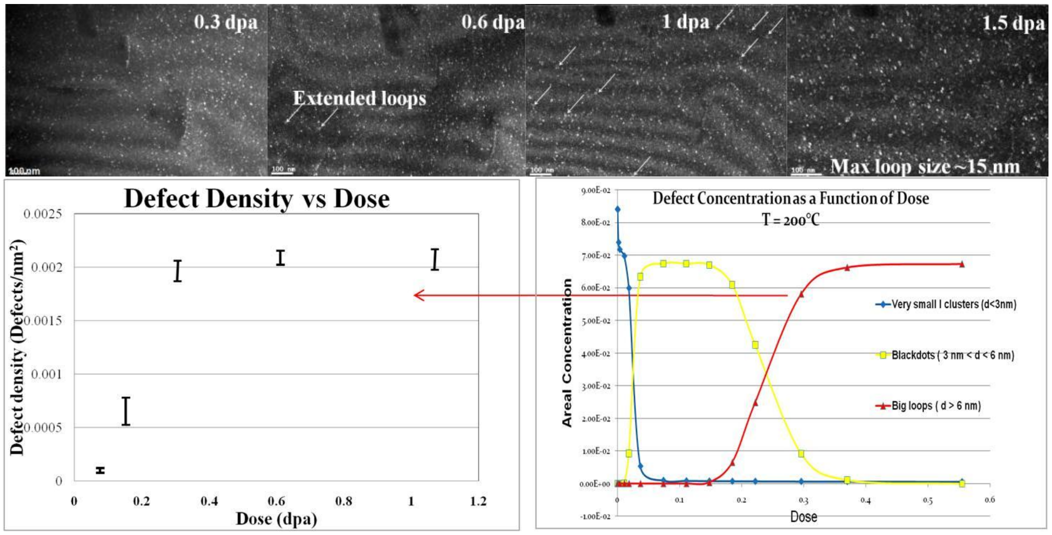

FIG. 1: In-situ TEM observations of irradiation of a model F/M steel at 200C: showing the same irradiated area at successive fluences. Below, measured and calculated defect density (visible defect density in red) [1]