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Comparative study of the behavior of different highly alloyed steels in liquid sodium

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HIGHLIGHTS

- Sensitivity of 8 different steels to liquid sodium embrittlement was studied.
- SPT in sodium at different temperatures, displacement speeds were performed.
- No direct link between the microstructure, the hardness and the LME sensitivity.
- Inhomogeneous stress distribution could promote LME.

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ABSTRACT

By small punch tests performed in air and in liquid sodium at different temperatures and strain rates, the sensitivity of different structural materials to liquid sodium embrittlement was studied: martensitic 9Cr steel, ferrito-martensitic 14Cr steel, ferritic 18Cr steel, austenitic 316LN and 15-15Ti steels, martensitic 9Cr oxide-dispersion strengthened (ODS) steel, ferritic 14CrODS and 18CrODS steels. All materials present ductile mechanical behavior in air. No effect of liquid sodium has been detected for the two 9Cr martensitic steels and the ferritic 18Cr steel. A minor effect of sodium without change in fracture mode or with local brittle fractures has been observed for the 15-15Ti and 316LN austenitic steels, for the ferritic 18CrODS steel and for the ferrito-martensitic 14Cr steel. The ferritic14CrODS steel exhibited a marked liquid metal embrittlement.

1. Introduction

New generation of fission reactors is presently under research in Generation IV plan. Sodium Fast Reactor (SFR) appears to be one of the most credible options to be operated at short time. To achieve the goals fixed by the GEN IV forum in terms of safety, efficiency, competitiveness, life duration (60 years), researches concerning the choice or the development of structural materials are in progress. So, structural stability during ageing and under irradiation, corrosion resistance, mechanical properties at low and high temperatures including resistance to creep and creep fatigue, ability to be fabricated and welded need to be considered for the design of the components. Furthermore, the study of the behavior of structural materials in contact with the liquid sodium is essential to guarantee the integrity and the life-time of the SFR. The compatibility of liquid sodium with structural materials includes the study of liquid metal corrosion, liquid metal embrittlement (LME), and liquid metal accelerating damage (LMAD). While liquid metal corrosion is the degradation of a solid material by the environment alone without mechanical stress, LME and LMAD occur in the presence of mechanical stress. LME is the loss of ductility (total or partial) of a ductile metal or metallic alloy when deformed in a liquid metal (Fernandes et al., 1994; Fernandes and Jones, 1996; Joseph et al., 1999). In this case, fractography points out brittle fracture, partial or total, in the fracture surface. LMAD is the modification of the mechanical resistance of the materials without change in fracture mode. LME is not easily predicted since it occurs for a given set of conditions including material, environment, temperature and stress which, if this set is encountered, restricts the use of the material for the given application. However, pointing out the conditions of LME occurrence in a material has the advantage to increase the reliability of the use of this material and does not mean a total reject of it.

For SFR developed in Generation IV plan, austenitic steels are planned to be used for vessel, exchangers, pipes, pumps …. Ferritic/martensitic steels or oxide dispersion strengthened (ODS) steels are preferred for cladding because of their resistance to high

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temperature creep and their irradiation swelling resistance. Both austenitic stainless steels (such as 304 and 316 types) and martensitic (9 Cr type) are ductile materials under mechanical loading in air, in a temperature range from room temperature to 600 °C. To point out any possible liquid sodium effect on the mechanical response, studies have been performed within the last 30 years by taking into account the chemistry of the liquid metal, the test temperature, the loading conditions and a pre exposure in liquid sodium (Chopra, 1983; Hamdane et al., 2011; Hamdane et al., 2014; Hemery et al., 2013; Kannan et al., 2009; Kannan et al., 2011; Mathew et al., 2013; Sivai Bharasi et al., 2012; Skeldon et al., 1994). A key parameter that avoids degradation of mechanical properties is a low oxygen (in the range of 0–4 ppm) content in sodium (Mishra et al., 1993). It is very classically observed that the strength in liquid sodium is higher than in air as it is the case for T91 steel under low cycle fatigue (Kannan et al., 2009; Mathew et al., 2013), for 316LN under creep (Mathew et al., 2013; Mishra et al., 1993; Ravi et al., 2013) or under creep fatigue (Date et al., 2008; Mathew et al., 2013; Sivai Bharasi et al., 2012). The beneficial effect of pure sodium is attributed to the absence of oxidation of the solid materials and has been taken into account for a long time ago in nuclear plants. Nevertheless, detrimental effects of pure sodium must not be underestimated. Indeed, leaching of alloying elements due to exposure of the steel in the liquid sodium may occur resulting in surface degradation. This effect is particularly noticeable in Cr-Ni austenitic stainless steels where ferrite phase has been identified at the surface of the material (Ravi et al., 2013; Sivai Bharasi et al., 2012). A decrease in ductility with partial brittle fracture has been observed for T91 steel in the standard metallurgical state after pre exposure in liquid sodium for short time at 450 °C (Hemery et al., 2013). This was the result of promoted wetting of the liquid metal with the steel surface, which is a prerequisite for LME. Therefore, the second parameter also connected with the operation condition of nuclear plants is the immersion conditions (time, temperature, circulating fluids) of the steel with liquid sodium. Besides, there are other factors that promote degradation of properties due to liquid sodium. A modification in the mechanical loading can shift the beneficial effect of sodium to a detrimental one. The addition of a tensile-hold time in a continuous cyclic loading has a negative effect on the fatigue lives of T91 (Kannan et al., 2011; Natesan et al., 2009). Finally, liquid sodium degradation is very sensitive to variation in microstructure. 316 FR stainless steel (0.01%C and 0.076%N) is better resistant than 316 stainless steel (0.045%C and 0.04%N) to fatigue in liquid sodium (Date et al., 2008). T91 steel is more sensitive than 316 type austenitic stainless steel. Indeed, this material has to receive a recommended heat treatment comprising a water quench from 1050 °C followed by a tempering between 750 °C and 780 °C. With this heat treatment, the general tendency is that mechanical strength obtained from standard test conditions are not so much deteriorated by liquid sodium in a temperature range between 180 °C to 600 °C (Hamdane et al., 2011; Skeldon et al., 1994). A clear ductile to brittle transition due to liquid sodium is observed when T91 steel is tempered at lower temperatures. In this case, sodium plays a role as well on the crack initiation step by weakening grain boundary strength as on the crack propagation step by promoting transgranular crack (Hamdane et al., 2011). According to the microstructure of the T91 steel, sodium penetrates prior austenitic grains boundaries and induces brittle crack initiation under a plastic deformation as shown by Secondary Ions Mass Spectroscopy (SIMS) analyses (Hamdane et al., 2014).

ODS ferritic or martensitic steels have been developed to resist at high temperature and at high irradiation doses. Fe-9/12Cr martensitic ODS alloys and Fe-14/18Cr ferritic ODS alloys reinforced generally by yttrium oxide (Y_2O_3) were considered (Dubuisson et al., 2012; Steckmeyer et al., 2010; Yoshida and Kato, 2004). A large part of mechanical tests were performed in air, and a minor one in liquid sodium. No significant effect of liquid sodium has been observed.

Therefore, a lot of data concerning the behavior of various steels in liquid sodium is available but it is difficult to make clear comparison between them because the experimental conditions are different from one material to another one. The present paper is a comparative study of the behavior in liquid sodium of a set of eight highly alloyed steels (austenitic, martensitic, ferritic, ferrito-martensitic) while maintaining the same experimental conditions. It aims at pointing out their sensitivity or immunity to liquid sodium embrittlement and at highlighting the eventual role of the microstructure, the hardness, the chemical composition. The Small Punch Test (SPT) technique has been employed to assess the mechanical response in liquid sodium of all the considered steels since earlier investigations have shown that it appeared very sensitive to evidence LME (Serre and Vogt, 2007). Because LME sensitivity depends on temperature and strain rate (Fernandes and Jones, 1996; Hamdane et al., 2011; Joseph et al., 1999; Skeldon et al., 1994), different temperatures and strain rates were considered to evaluate their eventual effect on LME sensitivity. The mechanical response was associated with Scanning Electron Microscopy (SEM) fractographies.

2. Experimental

2.1. Materials

Eight different materials were considered in this study:

- two austenitic stainless steels (316LN and 15-15Ti)
- three chromium steels (noted 9Cr, 14Cr and 18Cr)
- three chromium steels (9Cr, 14Cr and 18Cr) reinforced by yttrium oxide (Y_2O_3) noted 9CrODS, 14CrODS and 18CrODS

The last six chromium steels were elaborated in the French Alternative Energies and Atomic Energy Commission (CEA-France) by powder metallurgy and by hot extrusion at 1100 °C.

The chemical composition of all materials is given in Table 1. The studied 316LN is the steel used for SuperPhenix (SPX) reactor and only the chemical composition given in the table refers to the standard.

The microstructure of the materials has been studied by optical microscopy, SEM, X-ray diffraction. Vickers macro-hardness measurements were performed at room temperature. The data are given in Table 2 and Fig. 1. Note that the 9Cr and 9CrODS steels are martensitic, while the 18Cr, 14CrODS and 18CrODS steels are ferritic. The 14Cr steel presents a duplex ferrito-martensitic microstructure.

2.2. Small Punch Test

A set-up was designed to perform tests in air and in liquid sodium at temperatures up to 550 °C (Hamdane et al., 2011; Serre and Vogt, 2007) (Fig. 2a). It consists of a specimen holder, a pushing rod and a ball. The specimen holder includes a lower die, an upper die which is also used as the tank for the liquid sodium and four clamping screws. The load is transferred onto the specimen by means of a pushing rod and a 2.5 mm diameter tungsten carbide ball in contact with the lower surface of the specimen. In this way, the upper surface of the specimen is in contact with the liquid metal and is submitted to tensile loading. A heat ring surrounded the setup with the specimen and the liquid metal to guaranteed a homogeneous temperature for the different elements: lower die, upper die, specimen and environment. The tem-