Fusion Engineering and Design 126 (2018) 5–9

Contents lists available at ScienceDirect

Fusion Engineering and Design

journal homepage: www.elsevier.com/locate/fusengdes

Full Length Article

Magnetic characterization of ODS-Eurofer steel: Remanent magnetization and magnetostriction behavior

M.J.R. Sandim[a], R.A. Renzetti[a,b], C. Bormio-Nunes[a], H.R.Z. Sandim[a,*]

[a] Lorena School of Engineering, University of Sao Paulo, 12602-810, Lorena, Brazil
[b] Federal University of Itajubá – UNIFEI, 35903-087, Itabira, Brazil

ARTICLE INFO

Keywords:
ODS-Eurofer steel
Magnetic properties
Magnetostriction
Remanent magnetization
Microstructural evolution

ABSTRACT

Oxide dispersion strengthened ODS-Eurofer steel is a promising candidate for structural applications in future nuclear fusion reactors. Samples of 9Cr-ODS-Eurofer steel were cold rolled up to 80% thickness reduction and annealed up to 1350 °C for 1 h. The microstructural changes of the annealed samples were followed by magnetic measurements taken at room temperature. In comparison with the coercive field (H_c), the remanent magnetization (M_r) and longitudinal magnetostriction (λ_{long}) behaviors can be explained only if we assume some interaction between pinning sites and residual stresses in the material. It was found that H_c, M_r, λ_{long}, and hardness display the same trend. At the expected fusion DEMO reactor operating conditions (around 650 °C and maximum magnetic field of 6 T), the magnetostrictive deformation would not surpass 15 ppm (15 μm in 1 m).

1. Introduction

There has been considerable interest in reduced-activation ferritic-martensite (RAFM) steels since these materials are potential candidates for structural applications in future nuclear fusion reactors, in particular the DEMO (DEMOnstration Power Plant) version of this novel technology [1–3]. They possess a unique combination of high mechanical strength, moderate-to-good ductility at room temperature, high irradiation resistance, and low neutron-induced radioactivity [2,4]. RAFM steels developed in the framework of European Union (EU) fusion technology program are termed Eurofer. In this scenario, the microstructural evolution of RAFM-9%Cr oxide dispersion strengthened (ODS) Eurofer steel has been widely investigated under non-irradiation conditions and reported in a series of papers [5–10].

Regarding its microstructural stability, ODS Eurofer steel is intended to operate at temperatures up to about 650 °C [4,11]. This steel has a ferromagnetic character and it would be exposed not only to high thermal and neutron fluxes, but also to high magnetic fields [12,13]. It is worth mentioning that under normal operation conditions ODS-Eurofer steel would be subjected to magnetic fields of about 6 T [13]. This implies that Lorentz forces (electromagnetic loading) exerted on ferromagnetic materials cannot be disregarded. Therefore, a detailed characterization of the magnetic properties of this material gains relevance.

When a magnetic field is applied to a given ferromagnetic material, a reversible net strain parallel to the applied field occurs, the so-called magnetostriction effect [14]. Quantifying the magnetostriction effect and its relationship with the microstructure is a relevant topic for mechanical engineers to calculate clearances and corresponding dimensional changes in vacuum-tight vessels, such as those required for steady operation of fusion reactors. In addition, magnetic measurements are non-destructive in nature and have high sensitivity to detect bulk microstructural changes in ferromagnetic materials [5,15]. For this goal, one of the most investigated magnetic parameters is the coercive field H_c. This parameter mirrors the strength of pinning exerted on domain walls and is strongly dependent on grain size (d) and dislocation density (ρ). For ferritic steels it is well known that H_c is proportional to the square root of ρ ($H_c \propto \rho^{1/2}$) and directly proportional to the inverse of d ($H_c \propto 1/d$) [15,16]. A few works reported about magnetic properties of RAFM Eurofer steels [5,7,12,13,17–19]. Some of these works described the temperature dependence (up to ~ 627 °C) of magnetic parameters such as coercive field (H_c), remanent magnetization (M_r), and saturation magnetization (M_s) in Eurofer steels [13,17,18]. The correlation between microstructure and coercive field of isothermally annealed Eurofer steels was reported in refs. [5,7,19].

The annealing effects on microstructure and H_c (obtained at room temperature) of RAFM-9%Cr ODS Eurofer steel over a wide range of temperatures up to 1350 °C for 1 h were reported in refs. [5,7] and their main findings are presented as follows. Concerning thermal stability, within the ferrite phase field (below about 850 °C) only a small amount of softening is noticed. Annealing at 800 °C for 1 h caused a small drop in hardness of only 7%. No change in grain size was observed when

* Corresponding author. University of Sao Paulo – USP, Brazil.
E-mail address: hsandim@demar.eel.usp.br (H.R.Z. Sandim).

http://dx.doi.org/10.1016/j.fusengdes.2017.11.003
Received 21 November 2016; Received in revised form 22 September 2017; Accepted 1 November 2017

annealing is performed up to this temperature. These overall characteristics were attributed to the presence of nanosized oxide particles dispersed in the metal matrix that prevent static recrystallization due to strong Smith-Zener pinning forces [5,7]. In other words, in spite of the presence of a few recrystallized grains, the small softening observed up to annealing at 800 °C was mostly attributed to static recovery reactions such as dislocation annihilation (dipoles) and subgrain growth. The term "static" means that the material undergoes recovery during annealing under no mechanical stress; i.e.; after cold rolling. Regarding crystallographic texture (anisotropy), the sample annealed at 800 °C retains most of the rolling texture components with a moderate intensity, about 7.8 times random [5].

Above 850 °C, martensitic transformation occurs even upon air cooling. Such transformation weakens texture intensities, being equal to 3.6 and 3.4 times random for the samples annealed at 1100 and 1350 °C, respectively [5]. On the other hand, as a result of martensitic transformation, the material experiences strong internal stress fields and becomes harder due to the increase of both, dislocation density and interface density (grain boundaries and martensite laths) [5,7]. Hardness remains almost unchanged when annealing is performed between 900 °C and 1250 °C but experiences a sharp drop at 1350 °C. A first attempt at explaining this effect was attributed to the occurrence of intense austenite grain growth. According to Renzetti et al. [5] when annealing is performed at such high temperature, nanosized Y_2O_3 particles become less effective to retard grain boundary migration. A recent study has shown experimental evidence of coarsening of Y-rich particles for ODS-Eurofer steel annealed at 1350 °C [20]. In consequence, lower amounts of solute in the matrix diminish both solute drag and Zener pinning forces (in case of second-phase particles) easing boundary migration. Such a feature explains the occurrence of austenite grain growth at such high annealing temperature [20].

Prior described microstructural changes are also mirrored in the changes of the coercive field of ODS-Eurofer steel upon annealing. Values of H_c and Vickers microhardness display similar trends in this material [5,7]. Going further in the analysis concerning the relationship between microstructure and magnetic properties of 9%Cr ODS-RAFM Eurofer steel, in this work we focus on both remanent magnetization and magnetostriction behaviors of such material using the same set of samples investigated by Renzetti et al. [5]. With regard to ferromagnetic materials, among several metallurgical factors, it is known that crystallographic texture and level of residual stresses affect both their remanence and magnetostriction behavior [14,21]. To the best of our knowledge, no previous works reported about the relationship between the metallurgical state, magnetostriction, and remanent magnetization of 9%Cr ODS-RAFM Eurofer steel annealed over such a wide range of temperature (200–1350 °C) and related microstructural changes.

2. Experimental

The nominal composition of the ODS-Eurofer steel slab was 9Cr–1W–0.08Ta–0.2V–0.07C–0.4M–0.3$Y_2O_3$0.3Y_2O_3 (wt.%). This material was kindly supplied by Prof. A. Möslang (KIT, Germany). The steel was supplied in the tempered condition (as-received), i.e. finishing rolling temperature of 980 °C in the austenite phase field followed by tempering at 750 °C for 2 h. Following, it was cold rolled up to 80% thickness reduction in multiple passes. Samples were sealed in quartz-glass in vacuum and annealed for 1 h (holding time) at several temperatures ranging from 200°C up to 1350 °C 1 h followed by air cooling.

The magnetic measurements were performed using a vibrating sample magnetometer (VSM) from EG & G Princeton Applied Research. The hysteresis loops were carried out at room temperature and the maximum applied field was 16 kOe. Samples were cut into approximate dimensions 5 mm × 3 mm × 1.5 mm, with the larger dimension taken parallel to the rolling direction (RD). The magnetic field was applied parallel to the RD. More details of the measurement protocol are given elsewhere [5]. Values of remanent magnetization (M_R) were taken from the same set of hysteresis loops obtained by Renzetti et al. [5], with an accuracy of 2%. Hysteresis loops were not corrected for demagnetization effects. The demagnetizing factor (N_m) depends both on the body shape and material magnetic susceptibility [22,23]. In the present case, samples present both similar dimensions and susceptibility values, which minimize differences among them concerning N_m values. In addition, the values of H_c reported by Renzetti et al. [5] for the same set of samples are included for comparison purposes. Such H_c values were determined with an accuracy of ± 5 Oe.

Magnetostriction measurements were made using a miniature capacitance dilatometer with absolute resolution of 0.1 nm [24] and a Lakeshore electromagnet generated the applied field. Experiments were carried out at temperatures close to 300 K for fields up to 0.95 T. An error of about 2 ppm can be considered in magnetostriction values, attributed to thermal fluctuations of about ± 0.1 K during measurements [24]. For these measurements, samples were cut into approximate dimensions 2.6 mm × 1.4 mm × 1.9 mm, with the larger dimension taken always parallel to the RD. For longitudinal (λ_{long}) and transversal (λ_{trans}) magnetostriction measurements, a magnetic field parallel and perpendicular to the larger dimension of the sample was applied, respectively. Fig. 1 depicts a schematic drawing of the magnetostriction measurement set up, showing the electromagnet coils and the capacitance cell for λ_{long} and λ_{trans} measurement configurations. A basic description of the capacitance cell is as follows. The cell is composed roughly of two silver parallel plates of area A and the ferromagnetic sample is positioned in between the plates. A capacitance bridge applies a fixed voltage to the plates and a capacitance is measured. Capacitance (C) varies as a function of the distance (L) between the plates following the physical law for a parallel plate capacitor, $C = \frac{\varepsilon_0 A}{L}$, in which ε_o is the electric permittivity of the vacuum. Therefore, after positioning the sample between the plates, the initial gap created before applying the magnetic field determines the initial length (L_o) of the sample. In fact, our capacitance cell has an inclined type of the plates, but only geometric corrections to the equation need to be made [24]. Following the magnetic field application, parallel or perpendicular to the initial cell plates of gap L_o, the gap length changes to L due to sample dilatation or contraction and a corresponding change in capacitance is acquired. The resulting deformation $\frac{L-L_0}{L_0} = \frac{\Delta L}{L_0}$ is the magnetostriction, $\left.\frac{\Delta L}{L_0}\right|_{long} = \lambda_{long}$ and $\left.\frac{\Delta L}{L_0}\right|_{trans} = \lambda_{trans}$.

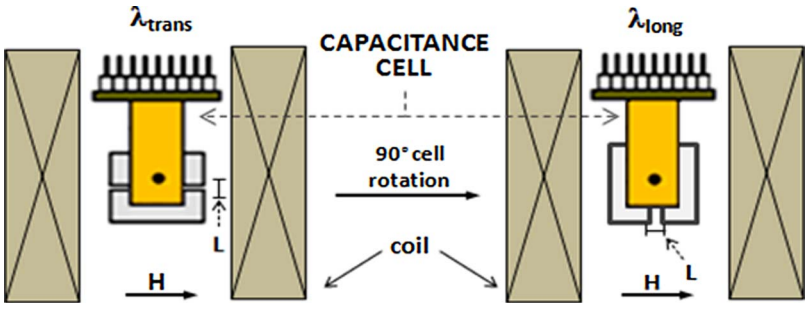

Fig. 1. Graphical representation of the magnetostriction measurement system set up. Adapted from [25].