Mechanical behaviour and fracture characterization of heat treated T91 martensitic steel in liquid sodium environment

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Abstract

The paper deals with the mechanical behaviour of modified 9Cr1Mo steel in air and in liquid sodium. The tests were carried out on small-sized specimens using the small punch test and the three points bending test. It is shown that the steel exhibits high degree of ductility in most of cases. A ductile to brittle transition induced by the liquid sodium has only been observed when the material was subjected to a hardening heat treatment. In this case, the fracture surface comprised both inter and transgranular fracture. Calculation showed that liquid sodium decreases the J value toughness by a factor of 80%.

Introduction

Due to their excellent properties of ductility, metallic alloys are the most widely employed for structural components in all industrial fields. Combination of ductility properties and of high mechanical strength is often asked while these properties tend to vary in opposite directions. Among the different structural alloys, martensitic steels are very sensitive to heat treatments which permit to adapt their hardness. This is the case of 9Cr1Mo steel family, one of it being the modified grade T91 for which a recommended heat treatment has to be applied. The alloys however can undergo a ductile to brittle behaviour under the action of an agent which affects either the bulk of the material or the surface or reduced amount of materials. Typically, temperature or irradiation act at the scale of the bulk while the effect of environment tends to occur at the surface or at crack tip.

One of the most striking but not yet very well understandable cases is the liquid metal embrittlement (LME). When strained in liquid metals, an otherwise ductile material can exhibit a loss of ductility and, sometime a brittle fracture. Many metals, pure or alloyed, are known to be sensitive to LME e.g. Al by Ga [1], Ni by Li [2], Cu by Bi [3], T91 by PbBi [4, 5]... Generally speaking, it can be stated that no material can be considered as immune against LME.

The presence of a liquid metal on a solid metal can be accidental e.g. during cryogenic distillation for gas processing [6] or simply because the liquid has the function of cooling. Thermal solar plants, fusion reactor, electronic devices… can be cooled by liquid metals such as Pb, Pb-Bi, Pb-Li, Na, Ga…

The present paper deals with the mechanical behaviour of the T91 steel in presence of liquid sodium. Because high strength materials are more prone to liquid metal embrittlement, the T91 steel was investigated after having received its standard heat treatment and after being subjected to a hardening heat treatment. This procedure has been done essentially in a comprehensive goal. In this paper, attention is also paid on the experimental procedure which involves small-sized specimens.

Materials

The chemical composition of T91 is given in Table 1.

Table 1. Chemical composition of T91 steel (wt %).

Element	C	Cr	Mo	Nb	V	Si	Mn	Ni	Fe
[wt %]	0.11	8.80	1.00	0.07	0.25	0.41	0.38	0.17	Bal

The material was subjected to the following heat treatments:

- The standard heat treatment which consists of an austenitisation at 1050 °C during one hour followed by air cooling and tempering at 750 °C for one hour noted “TR750”.
- The hardening heat treatment which consists of an austenitisation at 1050 °C during one hour followed by air cooling and tempering at 550 °C for one hour, noted “TR550”.

After heat treatments, both alloys had the same prior austenitic grains size of 20 μm, and the same tempered martensite laths size of 0.5 μm and 7 μm medium width and length respectively. The major difference in the microstructure concerned the dislocation structures, and the size and distribution of precipitates. TR750 alloys contained coarse $M_{23}C_6$ carbides (250 nm) along prior austenite and lath boundaries and MX carbonitride precipitates within the laths. The high tempering temperature of 750 °C allowed the dislocations to be recovered and entangled around MX carbonitride precipitates forming a sub structure inside laths. By contrast, TR550 exhibited very fine MX precipitates of 20 nm, not along the laths boundaries this time but within the laths, entangling dislocations and making the material much harder than TR750.
The main mechanical properties of TR750 and TR550 materials are shown in Table 2.

Table 2. Mechanical properties of the materials

	Yield Stress [MPa]	Ultimate Tensile Strength [MPa]	Vickers Hardness [HV]
T91-TR750	340±10	650±30	250
T91-TR550	660±25	1200±60	450

Experimental procedure

Two different mechanical set up have been designed and adapted to allow testing small-sized specimens in liquid sodium in a temperature range of 150°C – 550°C. These were the small punch test (SPT) and the three points bending test (TPBT).
Because sodium is very reactive with oxygen and water, the SPT and TPBT experiments must be carried into a cell where the environment is controlled. The cell made of plexiglas comprised a test zone and an airlock. An argon flow spread out continuously the test zone. The solid sodium and the specimen were introduced in the cell through the airlock. Solid sodium was then sliced into small pieces, deposited and gently pressed at the specimen surface. The oxygen content and humidity content were reduced as low as possible (80-100 ppm and 50 ppm respectively) by argon sweeping.

For loading the specimen, an electro mechanical tensile machine was employed at a controlled cross-head speed of 0.05 mm/min.

Small punch test (SPT)

The description of the STP set up is described elsewhere [5]. The specimen holder included a lower die, an upper die which was also used as the tank for the liquid sodium and four clamping screws. The load was 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 puncher being under the specimen (unlike usual small punch test setup), the upper surface of the specimen was in contact with the liquid metal and was submitted to tensile loading.

Specimen had a squared shape 10x10 mm. Before testing, the sample surfaces were mechanically polished with SiC paper up to 1200 grade and then electro polished, in order to avoid effects due to the roughness of the surface and residual stresses developed during the mechanical polishing. The final thickness was 500 ± 20 µm.

The SPT was performed at 150 °C, 200 °C, 300°C, 450°C and 550°C, in air and in liquid sodium.

Load – crosshead displacement curves were recorded during the tests. From the curve [5], it was possible to extract the typical values such as the yielding load Fe, the maximum load Fm, the fracture energy (derived from the area under the load versus displacement curve) J_f and the displacement to fracture d_f.

A ductility factor (D.F.) has been defined as:

D.F. = J_{nfNa} / J_{nfAIR} (1)

where J_{nfNa} is the normalized fracture energy (fracture energy divided by thickness of specimen) measured from test in liquid sodium and J_{nfAIR} the normalized fracture energy measured from test in air.

Three points bending test (TPBT)

The TPBT were carried out in air and in liquid sodium at 200°C and 300°C on specimens prepared according to requirements of ASTM E1820-01 standard [7]. The specimens were first cut using the load controlled abrasive cutoff with an alumina cutting wheel. Then, they were notched thanks to a thin diamond cutting wheel, turning with a very low speed at a very low load in order to avoid cold work hardening of the notch tips. The dimensions of the specimen are reported in Figure 2.

After cutting and dimensioning, all of the specimens were mechanically polished with SiC paper up to 2400 grade, and then electro polished in order to reduce surface roughness and residual stresses.

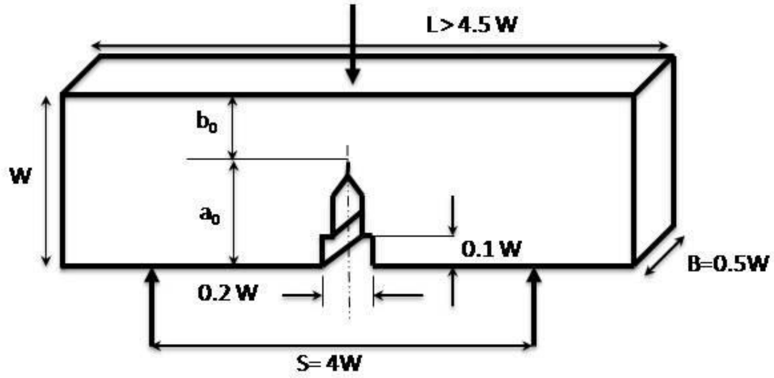
The bending test took place in a special set up which consisted of two specimen holders where the specimen was maintained and submitted to bending, while being in contact with liquid sodium inside a container.

The stress intensity factor K was calculated from the following formula:

$$K=(PS/(BB_N)^{1/2}W^{3/2})3[(a/W)^{1/2}(1.99-(a/W)(1-a/W)(2.15-3.93(a/W)+2.7(a/W)^2)]/2[1+2(a/W)(1-a/W)^{3/2}]$$

Where S, W, B are dimensions of specimen, a the crack length and P the load.

Fig.2. Bending test sample description



with 1 mm < B < 1.6 mm and 2.1 mm < W < 2.4 mm

During the tests, load–cross-head displacement curves were recorded for further calculation of toughness.

Liquid metal environment
The sodium had the chemical composition given in Table 3.

Table 3. Chemical composition of sodium.

Impurities [ppm]	C	Cl^-, Br^-	Sr	Ag	Al	B	Fe
	2-10	2-4	2-5	<0.5	0.5-1	<1	<4

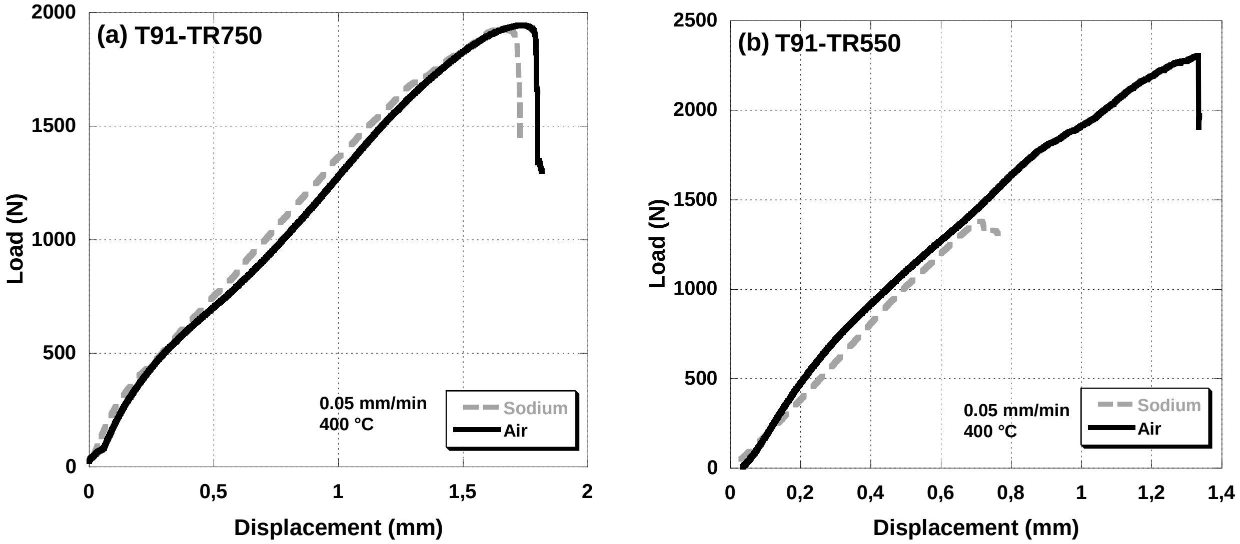
It was not possible to measure experimentally the hydrogen and oxygen contents in the considered sodium bath during SPT and TPBT tests. This was estimated from Noden's calculations for the hydrogen content and from Whittingham's calculations for the oxygen content [8, 9]. It was found that when the liquid sodium was in contact with saturated oxygen air, at 200 °C and 300 °C, the maximum hydrogen and oxygen contents were respectively 1.2 ppm and 12 ppm at 200 °C, and 15 and 96 ppm at 300 °C. Since the oxygen content in our experimental cell was very much less than 150 ppm, one can expect much lower oxygen content in the sodium bath for each test temperature.

Results and discussion

Occurrence of liquid sodium embrittlement

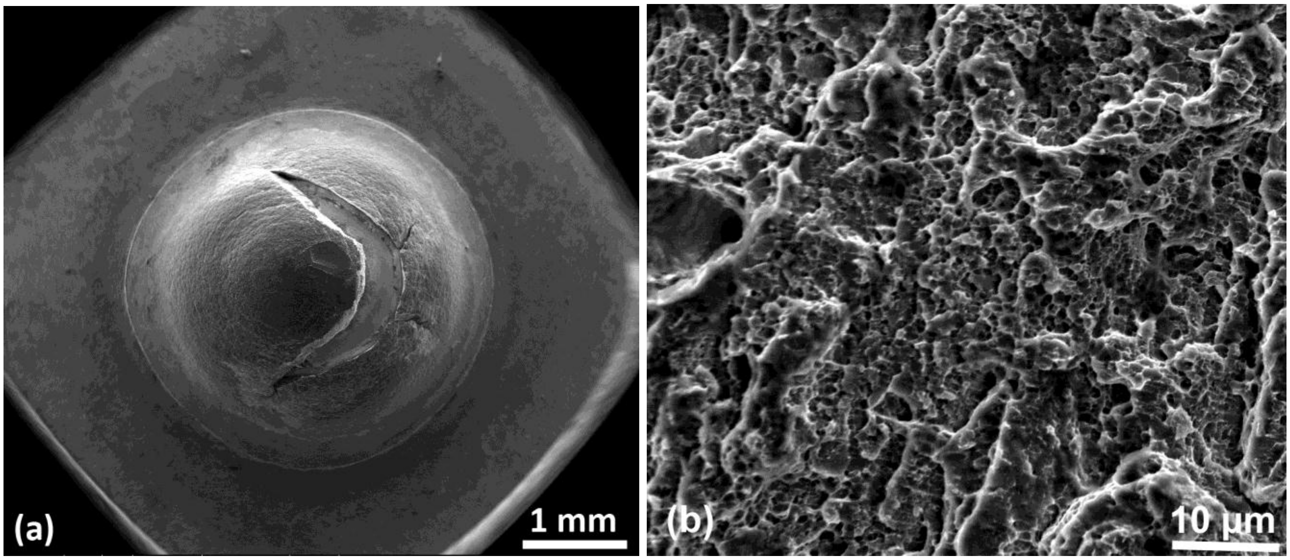
The standard T91-TR750 steel shows a similar mechanical behaviour in air and sodium environment at 400 °C, and a cross head displacement of 0.05 mm/min (Fig. 3.a). However, TR550 steel exhibited a load–displacement curve which was different according to the material was tested in air or in liquid sodium. In air, TR550 exhibited high mechanical resistance involving F_m value of 2385 ± 40 N, and displacement to fracture d_f of 1.35 mm (Fig. 3.b). In liquid sodium, the curves superimpose at the beginning of test, in the elastic part and diverge in the plastic domain. The maximum load F_m as well the corresponding value of displacement are strongly reduced up to 1355 N and 0.75 mm respectively (Fig. 3.b).

Fig.3. SPT curves in air and in liquid sodium at 400°C, at 0.05 mm/min of: a) TR750 steel, b) TR550 steel



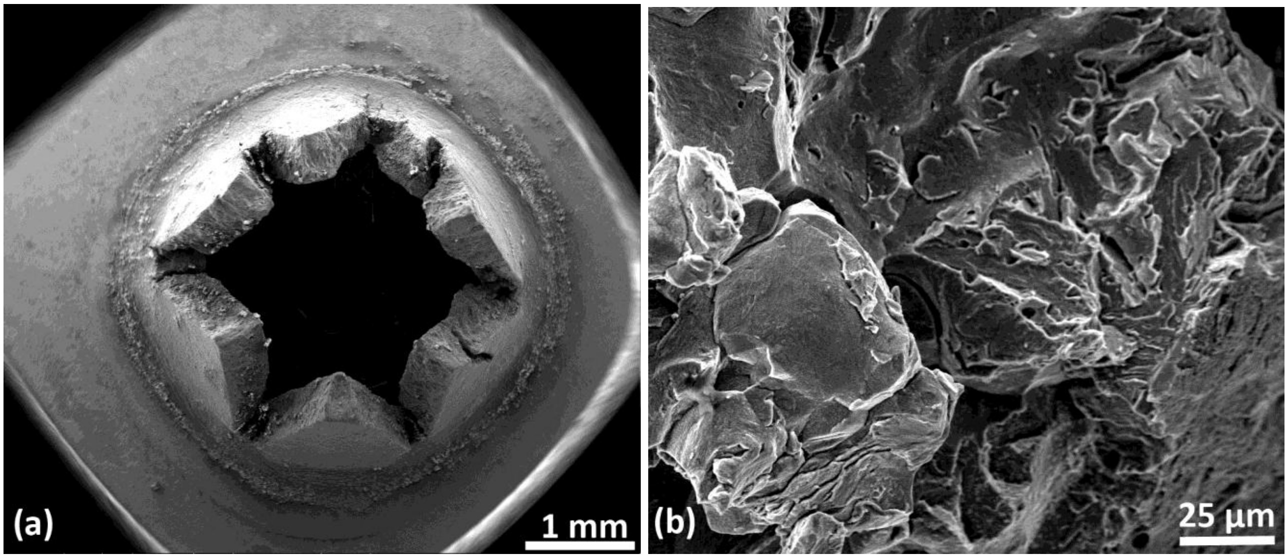
Scanning Electron Microscopy (SEM) observations at low and high magnifications of the fractured specimens revealed also a difference depending on whether the test was performed in air or in liquid sodium. For each material fractured at 400°C in air but also for the TR750 steel fractured at 400°C in liquid sodium, the specimen dome contained a circular crack (Fig. 4.a) and the fracture surface contained dimples typical of ductile rupture (Fig. 4.b).

Fig.4. Fracture in air of TR750 specimen a) Principal crack, b) Ductile fracture surface



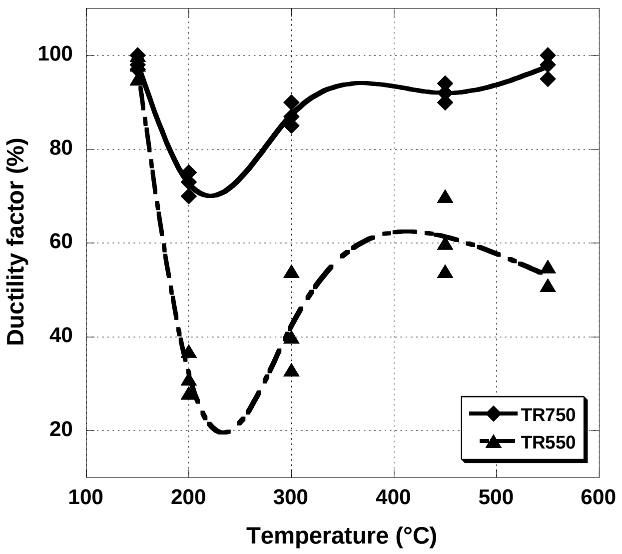
The general tendency for TR550 material fractured at 400°C in liquid sodium is that cracking contained very limited circular parts with large radial cracks (Fig. 5.a). The weak mechanical properties of this material were associated with a fully brittle fracture mode. The brittle fracture surface comprised an intergranular zone (Fig. 5.b) close to the external surface in contact with the liquid sodium which changed for transgranular one in the rest of the specimen.

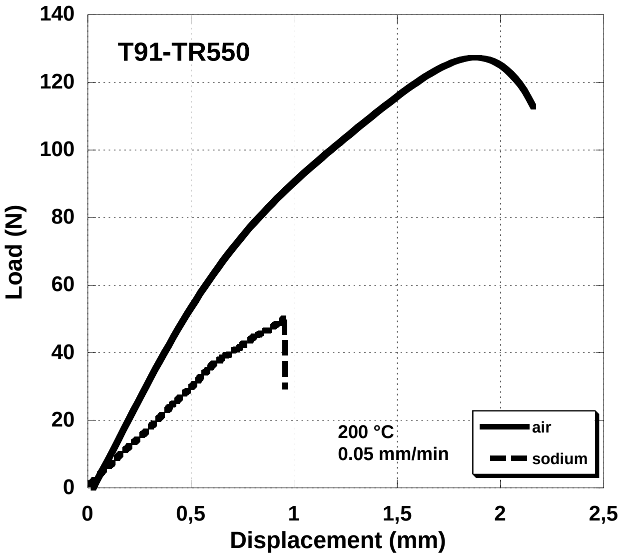
Fig.5. Fracture in liquid sodium of TR550 specimen c) Principal crack, d) Brittle fracture surface, showing intergranular decohesion near the exposed surface



LME being a temperature dependent phenomenon, TR750 and TR550 steels have been tested at 150, 200, 300°C, 450°C and 550°C. The ductility has been used to point out the temperature effect (Figure 6).

Fig.6 (left): Evolution of ductility factor with test temperature of TR750 and TR550 materials tested in sodium at a cross head speed of 0.05 mm/min and
Fig.7 (right) Load-Displacement curves of T91-TR550 notched specimen tested at 200 °C in air and in liquid sodium





This figure shows the existence of a ductility trough located at about 250 °C. The location appears to be the same for both alloys. However, the width and the depth of the ductility-trough increase with increasing the strength of the material.

Effect of liquid sodium on toughness

The TPBT performed on TR550 steel in sodium and air at 200 °C and at 300 °C confirmed the SPT results. In air, TR550 material combined high strength with wide domain of deformation. However, it can be seen that TR550 material failed very early (in the beginning of plastic regime) by sudden, unstable crack extension when they were exposed to sodium. The curves indicate a typical brittle fracture behavior (Fig.7).

The fracture toughness was estimated by two ways. The first way was by calculating the critical value of the stress intensity factor K_{IC} and by calculating the J integral in respect to ASTM E1820 standard requirements.

Both approaches indicate that TR550 has low fracture toughness in liquid sodium at 200 °C. The value of K_{IC} at 200 °C is 63 ± 9 MPa.mm$^{1/2}$ which is rather low for such an alloy. The J-values evaluated from the load displacement curves at 200 and 300 °C are about 214 ± 26 kJ/m^2 and 241 ± 10 kJ/m^2. Comparing these values with those obtained in liquid sodium, 27 ± 1.5 kJ/m^2 at 200°C and 44 ± 5 kJ/m^2 at 300 °C, one can see that the presence of sodium results in a significant decrease in the fracture toughness of TR550 specimens (drop up to 80 %).

Conclusions

By control of the heat treatment of modified 9Cr1Mo steel, in particular the tempering temperature, it has been possible to obtain two materials: the standard TR750 material and the hardened TR550 one.

Both materials have been tested in air and in liquid sodium between 150 °C and 550 °C.

The following points are worth mentioning:

- Both TR550 and TR750 materials were ductile in air whatever the test temperature and their fracture surfaces were ductile with dimples
- TR750, with the recommended heat treatment, was ductile in sodium whatever the test temperature and failed by ductile mode
- Brittle fracture occurred in the TR550 material when deformed in liquid sodium between 200 and 550 °C and the fracture surface comprised both inter and trans granular fracture
- Liquid sodium decreases the J value toughness of the TR550 material by a factor of 80%

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