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ESTRUCTURES BAINÍTIQUES EN ACERS HSLA DE BAIX CONTINGUT EN CARBONI: INFLUÈNCIA DEL CONTINGUT EN MICROALEANTS I DEL TRACTAMENT TÈRMIC SOBRE L'ESTRUCTURA I PROPIETATS

Memòria presentada per **Silvia Illescas Fernández** per a optar al grau de Doctor per la Universitat de Barcelona sota la direcció del Professor Josep Mª Guilemany Casadamon i del Professor Javier Fernández González.

Barcelona, Maig 2007

III.5. PROPIETATS MECÀNIQUES DE LES ESTRUCTURES BAINITÍQUES-FERRITA ACICULAR

III.5.1. ESTUDI DE LES PROPIETATS MECÀNIQUES

S'han determinat les propietats mecàniques de cada una de les estructures caracteritzades en l'apartat anterior amb la finalitat de comparar entre els diferents tractaments tèrmics i acers, i així poder establir la relació composició-estructura-propietats.

Els resultats obtinguts per a la propietats mecàniques de les estructures de bainita-ferrita acicular obtingudes per als acers 16MnNi4 i 16Mn4 es recullen en l'article "Influence on the vanadium content in the mechanical properties of low carbon content HSLA steels" presentat en la revista Materials Science and Engineering A Al Febrer del 2007, i pendent d'acceptació.

En aquesta publicació es mostren els resultats de duresa, tenacitat a l'impacte i resistència a la tracció per a cada mostra estudiada, i s'arriba a establir una relació dels valors obtinguts en les propietats mecàniques amb la composició de cada acer i l'estructura final ja caracteritzada. Així es pot observar la influència del contingut en vanadi sobre la formació de ferrita acicular el que comporta uns menors valors de duresa i resistència en l'acer 16MnNi4 de menor contingut en Vanadi.

A més s'observen diferents valors de propietats entre bainita superior i inferior, observant-se com per exemple per a les temperatures de tractament per sota de 500ºC, en les que s'obté bainita inferior, el valor de duresa del material presenta majors valors que per als tractaments que generen bainita superior.

S'han trobat les condicions òptimes de tractament tèrmic per a l'obtenció de les millors propietats mecàniques en els dos acers. Aquestes condicions són: 400ºC 30 min per a l'acer 16MnNi4 (V+Nb), i 450ºC 60 min per a l'acer 16Mn4 (V). La Taula 1 recull les esmentades propietats.

	HVN	R (MPa)	LE (MPa)	A (%)	Tenacitat: Tª Assaig (ºC)	Tenacitat: $E_{absorbida}$ (J)
16MnNi4, 400ºC 30 min	235	593	428	25	20 0 -20	193 154 122
16Mn4, 450ºC 60 min	208	545	378	32	-10 -50	216 110

Taula 1. Propietats Mecàniques per a les condicions de tractament òptimes per a l'acer 16MnNi4 i 16Mn4.

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Abstract: Two HSLA steels that present the same composition except in microalloyed content were studied. The purpose of the study was to determine the influence of the V in the microstructure and the mechanical properties of bainite in each of the steels. For this reason, standard tests were conducted to determine hardness, toughness, tensile and yield stress of the different bainite-acicular ferrite structures in both steels.

INFLUENCE OF THE VANADIUM CONTENT ON THE MECHANICAL PROPERTIES OF LOW CARBON CONTENT HSLA STEELS

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Abstract

Two HSLA steels that present the same composition except in microalloyed content were studied. The purpose of the study was to determine the influence of the V in the microstructure and the mechanical properties of bainite in each of the steels. For this reason, standard tests were conducted to determine hardness, toughness, tensile and yield stress of the different bainite-acicular ferrite structures in both steels.

Keywords: HSLA steel, Bainite, acicular ferrite, mechanical properties.

1. Introduction.

In markets such as the automobile component sector there is a need to increase the strength of steel and, at the same time, to reduce its weight. The combination of strength and resistance to impact is also important [1]. High strength low alloy (HSLA) steel, with an excellent combination of strength, toughness and weldability has been developed to replace conventional medium carbon steels over the last two decades [2,3]. The most typical microalloying elements are titanium, niobium and vanadium for grain refinement and carbide precipitation hardening [4].

Bainite and acicular ferrite are formed in the same range of temperatures: below the temperatures where allotriomorphic ferrite and pearlite form, and above the martensite-start temperature. The mechanism of formation of these phases is still subject to discussion [5–9]. It is the same for both structures, the main difference between both phases being the nucleation site: bainite is nucleated at austenite grain boundaries, and acicular ferrite is intragranularly nucleated at the non-metallic inclusions present in the steel [10-11]. The nucleated ferrite of bainite forms sheaves of parallel plates with the same crystallographic orientation, while acicular ferrite microstructures lead to a chaotic arrangement of plates. In particular, low temperature, low acicular ferrite is characterized by fine-grained interlocking morphologies [5,10–14].

Previous studies on the factors enhancing acicular ferrite formation have indicated that a reduction of the austenite grain boundary surface per unit of volume favours the formation of acicular ferrite, due to a reduction of bainite nucleation sites [15, 16]. A similar effect is obtained by increasing the number of inclusions present in the steel [17, 18] leading to bigger ferrite platelets nucleated onto the former particles.

2. Experimental Procedure.

Two low carbon HSLA steels were studied. Table 1 shows the chemical composition of 16MnNi4 and 16Mn4 steels. The steels were supplied in the form of hot-rolled plate of 11mm thickness.

Three different types of samples were prepared: the first ones for light optical microscopy (LOM) from 10x10x20 in mm, others for tensile test in accordance to ASTM E8M-89 standard and the last ones for impact toughness test, following E23-88 standard. All the samples were analysed in a transverse plane.

The 16MnNi4 (V-Nb) and 16Mn4 (V) samples were austenitized at 1050ºC for 30 minutes and 1000ºC for 15 minutes receptively, subsequently bainite was formed by immersing the samples in a molten salts bath (KNO_3 / $NaNO_3$, 1:1) at holding temperatures between 350ºC and 500ºC for times ranging from 30 to 60 minutes.

The bainitic structures were observed by LOM (OLYMPUS BH2-UMA), and SEM (JEOL 5510). The LOM and SEM observations were carried out in etched samples with 5%picral to reveal the bainitic structure. The samples were observed in a transverse plane.

Micro-hardness measurements were performed in accordance with ASTM 384-89 (UNE-EN ISO 6507-2) standard with a MATSUZAWA MXT-1 microindenter on a polished surface. The 0.2 percent offset yield stress and the tensile strength were determined according to the ASTM E8M (EN 1002) standards with a universal tensile test machine model SHIMAZDU AUTOGRAPH AG-100KNG at a constant ram velocity of $1mms^{-1}$ for the present work. Finally, toughness was evaluated using the Charpy test ASTM A370-8a (E23-88, EN 100045). A Néstor impact test machine 300 J type C was used.

3. Results and Discussion.

The starting materials were two hot rolled plates with a ferrite plus pearlite microstructure. It is noted that for both steels in the as-received condition a heavily pearlitic banding was observed in both longitudinal and transverse planes (Figure 1).

Bainitic plus acicular ferrite structures after the specified heat treatments were obtained. The micrographs in Figure 2 show the microstructure with upper and lower bainite with the presence of acicular ferrite for each steel.

The volume fractions of the constituents found after heat treatment were evaluated by the point count method and the results for the volume fraction of acicular ferrite are presented in Figure 3. Six micrographs were used to determine the acicular ferrite (%vol). The standard deviation is shown by error bars.

The graph shows that the V steel presents a higher ferrite acicular content than the V+Nb steel, for all the heat treatments. This can be attributed to higher vanadium content in the V steel, since this element promotes the formation of acicular ferrite [19].

Figure 4 shows the microhardness values for the two steel compositions after isothermal treatments for 30 and 60 minutes.

This figure shows a decrease of microhardness with increasing isothermal temperature. This result is consistent with the development of lower bainite at temperatures below 500ºC as reported in a former work [20].

The graph also shows that the hardness values in the V bearing steel are somewhat lower than in the V+Nb steel. The result is consistent with the observations by Babu and Bhadeshia [21] for which an initial low austenite grain size favours the development of a high volume fraction of lower bainite due to the high number of sites available for nucleation. It was observed that reciprocally high V contents enhance the occurrence of a higher proportion of acicular ferrite, thus producing a softer material.

It is a common feature that a peak in hardness is reached for both steels with 60 minutes of treatment. A convergence in hardness values at 500ºC is observed for the two steels.

The hardness values obtained for the V+Nb steel are independent of the time of heat treatment. However the results at 30 minutes and 60 minutes of heat treatment are different for the V steel.

Charpy tests were carried out to measure toughness. First, the ductile-brittle curve was constructed for the initial material in the as-rolled condition. Figure 5 shows the ductile-brittle curve for each steel and allows us to identify the ductile-brittle temperature transition.

Impact Fracture testing was conducted to ascertain the fracture characteristics of the material. The Charpy V-notch test (CVN) technique was selected to measure the impact energy of the as-received material and also after heat treatments at different temperatures.

Figure 5 shows the temperature dependence of the Charpy V-notch impact energy for both the V+Nb and the V steel in the as-rolled condition. They both exhibit high values of absorbed energy in the ductile region: 225 J for the V+Nb steel and around 175 J for the V steel. Also the V steel shows a clear ductile-brittle transition with fully brittle behaviour at –50ºC and below, with absorbed energy of around 40 J. The transition between both types of behaviour occurs at –40ºC approximately.

As for the V+Nb steel, the curve does not present the classical “S”-shape. It presents an upper shelf and an intermediate shelf, both corresponding to high absorbed energies in the ductile region, at test temperatures above –60ºC.

Charpy tests for the bainitic-ferritic acicular structures were also performed once ductile-brittle transition had been established. The V+Nb steel was tested at -20, 0 and 20ºC, and the V steel at -50, -20 and -10ºC. The results obtained for the different heat treatments are presented in Figure 6.

The energy values in the steel V+Nb do not present differences between the different heat treatments or even the different test temperatures.

A big difference in absorbed energy values at –20ºC and –10ºC was observed for the V steel. However there were no differences when different heat treatments were compared. Bigger differences in absorbed energy versus heat treatment were shown with longer times (60 minutes).

The absorbed energy decreased in the V+Nb steel for all the heat treatments when compared with the as-rolled steel (for example, at 20ºC the absorbed energy is a 15% lower than the initial material). However, for the V steel at -10ºC the absorbed energy increased by 24% with respect to the as-rolled material tested at the same temperature.

In general, a decrease in the absorbed energy values around 14% was also observed for all the samples and for both steels when the time of heat treatment was increased. This is due to the growth of carbides present in the bainitic samples. An exception to this behaviour is the V steel treated at 450ºC, which shows an absorbed energy independent of the time of heat treatment.

Comparing the results at different test temperatures, it can be seen that absorbed energy decreases when the test temperature increases for V+Nb steel, but this decrease is low as in the case of the ductile-brittle curve for the as-rolled steel. The samples that have been obtained at 500ºC show a bigger decrease in absorbed energy values when the test temperature increases (due to the high content of upper bainite). However, the decrease is low and gradual in the samples treated at 350ºC, which should be related to a structure with a high content in lower bainite, which is a constituent with a high degree of toughness.

An abrupt decrease in the absorbed energy was observed at -20ºC for the V steel, obtaining similar values in the samples tested at lower temperature. The steel in all the heat treatments presents worse behaviour at -20ºC, but a higher absorbed energy at -50ºC, comparing with the as-rolled material.

The treated samples show a ductile-brittle fracture as can be observed in Figure 7.

The standard tensile test was made for the initial material and the different bainitic samples. The results for the initial material are shown in Table 2. Figure 8 represents the results obtained for the bainitic samples.

In the V+Nb steel, the tensile strength and yield stress increased and the total elongation decreased for all the heat treatments. Moreover, an increase in the time of heat treatment produced better ductility and worse yield stress, except for the sample at 350ºC. The condition of maximum ductility after heat treatment for the V+Nb steel is for the heat treatment at 500ºC where the elongation values are similar to those obtained for the as-rolled steel and the tensile strength and the yield stress are better, showing an improvement in the behaviour of static toughness (total area below the tensile curve).

This improvement in tensile strength and yield stress is smaller for the V steel than for the V+Nb, and the ductility is practically constant with the heat treatment. The maximum plastic deformation strength was obtained for the sample treated at 350ºC during 60 minutes and in agreement with a low decrease of the elongation.

The decrease of ductility after the heat treatment is greater for the V+Nb steel than for the V steel and generally the samples after heat treatment show more toughness for the V steel than for the V+Nb steel.

The resistance values between low and high temperatures are different in the V+Nb steel, which presents a Vanadium content of 0.026% wt due to the presence of upper bainite at temperatures higher than 500ºC, as observed in previous studies [20]. This upper bainite, due to its carbide distribution, leads to lower resistance in steel than where there is lower bainite. Also, this is due to the high acicular ferrite content observed at high temperatures, as is shown in Figure 3.

For the V steel with a 0.0513% wt Vanadium content no significant differences were found. The values were always lower compared to the V+Nb steel. He and Edmonds [19] demonstrated that vanadium additions may enhance the acicular ferrite microstructure. This acicular ferrite improves toughness but promotes a decrease in the resistance of the steel.

4. Conclusions.

In accordance with the information given above, the following conclusions may be drawn:

(a) The presence of Vanadium in steel promotes the formation of acicular ferrite.

(b) The V steel which presents high acicular ferrite content presents low values in hardness and in tensile strength, but with an improvement in toughness.

(c) The V+Nb and V steels present different ductile-brittle curves. The ductile-brittle temperature transition for V+Nb steel is higher than for V steel. However, the total amount of absorbed energy is higher than for the V steel.

(d) The Charpy test shows a decrease in absorbed energy values for V+Nb steel and an increase for V steel compared to the initial as-rolled steels. A decrease of this energy value was also observed when the time of heat treatment was increased.

(e) The presence of upper bainite at temperatures of 500ºC leads to a decrease in strength, compared with lower bainite.

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	C	Mn	Si	Cr	Ni	Mo	V	Nb	Al	Ti	Cu	P	S	N
16MnNi4 (V-Nb)	0.165	1.11	0.23	0.02	0.34	0.001	0.026	0.0135	0.0244	0.0014	0.184	0.015	0.0022	0.0059
16Mn4 (V)	0.166	1.24	0.16	0.02	0.02	0.001	0.051	0	0.0309	0	0.186	0.015	0.0151	0.0027

Table 1. Chemical composition of studied steels (wt.%)

	(V-Nb) steel			(V) steel		
	TS (MPa)	YS (MPa)	A (%)	TS (MPa)	YS (MPa)	A (%)
Initial Material	548	385	34	544	385	30

Table 2. Results of the standard tensile test for the as-rolled V+Nb and V Steels.

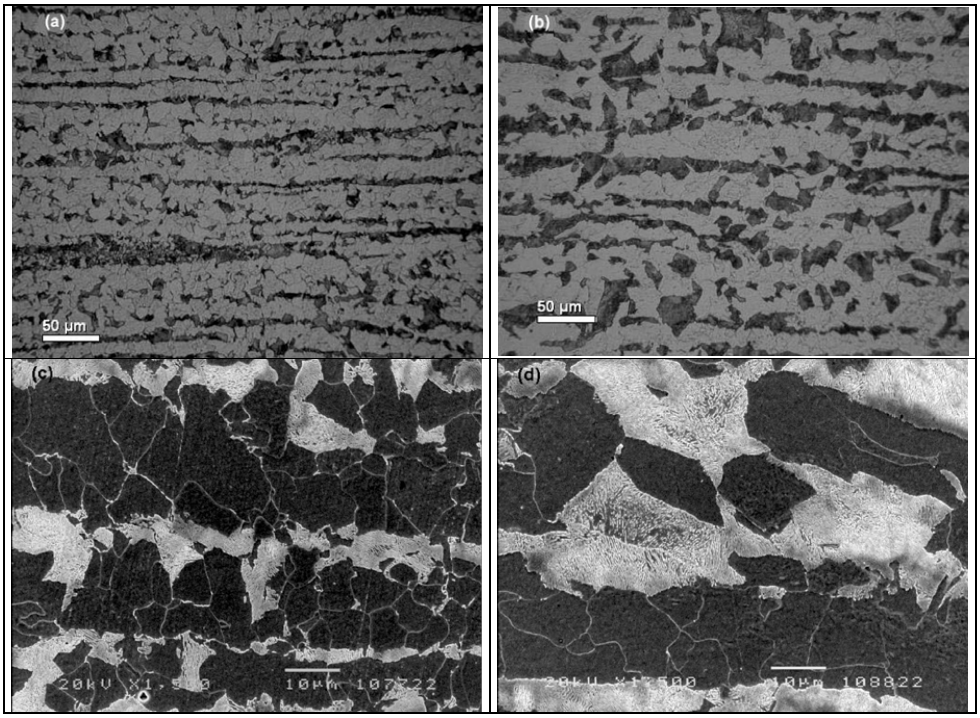

Figure 1. Initial microstructure in the transversal plane: Ferrite plus pearlite microstructure showing heavily pearlitic banding. (a) LOM micrograph of the V+Nb steel (b) LOM micrograph of the V steel, (c) SEM micrograph of V+Nb steel, (d) SEM micrograph of V steel.

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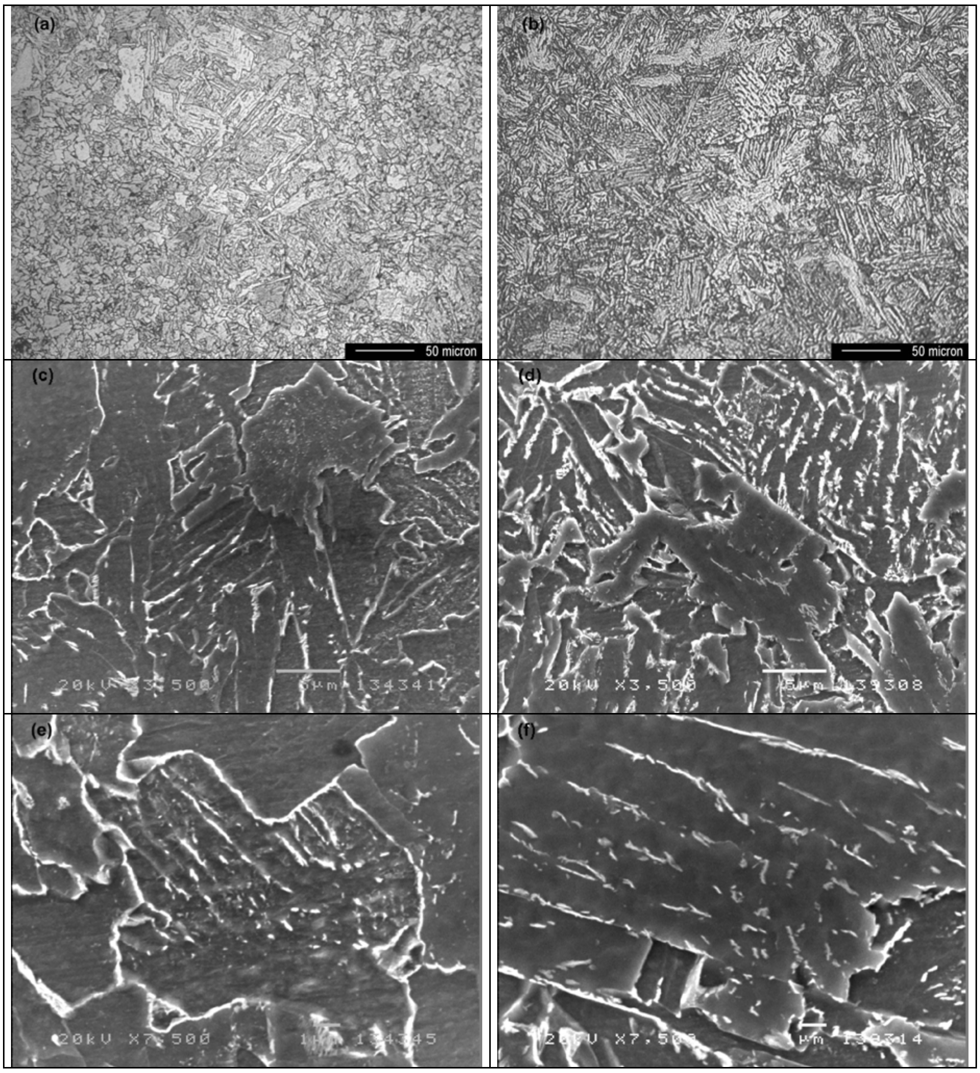

Figure 2. Microstructures with upper and lower bainite with the presence of acicular ferrite: (a) LOM micrograph of V+Nb steel treated at 450ºC during 30 min, (b) LOM micrograph of V steel treated at 450ºC during 30 min, (c) and (e) SEM micrograph of the V+Nb steel treated at 450ºC during 30 min, (d) and (f) SEM micrograph of V steel treated at 450ºC during 30 min.

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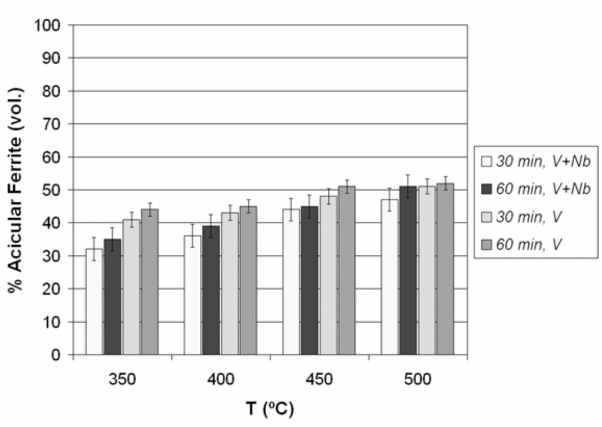

Figure 3. Volume fraction of acicular ferrite for the different heat treatments and steels.

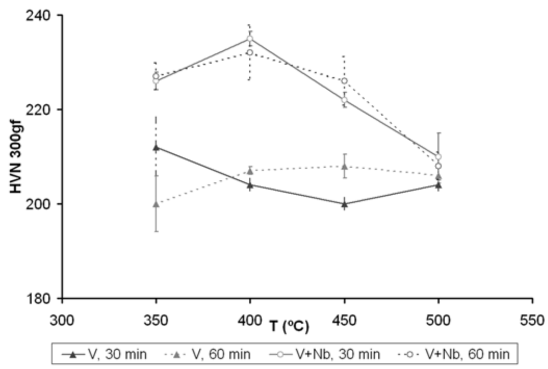

Figure 4. Evolution of the Microhardness values at 300gf after different heat treatments.

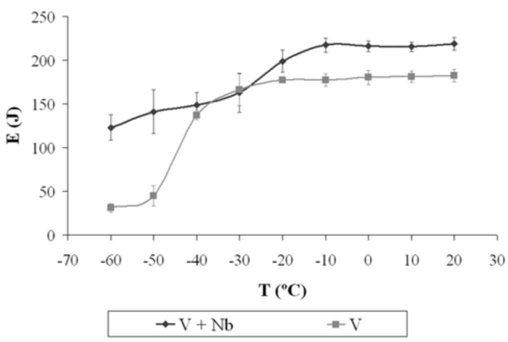

Figure 5. Ductile-brittle curve for both steels.

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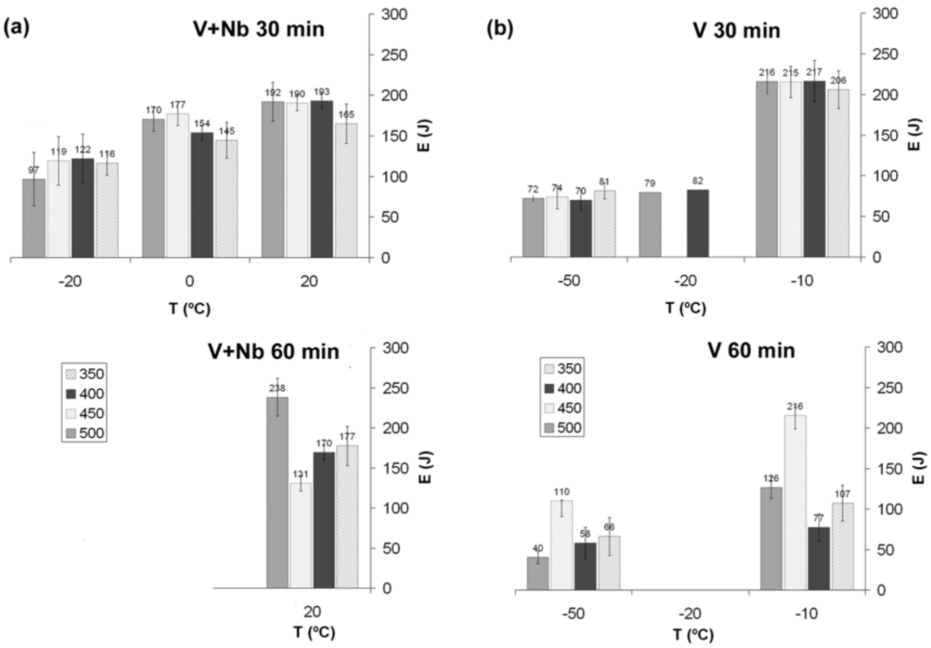

Figure 6. Charpy test Results. (a) V+Nb Steel and (b) V Steel.

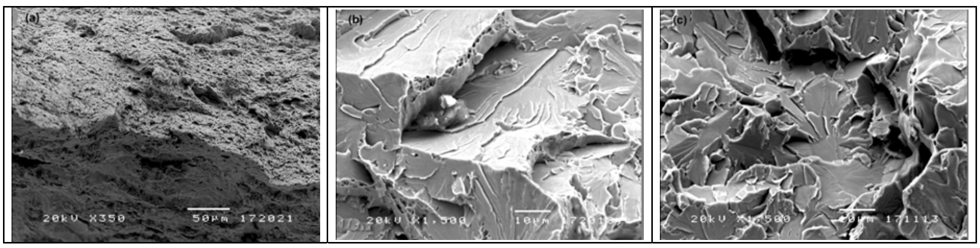

Figure 7. SEM micrographs showing ductile-brittle fracture: (a) Ductile fracture near the V-notch for V+Nb steel treated at 350ºC during 30 minutes at test temperature of 20ºC, (b) Ductile-brittle fracture for V+Nb steel treated at 350ºC during 30 minutes at test temperature of 20ºC, (c) Brittle fracture for V steel treated at 350ºC during 30 minutes at test temperature of -10ºC.

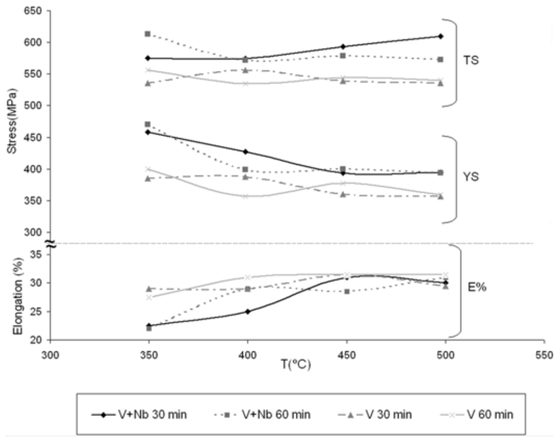

Figure 8. Tensile Strength (TS), Yield Stress (YS) and Elongation (E%) results of the standard tensile test for the bainitic structures of V+Nb and V Steels.

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III.5.2. CONCLUSIONS PARCIALS

A partir dels resultats obtinguts per a la determinació de propietats mecàniques de les estructures bainítiques en cada acer, s'han obtingut les conclusions parcials:

1. La presència de Vanadi en la composició de l'acer afavoreix la formació de ferrita acicular. D'aquesta manera les estructures obtingudes per a l'acer 16Mn4 (V) que presenten un major contingut en V, i per tant un major percentatge en ferrita acicular, presenten menors valors en un 12% de duresa i resistència mecànica, encara que s'obté un 12% de millora en la tenacitat a l'impacte.

2. Les mostres amb estructura de bainita superior presenten valors de duresa i resistència a la tracció menors (un 8% i un 6% respectivament) que les que contenen bainita inferior. A més la bainita superior presenta un millor comportament tenaç (un 14% comparant els valors d'energia absorbida per a les mostres tractades a 500ºC) respecte a la bainita inferior, a causa del marcat caràcter acicular grosser dels seus carburs i la seva globulització per permanència prolongada.

3. Les corbes de transició dúctil-fràgil obtingudes per al material brut de laminació, mostren una transició més marcada (d'un 83% entre els valors en la zona dúctil i els de la zona fràgil) per a l'acer 16Mn (V), mentre que per a l'acer 16MnNi4 (V+Nb) aquesta transició no és tan gran (d'un 57%). A més s'observa com l'acer 16MnNi4 (V+Nb) presenta majors valors d'energia absorbida (16% per a la zona dúctil i 65% per a la zona fràgil).

4. Respecte al material en estat inicial (laminat), les mostres per a l'acer 16MnNi4 (V+Nb) després de tractament tèrmic mostren un descens de fins al 15% en el valor de l'energia absorbida en l'assaig d'impacte Charpy, mentre que per a l'acer 16Mn (V) es dóna un increment de fins al 23% en la seva magnitud.

5. A partir dels assajos de tenacitat Charpy, per a l'acer 16MnNi4 (V+Nb) s'observa un descens de l'energia absorbida al disminuir la temperatura d'assaig que no supera el 13%. Tots els valors són indicatius de ruptures de tipus dúctil, tal com es corrobora per observació microscòpica de les mostres assajades. Per a aquest acer es pot discernir entre dos comportaments: per un costat les mostres tractades a 500ºC experimenten un descens de l'energia absorbida més abrupte per efecte de la presència de bainita superior, mentre que en els tractaments a menors temperatures, com a 350ºC, la disminució d'energia absorbida és lleu i gradual, indicatiu d'una estructura amb abundància del constituent tenaç de bainita inferior.

6. A partir dels assajos de tenacitat Charpy, per a l'acer 16Mn4 (V) s'observa com per als assajos a –10ºC els valors d'energia obtinguts superen en un 24% als obtinguts per al material de partida. A més hi ha una caiguda abrupta en els valors d'energia absorbida per als assajos a –20ºC que resulten ser semblants als obtinguts en els assajos a –50ºC. Aquestos valors obtinguts a –20ºC i –50ºC formen part de la transició dúctil-fràgil de l'acer en estat de recuit isotèrmic i suposen una caiguda més abrupta que en el cas del material brut de laminació (a –20ºC el material inicial presenta millors valors que el tractat tèrmicament, mentre que a –50ºC l'energia absorbida és major en el cas de les mostres sotmeses a tractament tèrmic).

7. En tots els casos, l'efecte que genera el temps de tractament tèrmic sobre la tenacitat del material, indica com l'augment en aquest valor repercuteix en una disminució del valor d'energia absorbida (arribant a ser del 40%), és a dir, un empitjorament del comportament tenaç, per efecte d'engrossiment de carburs a l'augmentar el temps de tractament tèrmic.

8. Com a mitjana, els materials després de tractament tèrmic són un 7-12% més tenaços en el cas de l'acer 16Mn4 (V) que en el cas de l'acer 16MnNi4 (V+Nb).

9. Els resultats de l'assaig de tracció per a l'acer 16MnNi4 (V+Nb) mostren que tots els tractaments augmenten al voltant d'un 2-10% els valors de resistència i límit elàstic, i disminueixen en un 10 % l'allargament total a ruptura, observant-se el màxim augment per al tractament a 350ºC durant 60 minuts. En el cas de l'acer 16Mn4 (V) les millories observades resulten molt tènues (1-2%), així com la ductilitat a penes es veu modificada, encara que en coincidència amb l'acer 16MnNi4 (V+Nb) es troba que el tractament tèrmic que aporta un major increment és també el realitzat a 350ºC durant 60 minuts. Cal destacar que la pèrdua de ductilitat després de tractament tèrmic és major en l'acer 16MnNi4 (V+Nb) que en l'acer 16Mn4 (V).

10. Sospesant el conjunt de propietats mecàniques estudiades, s'arriba a establir que l'acer 16MnNi4 (V+Nb) presenta una milloria del 7-12% a comportament a tracció respecte a l'acer 16Mn4 (V), mentre que en el cas de la tenacitat, s'observa com a pesar que la corba de transició dúctil-fràgil presenta valors més elevats per a l'acer 16MnNi4 (V+Nb), l'efecte del tractament tèrmic ocasiona que sigui l'acer 16Mn4 (V) el que presenta els valors més elevats (7-12%) dels assajos realitzats.

11. S'estableixen com a condicions òptimes de tractament per a l'obtenció de l'estructura amb millor combinació de propietats: 400ºC 30 min per a l'acer 16MnNi4 (V+Nb), i 450ºC 60 min per a l'acer 16Mn4 (V).