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Ion irradiation combined with nanoindentation as a screening test procedure for irradiation hardening

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HIGHLIGHTS

- A screening test for irradiation hardening based on ion irradiation and nanoindentation was applied.
- A new elastic-modulus-based correction of the contact area was proposed.
- The procedure was validated by comparing results for ion- and neutron-irradiated T91.

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ABSTRACT

Ion irradiation has long been recognized as a means to efficiently approximate neutron damage in structural materials. Likewise, nanoindentation has long been recognized as a tool to probe the mechanical behaviour of thin layers. The combination of both techniques in order to establish a screening test procedure for the resistance of ferritic/martensitic (f/m) steels to neutron damage in terms of hardening requires consideration of a number of details. The objective is to specify one among several possible variants of such a screening test. Important constituents of the approach include: (1) the design of the ion irradiation experiments, e.g. using the Monte Carlo binary collision code SRIM, (2) nanoindentation testing over a large range of indentation depths, and (3) proper consideration of the indentation size effect, the substrate effect and the pile-up effect. An elastic-modulus-based correction of the contact area was rationalized. A version of the overall approach sketched above was applied to unirradiated, self-ion-irradiated and neutron-irradiated samples of the 9% Cr f/m steel T91. Apparently, this is the first direct comparison of nanoindentation results obtained for samples of the same f/m steel irradiated with ions and neutrons at the same temperature (200 °C) and up to about the same fluence (2.5 dpa versus 2.31 dpa). The findings indicate that the indentation hardness increase is significant and agrees within the range of errors.

1. Introduction

Future nuclear applications like GEN IV fission and fusion reactors present new materials challenges in terms of higher operating temperatures and higher neutron exposures with respect to existing technologies [1–4]. The development and qualification of new structural materials for these applications require a profound understanding of the mechanisms of neutron damage achieved by fundamental research on and screening of a considerable number of materials to identify the most promising candidates.

One aim of the European FP7 Project MATTER (MATerials TEsting and Rules) was to complement the fundamental materials research by providing best-practice guidelines for materials screening tests. Suitable screening tests generally should limit the need for expensive experiments and testing procedures, provide maximum comparability of the experimental data produced by applying equivalent procedures and allow for fast access to experimental results.

Within the framework of MATTER, a number of candidate testing methods for screening tests addressing different phenomena relevant for structural materials in nuclear applications were considered, e.g. slow strain rate test to measure the material susceptibility to environmentally assisted cracking as well as small-punch and indentation tests as screening procedures for environmental and irradiation effects on mechanical properties. The present paper focusses on the combination of ion irradiation

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experiments and nanoindentation testing as a screening test procedure for irradiation hardening in structural materials.

Ion irradiation has long been recognized as a means to efficiently approximate neutron damage in structural materials [5–8]. Likewise, nanoindentation has long been recognized as a tool to probe the mechanical behaviour of thin layers [9–12]. The combination of both has been extensively used to study irradiation hardening in structural materials [8,13–28]. Clearly, there is a need to harmonize several versions of this approach developed in different labs.

The attractiveness of ion irradiation as a substitute for neutron irradiation in the framework of a screening procedure for the resistance to neutron damage in terms of hardening is based on the short irradiation times (hours and days compared to years), the avoidance of radioactive material, the comparably low costs and the relatively easy variability of irradiation parameters (e.g. irradiation temperature, fluence). One serious drawback of ion irradiation is the limited penetration depth of ions, which depends on the type and energy of the ions used. While it is possible to reach penetration depths in the range of a few 10 μm with light ions, the penetration depth of self-ions in Fe-based materials is significantly smaller and in the range of a few micrometers or less. Because of the small penetration depth, the characterisation of irradiation-induced changes of the mechanical properties of ion-irradiated layers requires the application of nano-/micromechanical testing methods, e.g. nanoindentation. Therefore, the acceptance of ions as a neutron surrogate is accompanied by the introduction of a new transferability issue related to the probing of thin layers versus bulk.

Important constituents of an approach based on ion irradiation and nanoindentation as a screening test procedure for the irradiation response of structural materials exposed to heavy neutron irradiation are: (1) the design of the ion irradiation experiment, e.g. using the Monte Carlo binary collision code SRIM, (2) nanoindentation testing over a large range of indentation depths, and (3) pile-up correction as well as consideration of both the indentation size effect and the substrate effect. The constituents will be briefly described in the following.

(1) Design of the ion irradiation experiment

In order to derive a quantitative measure of irradiation hardening, a sufficient layer thickness, an approximately homogeneous layer and a homogeneous lateral exposure of the surface are necessary. Experimental approaches to produce an approximately homogeneous layer include the sequential irradiation with ions of different energies [21,23,27], the use of a degrader [13,15] or the irradiation with varying angles of incidence [29]. A homogeneous lateral exposure can be achieved by scanning the beam over the surface of the sample.

Ion ranges and dpa-profiles are commonly calculated using the Monte Carlo code SRIM [30]. The SRIM code offers different ways (e.g. quick calculation mode) to calculate the dpa-profile which can produce significantly different results in some cases. It is advisable to follow the recommendations of Stoller et al. [31] and to document the full set of irradiation parameters as well as the calculation method.

(2) Nanoindentation testing

The calibration of the stiffness of the machine and the area function of the indenter, for example based on reference materials with known elastic modulus (e.g. fused silica, sapphire), is an important prerequisite of the measurements [32]. The known elastic modulus of the material to be investigated can serve as an additional indication that the calibration is appropriate [33].

According to the present approach, it is necessary to collect hardness data over a sufficiently large range of indentation depth from less than 10% to about 100% of the layer thickness to be able to judge and account for the indentation size effect and the substrate effect. This can be done using hardness measurements at different loads or to different maximum indentation depth [21,24], by extracting the hardness data from the load–displacement curve exploiting the full unloading curve to determine the contact stiffness at each load [16,34] or more conveniently by applying the CSM (Continuous Stiffness Measurement) [35] or QCSM (Quasi Continuous Stiffness Measurement) [36] method. QCSM is applied in the present work as specified in the experimental section.

(3) Analysis of nanoindentation data

The load–depth curves provide information on the elastic and plastic properties of the sample and can be analysed by means of a method developed by Nix and Doerner [9] and improved and adapted to pyramidal indenters by Oliver and Pharr [12,37]. This method became a standard method for the analysis of nanoindentation testing results. One important limitation of this method is that it does not account for pile-up of material around the indent [12] which can lead to an underestimation of the contact area and consequently an overestimation of the indentation hardness. Pile-up is favoured by a large E/σ-ratio and little work hardening [12,38]. The pile-up behaviour can change with irradiation due to changes in both hardness and work hardening rate. An irradiation-induced change in pile-up has been observed by Armstrong et al. in a W-5wt%Ta alloy [39] and later by Hardie [40,41] in Fe–Cr alloys. The actual contact area is needed to account for the pile-up effect. The actual contact area can be estimated by means of AFM [39–41] and SEM [40,41] on the residual indents. Pile-up correction (PUC) can then be carried out using the ratio of the actual contact area, A_{actual}, to the (pile-up unaffected) corner-to-corner area, A_{cc} (PUC factor, $C_{PUC} = A_{actual}/A_{cc}$), as described by Hardie [40]. In this work we propose an alternative PUC method that relies on the measured elastic modulus. In order to distinguish it from the correction via direct measurement of the contact area, it is called elastic-modulus-based correction (EMC) below. This method does not require any additional measurements and is, therefore, attractive for the purpose of screening.

A size dependence of indentation hardness, H_{IT}, the so-called indentation size effect (ISE), is usually observed for low indentation loads and small depths (< 6 μm). A number of possible causes of the ISE is discussed in the literature [42–45]. Even after full elimination of test-related effects and for perfectly homogeneous material, an ISE remains due to the presence of geometrically necessary dislocations [46] required to accommodate the surface profile of the indent. The density of these dislocations is proportional to $1/h_c$ (h_c is the contact depth), in case of pyramidal indenters [42,45], i.e., the dislocation density increases with decreasing size of the indent and poses an increasing resistance to further plastic deformation, i.e. the hardness increases. This hardness increase has to be separated carefully from the irradiation-induced hardness increase.

The most commonly applied model to describe the ISE is the Nix–Gao-model [42], Eq. (1), where h^* is a characteristic length for a given indenter and material and H_0 is the bulk-equivalent ISE-free hardness.

$$\frac{H_{IT}^2}{H_0^2} = \frac{h^*}{h_c} + 1 \tag{1}$$

For a layer-substrate system, the ISE is superimposed by the