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Indentation size effects of ferritic/martensitic steels: A comparative experimental and modelling study

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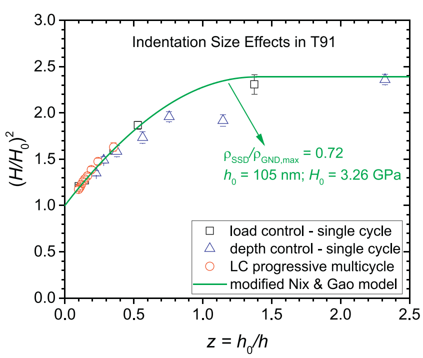
HIGHLIGHTS

- Quasi-static nanoindentation properties are measurement mode independent.
- The parameters of dynamic oscillations influence the indentation size effect (ISE).
- A new modified Nix—Gao model accounts for the break-down of the ISE scaling regime.
- The new ISE model predicts the high dislocation densities of martensitic laths.

GRAPHICAL ABSTRACT



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ABSTRACT

The paper presents a comparative study of different nanoindentation methods as applied to the ferritic/martensitic steels T91 and Eurofer97, here investigated in the non-irradiated reference state, but envisaged as structural materials for nuclear fission and fusion applications, respectively. Depth-controlled single cycle measurements at various indentation depths, force-controlled single cycle, force-controlled progressive multi-cycle measurements, and continuous stiffness measurements (CSM) using a Berkovich tip at room temperature have been combined to determine the indentation hardness and the elastic modulus, and to assess the robustness of the different methods in capturing the indentation size effects (ISE) of those steels. The Nix—Gao model is found inappropriate because it does not account for the breakdown of the scaling regime at small indentation depths that is linked to the extremely high density of dislocations associated with martensitic lath boundary misorientation. A generalization of the Nix–Gao model is therefore developed which allows the prediction of the dislocation densities in the lath structure in accordance with neutron diffraction results. Amplitude and frequency of the CSM oscillations influence the ISE observed. Differences of the microstructure-based parameters describing the ISE of quasi-static and dynamic measurements on T91 and Eurofer97 may reflect differences in the associated deformation mechanisms and histories.

1. Introduction

High strength ferritic/martensitic (F/M) steels are candidate materials for structural components in next generation nuclear fission and fusion reactors owing to their superior corrosion and irradiation resistance, as well as their good mechanical performance in terms of creep

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Table 1
Chemical composition of the T91 and Eurofer97 materials (in wt%; Fe balance).

	Cr	Mo	Mn	Si	V	Ni	Nb	Cu
T91	8.873	0.871	0.386	0.218	0.195	0.115	0.077	0.080
Eurofer97	8.87	<0.001	0.42	0.06	0.19	0.0075	<0.001	0.021

	Al	C	N	P	S	Sn	O
T91	0.009	0.097	0.0440	0.020	0.0005	-	-
Eurofer97	0.008	0.12	0.018	0.004	0.003	<0.005	0.001

	W	Ta	Ti	Co	As	Sb	Zr
T91	-	-	-	-	-	-	-
Eurofer97	1.10	0.14	0.008	0.005	<0.005	<0.005	<0.005

and fatigue life. Understanding their deformation behaviour under high irradiation dose and high temperature is important to predict the component lifetimes at operating conditions. Recent advances in small specimen and micromechanical testing have promoted the assessment of radiation damage, either ion- or neutron-induced, thanks to the capability of testing shallow depths and minimized volumes of activated materials [1–3]. Furthermore, small scale mechanical testing is used for validating mechanism-based computational material engineering, since the relevant length scales can be addressed by modelling and experiments, leading to improved understanding and extrapolation of the long-term performance of materials [4]. Amongst the micromechanical testing techniques explored by the scientific community, nanoindentation is the most widely developed and used so far. Continuous improvements in methodologies to record and analyse load-displacement curves are promoting nanoindentation to complement conventional mechanical testing in materials selection and design [5]. In addition, small indentations are virtually non-destructive for larger components, while, through sectioning, the technique allows the testing of positions with varying properties across the thickness, such as graded ion-irradiated layers or welded joints, the assessment of which is of interest to nuclear safety and beyond.

Nowadays, nanoindentation devices offer the possibility to continuously probe the stiffness of a material at increasing depths, and thereby derive hardness and elastic modulus, by superimposing a small oscillating force on the quasi-static load cycle applied in traditional nanoindentation. However, an oscillation softening effect generally occurs in metals [6], and the derived mechanical properties depend on the magnitude and frequency of the oscillation [7,8]. Therefore, further studies are needed to understand if and to what extent dynamic measurements in continuous stiffness measurement (CSM) mode, influence the outcome of the tests.

While the measured indentation hardness depends on the material properties, like elasticity, yield stress and strain hardening behaviour,

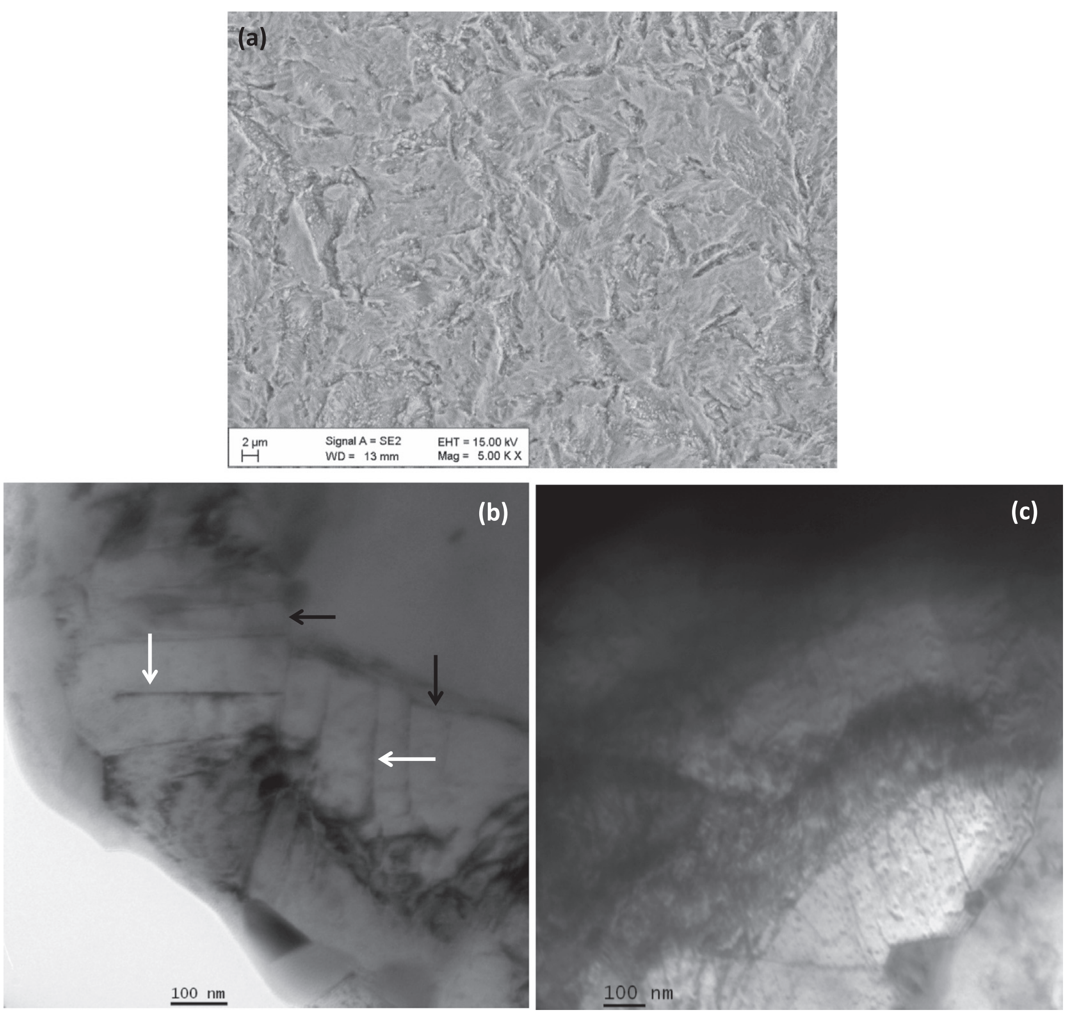

Fig. 1. Microstructure of T91 imaged by SEM (a) and TEM (b, c) evidencing dislocation arrays (white arrows in b) transverse to lath boundaries (black arrows) and tangles of high dislocation density inside martensite laths (c).