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Fracture behaviour of the 14Cr ODS steel exposed to helium and liquid lead

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HIGHLIGHTS

- We compared the impact energy curves of as received, isothermally aged and He/Pb exposed ODS steel samples.
- The highest transition temperature showed the ODS steel exposed to liquid Pb at 650 °C for 1000 h.
- We observed the higher tendency of the He exposed samples to crack arrester delamination than the Pb exposed ones.
- The crack arrested delamination induced apparent increase of impact energies.

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ABSTRACT

This work describes the fracture behaviour of the 14Cr ODS steel produced by mechanical alloying process, after high temperature exposures. Small specimens were exposed to helium gas in a furnace at 720 °C for 500 h. Another set of specimens was exposed to flowing liquid lead in the COLONRI II loop at 650 °C for 1000 h. All specimens were tested for the impact and tensile behaviour. The impact test results are compared to other sets of specimens in the as received state and after isothermal annealing at 650 °C for 1000 h. The impact curves of the exposed materials showed positive shifts on the transition temperature. While the upper shelf value did not change in the Pb exposed ODS steel, it significantly increased in the He exposed one. The differences are discussed in terms of surface and subsurface microscopy observation. The embrittlement can be explained as the effect of a slight change in the grain boundary and size distribution combined with the depletion of sub-surface region from alloying elements forming oxide scale on the surface.

1. Introduction

Oxide dispersion strengthened (ODS) 9–14% Cr steels are considered as promising candidates for blanket and divertor components in fusion reactors and as fuel cladding for next generation nuclear systems (such as lead- and gas cooled fast reactors, LFR resp. GFR) due to expected superior tensile and creep strength at high temperatures as well as enough high temperature oxidation performances, swelling resistance and reduction of radiation embrittlement [1–4]. Nevertheless in systems mentioned above, an operation at temperatures higher than 550 °C and in aggressive environments are envisaged, therefore thermal ageing, high temperature fracture resistance and compatibility related issues need to be studied further [5]. It is worth verifying if the steels keep these properties even in the long-term contact with cooling media.

It is well known that the thermal aging of high Cr (>10%) ferritic steels at 300–550 °C stimulates α (Fe-rich) and α' (Cr-rich) phase separation [6]. It occurs at the nanometric scale and induces hardening and degradation of impact properties. The degree of the embrittlement increases with the Cr content [6,7]. ODS materials were invented to overcome these changes and to stabilize the microstructure for long-term operation, but the development of their optimal composition and fabrication process continues. In fact, a very moderate start of the Cr phase separation process had been indicated for 14Cr-4Al ODS at 440 °C after 320 h [8]. Above

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550 °C, precipitation of σ intermetallic Laves phase further increases the severity of the embrittlement [9]. Likewise, high temperature ageing can influence thermal stability of nanoclusters [10]. In a new study of long-term ageing stability of 16Cr-1.7W-0.4Ti ODS containing large-sized Ti-bearing precipitates (about 400 nm), Cr-rich phase segregation was hardly detected after ageing for 5000 h at 700 °C [11]. Moreover, it was indicated that the Y_2O_3 particles did not change much owing to the ageing. This is showing that because these materials are under development, there are results in disagreement as a result of different manufacturing conditions.

This work focuses on the behaviour of the ferritic 14% Cr ODS steel, ODM401 (14Cr-0.8Ti-0.3Mo), at upper temperature limit of LFR operation, i.e. below 700 °C, and in the lower range of the gas cooling systems, i.e. above 700 °C. The 14Cr ODS steel was selected because both good oxidation and acceptable fracture behaviour are expected [12]. Whereas the LFR and DCLL/GFR temperature ranges do not overlap, two different temperatures and cooling environments condition were selected for this study. The first test temperature is 720 °C in helium gas, based on the reference cooling condition of the US Dual Coolant (He and PbLi) Lead Lithium (DCLL) test blanket module (TBM) concept and close to GFR concept. The second temperature is 650 °C in liquid Pb representing an upper limit for the LFR technology. The oxidation behaviour of the steel in the two environments is a function of temperature and oxygen content: in the gaseous environment, the oxygen was kept to traces level; in liquid lead the amount of oxygen was significantly higher; the maximum amount, for comparison, was in air (beyond the scope of this article). It is noteworthy to investigate more in detail the oxide layers that can be formed on the ODS steel in the two media (He gas, liquid lead), as well as to compare the fracture response after long-term exposure in environments with the oxide preventing the direct contact between steel and environments.

To obtain sufficient information in a wider spectrum of experimental conditions, a compromise was reached respecting both temperature influence and the contact with the environments. Two different exposure periods were chosen: 1000 h exposure is the minimum time to observe relevant oxidation and the temperature effect in liquid Pb. However, according to the Hollomon-Jaffe relationship [13], the pure temperature effect on the bulk behaviour (microstructure, mechanical properties) of650°C/1000 h corresponds to the parameters 720°C/5 h, which is a very short period to study the oxidation in the helium gas. Therefore, the exposure in the helium gas was increased to 500 h for the study, regardless of the fact that 720°C/500 h correspond to the pure temperature effect of 650°C/150000 h.

Although the high-temperature oxidation process for the ODS steel in various coolant media has not been widely described in the literature yet, some papers have been published on the oxidation in steam. The advanced steels including the ferritic 14YWT (14Cr-0.4Ti-3W-0.25Y_2O_3) ODS and APMT (22Cr-5Al-3Mo), showed an outstanding resistance to reaction with the steam (100% superheated steam, 800 °C/50 h) by developing stable and protective oxides; the 14YWT weight change was 0.2 mg/cm^2 [14]. Another 16Cr-4Al ODS steel established the excellent oxidation resistance in supercritical water (510 °C/1800 h), which was attributed to early formation of the protective α- Cr_2O_3 scale and Al_2O_3 oxide at the matrix and scale interface [14,15]. However, the steels with Al content possess very low fracture resistance and thus less perspective. Moreover, it is not known very much about the interaction of the ODS steel with the liquid lead. In the studies [12,16] were obtained first data of the corrosion behaviour of ODS steels (12Cr-Ti-W, 19Cr PM2000 and 22Cr MA956) at 650–700 °C in stagnant/flowing lead with oxygen contents of 10^{-5} - 10^{-7} wt%. For the environment with higher oxygen content (10^{-5} wt%) no signs of oxidation or dissolution appeared up to 2000 h of exposure; however, in the environment with lower oxygen content (10^{-7} wt %), there were observed some zones of dissolution in PM2000 [16].

In addition to concentration of alloying elements and distribution and composition of nano-sized particles, the anisotropy of the material properties plays a key role in the high temperature properties of ODS steels [17]. The typical pronounced morphologic and crystallographic texture of the selected 14Cr ODS steel, hot extruded into a rod, led to a strong anisotropy in the impact properties and the fracture appearance, which were referred elsewhere [18]. It has been shown, that the extrusion shape plays a major role on the anisotropy of the properties. For example, the impact properties of the ODS materials extruded as a rod present a higher upper shelf energy (USE) and a lower ductile to brittle transition temperature (DBTT) compared to the materials extruded as plates [19]. Moreover, in the study [19], focused on the fracture properties of the 9-18Cr–W–Ti–Y_2O_3 ODS steels, extensive delamination of fracture surface was evidenced and the orientation of the fracture plane towards the extrusion direction significantly modified the impact response. According to terminology referred elsewhere [20], two types of delamination were observed: “The crack arrested delamination”, i.e. kinking of the fracture at 90° and continuing the fracture path on a parallel plane, and “the crack divider delamination”, i.e. splitting the fracture surface into parts perpendicular to the fracture and parallel to the crack growth direction. Both types can be found in the ODS steels [9,19,21–25]. Fracture modes are highly linked to the grain boundaries [21,22]. According to a model for the nanostructured steel fracture [25], the tendency for the crack delamination, the out of plane cracking, is likely due to the existence of an easy cleavage system oriented to crack propagation in the extrusion direction [9].

In this work, the 14Cr ODS steel degradation of the impact fracture properties (energy and fracture appearance) owing to the long-term exposure in the liquid lead is compared to the behaviour of the material in the original state, after ageing and exposed to helium. This paper aims to give more insides on the correlation of the environment effect at the surface layer and the behaviour of the bulk material. This was done by using two different temperatures, exposure times and several environments, highlighting cross-cutting issues in parallel with the peculiar characteristics related to the specific environment.

2. Experimental

2.1. Material

The ferritic 14Cr ODS steel (ODM401) was produced by the mechanical alloying process by Dour Metal. The chemical composition of the steel is presented in Table 1. Commercially available atomized powders were mixed in exact proportions processed in the high energy ball mills for 24 h in a low pressure air atmosphere. The degassed powder was cold pressed to a compact pellet. Consequently a rod of diameter 30 mm was hot-extruded at 1150 °C from the pellet. This state is here referred to as the as-received state (AR).

Afterwards several temperatures and environments were applied to the AR material to evaluate the temperature stability and corrosion resistance, see the chart in Fig. 1. The isothermal ageing at

Table 1
Chemical composition of 14Cr ODS (ODM401) [wt. %].

Element	Fe	C	Cr	Ti	Mo	Y_2O_3	Al	O	N
14Cr ODS	Bal.	0.024	13.6	0.85	0.29	0.25	0.06	0.182	0.048