Thermomechanical testing under operating conditions of A516Gr70 used for CSP storage tanks

Cristina Prieto[1], Camila Barreneche[2], Mònica Martínez[2], Luisa F. Cabeza[3], A. Inés Fernández[2,*]

[1]Abengoa Research, C/ Energía Solar nº 1, Palmas Altas,41014-Sevilla

[2] Department of Materials Science & Physical Chemistry, Universitat de Barcelona, Martí i Franqués 1, Barcelona 08028, Spain.

[3] GREA Innovació Concurrent, INSPIRES Research Centre, Universitat de Lleida, Pere de Cabrera s/n, 25001, Lleida, Spain.

*Corresponding authors: ana_inesfernandez@ub.edu

Abstract

Thermal energy storage (TES) in molten salts is the storage dominating technology in solar power applications today. In two-tank molten salt storage systems energy density ranges from 30 to 70 kWh/m^3 are achievable. The salt material used is a binary system, composed of 60% $NaNO_3$ and 40% KNO_3. In the 8 MWh_{th} pilot plant built and tested by Abengoa, the storage tanks were made of steel A516Gr.70 using the Appendix M of code API 650 for their design. A specific testing device was developed to evaluate thermo-mechanical properties, and a study was conducted in order to evaluate tensile properties of A516Gr.70 specimens under operation conditions for the hot tank at the pilot plant that is in contact with molten salts at 380 °C. Results confirmed the outcomes of the work: the reduction of the yield limit, elongation before fracture, and Young modulus at 380 °C after having been 5 minutes immersed in molten salts. Moreover, after a creep-test simulating operating 7 days conditions during, an additional reduction of the yield limit was measured.

Key-words: *thermomechanical properties, concentrated solar power (CSP), thermal energy storage (TES),carbon steel, molten salts, thermal treatment*

1. Introduction

The global crisis of energy is one of the biggest challenges for the near future for researchers and policy makers in order to achieve a sustainable solution that deal with this energy crisis [1-2]. Solar energy is the highest power source of the world. Thereby, solar power technologies are able to produce high amounts of sustainable power. Concentrated solar power (CSP) plants [3-4] are systems able to concentrate a large amount of solar energy using different types of collectors; several characteristics of solar power plants are described by Desideri et al. [5].

Nowadays, thermal energy storage (TES) is a promising technology to be applied as a complement of CPS plants in order to reduce the gap between energy supply and energy demand [6]. Therefore, TES systems are implemented in CSP plants successfully by producing electricity several hours after sunrise [7]. Moreover, TES will help in making CSP much more viable and feasible from the economic and technical point of view. But the proper selection of materials has a great significance not only to deal with a proper performance of the plant but also for the economic interest of TES units.

A516Gr.70 steel was used to manufacture the storage tanks in the pilot plant of nitrate molten salts with 8 MWh_{th} built by Abengoa [8]. A516Gr.70 is the main used steel in commercial CSP plants due to its high mechanical performance efficiency under stress and its relative low cost. Mechanical design typically uses the yield strength as one of the main design parameters. However, it is well known that the mechanical properties of steels as the yield strength or the modulus of elasticity decrease with increasing temperature. In this study, this change is of extremely importance because during operation of both storage tanks, the cold one is designed to operate at 288 °C and the hot one at 388 °C. Moreover, the maximum service temperature is the highest temperature at which the material can be used for an extended period without significant problems [9]. For the majority of commercial carbon steels, this temperature ranges from 270 to 360°C and only few grades of steel are certified to work at higher temperatures like the operation conditions of the hot tank of molten salts storage systems. Prieto et al. [8] previously reported a detailed description of the tanks design. Both tanks were built with steel A516 and two grades with different grain size. Thus, grade 60 with the smallest grain size was used for the tank cover and grade 70 for the rest of the tank. Parameters used for the design were those of the Appendix M of code API 650, although it only gives the requirements for working temperatures between 90 ºC and 260 °C.

The study of the evolution of the mechanical properties with temperature may be performed varying temperature with special testing devices adapted for such purpose, and following international standards for testing [11]. Nevertheless, the working conditions of this material in operation, which are the temperature (388 °C) and the environment (in contact with nitrate molten salts), are not considered in any standard. For this purpose, a study was conducted in order to evaluate tensile properties under operation conditions by testing in a device developed at the University of Barcelona, where a series of mechanical experiments were performed with A516 gr70 carbon steel specimens in contact with molten salts at 380 °C. Prior and after the mechanical testing, a metallographic study was performed.

2. Methods and materials

2.1. Materials

The specimens for this study were obtained from a plate of the same carbon steel used in the construction of the tanks at the Abengoa plant. Table 1 summarizes the main characteristics collected from the CES Selector database for the reference ASTM A516 Grade 70 Carbon Steel Plate for Boilers and Pressure Vessels.

Table 1. Composition of Carbon steel A516 Grade 70 [12].

Composition	**Content (% wt.)**
Fe (Iron)	Base Element (>98)
C (Carbon)	0.27
Mn (Manganese)	0.79-1.3
S (Sulphur)	0.025
Si (Silicon)	0.025
Mechanical properties	
Ultimate tensile strength (UTS)	485 – 620 MPa
Yield Strength	260 MPa (minimum value)
Elongation	15% (minimum value)

Specimens for mechanical testing were machined from the original plate by laser cutting. The size of the specimens was calculated following the standard from the plate thickness [11]. Dimensions (in millimetres) and design of the specimens are shown in Figure 1.

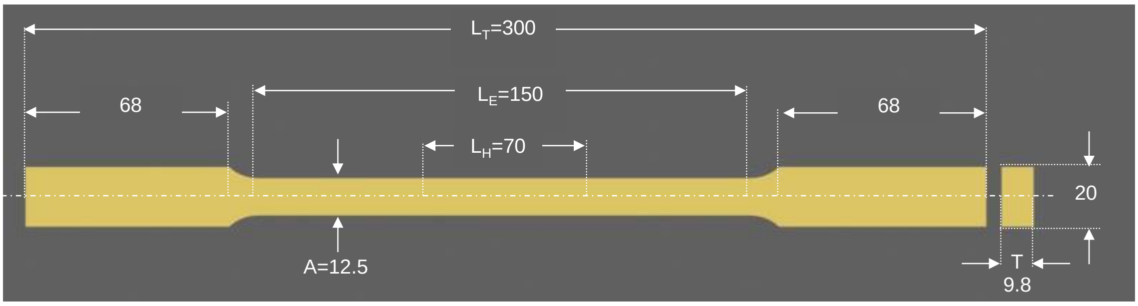

Figure 1 - Dimensions of the specimens machined from A516Gr70 plate [11].

2.2. Experimental set up

A small furnace was designed and built at University of Barcelona to perform the tests at the desired temperature (in this case 380 °C), having the specimen in contact with nitrate molten salts during the mechanical testing. The mixture selected is the so-called *Solar salt* reported in the literature with a composition 60:40 $NaNO_3$ and KNO_3 by weight, close to the eutectic composition.

The cylindrical device was made of quartz, and its size allows testing the central part of the samples. A specific opening was designed to introduce a thermocouple to monitor the molten salts temperature as shows the scheme of Figure 2.(a). The top and the bottom of the device are designed so that a refrigeration circuit using water as heat transfer fluid may be coupled, to prevent molten salts leakage. Furthermore, it also has the central part with a reduced section in which an electrical resistance was coiled around, see Figure 2.(b), and then fully thermally insulated, see Figure 3.

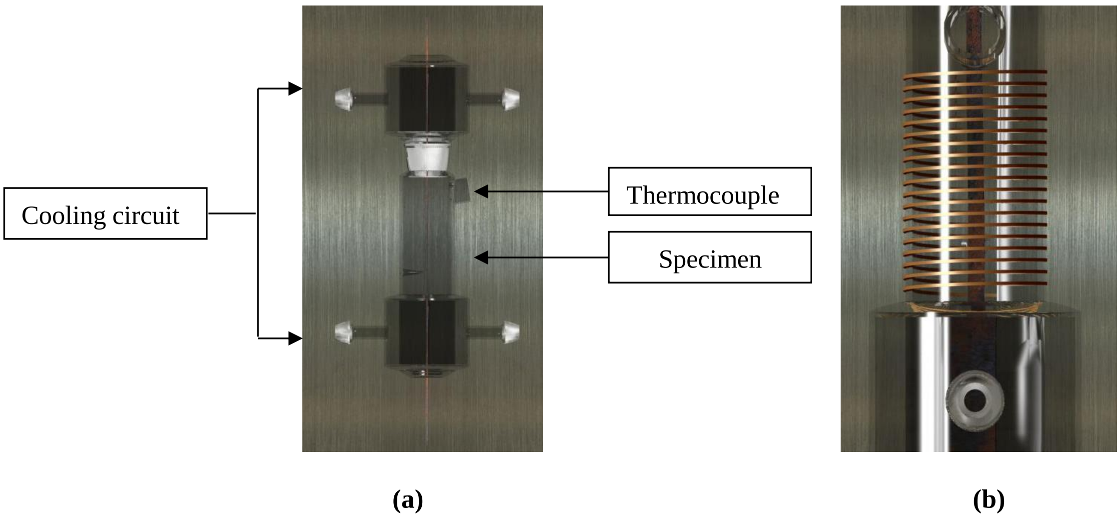

Figure 2. (a) Small furnace devise; (b) Heating source

The insulated system keeping the length of reduced section of the specimen in contact with molten salts is presented in the upper right part of Figure 3. The sample is then fixed through the jaws to the mechanical testing machine Incotecnic MUTC-200.

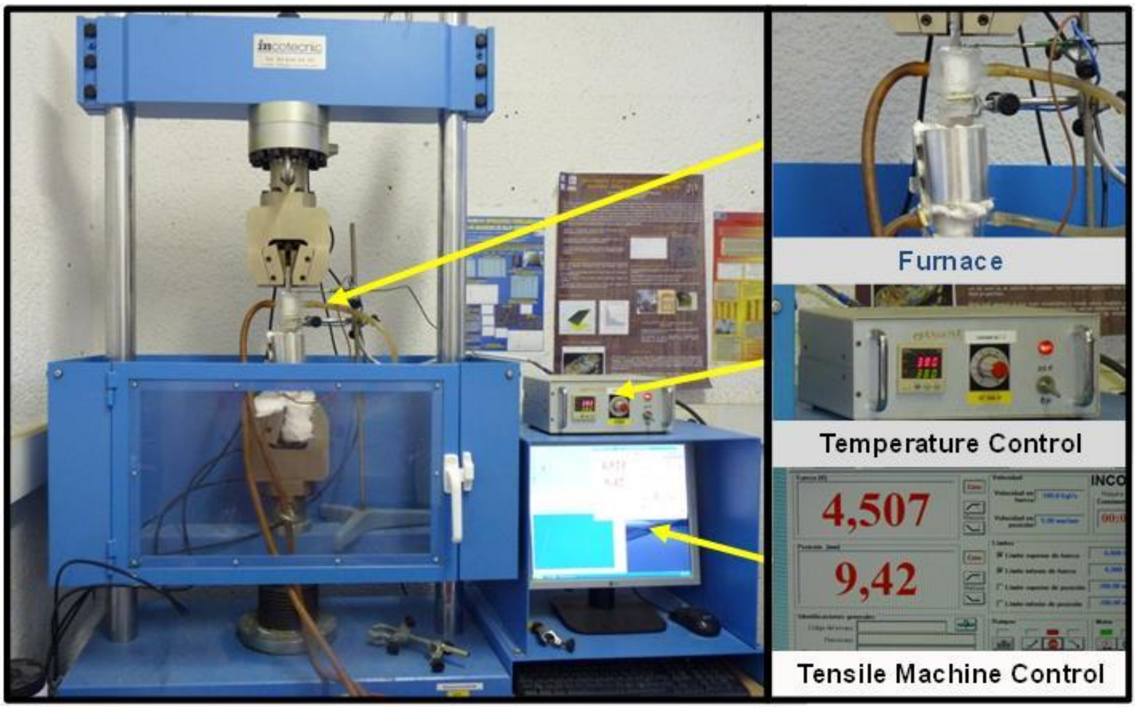

Figure 3 - Picture of the experimental set-up

In order to evaluate the effect of temperature on the mechanical behaviour, three different experiments were performed.

First experiment: the material under study was first tested at room temperature. The tensile test was carried out at a loading rate of 5 mm·min^{-1} until failure. Then, the conventional diagrams plotting stress (σ) vs. % strain (ε%), where stress and strain are defined with Eq. 1 and Eq. 2, were calculated:

$$\sigma\,(MPa) = \frac{F\,(N)}{S\,(mm^2)} \qquad \text{Eq. 1}$$

$$\%\,\varepsilon = \frac{\Delta l\,(mm)}{l\,(mm)}\,x100 \qquad \text{Eq. 2}$$

where *F* is the applied force (in *N*) and *S* is the area perpendicular to the application of force, in the case of the specimens tested it is 122.5 mm^2 and to calculate it, *l* is fixed to 205 mm, being the distance between jaws.

The yield strength obtained from the conventional diagram was used as a reference yield strength to perform the next mechanical tests at higher temperature.

Second experiment: the effect of the temperature on the mechanical properties was tested using the device described previously, having the central part with reduced section in contact with the molten salts at 380 °C. Notice that the sample was in contact with the molten salts not only during the mechanical-test but also during the heating processes (the heating rate applied was 5 $°C \cdot min^{-1}$). Once the temperature of the salts reached 380 °C, the sample was left five more minutes to homogenize the temperature of the specimen. Afterwards, the specimen was tested at 380 ºC under a loading rate of 5 $mm \cdot min^{-1}$ until the sample fractured in order to observe if the temperature affected the mechanical properties. It is important to remark that the main objective of performing these tensile strength tests (out of standards) is to determine the temperature effect instead of determining high precision values for mechanical properties. Even though, the obtained results can be used to perform a comparison in order to differentiate mechanical behaviours, being this comparison semi-quantitative.

Third experiment: as seen so far, the mechanical behaviour of the steel tested change when working at high temperature. In order to determine a possible variation of the properties under working conditions (load, temperature, and contact with molten salts) a specific experiment was designed. It consists in applying a load (39200 N) and keeping the specimen contact with molten salts during 7 days under a constant elongation (4.79 mm): this test is known as creep-test in materials engineering field.

The experimental procedure consisted on applying an initial load. This load has to be below the one obtained by measuring yield strength of a A516Gr70 steel sample immersed in molten salts at 380 °C (second experiment).. Therefore, regarding the load a safety factor of 1.25 was used to perform thermo-mechanical experiments, which is commonly used for exceptionally reliable materials used under controllable conditions [9]. After the creep-test, a tensile strength test was performed at 380 ºC (inside the device) under a loading rate of 5 $mm \cdot min^{-1}$ until the sample fracture too.

2.3. Metallographic study

After the failure, different zones of the tested specimen at 380 ºC were cut and prepared for the metallographic study. Sample preparation follows the ASTM standard [13] for steel samples, and the etching agent used to reveal the microstructure was Nital (98%v/v ethanol, 2%v/v HNO_3).

For each sample, the microstructure was observed in two different zones: zone F is the failure zone and zone O is a zone of the reduced section far from the failure. Moreover, the microstructure was observed from both applied force directions, longitudinal and perpendicular to the application of stress as Figure 4 shows.

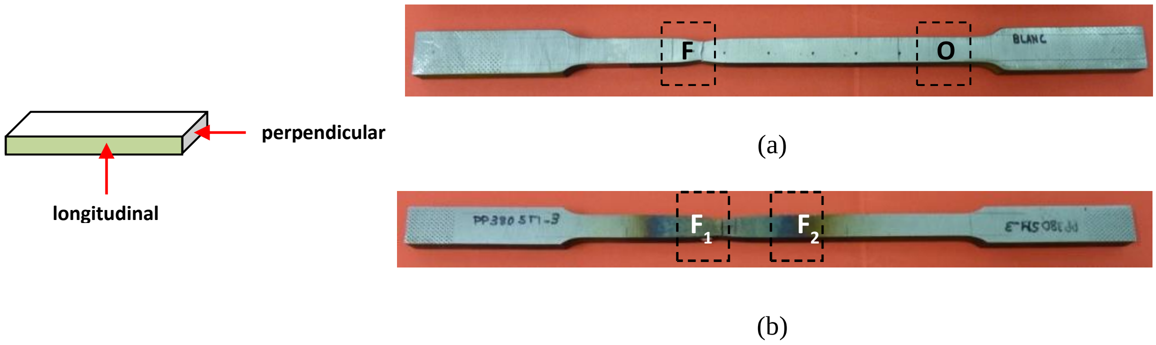

Figure 4 - Zones where the metallographic study was performed with (a) samples measured under 380 °C and (b) sample tested under 380 °C after 7 days

Notice that the temperature of F, F1 and F2 zones were at 380 ºC because these zones are inside the furnace and 25 ºC is the temperature outside the furnace (O zone).

3. Results and discussion

3.1. Mechanical testing

Mechanical properties obtained from the tensile strength test under room temperature conditions are shown in Figure 5 (blue line). The yield strength obtained ($\sigma_{0,2}$) is ~400 MPa and the elongation 17%. Comparing these results with the specifications for a steel of this grade, the yield obtained in the test performed is well above the minimum specified for this material (260 MPa), while the measured elongation is slightly higher than the minimum specified (15%).In addition, tensile strength test results for the sample tested at 380 ^{0}C in contact during 5 minutes with molten salts are also shown in Figure 5 (orange line). In this case, the yield strength ($\sigma_{0,2}$) is about 340 MPa and the strain percentage 13%, showing lower plastic deformation. To calculate the strain the initial length used was L_0 = 205 mm in order to use the same one than in the test before (ambient temperature). In this test, the reduction of the Young Modulus with temperature is also noticeable. The change of this and other properties is described following the equation [14]:

$$P \approx P_0\left(1+\beta\frac{T}{T_m}\right) \qquad \text{Eq. 3}$$

where P is the property at a given temperature T (K), P_o is the property at 25ºC, β is a constant (β ≈–0.5 for metals), and T_m is the melting temperature (K). This equation predicts a reduction of the Young Modulus of around 19% that should be also considered during the mechanical design. But in the experiments presented, 40% reduction of Young Modulus was calculated from the obtained curves. A more accurate Young Modulus values could be expected with the use of an extensometer, but this device is not available.

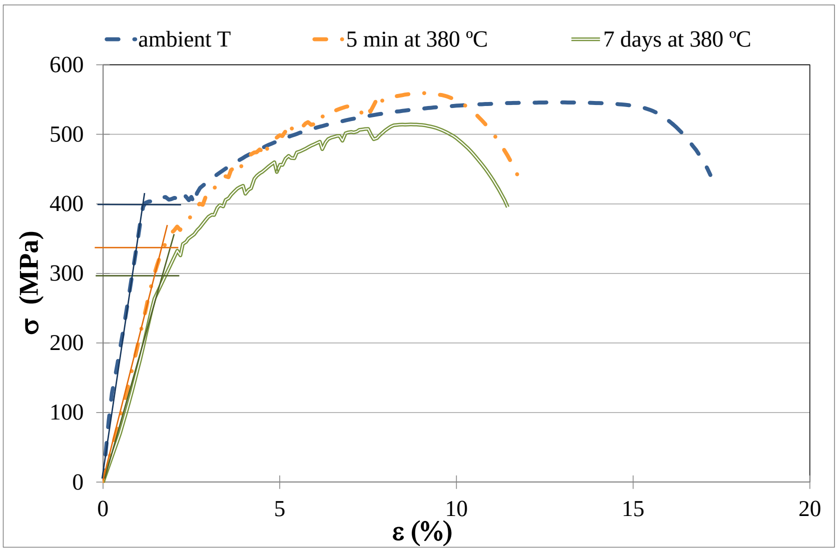

Figure 5 - Conventional diagram of A516GR70 specimen tested at room temperature (blue line), tested at 380°C (orange line), and tested at 380 ºC after 7 days creep-test (green line)

The part of the specimen in contact with the molten salts at 380 °C begins to deform plastically at a lower load, while the rest of the specimen is at a lower temperature, still working in the elastic range. This assumption is verified by measuring the dimensions of the specimens after the test.

In the sample tested at 380 °C, dimensions change only in the region in contact with molten salts, whereas in the specimen tested at room temperature, all the central part with a reduced section changed its dimensions.

In the sample tested at room temperature (blue line), when the stress is increased, the strain is slightly increased (steep slope) until a critical stress value (yield strength). After that, low increments in load produced high elongation (yield zone). The behaviour shown in this area is due to oblique surface slides caused by shear stress (Luders bands). However, when the material is tested under 380 ºC, the yield zone disappears because the temperature disables sliding surface mechanisms [15] or the temperature decreases the yield strength.

Finally, Figure 6 shows the specimens that have undergone necking in the area of rupture, showing a ductile failure with plastic deformation in both cases (room temperature and under 380 ^{0}C).

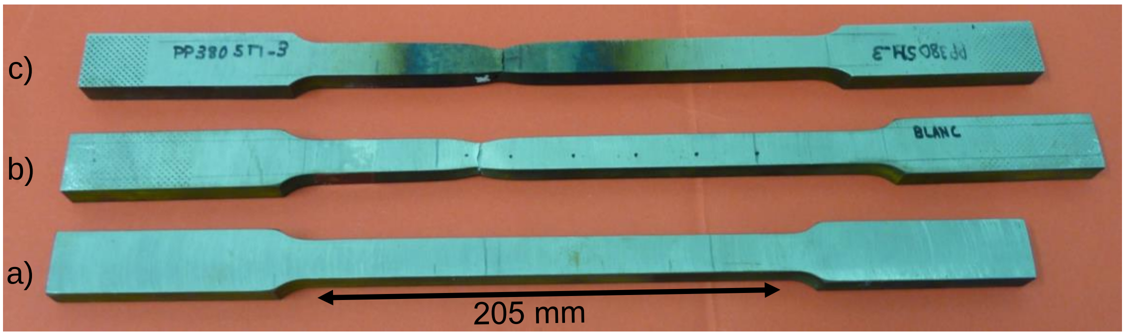

Figure 6 - Specimen of A516GR70 (a) before the tensile strength test, (b) after the tensile test at room temperature, (c) after the tensile test at 380 °C

In order to evaluate the performance under operational conditions, the sample was tested at 380 ºC in contact with molten salts during 7 days by applying an initial load of 320 MPa (below the yield strength measured at 380 ºC). The elongation produced by applying this load was 4.79 mm. Then, this elongation was kept constant during the 7 days of test and load was registered over time. Notice that the stress required to keep a constant elongation decreases over time to an asymptotic value (approximately 265 MPa) due to stress relaxation, as Figure 7 shows.

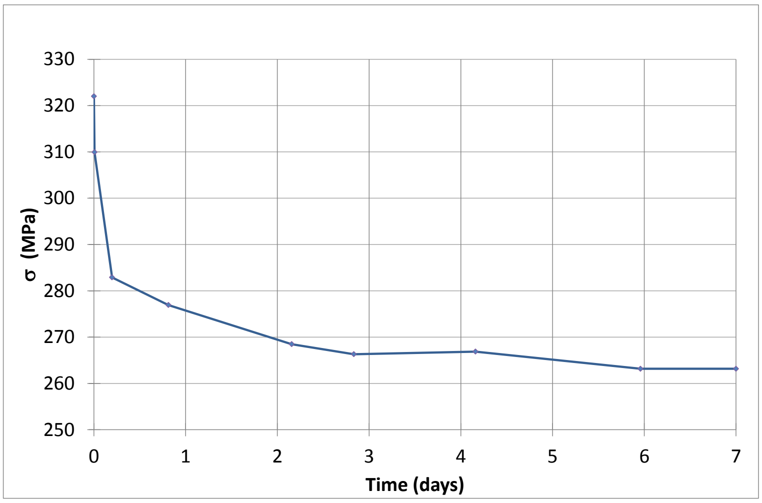

Figure 7 - Change in the stress needed to keep a constant elongation of 4.79 mm at 380 °C

Once 7 days have elapsed, a new tensile test was performed on the sample and the conventional diagram obtained is also shown in Figure 5. Thereby, the yield strength is less than 300 MPa, about 40 MPa lower than that obtained when the specimen had only been 5 minutes at 380 °C (Figure 6). The elongation at fracture was very similar while the measured yield strength was higher than the 260 MPa accepted as minimum value in the standard. The material anneals from the beginning and this annealing continues during this period of 7 days of performance under load, at 380 °C and in contact with molten salts.

Therefore, when a creep test is performed (7 days, 380 ºC, 320 MPa) the sample is plastically deformed, and hence, in a posterior tensile strength test the deformation mechanisms are disabled due to the previous plastic deformation [14] and the ultimate tensile strength also decreased.

3.2. Metallographic study

Since there were differences in the mechanical response upon the testing conditions, the study of the microstructure prior and after each step was done to complement the obtained results.

The longitudinal section of the initial microstructure at 50x magnification is shown in Figure 8. The grains are equiaxially oriented following the shaping direction of the sheet.

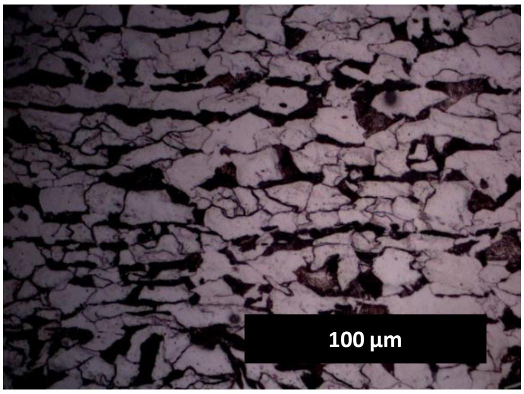

Figure 8- Initial longitudinal microstructure of A516Gr70 carbon steel sample

The microstructure of samples tested at room temperature is shown in Figure 9 applying 50x magnification. Figure 9.(a) and 9.(b) show a microstructure with oriented equiaxial grains, due to the shaping of the sheet. In the image of the longitudinal failure zone shown in Figure 10.(c), the grains deformed in the direction of application of force during the tensile test are observed. Moreover, notice that the grains became smaller due to the force applied, as Figure 9.(d) shows.

On the other hand, Figure 9.(e) and Figure 9.(f) show the areas where the microstructure and the longitudinal and cross direction of samples tested at 380 ºC during 7 days can be seen. The longitudinal failure zone (F_1) observed in Figure 9.(e) shows that the grains are deformed in the direction of the applied force (↑). Moreover, the cross-section of the broken zone (F_1) is shown in Figure 9.(f) where the grains are highly deformed losing their initial form. On the other hand, the grains are slightly affected and preserve almost the same form as the original one in the longitudinal section outside of the failure zone (F_2), as shown in Figure 9.(g).

Even though the operational conditions are reported as corrosives, corrosion evidences appear at longer exposure times and there is no evidence of intergranular corrosion in the metallographic analysis of samples under load at 380 ºC, in contact with molten salts for 7 days.

The reduction of yield strength and elongation is attributed to the annealing of the sample and this fact is corroborated with the metallographic study as shown in the deformation in concordance with the direction (see Figure 9).

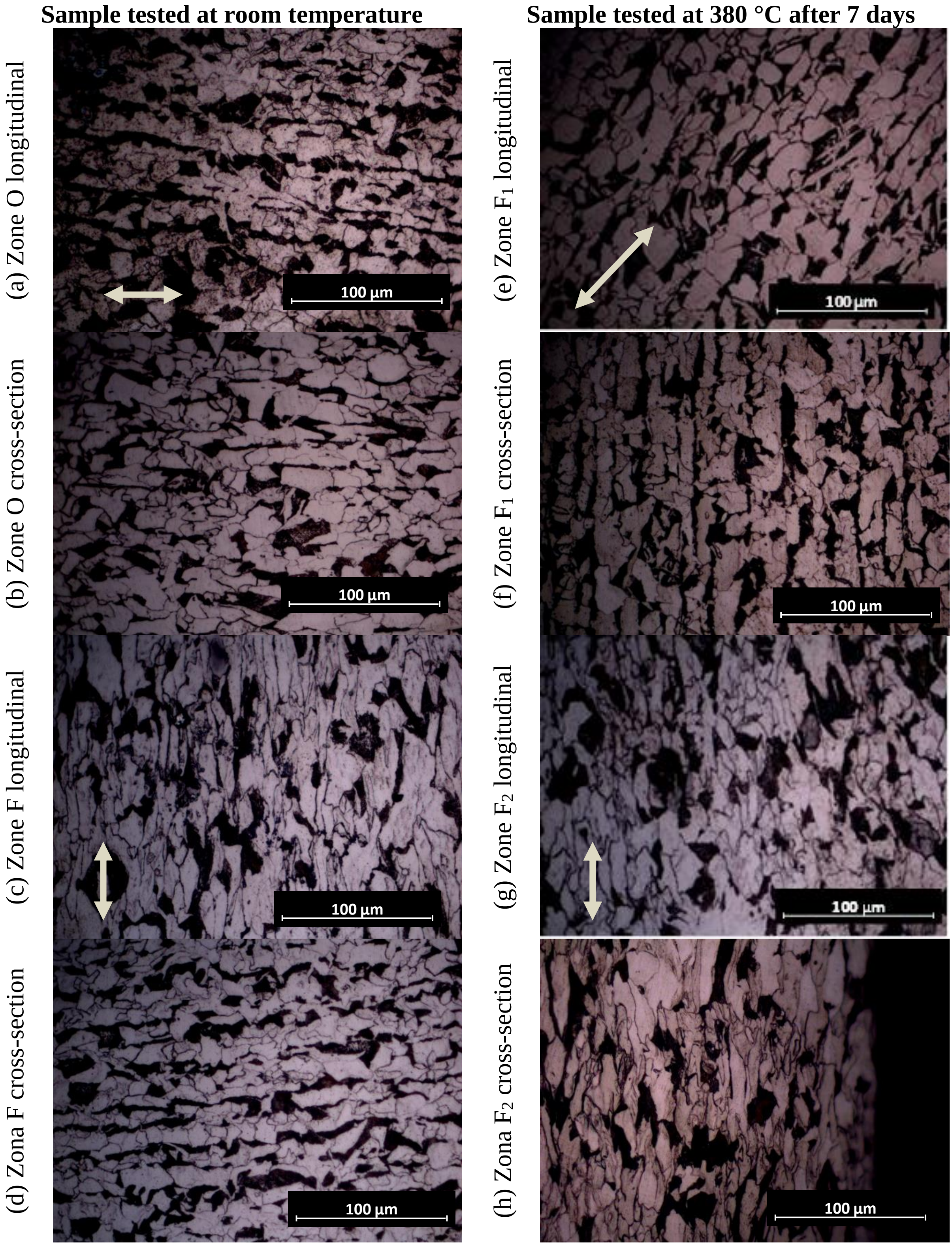

Figure 9- Microstructure of the samples tested under the following conditions: (a) Longitudinal section outside of the broken part of the samples tested at room temperature; (b) Cross-section outside of the broken part of the samples tested at room temperature; (c) Longitudinal section outside of the broken part of the samples tested at room temperature; (d) Cross-section outside of the broken part of the samples tested at room temperature; (e) Longitudinal section in the broken part of the samples treated at 380 ºC during 7 days; (f) Cross-section in the broken part of the samples treated at 380 ºC during 7 days; (g) Longitudinal section outside of the broken part of the samples treated at 380 ºC during 7 days; (h) Cross-section section outside of the broken part of the samples treated at 380 ºC during 7 days

*** Notice that arrows indicate the direction load applied during the tensile strength test**

4. Conclusions

An experimental setup to perform the mechanical testing of steel A516Gr70 under operational conditions at 380 ºC in contact with molten salts was designed and built.

Mechanical testing under operational conditions confirmed the reduction of yield strength and Young Modulus as well as the elongation compared with testing at room temperature. The measured yield strength is above the limit defined in the ASTM standard.

A seven days creep-test experiment maintaining the sample under working conditions and a load below the yield strength was performed. Changes in mechanical properties after the creep-test are confirmed: the reduction of yield strength and elongation are attributed to the annealing of the sample and this fact is corroborated with the metallographic study carried out.

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