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Improvement of reduced activation 9%Cr steels by ausforming

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ABSTRACT

For improved performance of the components in a fusion reactor, an increased application temperature for structural materials such as 9%Cr reduced activation steels is crucial. The improvement of the current generation of 9%Cr steels (i.e. EUROFER) is one of the aims of the current EUROfusion programme for advanced steels. The goal of this work is to determine the most effective thermo-mechanical treatment of reduced activation ferritic martensitic steels with respect to high-temperature strength. Compatibility of these treatments with industrial production processes is essential.

In the present study, two different batches of EUROFER-2 were prepared with a thermo-mechanical treatment. The materials were solution annealed at 1250 °C and then slowly cooled to the rolling temperature, which was varied between 600 and 900 °C. Hot-rolling was performed in the austenite regime with a subsequent rapid cooling to form the ferritic-martensitic structure. The characterization of the materials was done in as-rolled state and after a subsequent tempering at 750 °C.

The materials characterization was performed by tensile and Charpy impact tests using miniaturized specimens. The microstructure was characterized by scanning electron microscopy (SEM) backscatter images and electron backscatter diffraction (EBSD) maps. All the results were compared to those of conventionally processed EUROFER-2 alloys.

The first results show a gain in tensile strength of approximately 50 MPa at temperatures above 600 °C compared to conventionally treated EUROFER alloys. Microstructural investigations reveal a fine and homogeneous distribution of the martensitic laths, while the prior austenite grains are about one order of magnitude larger. This can be explained by the exceptionally high austenitization temperature compared to the as-received state.

Introduction

Reduced activation ferritic martensitic (RAFM) 9%Cr steels have been the subject of research within the fusion community for years. For increased performance of the components in a fusion reactor, an increase of the application temperature for structural materials such as 9%Cr reduced activation steels is crucial. The improvement of the current generation of RAFM 9%Cr steels (i.e. EUROFER) is within the scope of the current EUROfusion programme for advanced steels.

These steels are members of a class of Fe-based alloys with varying and carefully balanced amounts of alloying elements such as Cr, W, Ta, and V [1]. They show an improved void swelling even under high irradiation doses [2]. The amount of carbon and nitrogen needs to be adjusted with caution in order to form strengthening secondary phases. The distribution and nature of the carbides and nitrides which form after heat treatments have been well studied [3,4]. However, a possible improvement of the alloys could be achieved by adjusting the chemical composition and distribution of these secondary phases inside the material. In particular, the amount and size of $M_{23}C_6$-type carbides need to be controlled. Coarsening and agglomeration of this phase can have a detrimental effect on the impact properties [5].

The distribution of the secondary phases depends on the number of available heterogeneous nucleation sites within the material during precipitation. A large number of these sites will lead to a finer distribution. The approach of this work follows a scheme proposed by Klueh et al. where dislocations are used as precipitation sites [6]. However, the dislocations need to be created before precipitation occurs. Therefore the materials are mechanically deformed (rolled) and in an undercooled (metastable) austenite regime. A subsequent quenching in air is followed by a tempering.

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Experiments concerning TMT on conventional Grade 91 steel (e.g. P91) have been performed in the recent years [7]. Early studies on low alloy and carbon steels date back to the 1960s where an additional hardening of the martensitic structure was shown after ausforming [8]. The main difference to EUROFER and other RAFM steels are the precipitate-forming elements like Niobium and Molybdenum which are left out of steels for fusion applications. While conventional industrial production of EUROFER consists of rolling at high temperatures in a stable austenite range, the additional dislocations created by the lower rolling temperatures may create a different distribution of the secondary phases inside RAFM EUROFER steels.

The TMT of EUROFER may raise the high temperature operation limit of the steel. The rolling in the austenite regime may refine the microstructure and provide a nano-scale distribution of the secondary phase precipitates [6]. TMT-9Cr steels could form a strengthened microstructure similar to ODS alloys which up to day need to be produced by the costly mechanical alloying process [6,9,10]. Although the strengthening effect will be less compared to ODS particles, the possibility of higher creep strength and fatigue behavior are still worth the effort of TMT [8].

The goal of this work is to determine the most effective thermo-mechanical treatment of RAFM steels with respect to high-temperature strength. Compatibility of these treatments with industrial production processes is essential. High-temperature tensile strength and creep strength are especially important for future applications in a DEMO fusion reactor [11]. All results are compared to those of a conventionally treated EUROFER97/2 plate. The results of this study give direct input regarding the new experimental alloys proposed within EUROfusion.

Materials and methods

Plates of two different EUROFER97-2 batches with a thickness of 25 mm were processed at OCAS, Gent. The chemical compositions of the alloys are given in Table 1. Only minor differences between the two batches exist.

The thermo-mechanical treatment consisted of a solution and austenitization treatment at 1250 °C in air followed by cooling in a second furnace. The temperature of the second furnace was set to the desired rolling temperature (namely 600, 700, 800, and 900 °C). To ensure temperature control during the whole treatment, a dummy plate of the same material was equipped with thermocouples and processed in the same way as the other materials. The temperature was kept constant during the rolling, with reheating if necessary. Prior to any testing and/or microstructural characterization, all materials were annealed at 750 °C for two hours. As-received state refers to EUROFER97/2 materials after treatment at 960 °C for 1.5 h with quenching in oil followed by 4 h at 750 °C with air cooling.

Mechanical tests were performed on miniaturized cylindrical tensile specimens with a gauge length of 7.6 mm × 2 mm taken out in the rolling direction [12]. The tensile tests were performed between RT and 700 °C under vacuum with a strain rate of $1.6 * 10^{-6}$ ms^{-1}. KLST-type specimens with a size of 3 × 4 × 27 mm^3 were used for the Charpy impact tests. The orientation was LS type with a 1-mm notch. All specimens were cut using electro-discharge machining (EDM). A tanh fit was applied to the data to determine the DBTT [13].

Table 1.
Chemical composition of EUROFER97-2, all values given in wt%.

Batch	W	Cr	V	N	Ta	C	Fe
EUROFER97-2 Batch 993402	1.06	8.9	0.18	0.04	0.15	0.1	Balanced
EUROFER97-2 Batch 993391	1.08	8.83	0.2	0.02	0.12	0.1	Balanced

Microstructural characterizations were mainly done using transmission electron microscopy (TEM, Jeol JEM 3000F) on thin foils in order to determine the effect of the treatments to the chemical compositions of the secondary phases and on the martensite microstructure (packet size, lath size, dislocation density). The precipitate microstructure (nature of precipitates, size distribution) was measured mainly on extraction replicas (including energy filtered TEM, EDX). Bright field and STEM mode with a medium-angle dark field detector (LAADF) were used for imaging.

Electron backscatter diffraction (EBSD) maps were measured on a Zeiss Merlin field emission gun scanning electron microscope (SEM) equipped with an EDAX Hikari high-speed camera operating at 20 kV. The measured data were further processed by OIM Analysis Software v7.2. A step size of 300 μm was used. All data points with a confidence index below 0.1 were discarded. Apart from a Confidence Index Standardization, no cleanup algorithms were performed on the maps.

Thermodynamical calculations were performed using JMatPro-v5 with the general steel database. All possible phases were considered in the model. M inside the secondary phases refers to either Tantalum, Vanadium, Chromium and Iron or mixtures of these elements. The correct compositions were determined by TEM replica.

Results

The tensile tests of the materials showed only minor variations between the different materials (Fig. 1). While the material treated at 900 °C shows lower strength at room temperature, in the range of the operation window (550–700 °C) within the error range, no differences can be observed. The thermo-mechanical treatment (TMT) brought a general increase in strength to the materials. The yield strength is approximately 50 MPa higher than the as-received condition throughout the whole tested temperature range.

The alloys performed worse compared to the as-received condition in the Charpy-impact tests. A shift of the ductile-to-brittle transition temperature (DBTT) to temperatures approximately 30 °C higher can be seen (Fig. 2). The as-received condition shows a DBTT of −90 °C. Inhomogeneities and the large prior austenite grain (PAG) sizes in the TMT materials caused a large scatter in the values of the impact energies. Therefore the curves do not show an abrupt drop in energy. Tanh fitting gives values for the DBBT of −37.7 °C (TMT@800 °C, standard deviation = 7.11 °C) and

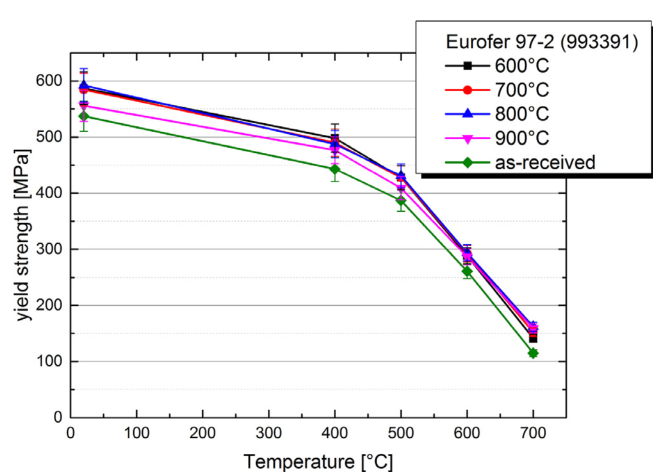

Fig. 1. Results of the tensile testing after TMT and in as-received condition (993391).