

# Evaluation of the mechanical properties after thermal treatment of a structural hot rolled multiphase steel<sup>(·)</sup>

J. Asensio-Lozano\* and J.T. Panta-Mesones\*\*

## Abstract

The present paper corresponds to the experimental study conducted on a hot rolled (HR) multiphase (MP) steel, in which hardness, tensile and toughness properties were measured after the application of a series of subcritical and intercritical heat treatments (HT) to the hot rolled stock. The aforementioned values were compared to the corresponding ones in the as-rolled state and after normalizing. The microstructure in the longitudinal plane of the strip was analyzed by light optical microscopy in the as-rolled state and in the HT samples. Longitudinal (L) and transverse (T) tensile and toughness specimens were cut to characterize every condition studied. Toughness properties were evaluated by means of Charpy V-notch tests conducted at 20 °C, 0 °C, -20 °C, -40 °C, -60 °C and -80 °C. It was observed that the yield stress increased with the increase in the heat treatment temperature in the subcritical range, while the tensile strength decreased slightly over the same range of temperatures. Uniform and total elongation only showed a slight improvement when the treatment was conducted at 620 °C and 700 °C, while the best toughness response corresponded to the sample treated at 500 °C for operating temperatures comprised between -40 °C and room temperature (RT).

## Keywords

Multiphase steels. Heat treatment. Tempering. Normalizing. Mechanical properties. Hardness. Tension test. Charpy impact toughness.

# Evaluación de las propiedades mecánicas tras tratamiento térmico de un acero multifase estructural laminado en caliente

## Resumen

El presente estudio corresponde al trabajo experimental desarrollado en un acero multifase laminado en caliente, en el que se evaluaron las propiedades de dureza, tracción y tenacidad a impacto, tras realizar tratamientos térmicos subcríticos e intercríticos al material laminado en caliente. Los valores precedentes se comparan con el material de partida laminado en caliente y tras tratamiento de normalizado. Se analiza la microestructura en microscopía óptica de reflexión, en el plano longitudinal tanto en el estado laminado como en las muestras tratadas térmicamente. Se estudiaron los comportamientos longitudinales y transversales en tracción y frente a impacto de todas las condiciones de material. Las propiedades de tenacidad al impacto se evaluaron mediante ensayo Charpy con entalla en "V" a las temperaturas de ensayo de 20 °C, 0 °C, -20 °C, -40 °C, -60 °C y -80 °C. Se ha podido observar que el límite elástico aumenta con la temperatura de tratamiento en el rango subcrítico de temperaturas, en tanto que la carga de rotura disminuye levemente en ese mismo dominio de temperaturas. Se detectó una leve ganancia del alargamiento longitudinal y transversal en muestras tratadas a 620 °C y 700 °C; en tanto que la mejor respuesta frente a tenacidad al impacto se obtuvo tras tratamiento a 500 °C para temperaturas de operación entre -40 °C y temperatura ambiente.

## Palabras Clave

Aceros multifase. Tratamientos térmicos. Revenido. Normalizado. Propiedades mecánicas. Dureza. Tracción. Tenacidad al impacto Charpy.

## 1. INTRODUCTION

The studied steel initially typified as HR multiphase steel falls into the category of high strength structural steels<sup>[1]</sup> and has been successfully used at industrial

scale in the construction of demolition crane arms. Its attractiveness resides in the optimum combination of resistance to plastic deformation, good ductility and high toughness measured at low temperatures (KCV at -20 °C and -40 °C). After cutting the HR

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\* Departamento de Ciencia de los Materiales e Ingeniería Metalúrgica, Escuela de Minas, Universidad de Oviedo, c/Independencia nº 13, E-33004 Oviedo, SPAIN (ESPAÑA).

\*\* Departamento de Ingeniería de Minas, Metalúrgica y Materiales, Facultad de Ingeniería, Universidad Nacional de Trujillo, Av. Juan Pablo II s/n, Trujillo-La Libertad, PERU (PERÚ).

strip to the specified plate dimensions, the steel pieces may be subjected to severe bending operations due to the uniform elongation that the as-rolled material possesses, followed by welding. The weldability of this steel falls into the fair-poor category, requiring a post-weld heat treatment (PWHT) of the heat affected zone (HAZ) affecting an entire portion of the assembled component. This is due to the presence of alloying elements leading to a medium-high equivalent carbon content (Ceq) of approximately 0.5 % wt. As a result, impact properties in the HAZ after PWHT represent a key issue for safety operation, and often lead to high energy absorbed values being specified within the acceptance safety standards. For instance, the Charpy values for demolition crane arms are restricted to steels complying with: KCV (-20 °C) ≥ 40 J and KCV (-40 °C) 27 J. The usual tensile properties of the as-treated plate are given by the 0.2 percent offset yield stress (0.2 %YS) varying in the 640-670 MPa range, with a tensile strength (TS) value in excess of 800 MPa, and a total elongation (A<sub>T</sub>) equal to or greater than 18 %<sup>[2]</sup>. These results are typical of ferrite-bainite low carbon steels<sup>[3]</sup>.

The aforementioned PWHT specification stimulated the investigation of the mechanical properties in the subcritical temperature range, intercritical and supercritical temperatures applied to the as-rolled material. One of the goals was to explore the mechanical behavior of the as-rolled stock after being subjected to HT cycles in which the chosen treatment temperatures were in the subcritical region and normalizing. The exploration of the HT response after tempering of the as-received material is due to the interest in quantifying the mechanical effect in the tension of alloy carbides, precipitated in the subcritical region and contributing to secondary hardening. The increase in strength is often accompanied by the development of ductility and/or toughness troughs which must be assessed<sup>[4]</sup>. A complementary goal was to investigate the possibility of applying low temperature PWHT in the subcritical region in order to replace the expensive normalizing process which is often mandatory to restore an acceptable toughness response in the HAZ, and to verify the potential development of new steel qualities

derived from simple heat treatments applied to the as-rolled strip<sup>[5 y 6]</sup>.

## 2. EXPERIMENTAL MATERIAL AND PROCEDURE

The as-received stock corresponds to a 7 mm thick strip processed by controlled rolling followed by accelerated cooling and subsequent coiling at the hot strip mill facilities that the ARCELOR-MITTAL steelmaking group has at its factory located in Avilés, northern Spain. The steel is categorized as a high strength steel (HSS) microalloyed with Mo-Nb-Ti and B.

The experimental composition of the HR strip expressed in weight per cent is given in table I. The recorded values for S and C were analyzed by combustion in a LECO CS 444 analyzer. Nitrogen was analyzed using a LECO TC 136 analyzer. For the remaining elements, chemical analysis was conducted with an ARL 4460 optical emission spectrometer. The results presented in table I are the average of 6 determinations.

As part of the experimental procedure, a series of HT were applied to rectangular samples of 400 × 30 × 7 in mm cut from the HR strip at 0° (longitudinal) and 90° (normal) to the rolling direction (RD).

Several plates were placed in a HERAEUS K-1253 model furnace. Those placed immediately above and below the sample of interest had a K-type Chromel-Alumel thermocouple inserted to monitor temperature changes during the thermal cycle of the sample of interest placed in between. Two sets of treatments were chosen. An initial series of independent treatments were carried out at 300, 400, 500, 620 and 700 °C for 2 h at temperature. The second series consisted of a single normalizing treatment at 920 °C for 30 min. In all cases, the samples were heated from room temperature (RT) at a heat-up rate of 10 °C × min<sup>-1</sup> and, on concluding the soaking process, were removed from the furnace and allowed to cool in air.

The hardness test was executed in accordance with the UNE-EN ISO 6507-1 standard<sup>[7]</sup>, which describes the Vickers hardness (HV) method. The load employed

**Table I.** Chemical composition of the steel expressed in weight percent.

*Tabla I. Composición química del acero expresado en porcentaje en peso.*

C	Mn	Si	S	P	Cu	Cr	Ni	Al	Nb	Ti	Mo	B	N
0.085	1.859	0.081	0.004	0.013	0.018	0.020	0.017	0.025	0.056	0.049	0.295	0.001	0.007

was 10 Kg. Hardness specimens of  $30 \times 30 \times 7$  in mm were cut from the heads of the tensile test specimens, and the longitudinal and transverse planes were surface ground and polished to give orthogonal planes. Five tests were conducted for every specimen in both the longitudinal and transverse plane in the center of the thickness. An EMCO model M4U hardness tester was used for the determinations.

On completing the HT schedules, these plates were then machined to give tensile specimens with a calibrated length of 80 mm, a gauge length of 60 mm, 17 mm width and 7 mm thickness, in accordance to the UNE-EN 10002-1 standard<sup>[8]</sup>. The specimens were tensile tested with an Instron 4488 - H1897 series universal tensile test machine equipped with a 60 T load cell, at a fixed crosshead speed of  $2.5 \text{ mm} \times \text{min}^{-1}$ . The parameters evaluated were: 0.2 % offset engineering yield stress (0.2 % YS); engineering tensile strength (TS); uniform engineering elongation (% $e_u$ ); and total strain at fracture (% $e_t$ ).

Charpy V-notch test were conducted in accordance with the UNE-EN 10045-1 standard<sup>[9]</sup> on longitudinal and transverse specimens at 20 °C, 0 °C, -20 °C, -40 °C and -80 °C to describe the ductile-brittle transition curve on an energy versus test temperature plot for every HT studied.

The research study was completed with the characterization of the microstructure of the steel by light optical metallography (LOM). For this purpose, samples were cut from the HR stock, normalized samples at 920 °C, and 620 °C treated samples as representative of the highly tempered steel, and 700 °C treated samples after mounting them in bakelite. After conventional grinding and polishing, the samples were etched with nital-10 and also with LePera, the latter showing the martensite plus retained austenite in white, the bainite constituent in brown and the ferritic matrix in a dark grey background<sup>[10]</sup>. Micrographs were taken with a ZEISS-AXIOVERT 25 LOM connected to a digital camera and archiving software equipment.

### 3. RESULTS AND DISCUSSION

The chemical analysis of the steel corresponds to a low carbon steel with high Mn, and the presence of microalloying elements for grain size control such as Al, Nb and Ti, together with elements such as Mo and B used to provide the required hardenability in order to promote the transformation of residual austenite into a bainitic aggregate and also into martensite plus retained austenite (M-A) islands.

The as-rolled material observed under LOM shows

fine, elongated ferrite grains (Fig. 1 a)) with the presence of dispersed constituents.

After LePera etching (Fig. 2 a)), the disperse constituents are revealed as bainitic aggregates and M-A islands.

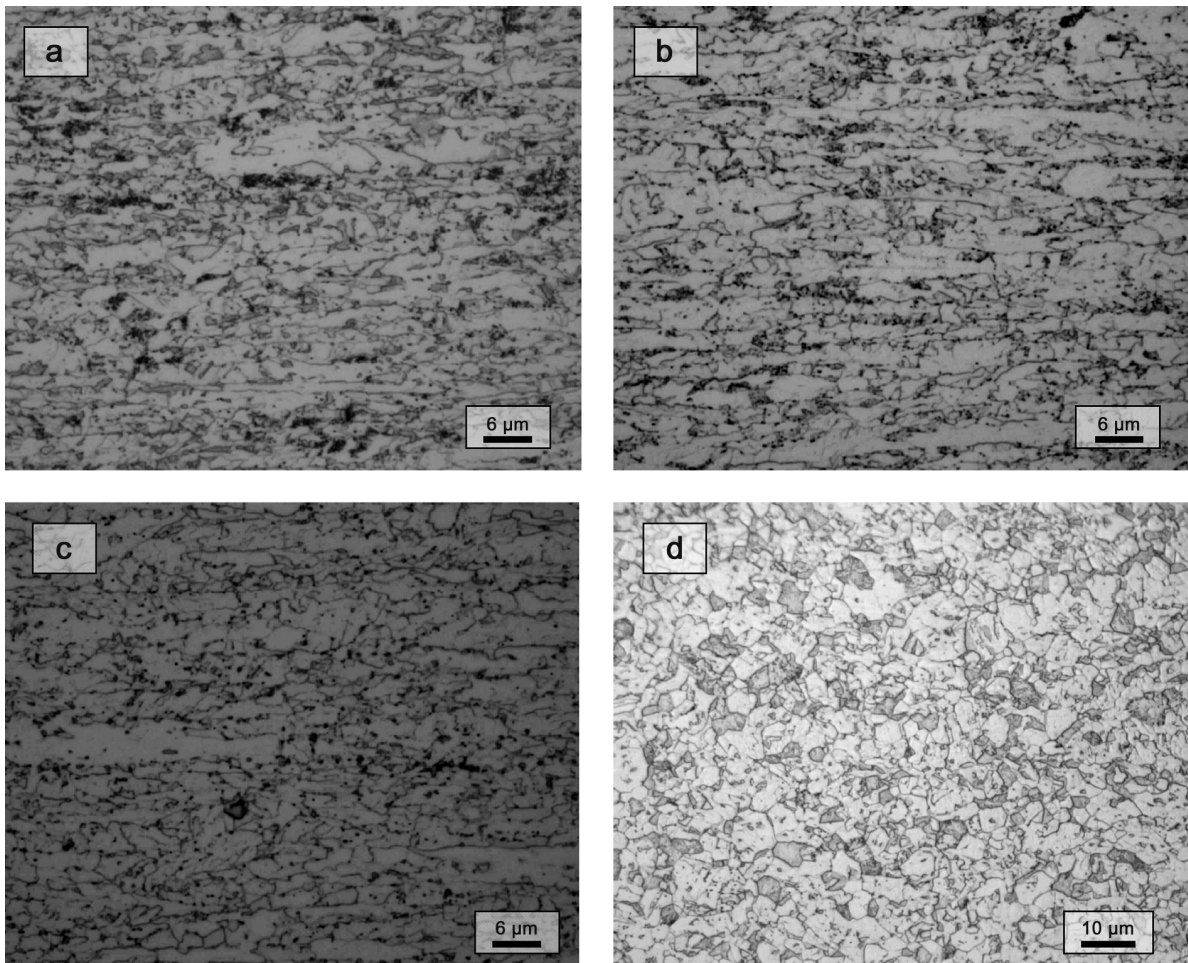
With increasing temperatures in subcritical HT, the volume fraction of M-A gradually decreases from 8 %-vol. in the as-rolled state to almost 1 %-vol. after the 620 °C treatment (Figs. 1 b) and 2 b)). In addition, the M-A island sizes becomes reduced from 2  $\mu\text{m}$  to 1  $\mu\text{m}$ , corresponding to the as-rolled and 620 °C treated samples, respectively<sup>[5]</sup>.

The sample treated at 700 °C (Figs. 1 c) and 2 c)) corresponds to the studied HT, in which the evaluated M-A volume fraction is smallest, around 0.3 %-vol., the size of the M-A being estimated to be very fine, under 1  $\mu\text{m}$ , and well dispersed over the ferritic matrix<sup>[5]</sup>.

The normalizing HT at 920 °C gives rise to a ferritic-martensitic structure, as seen in figure 2 d), with the absence of bainite. It is worth noting that the volume fraction of M-A reached approximately 9 %-v., exceeding that of the as-rolled state, with a somewhat smaller island size ( $\sim 0.8 \mu\text{m}$ ), though well dispersed over the entire thickness. After nital etching, the ferritic grain size presents a more equiaxed habit and is appreciably larger in size than that of the start-out as-rolled material (Figs. 1 a) and 1 d)). In summary, the microstructure after normalizing corresponds to that of a DP steel (Fig. 2 d)).

The Vickers hardness values measured in a longitudinal and transverse plane are represented in figure 3. These show the existence of a peak value due to precipitation hardening at 620 °C, and the development of two softer conditions, one at 400 °C and the other at 700 °C. These are indicative of the redissolution of cementite prior to the secondary hardening resulting from the dispersion of alloy carbides. The increase in temperature above 620 °C leads to coarsening and solution of precipitates, with the subsequent loss of their hardening effect<sup>[11]</sup>.

The stress-strain tensile curves corresponding to the as-received steel (Fig. 4) show a continuous elastic to plastic transition, and the yield stress (612-616 MPa) and tensile strength (831-838 MPa) are almost equal in the longitudinal and transverse direction, only showing anisotropic behaviour for the total elongation (22.4-25.5 %) and uniform elongation (12.6-15 %). Increasing the HT temperature makes the elasto-plastic transition become more pronounced. At 500 °C, the yield point phenomenon becomes incipient and most pronounced at 620 °C in the subcritical range of tested temperatures. The treatments at 700 °C and normalizing at 920 °C define a set of curves characteristic of dual-phase



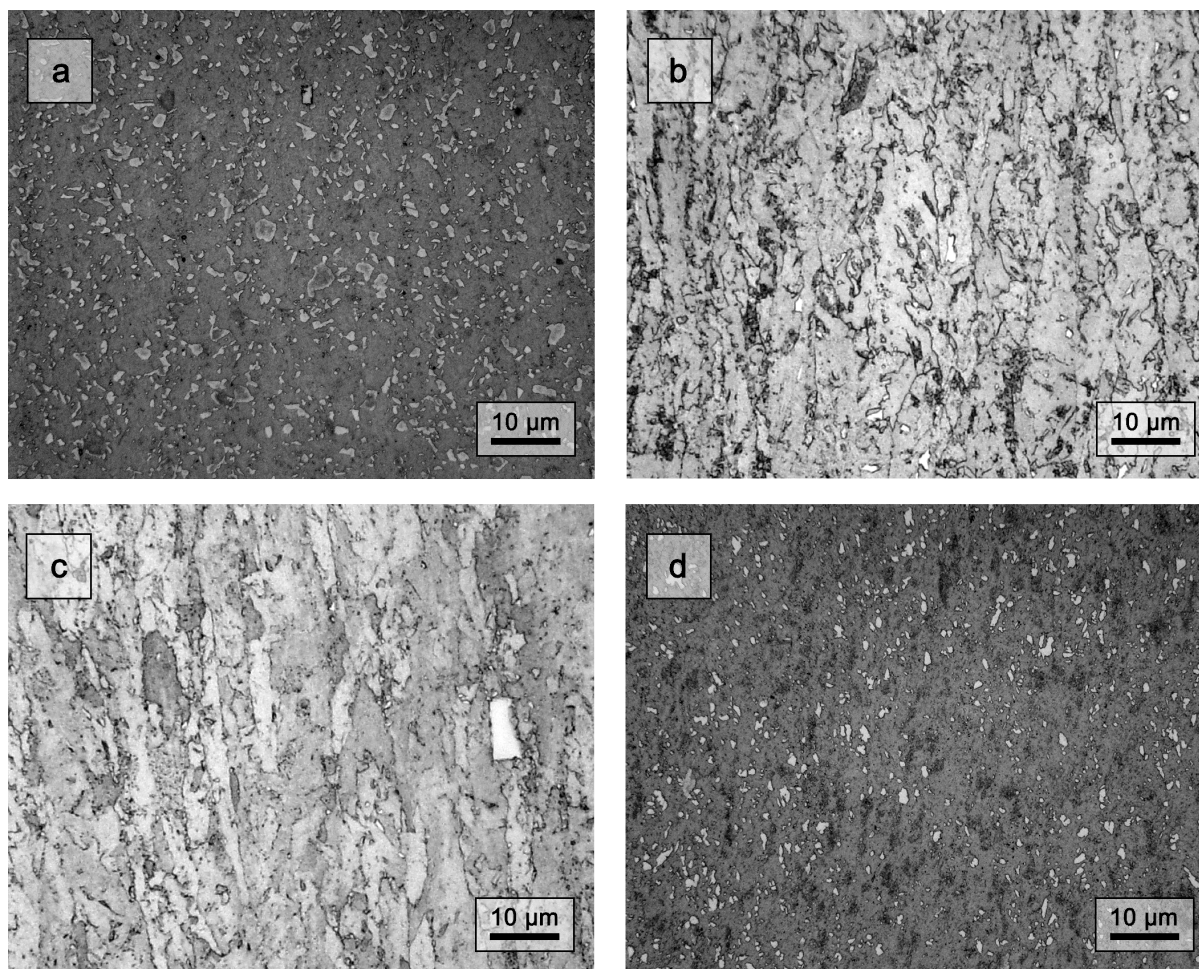
**Figure 1.** Light optical micrographs of the longitudinal plane after nital-10 etching of: (a) as-rolled material, and after HT at (b) 620 °C; (c) 700 °C; and (d) normalized at 920 °C.

*Figura 1. Micrografías ópticas tomadas en el plano longitudinal tras ataque con nital-10 de: (a) acero en estado bruto de laminación en caliente, y tras tratamiento térmico a (b) 620 °C; (c) 700 °C; y (d) normalizadas a 920 °C.*

steels, with a distinctive combination of low yield stresses together with the total absence of the yield point effect and elevated elongation values (Fig. 5).

The yield stress shows very little anisotropy between the longitudinal and transverse directions, though it shows values that increased with the increase in temperature, reaching a maximum stress level at 620 °C as seen in figure 6. The lowest 0.2 %YS value corresponded to the normalized value. As for the tensile strength, this is less sensitive to the treatment temperature than the yield stress. Furthermore, despite the decreasing trend shown by this parameter with increasing treatment temperatures in the tempering range represented in figure 6, it shows a peak at 700 °C close to that of as-rolled stock, in addition to the lowest value in the normalized condition. The anisotropy of the tensile strength is quite small compared to the yield stress.

Both uniform and total elongation for the different temperatures studied are represented in figure 7. As for uniform engineering ductility, an increase in the treatment temperature gradually reduces the anisotropy between the longitudinal and transverse direction. Ductility has a trough at 300 °C where it reaches a minimum of 12-13 % elongation; and from 400 °C to 700 °C the average gain in uniform elongation can be estimated at 0.63 % per 100 °C increase in temperature. The normalizing treatment renders a material with similar uniform elongation to the as-rolled stock, though completely isotropic. Total elongation shows a degree of anisotropy that increases with the increase in temperature in the subcritical range of temperatures up to 620 °C, where a maximum in total elongation was obtained. At 700 °C and 920 °C, both anisotropy and total ductility are reduced, while the normalizing treatment



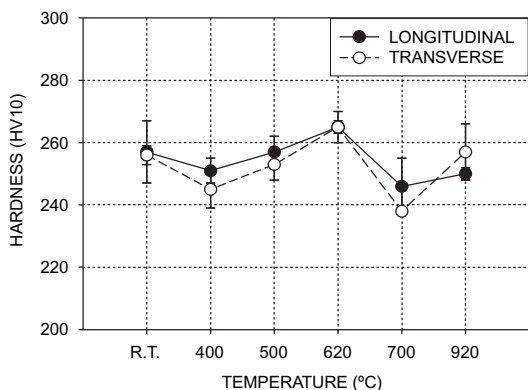
**Figure 2.** Light optical micrographs of the longitudinal plane after LePera etching of: (a) as-rolled material, and after HT at (b) 620 °C; (c) 700 °C; and (d) normalized at 920 °C.

*Figura 2. Micrografías ópticas tomadas en el plano longitudinal tras ataque con reactivo LePera de: (a) acero en estado bruto de laminación en caliente, y tras tratamiento térmico a (b) 620 °C; (c) 700 °C; y (d) normalizadas a 920 °C.*

achieves a total elongation value of 24 percent. The ratio of yield stress to tensile strength expressed by  $0.2YS/TS$  for the corresponding longitudinal and transverse values at the different HT temperatures investigated is represented in figure 8. This ratio is included among the rational criteria for the safe application of structural steels, indicating on the one hand that the safest structural application from a stress perspective can be obtained after tempering at 620 °C [12]. However, the most favourable forming conditions could correspond to either the 700 °C or to the normalized samples [13].

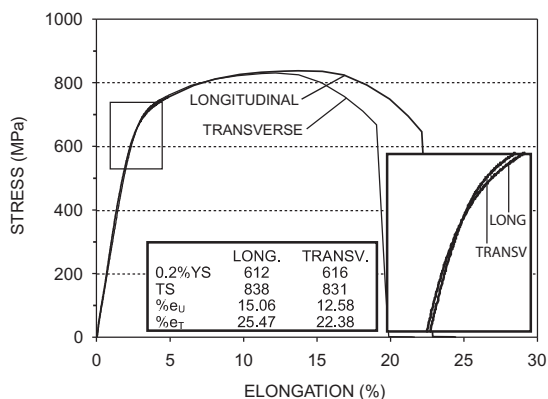
One of the most critical requirement for structural application is the subzero impact response. This is due to regular low operating conditions of structural components in movable structures that often operate under the risk of severe impacts, such as the arms of demolition cranes, etc. For this reason, the absorbed

energy in a Charpy V-notch test at +20 °C, 0 °C, -20 °C, -40 °C, -60 °C and -80 °C in the longitudinal and transverse direction was evaluated at each HT temperature, the results being represented in figures 9 and 10. In both energy plots, the as-rolled and the normalized curves were taken as reference material for comparison, since they respectively represent the upper and lower bounds of the absorbed energy versus test temperature curves. Figure 9 corresponds to the longitudinal values. It is worth noting that only the tempering at 500 °C improved the impact toughness from -40 °C up to RT. The plot corresponding to the steel tempered at 400 °C very closely follows the upper bound curve given by the as-rolled material. The tempering at 620 °C, however, renders absorbed energies that are lower than that corresponding to the normalized material at test temperatures equal to -40 °C and below. The



**Figure 3.** HV10kg Vickers hardness as a function of the HT temperature. Error bars indicate the degree of scatter corresponding to the average value plus minus the standard deviation.

*Figura 3. Valores de la dureza Vickers con carga de 10kg en función de la temperatura de tratamiento térmico. Las barras de error expresan el grado de dispersión correspondientes al valor medio mas / menos el valor de la desviación estándar.*



**Figure 4.** Engineering tensile test curves in the longitudinal and transverse directions of the as-rolled state. A numerical summary of the most relevant tensile properties and a graphical enlargement of the elasto-plastic transition are included in the graph.

*Figura 4. Curvas de tracción ingenieriles en probeta longitudinal y transversal del acero en el estado bruto de laminación en caliente. Se incluyen dos ventanas, una expresa los resultados numéricos mas relevantes y la otra es una ampliación de la transición elasto-plástica.*

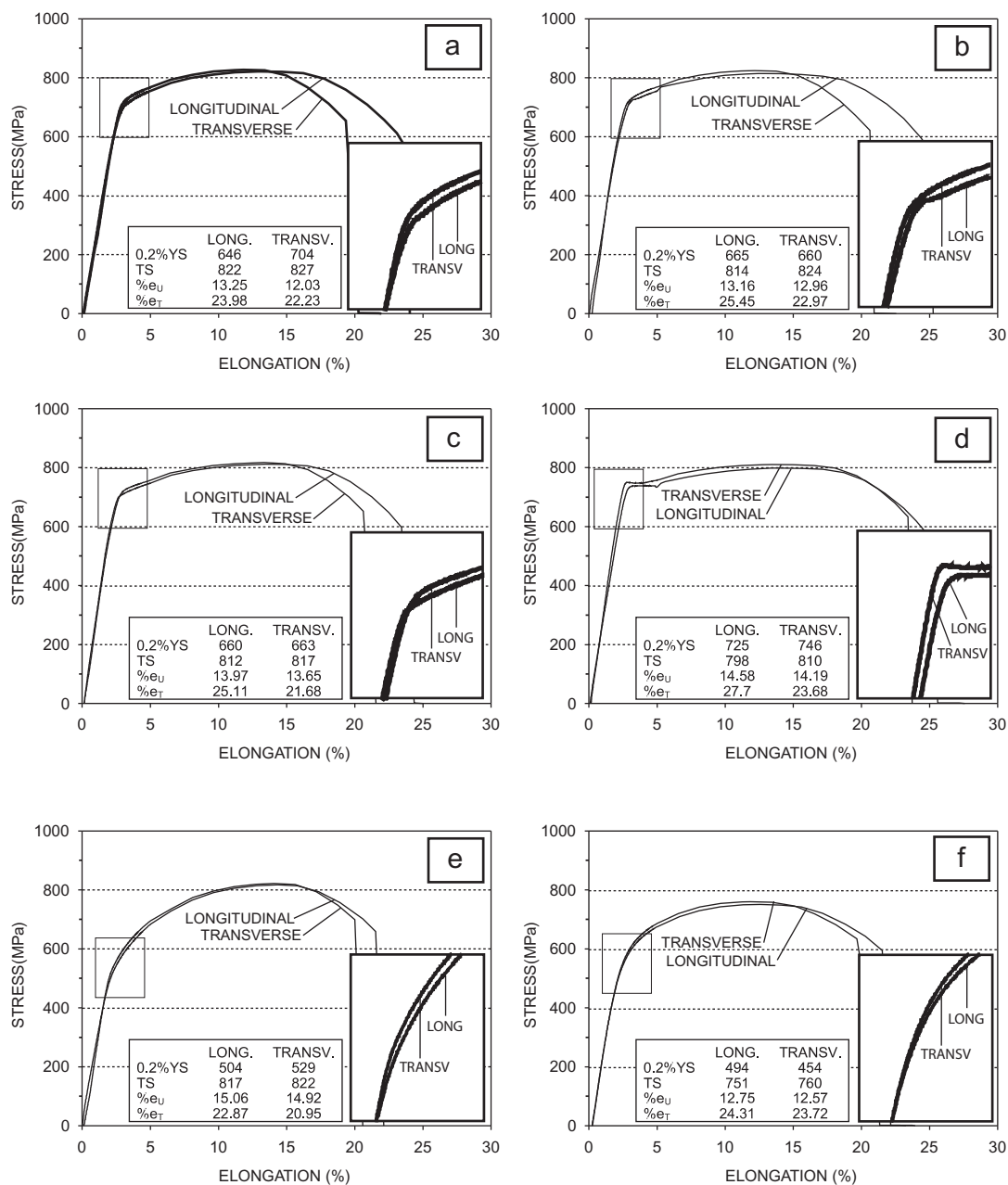
representation of the Charpy toughness values for transverse specimens at different test temperatures are shown in figure 10. The transverse values are

lower than the corresponding longitudinal ones; the as-rolled and normalized specimens are also represented as reference materials in this figure. The best result corresponds to the steel treated at 620 °C, followed by that treated at 500 °C. At 400 °C and 700 °C, the treated steel behaves similarly to the reference steels. A summary of the impact behaviour at selected operational temperatures is given in figure 11. KCV values at -20 °C and -40 °C are represented for all the studied conditions. These show a maximum for the 500 °C tempered steel, which is the only HT condition that improves that of the start-out as-rolled MP steel. There is a drop in impact toughness at 620 °C, while for the intercritical HT at 700 °C and the normalizing at 920 °C, the values are low and present the greatest anisotropy between the two studied directions. As a result of the standard set for the Charpy V-notch tests, KCV (-20 °C) 40 J and KCV (-40 °C) 27 J, only the as-rolled and the steels tempered at 400 °C and 500 °C satisfy the aforementioned prerequisite in both longitudinal and transverse directions. Only the steel treated at 620 °C does not satisfy any of the criteria.

#### 4. CONCLUSIONS

As regards the observed microstructure and the evaluation of steel hardness, it may be concluded that tempering at 620 °C of the hot rolled stock provides a peak in hardness values of 265 MN/mm<sup>2</sup> (HV 10 kg) as a result of the decomposition of bainite and M-A constituents leading to precipitation hardening strengthening by Mo and Cr alloy carbide formation in a ferritic matrix. It was observed that higher HT temperatures lead to lower hardness values, probably due to precipitate coarsening and further eventual solution. The observations for the 0.2 % YS coincide with the above hypothesis, as the 620 °C treated sample also leads to a maximum stress value, varying between 725 and 746 MPa in the longitudinal and transverse directions, respectively. This represents an increase of almost 10 % with respect to the as-rolled reference material. The TS, however, registers a slight decrease of around 2.5 % with respect to the as-rolled steel. This, however, has little influence, since the 0.2 YS/TS quotient still renders a value that confers the optimum ratio for static structural applications among all the analyzed conditions.

On the other hand, the material that exhibits the optimum combination of cold ductility without excessive loss of strength is the 700 °C HT stock. The values obtained for the engineering uniform and total elongation gave average longitudinal and transverse results of 15 % and 22 %. Due to the low



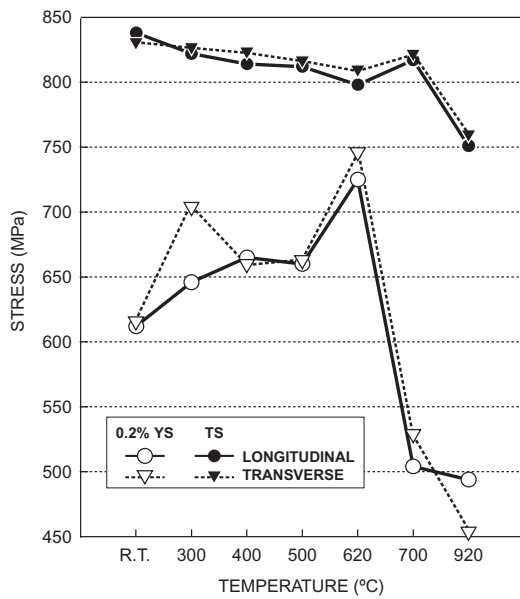
**Figure 5.** Engineering tensile test curves in the longitudinal and transverse directions for the different HT analyzed: a) 300 °C, b) 400 °C, c) 500 °C, d) 620 °C, e) 700 °C and f) normalized at 920 °C . It has been included the numerical summary of the most relevant engineering tensile properties together with a graphical enlargement window of the elasto-plastic transition in order to facilitate identification of the yield point phenomenon.

*Figura 5. Curvas de tracción ingenieriles en probetas longitudinales y transversales para los diferentes tratamientos estudiados: a) 300 °C, b) 400 °C, c) 500 °C, d) 620 °C, e) 700 °C y f) normalizada a 920 °C. Se incluyen dos ventanas, una expresa los resultados numéricos mas relevantes y la otra es una ampliación de la transición elasto-plástica que facilita la observación del fenómeno de fluencia en frío.*

anisotropy, this heat treatment may be considered the most ductile steel condition with a potential for structural applications among all the studied conditions.

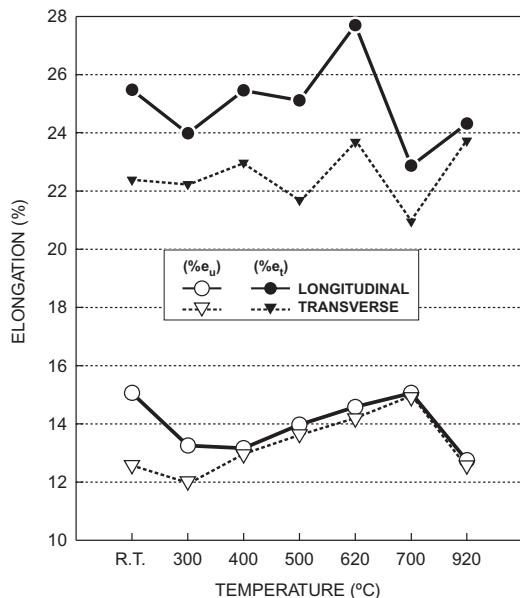
Finally, low temperature impact toughness is best

achieved by the 500 °C HT stock with values in excess to 90 J and 60 J for the energy absorbed in a longitudinal Charpy V-notch test at -20 °C and -40 °C, respectively, with an average 14 % improvement with respect to the as-rolled stock.



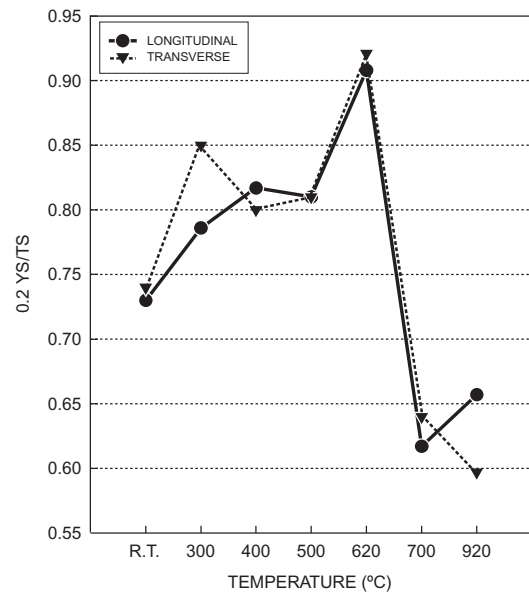
**Figure 6.** Effect of the HT temperature on the 0.2 percent offset yield stress and tensile strength values in the longitudinal and transverse directions.

*Figura 6. Efecto de la temperatura de tratamiento térmico sobre el límite elástico al 0,2 por ciento y la carga de rotura en dirección longitudinal y transversal.*



**Figure 7.** Evolution of both uniform and total engineering elongation as a function of the treatment temperature.

*Figura 7. Evolución de los alargamientos ingenieriles uniforme y total, en función de la temperatura de tratamiento térmico.*



**Figure 8.** Temperature dependence of the 0.2YS/TS ratio for the different HT analyzed conditions.

*Figura 8. Dependencia del cociente 0.2YS/TS con la temperatura para cada tratamiento térmico analizado.*

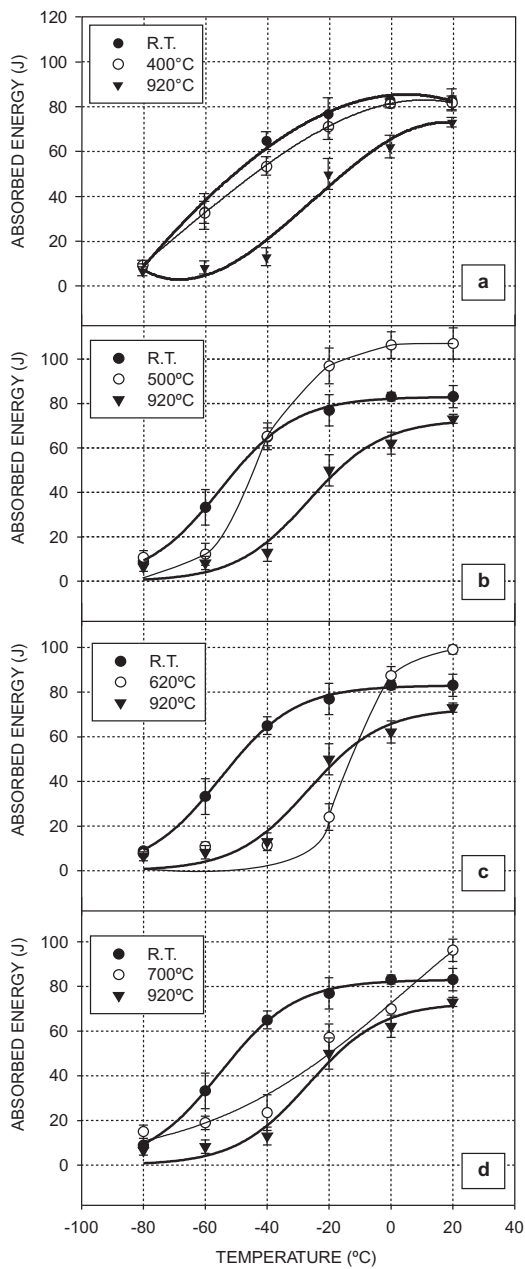
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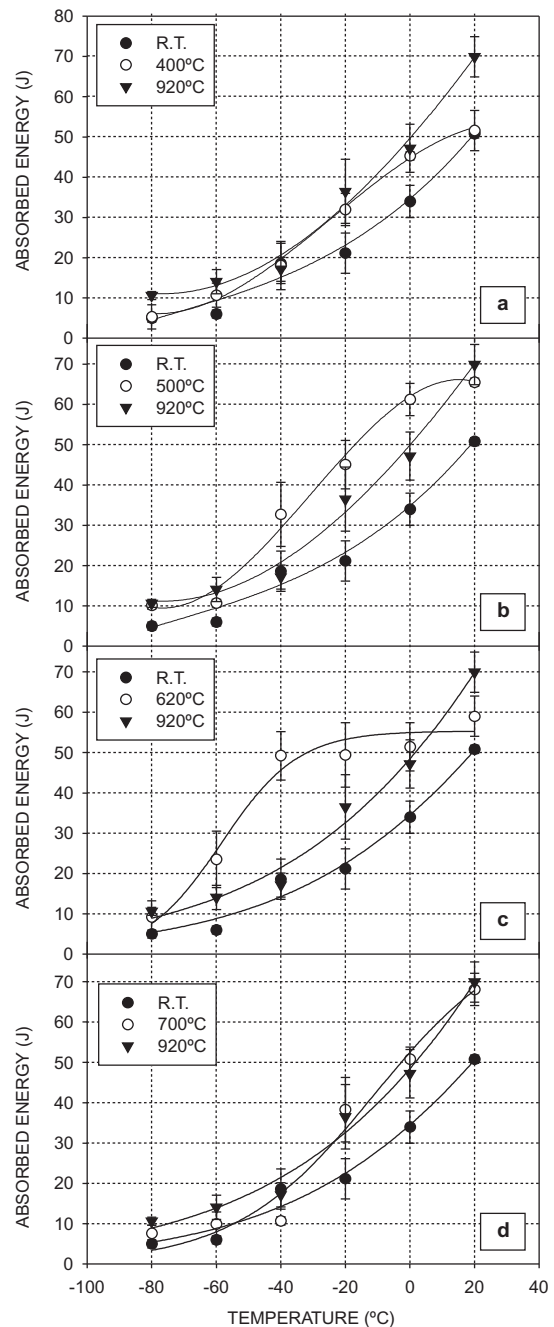
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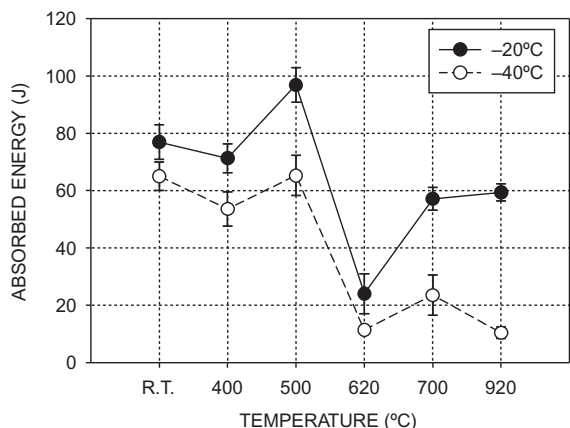
**Figure 9.** Temperature dependence of the longitudinal Charpy V-notch impact energy for the different steel conditions analyzed. The curves for the as-rolled material and after normalizing are included in plots a) - d) as upper and lower bounds of the material's response: a) 400 °C, b) 500 °C, c) 620 °C, and d) 700 °C treated samples.

*Figura 9. Dependencia de la energía absorbida en el ensayo Charpy con entalla en V en probeta longitudinal respecto de la temperatura de ensayo de los diferentes estados del acero estudiados. Las curvas correspondientes al estado laminado en caliente y normalizado se incluyen como límites superior e inferior aproximados de la energía absorbida para las muestras tratadas a: a) 400 °C, b) 500 °C, c) 620 °C, y d) 700 °C.*



**Figure 10.** Temperature dependence of the transverse Charpy V-notch impact energy for the different steel conditions analyzed. For the sake of comparison, the graph includes the as-rolled and normalized curves corresponding to: a) 400 °C, b) 500 °C, c) 620 °C, and d) 700 °C.

*Figure 10. Dependencia de la energía absorbida en el ensayo Charpy con entalla en V en probeta transversal respecto de la temperatura de ensayo de los diferentes estados del acero estudiados. Para facilitar su comparación se incluyen las curvas correspondientes al estado laminado en caliente y normalizado para: a) 400 °C, b) 500 °C, c) 620 °C, y d) 700 °C.*



**Figura 11.** Summary of the Charpy V-notch test values at -20 °C and -40 °C in the longitudinal direction for the as-rolled strip and for each HT studied.

*Figura 11. Resumen de los datos del ensayo Charpy a -20 °C y a -40 °C en probeta longitudinal del acero en estado bruto de laminación en caliente y para cada tratamiento térmico estudiado.*

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