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An analytical method to extract irradiation hardening from nanoindentation hardness-depth curves

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Key words: Fe alloys, ion-irradiation, nano-indentation, irradiation hardening

1. Introduction

The Generation IV nuclear reactor initiative provides the potential for new nuclear energy systems that will significantly improve safety and reliability, sustainability, reactor life, proliferation resistance and profitability [1]. In these designs, higher operating temperatures ($>900^{\circ}\text{C}$) and much higher neutron doses (30-100 dpa) than in current commercial reactors are expected [2][3]. Ferritic/martensitic and ODS steels are candidate materials for the structural components of these reactors due to their high yield strength, reduced activation and higher swelling resistance [4]. Metals exposed to irradiation are known to harden due to the generation of Frenkel pair defect clusters that act as obstacles to dislocation motion under an applied stress [5]. This hardening increases the yield strength, σ_y , of the material but reduces the ductility and causes embrittlement [6][7][8]. Therefore characterising the mechanical properties and quantifying the changes observed in the mechanical properties post irradiation is essential for the safe design of these reactors.

Traditional methods of mechanical testing, using millimetre- or centimetre- size specimens can be costly and time consuming when considering radioactive materials due to the stringent regulations associated with handling active materials and thus the need for specialist facilities. Ion-irradiation has successfully been used as a surrogate to emulate neutron irradiation damage, providing highly damaged samples that are non-radioactive in much shorter timescales (i.e. a number of days rather than years) [9]. The resulting ion-irradiated microstructure contains a high density of irradiation-induced defects, primarily dislocation loops, comparable to the damage observed in neutron-irradiated material [10][11]. A major drawback associated with using ion-irradiation is that the volume of damaged material obtained is restricted to a shallow surface layer (less than a few microns) and the extent of damage or the dose, measured in displacements per atom (dpa), varies strongly through the thickness of this layer. The use of high-energy ion beams, or proton irradiation, can increase the depth of this layer to some extent, up to a few tens of microns; but measurement of the mechanical properties targeting material irradiated to a single specific dose is complicated because a wide range of doses will always be sampled. Small-scale mechanical test methods still sample the properties of a range of non-uniform dose [12].

Nanoindentation is an established small-scale mechanical test that has been extensively used to characterise the hardening of materials exposed to irradiation [13][14]. The hardness, H , measured from the test can be used to approximate the yield stress, σ_y , of the material through the relation $H = 3\sigma_y$ [15]. Nanoindentation of the ion-irradiated surface allows the relative increase in hardness due to irradiation to be measured. Typically, the hardness of the irradiated material is compared to the hardness of the virgin material, using the same experimental conditions and equipment [9]. However, for a single indentation depth the plastic deformation beneath the indentation, which typically fluctuates with varying amounts of plastic strain, extends to a depth ~ 2 -5 times that of the indentation itself; that volume of material (the indentation ‘plastic zone’) spans a range of irradiation doses through the damage profile. This is known as the ‘Damage Gradient Effect’ [16][17][18][19]. As a result the single measured hardness value is characteristic of a dose range and not a particular dose of interest [20]. Indentation into a cross-section of the irradiated sample can reduce this problem as the depth, and therefore dose, of interest from the irradiated surface can be selected as the site for indentation [21], though for typical nanoindentation sizes (50 – 2000nm) and ion-irradiation depths (0.5 – 5 μm), the plastic zone will still sample a range of doses. This cross-sectional technique is more useful when depth of damage from the irradiation is large with respect to the indentation size; e.g. proton irradiation, where damage profiles can extend tens of micrometres into the material. Heavy-ion-irradiated materials usually have much shallower depths of damage; in addition to rapidly varying dose

with depth, it is technically difficult to prepare a flat cross-section close to the edge of the sample and indents made close to the free surface of the edge may yield invalid data.

The well-known indentation size effect (ISE), where the measured indentation hardness increases with decreasing indentation depth, has significant implications when using nanoindentation to measure the radiation induced hardening of materials. The effect is particularly strong in the size range of nanoindentations used to test irradiated layers, with measured hardness values increasing two or three fold relative to the ‘bulk’ value as the indentation scale falls below $\sim 1\mu\text{m}$ [22]. Indentation perpendicular to the irradiated layer, using a shallow indentation depth, will give a hardness increase that is a combination of the indentation size effect and the irradiation induced hardening over the varying range of doses sampled. Separating these effects is not trivial. When using nanoindentation in the cross-section of irradiated materials, a constant indentation depth can be maintained across the damage profile; however it is necessary to use a shallow indentation depth to minimise the dose range being sampled, hence hardness values obtained will be strongly influenced by the ISE.

The Nix and Gao model [22] is commonly used to quantify the indentation size effect in both irradiated and unirradiated materials [12][16][21][23] and is based on the density of geometrically necessary dislocations (GNDs) contained within the hemispherical plastic zone beneath the indentation. To apply the model, given by Equation 1, the hardness vs. indentation depth must be measured experimentally to obtain the size effect parameters H_0 (hardness at an infinite depth) and h^* (a characteristic length scale depending on the material and the indenter geometry). The model is applicable for indentation depths larger than 100nm; below this depth the shape of a real indenter is not self-similar due to tip blunting and the model breaks down. The parameters H_0 and h^* are not independent and are specific to the material being tested [22].

$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h}}$$

Equation 1

The region of plastically deformed material beneath the indentation extends significantly deeper into the material than the displacement of the indenter [8]. It is assumed for the Nix and Gao model, and elsewhere, that the plastic zone is a hemisphere with a radius of 5-10 times the indentation depth [24]. In an ion-irradiated material with a shallow depth of damage, it is thus probable that for indentation depths larger than one fifth of the thickness of the irradiated layer, part of the plastic zone will be in the underlying, unirradiated material. As the indentation depth increases, and the portion of unirradiated material within the plastic zone increases, the hardness of the material will decrease until the measured hardness approaches the hardness of the unirradiated material. This is often referred to as the ‘substrate effect’. To describe this effect, it was proposed [23] that the irradiated layer could be modelled as a hard film on a softer substrate. The measured hardness is modelled as an average of that of the film and that of the substrate, weighted according to the volume of the plastic zone assumed to be in each. This is usually based on the plastic zone being hemispherical or of some other simple geometry [16][25][26].

A problem with this type of approach is that it is assumed that the hardness of the irradiated layer is uniform throughout the thickness, which due to the varying dose profile is not the case. For use in irradiated materials, a rule-of-mixtures type volume fraction approach has been used to extract the hardness of the irradiated layer [20][27]. A study by Saleh et al. [28] used a volume fraction model that separated the estimated plastic zone into segments. The hardness of each segment was assumed to be given by the equation $H = H_0 + C\sqrt{D}$, where D is the dose in dpa and C is a constant, analogous to form used to estimate the increase in yield stress. However, this simple analytical approach was not able to accurately model the irradiation hardening of the He^{2+} implanted austenitic SS316 steel used in the study. A more complex mechanistic model has been proposed by Xiao [17] to describe the

indentation hardness of irradiated materials using the predicted defect density due to irradiation damage, within an estimated plastic zone volume. The model produces a number of parameters that describe the irradiation hardening obtained through data fitting from indentations in the irradiated region. Kim [27] developed a model that uses a rule-of-mixtures type approach and an estimation of the plastic zone size in order to separate the measured hardness into hardness of the irradiated layer and hardness of the substrate.

While some of the irradiation-plastic zone models have some success, they often fail to predict the measured indentation hardness. The Kim model [27], for example, assumes that one needs to know the anticipated yield strength of the material to adjust the plastic zone size and therefore is not suitable for materials where this is not known. Therefore, a simple analytic approach may be of interest to describe radiation induced hardening as a function of dose. In this paper, hardness vs. displacement curves, from indentations made perpendicular to an irradiated surface, are decomposed to isolate the indentation size effect, the damage gradient effect and the substrate effect. The aim of the study reported here is to calibrate a function that describes irradiation hardening as a function of dose using a simple, analytic approach that can be applied through post processing of indentation data. A volume fraction model similar to that used in [28] is used along with a Nix and Gao analysis to describe the indentation size effect.

2. The volume fraction model

It is assumed that the plastically deformed material is contained within a hemisphere beneath the surface as suggested in [20],[24] and [29] and that the hardness is a volume-weighted average of the hardness of plastically deformed material in the irradiated layer and the unirradiated underlying substrate. As the hardness varies with dose through the thickness of the irradiated layer, the irradiated layer is decomposed further into segments, each with its dose-dependent hardness value, to calculate their individual contributions to the hardness.

The model assesses the contributions to measured hardness of radiation damage hardening, ISE and substrate hardness for a series of discrete indentation depths. For each indentation depth, the plastic zone is divided into segments of equal thickness, h , as shown in Figure 1; $r_1^2 = r^2 - a_i^2$ and $r_2^2 = r^2 - (a_i + h)^2$ where r is the plastic zone radius at the current indentation depth and a_i is the depth of the top of the segment from the surface. The volume of each segment is calculated for all segments within the plastic zone, for all indentation depths. In order to define h , the plastic zone radius at maximum indentation depth is divided by the number of discrete indentation depths included in the model, such that for the first indentation depth, the plastic zone contains one segment, the second data point contains two segments, and so on until the maximum indentation depth is reached.

Assuming that the plastic zone radius increases linearly with increasing indentation depth, the plastic zone radius, r for any indentation depth can be obtained from Equation 2 where R is a constant and h_c is the indentation contact depth.

$$r = Rh_c$$

Equation 2

An estimate for R can be obtained from experimental data by correlating the depth of maximum irradiation damage to the depth of the maximum measured hardening. The damage peak depth is predicted from Monte Carlo simulations using SRIM [30] software and the peak hardening is obtained experimentally from the difference between the irradiated indentation hardness and the unirradiated hardness, i.e. ΔH (See Figure 2). For example, if the Bragg peak from ion-irradiation is predicted from SRIM to be at a depth of 6100 nm from the surface, and the peak in the hardening curve occurs at 1000 nm, R is $6100/1000 = 6.1$. It is assumed that at a depth of 1000nm, the plastic zone is at the limit of the

irradiated layer, beyond which the plastic zone begins to sample unirradiated material and as a result, ΔH begins to decrease. (See Figure 2(a)).

The volume fraction model is given in Equation 3. H_c is the indentation hardness at a particular indentation depth, H_i is the hardness value for segment i and v_i is the volume of segment i .

$$H_c = \frac{\sum H_i v_i}{\sum v_i}$$

Equation 3

In the unirradiated case, the hardness value, H_i for each segment, i , consists of the hardness due to the indentation size effect, ΔH_{ISE} , and the material hardness at an infinite depth, H_0 . In the irradiated material, there is an additional hardening component due to the irradiation, $\Delta H_{irr(i)}$, therefore the hardness of each segment can be expressed as:

$$H_i = \Delta H_{ISE} + \Delta H_{irr(i)} + H_0.$$

Equation 4

A Nix and Gao approach can be applied to the unirradiated hardness data to obtain the Nix and Gao parameters H_0 , (the hardness at an infinite depth) and h^* (a characteristic length dependent on the material) [22]. ΔH_{ISE} is assumed to be constant for all segments at a given indentation depth, but changes as a function of indentation depth. Using these parameters, it is possible to use Equation 5 to predict $\Delta H_{ISE} + H_0$ as a function of indentation depth, h_c .

$$\Delta H_{ISE} + H_0 = H_0 \sqrt{1 + \frac{h^*}{h_c}}$$

Equation 5

Assuming that ΔH_{ISE} and H_0 are specific to the material these can be used in Equation 4 to model the irradiated material. It is necessary to assume a relationship between damage dose and radiation hardening; here, a simple power law relationship was assumed [32]. Each segment is assigned a dose from the SRIM damage profile and $\Delta H_{irr(i)}$ is estimated to be:

$$\Delta H_{irr(i)} = m(D_{(i)})^n.$$

Equation 6

Where D is the dose in dpa and m and n are constants. These $\Delta H_{irr(i)}$ values are used as inputs into Equation 4 and are subsequently used in Equation 3 to model the irradiated hardness. The constants m and n are adjusted until agreement is reached between the experimental and modelled hardness vs. depth curves. For simplicity, this approach assumes that the size effect in both the irradiated and unirradiated material is of the same form. In reality, this may not be the case as the dislocation density will be higher in the irradiated material and therefore produce a lower size effect according to the mechanistic model proposed by Nix and Gao [22].

3. Experimental

3.1 Irradiation experiments

Three samples were used in this study. Two samples were irradiated at the Centre for Accelerator Mass Spectrometry (CAMS) accelerator facility at Lawrence Livermore National Laboratory (LLNL) using a

10MV tandem accelerator. A sample of T91 ferritic/martensitic steel and a sample of austenitic 800H steel were irradiated with 70MeV Fe^{2+} to a fluence of 4.19×10^{20} ions/cm² at 452°C (averaged normalised infrared camera temperature during the irradiation). Steel compositions are given in Table 1. The irradiation temperature was monitored using an IR camera and a thermocouple behind the samples. The damage profile based on SRIM calculations, using the “Ion Distribution and Quick Calculation of Damage” method [30], is given in Figure 2 (a). Part of each sample was masked from the ion beam, allowing both an irradiated and unirradiated region of the sample to be examined post irradiation; the implanted regions were visibly identifiable due to discoloration on the sample surface. The third sample was a high purity Fe-10%Cr alloy irradiated at the Surrey Ion Beam Centre in the UK, with Fe^+ ions with multiple energies to produce the damage profile given in Figure 2 (b). For full details of this irradiation experiment see [31].

3.2 Nanoindentation

Nanoindentation experiments on the T91 sample and the Fe-10%Cr sample were carried out using an Agilent G200 nanoindenter fitted with a Berkovich tip and the continuous stiffness measurement (CSM) technique. The CSM technique, in which a cyclic load is superimposed in the monotonic loading sequence, allows a direct measurement of contact stiffness during the loading segment of the indentation test. This can be used to provide hardness and modulus as a function of indenter displacement during one indentation cycle [33].

Arrays consisting of 25 indentations were made in both the irradiated and the unirradiated regions of the sample; all indentations were conducted in displacement-control mode to a maximum displacement of 2µm with a displacement rate of 10 nm/sec. Indents were positioned 50µm apart to ensure that the plastic zones beneath the surface did not interact with each other.

A Micro Materials (MML) instrument, also fitted with a Berkovich tip, was used at the University of Berkeley in the quasi-static mode to measure the 800H sample. Load-displacement data from each indentation cycle was analysed using the Oliver Pharr Method [34]. Indentations were performed prior to ion-irradiation and after irradiation, using in each case a series of separate tests to a range of depths (10 indents at each depth in each irradiation condition) covering the depth range from 100nm to 1000nm; the hardness/depth curve for each irradiation condition was then assembled from the data from these tests. A separation distance of 5-10µm was used between the indents ensuring no plastic zone interactions.

Both instruments were calibrated for tip geometry and frame stiffness using a fused silica reference material.

4. Results

Experimental indentation modulus as a function of indenter displacement is shown in Figures 3(a), 3(b) and 3(c) for the irradiated and unirradiated regions of the T91, 800H and Fe-10%Cr materials respectively. As expected, the modulus is unaffected by irradiation damage. It has been shown that the elastic modulus of a material can be greatly affected by cavity defects, however, as long as the defects are small dislocation loops, the measured indentation modulus has been found to be the same for both irradiated and unirradiated material within the error bars of the experiment [35]. The T91 material shows a large amount of scatter in the modulus measurements. This due to the fine microstructure of the material; each indentation site is likely to be sampling different microstructural features such as martensitic laths or prior austenite grains. The amount of scatter is significantly reduced for the 800H and Fe-10%Cr samples; these materials had very large grains (the average grain size of 800H is 204µm) and in the Fe-10%Cr sample, all indentations were performed in a single grain in the same orientation.

Hardness as a function of indenter displacement is given in Figures 3(d), 3(e) and 3(f); an increase in hardness due to the ISE is observed for all samples. In all samples, the first 100nm of displacement shows a considerable increase in both the absolute values of hardness and an increase in the scatter in the data. The T91 unirradiated material has a higher hardness than the irradiated material at shallow displacements. Irradiation softening is not expected in this material and it is likely that the observed 'softening' is due to a layer of ion-beam contamination on the surface. Consequently, for all samples the first 100nm of data is likely to be unrepresentative of the irradiation hardening and will be ignored for the remainder of the analysis.

Experimental data are re-plotted as H^2 vs. $1/h$ in order to obtain the Nix and Gao parameters H_0 and h^* . This is shown in Figure 4. For the unirradiated material, a linear relationship is observed for data with depths greater than 100nm. Nix and Gao parameters are listed in Table 2. The irradiated T91 appears to show a bilinear relationship with a shoulder between $800\text{nm} < h < 1100\text{nm}$. The transition in this bilinear behaviour has previously been associated with the plastic zone extending into the unirradiated region of material, beneath the irradiated layer [16]. From our selection of R it is assumed that in the T91 the plastic zone reaches the limit of the irradiated layer at an indentation depth of 1000nm (see Figure 2a). This is consistent with the depth at which the bilinear transition occurs. In the 800H sample, a bilinear transition would be expected at an indentation depth of 763nm. This is not as clear as in the T91 sample due to the limited data points. In the Fe-10%Cr data a slight transition can be observed at 172nm. However, due to the irradiated layer being extremely shallow, the plastic zone extends into the unirradiated material at a very low indentation displacement.

The volume fraction analysis uses 95 indentation depths in increments of 20nm (i.e. to a maximum indentation depth of 1900nm). For each sample, R is estimated from the ratio between the peak in the damage profile and the peak in the ΔH curves (see Figure 2); the values of R used for each material are given in Table 2. The Nix and Gao parameters obtained from the linear fit to the unirradiated data plotted in Figure 4 were used to obtain $\Delta H_{ISE} + H_0$ for the 95 indentation depths. H_0 , ΔH_{ISE} and $\Delta H_{irr(i)}$ used in the volume fraction model are given in Figure 5; each segment i is assigned a dpa level using the SRIM profile. The constants m and n used in the power law relationship to describe ΔH_{irr} (see Equation 6) are listed in Table 2. The hardness obtained from the volume fraction model is given in Figure 3a), b) and c) for the three samples.

5. Discussion

This study focuses on the problems that can arise when using analysing nanoindentation data to study the hardness of shallow ion-irradiated layers with varying dose profiles through the thickness of the material. The volume fraction model proposed in this study allows the hardness to be separated into components of hardening specific to the dose at that depth of material. It is assumed that the measured indentation hardness is a weighted average of the individual volumes of individual segments within the total plastic zone.

This study uses the Nix and Gao parameters from the unirradiated hardness, to predict the hardening component due to the size effect of the irradiated material. The size effect parameter, h^* , is smaller in the irradiated T91 and 800H material compared to the unirradiated material indicating a weaker size effect. This is comparable to the work carried out in [22] whereby a weaker size effect is observed for materials with higher defect density. In the Fe-10%Cr sample, the h^* parameter is significantly larger in the irradiated material compared to the unirradiated material. The Nix and Gao analysis carried out in [20] also shows this result (i.e. a stronger size effect was observed in the irradiated material when compared to the unirradiated); it is suggested this is due to difficulties in deconvoluting the influence of the underlying unirradiated material. The thickness of the irradiated layer in [20] was limited to just over 1000nm, which is approximately the thickness of the damaged layer in the Fe-10%Cr sample used

in this study. In the Nix and Gao analysis carried out in this study on the Fe-10%Cr sample, a linear fit is applied to the entire data set, the majority of which have plastic zones extending into the unirradiated region. Therefore for this material, the obtained Nix and Gao parameters are not representative of the purely irradiated material; they represent a composite consisting of irradiated and unirradiated material, which appears to indicate a stronger size effect than in the unirradiated bulk material. The values of H_0 , however, are in good agreement. The high-energy ion-irradiation used for the T91 and the 800H sample allows a much larger volume of material to be sampled, thus the underlying material begins to influence the hardness measurement at a much deeper indentation depth. The Nix and Gao analysis for these samples has been applied to the data points whereby the plastic zone is contained within the irradiated layer, where we observe a reduction in the size effect parameter h^* , as might be expected for a material with a higher defect density. The size effect in the irradiated material takes a different form to that of the unirradiated material due to the differing density of defects present in the material. This indicates that using the size effect parameters for the unirradiated material to describe the size effect in the irradiated material, as in this model, will overestimate the contribution of hardening due to indentation size. However using the size effect parameters of the irradiated material in the analysis would not be suitable, as within the irradiated region the dose, and therefore defect density, is not uniform. This would suggest that the size effect parameters would change throughout the thickness of the damage profile, with it being the weakest in the Bragg peak where the defect density is expected to be largest. This complicates the problem even further; thus the output $\Delta H_{irr(i)}$ from this study contains some element of hardening due to the depth of the indentation, which is a function of dose.

It was assumed that $\Delta H_{irr(i)}$ follows a power law relationship $\Delta H_{irr(i)} = a(D_{(i)})^n$. This relationship is analogous to models used to describe the radiation-induced increases in yield strength based on barrier hardening theory. Early models were in the form of a power law expression $\Delta\sigma_y = h(\Phi t)^x$ in which the barrier density, and therefore strengthening, were assumed to scale with the fluence, Φt or dpa, where h and x are regression coefficients [32]. It was found that the regression coefficient x in the power law relationship is equal to 0.5 at low doses (up to approximately 0.05 dpa) but reduces to typically 0.1 at higher doses (above 0.05 dpa) due to saturation in irradiation hardening from cascade overlap [36][37]. The coefficient n used in the power law relationship to describe ΔH_{irr} is equal to 0.3 for the Fe-10%Cr sample and 0.1 for the T91 and the 800H samples. These values are rather lower than those found in the earlier studies [33,34]. Byun and Farrell suggested plotting $\Delta\sigma_y$ vs. dpa in the log-log format to reveal two distinct regimes, the high dose regime where a rapid hardening occurs and the low dose regime where the log-log plot shows a considerably reduced slope. In [38] authors concluded that the transition from “low-dose” to “high-dose” regimes in irradiation hardening is associated with strain localisation and strain softening, such that the tensile specimen undergoes prompt plastic instability. At these dose levels, the radiation defect density is approaching or has reached saturation, so that the rate of increase of hardening with further dose increase is small. Figure 2 shows that these test data reported and analysed here are in this high dose regime (above 0.05 dpa).

The volume fraction model assumes that the plastic zone beneath the material is hemispherical and that the plastic zone scaling factor is consistent throughout both the irradiated and unirradiated regions of the sample for simplicity. However, previous work has shown that the plastic zone radius is smaller for harder materials, and therefore smaller within the irradiated region with respect to the unirradiated region, giving a non-hemispherical plastic zone [27]. Hardie et al. investigated the size and shape of the plastic zone beneath indentations in heavy-ion irradiated model Fe-Cr alloys [39]. It was found that for shallow indentation depths, the plastic zone was restricted within the irradiated layer; as the indenter penetrated deeper into the underlying unirradiated material, the plastic zone very rapidly increased. Directly measuring the plastic zone size in the more complex alloys, such as T91 through use of TEM, Transmission Kikuchi Diffraction (TKD) or High-Resolution Electron Backscatter Diffraction (HR-EBSD), and mapping the irradiation damage through the plastic zone would eliminate the necessity of assuming the volume of the plastic zone is hemispherical. Further, this would aid the understanding of how the variation of dose within the plastic zone influences the measured hardening.

6. Conclusions

The volume fraction model described in this paper is a simple analytical tool that allows extraction of the irradiation hardening as a function of dose from one irradiated sample using nanoindentation perpendicular to the irradiated layer, where measured hardness data combine effects of a depth dependent indentation size effect and effects of the unirradiated substrate. The method is applicable for the analysis of hardness-depth data both from a succession of separate indentations to a series of depths (e.g. from the MML instrument used here for the tests on 800H steel) and from indentations where hardness-depth data can be determined continuously in individual indentations (e.g. from the G200 instrument used here for the tests on T91 steel and the Fe-10%Cr alloy). Some simplifying assumptions are made about the indentation plastic zone size and shape and the variation of hardening with irradiation dose within the plastic zone; further direct investigation of these would allow this analysis to be extended further.

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Tables

Table 1

Wt.%	Cr	Ni	C	N	Mn	Mo	Nb	Si	V	P	Al	Cu	Ti
T91	8.60	0.09	0.37	0.05	0.37	0.89	0.07	0.11	0.21	-	-	-	-
800H	20.42	31.59	0.07	-	0.76	-	-	0.13	-	0.04	0.51	0.42	0.57

Table 2 – P used to predict the plastic zone size in Equation 1, H_0 and h^* obtained from the Nix and Gao analysis in the irradiated and unirradiated regions on the sample and best-fit irradiation hardening constants a and n .

	P	H_0 /GPa	h^* /nm	H_0 /GPa	h^* /nm	a	n
		<i>unirradiated</i>		<i>irradiated</i>		<i>irradiated</i>	
T91	6.1	3.06	288	4.93	112	1.4	0.1
800H	8	1.72	746	3.24	217	1.2	0.1
Fe-10%Cr	5.8	1.19	458	1.13	1445	1.8	0.3

Figure 1 – Schematic showing the plastic zone beneath the indentation. For the volume fraction model the hemisphere is separated into segments with equal thickness, h and the volume of each segment is calculated. Here $r_1^2 = r^2 - a_i^2$ and $r_2^2 = r^2 - (a_i + h)^2$. a starts at zero and increases in increments of h depending on the segment number.

Figure 2 – damage profile as a function of depth into the surface calculated using SRIM [30] for 70MeV Fe^{++} (left) and the multiple energy irradiation of Fe^+ from [31] (right). Blue curves show ΔH for T91 (left) and Fe-10%Cr (right).

Figure 3 – Experimental modulus vs. displacement plot for a) T91 b) 800H and c) Fe-10%Cr, hardness vs. displacement plot for d) T91 e) 800H and f) Fe-10%Cr. The dotted lines in d), e) and f) show the hardness obtained from the volume fraction model. Error bars in a), c), d) and f) represent one standard deviation of the mean, c) and e) show all measurements taken.

Figure 4 – Experimental data from Figures 3a), 3b) and 3c) re-plotted as Hardness² vs. reciprocal displacement. The vertical dashed lines represent the depth at which the plastic zone extends into the underlying unirradiated material

Figure 5 – ΔH_{ISE} vs. indenter displacement, $\Delta H_{irr(i)}$ vs. depth of segment i , and H_0 used to determine H_i from Equation 4 for a) T91 b) 800H and c) Fe-10%Cr

Highlights

- A simple analytical post-processing tool is proposed to extract the irradiation induced hardening as a function of dose from nanoindentation hardness vs. depth curves in heavy-ion irradiated Fe-alloys
- The model is able to separate plasticity size effects, the effect of the underlying unirradiated material and utilise the varying dose profile through the thickness of the irradiated layer.