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Retained Austenite in the C.G.H.A.Z. of 0.1 wt% C Si-alloyed Steels

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Abstract. This investigation deals at first with the existence of retained austenite in the coarse-grained heat-affected zone (CGHAZ) of 0.1 wt% C steels, and subsequently with the mechanism of deterioration of the CGHAZ toughness by retained austenite. The study is achieved by X-ray diffractometry, Mössbauer spectrometry, transmission electron microscopy, instrumented Charpy V, CTOD and impact tensile tests. The work demonstrates the general increase of the concentration of carbon, and therefore of the stability of retained austenite with the 0.05 to 0.5 wt% silicon content of only Ti-microalloyed steels. These correlations are explained by the hindering effect of silicon on the formation of carbides. Otherwise, and according on the one hand to the test temperature, and on the other hand to their morphology, strength and localization, the particles of retained austenite are more or less completely transformed into martensite during an impact because of their stress raising power. The influence of the attendant stress concentrations on the fracture mechanism is further demonstrated with the consequence of the interpretation of the deleterious effect of retained austenite on the CGHAZ toughness. Finally, the whole of these results lead to the choice of an optimum chemical composition that is proved to ensure a favourable microstructure for the CGHAZ toughness of 0.1 wt% C microalloyed steels.

1. INTRODUCTION

At least in structural steels, the most brittle part of a weldment is commonly the coarse-grained heat-affected zone (C.G.H.A.Z.) [1]. In accordance with this fact, the authors have already investigated the effects of welding energy on the C.G.H.A.Z. microstructure and toughness of industrial heats of 0.1 wt% C high strength low alloyed (H.S.L.A.) steels with a silicon content between 0.270 and 0.500 wt% [2-4]. These works have established the predominant and harmful effect of retained austenite on toughness. The comparison of this result with the well-known stabilization of austenite by high levels of silicon close to 2 wt% in bainitic steels [5, 6] suggests the importance of a study of the effect of a reduction of the Si-content of H.S.L.A. steels under its usual 0.500 wt% value.

The aim of this paper is twofold. First, it answers the previous question by investigating the effect of the chemical composition, and more especially of a silicon content ranging from 0.05 to 0.5 wt% on the C.G.H.A.Z. toughness and microstructure of 0.1 wt% C microalloyed steels. Secondly, it elucidates the mechanism of action of retained austenite.

Finally we propose a judicious chemical composition that leads to an optimum in microstructure, and therefore in toughness for the C.G.H.A.Z. of 0.1 wt% C steels.

2. EXPERIMENTAL

Table 1 gives the chemical composition of the alloys. The 80-mm-thick plates of steels A and B were manufactured by controlled rolling and normalization treatments. On the contrary, the 12-mm-thick plates of steels C through F were experimental materials. They were first controlled rolled and then accelerated cooled at 3°C/s between 800°C and 550°C. These laboratory alloys are only Ti-microalloyed and differ only by their silicon content that ranges from 0.05 to 0.5 wt%.

The samples were machined in the long transverse direction, and 2 mm below the surfaces for the peculiar case of the thickest plates A and B.

Table 1: Chemical composition (10^{-3} wt%) of the plates.

steel	C	Mn	Ni	Ti	Nb	V	N	Si
A	105	1446	423	15	28	32	4	505
B	111	1526	418	<3	34	<3	4.2	452
C	75	1577	469	13	<3	<3	1.5	50
D	89	1580	469	13	<3	<3	1.8	104
E	86	1598	477	11	<3	<3	2.1	244
F	89	1581	471	11	<3	<3	2.1	498

The steels were investigated after thermal cycles that simulate the coarse-grained heat-affected zone (C.G.H.A.Z.) thermal history. The samples were heated by passing a high-amperage current through them while using a Gleeble 1500 thermomechanical simulator. The thermal cycles were computed from the three dimensional heat-flow equations of Hannerz [7]. These cycles comprise a 400°C/s heating stage up to 1350°C , followed by a cooling stage defined by a cooling time (Dt) between 700 and 300°C . In the present work, the cooling time (Dt) depends only on welding energy. Dt ranges from 10 to 300 s which corresponds to an heat input between 0.3 and 10 kJ.mm^{-1} . Except for the samples of impact tensile test, the width of the simulated CGHAZ is close to 5 mm.

The mechanical properties were characterized by impact tensile, Charpy V toughness and CTOD tests. The dimensions of the CTOD specimens were $20 \times 10 \times 100 \text{ mm}$ with total notch and crack length of 10 mm including 2 mm fatigue crack.

The microstructural analysis included both X-ray diffractometry, thin foil ($40 \mu\text{m}$) transmission Mössbauer spectrometry, light, scanning (SEM) and thin foil transmission electron microscopy (TEM). Some measurements of chemical composition were further performed by means of an X-ray energy-dispersive (EDX) spectrometer and using the 30 nm -diameter probe of a scanning transmission electron microscope (STEM).

3. RESULTS AND DISCUSSION

3.1. Effect of chemical composition on CGHAZ toughness

Figure 1 displays the 28 J Charpy V transition temperature (T_k) of the steels in their various heat treated conditions. It is worthy to note: 1) the better resilience of experimental alloys C through F compared to

Figure 1: Evolution of T_k with welding energy for: a) steels A and B, b) steels C through F.

that of the industrial batches A and B; 2) the unusual improvement of toughness of the laboratory casts with increasing welding energy [1-4]; and 3) the higher CGHAZ toughness, the lower the silicon content of steels C through F. These features are the consequence of the CGHAZ microstructure [2-4].

3.2. Effect of silicon on CGHAZ microstructure

In agreement with its aim, this paragraph deals only with steels C through F that differ by their content in silicon.

3.2.1. Overall microstructure

As proved by table 2, and in close accordance with the results obtained with numerous 0.1 wt% C microalloyed steels, among which steels A and B [3-4], the increase of Dt brings about the evolution of the CGHAZ microstructure of the laboratory alloys from a mixture of martensite (M) and lath upper bainite (LUB) to a predominant acicular ferritic structure. Acicular ferrite (AF) is a form of upper bainite composed of heavily dislocated and equiaxed grains characterized by a length to width ratio lower than 4 (see Fig. 2). Moreover, representative micrographs illustrating the modification of the microstructure with Dt already have been published [4, 8]. Table 2 exhibits also a slight increment of hardenability with the silicon content.

Table 2: Optical evaluation of CGHAZ phase amounts (in vol%)

Steel	C				D				E				F			
	10	30	100	300	10	30	100	300	10	30	100	300	10	30	100	300
Dt	45	15	0	0	50	20	0	0	55	35	0	0	55	35	0	0
M	50	70	20	10	45	70	25	15	40	60	40	20	45	60	35	20
LUB	0	5	60	70	0	0	55	65	0	0	45	60	0	0	50	60
AF	5	10	20	20	5	10	20	20	5	5	15	20	0	5	15	20
PF ¹

a)

b)

Figure 2: Typical thin foil electron micrographs displaying the predominant microstructural constituent in steel F: a) lath upper bainite (Dt= 30s), b) acicular ferrite (Dt= 300s).

¹ PF: Proeutectoid ferrite

3.2.2. Retained austenite

The TEM investigations further establish the existence of retained austenite (γ) in upper bainite (Fig. 2). Figure 2 shows that the particles of retained austenite are elongated in the boundaries of lath upper bainite, whereas they adopt a blocky morphology in acicular ferrite. X-ray diffractometry and Mössbauer spectrometry already have been proved very valuable for estimating the volume fraction of retained austenite in 0.1 wt% C microalloyed steels [2-4]. Figure 3 displays volume fractions of retained austenite ($\gamma\%$) of up to 5% in steels C through F.

Figure 3: Effect of cooling time and of silicon content on the X-ray estimates of the volume fraction of retained austenite.

Figure 4: Evolution of the weight percentage of carbon in retained austenite with welding energy and Si-content of the alloy.

The stabilization of the particles of austenite is due essentially to their high carbon content ($(C\%)_a$) (Fig.4), and to compressive strains induced by the ferritic transformation of their surroundings. The present conclusion is deduced from Mössbauer and STEM-EDX analyses that establish the lack of partitioning of the substitutional elements between ferrite and austenite [4, 8]. Moreover, it is important to note the increase of the proportion of austenite with the silicon content at any Dt, and on the other hand, the evolution of the quantity of austenite with Dt. These features will now be explained by the effects of silicon on both the formation of carbides and the partition of carbon between ferrite and austenite:

a) During the reaction of austenitization, silicon in solution in ferrite hinders the precipitation of cementite [9] with the consequence of an increase of the number of carbon atoms diffusing from ferrite to austenite. Moreover, this interpretation agrees well with TEM observations which establish an enhancement of the propensity toward intragranular precipitation of Fe_3C within the laths of bainite with a decrease of the Si-content of the steels [4, 8]. In addition, for the highest Si-alloyed steels E and F, the carbon content and volume fraction of retained austenite increase concomitantly with cooling time up to 30 and 100s, respectively (see Fig.3 and 4). These observations arise from the enhancement of the diffusion of carbon from ferritic to austenitic areas because of the reduction of cooling rate.

b) On the contrary, figures 3 and 4 display simultaneous maxima of $(C\%)_a$ and of the volume fraction of retained austenite for the highest Si-alloyed steels E and F, but continuous decreases of these two parameters with an increase of Dt for the less Si-bearing alloys (A and B). Moreover, it is worthy to note that an increase of the silicon content brings about: 1) the shift of the maximum of $(C\%)_a$ toward high cooling times and 2) an increase of the volume fraction of retained austenite at any Dt. All these evolutions arise from the partial decomposition of retained austenite into pearlite which is the slower, the higher the silicon content [4, 8] because of the hindering of the Fe_3C precipitation by the atoms of silicon in solid solution in austenite. This effect of silicon has been noted by some authors in not 0.1 wt% C microalloyed steels [10]. Two mechanisms are proposed for this retardation. First, the silicon in austenite impedes the diffusion of carbon [9]. On the other hand, the diffusion-controlled growth of carbides is counteracted by the presence of an Si-enriched layer around the precipitate nuclei [11,12].

3.3 Effect of retained austenite on toughness

3.3.1. Predominant action of retained austenite

Among the numerous parameters a priori likely to act on toughness (N interstitial content, prior austenite grain size, ferritic grain size, types of microstructural constituents...) the nature of the microstructure is the only one which exerts a significant influence in the present materials [2-4]. In this regard, figure 5a agrees with the usual reduction of the CGHAZ toughness due to the evolution of the microstructure from martensite to lower bainite and finally to lath upper bainite[11]. In addition, the beneficial effect for toughness of acicular ferrite is further noted for steel B at the longest cooling time. This latter feature has already been explained by the diminution of the effective grain size [2-4].

Figure 5: Correlation of T_k with the CGHAZ microstructure of: a) steels A and B, b) steels C through F.

However, it is worth emphasizing that the previous interpretation contradicts the parallel evolutions of T_k (Fig. 5b) and of the overall CGHAZ microstructure of steels C through F. This discrepancy arises

Figure 6: Effect of the volume fraction of retained austenite on T_k : a) ferritic side plates structure, b) acicular ferrite.

from the greater influence of retained austenite. The data obtained with the present and others 0.1 wt% C microalloyed steels lead to an harmful effect of retained austenite on T_k that amounts to $+14\text{ }^{\circ}\text{C}/\gamma\%$ in a predominantly ferritic side plates structure (martensite and/or lath upper bainite), but only to $+8\text{ }^{\circ}\text{C}/\gamma\%$ in prevalence of acicular ferrite (Fig. 6). For instance, the ductile-brittle transition temperature of alloys C through F is equal to $-50\text{ }^{\circ}\text{C}$ at the lowest heat input (see Fig. 1b). This constancy agrees with the only 3 percent estimate of the volume fraction of retained austenite in these four CGHAZ. In the same way, for the less Si-alloyed steels C and D, the continuous decrease of T_k with welding energy (Fig. 1b) correlates well with the concomitant diminution of the volume fraction of retained austenite (Fig. 3). Furthermore, this observation indicates a less harmful effect of pearlite than of austenite on toughness. By way of contrast, the increase of the volume fraction of retained austenite (Fig. 3) does not damage the toughness of the highest Si-alloyed steels E and F (Fig. 1b). In that case, the deleterious effect of austenite is offset by the increase of the volume fraction of acicular ferrite (Table 2). Moreover, it is noteworthy that this beneficial influence of acicular ferrite results from both a decrease of the effective grain size, and the smaller effect of retained austenite on toughness in acicular ferrite.

3.3.2. Mechanism of action of retained austenite

The instrumented Charpy V and CTOD data show that retained austenite promotes both crack initiation and crack propagation (see Fig. 7 and 8). A deeper understanding of both the effect and the behaviour of retained austenite during cracking is achieved by means of low temperature impact tensile tests which take the advantages of both a 10 mm-long heat treated gauge for the microstructural studies, and of an uniaxial deformation. Table 3 gives the results of this investigation together with the overall microstructure of the specimens. It is worth underlying that due to the type of upper bainite (LUB or FA), these samples differ by the morphology of the particles of retained austenite or more generally of MA constituent². They differ further by the size and the carbon content of these particles of second phases. Moreover, it is important to note that the microhardness of the MA constituent seems to increase linearly with its carbon concentration [14].

Figure 7: Effect of retained austenite on fracture initiation in steel A. E_a is the energy for crack initiation.

Figure 8: Effect of retained austenite on the energy of fracture propagation at $-30\text{ }^{\circ}\text{C}$ in steel A.

As illustrated by figure 9, the TEM observations establish a complete transformation of retained austenite into martensite during the $-196\text{ }^{\circ}\text{C}$ impact tensile tests. In accordance with the literature data [15], the martensite is twinned in steels A and B because of the higher than 0.4 wt% carbon content of

² The "MA constituent" appellation is frequently used so as to name retained austenite and its various products of transformation (martensite and carbides).

Table 3: Impact tensile tests data and microstructure of MA particles.

Steel	A	B	E	$\Sigma \%$ (3)	Fracture surface	Cracking mechanism
Dt (s)	100	300	100			
Overall microstructure (vol %)	50 LUB 45 AF 5 PF	100 LUB	45 LUB 40 FA 15 FP			
$\gamma\%$ before impact	7	8	3.2			
$(C\%)_a$	0.6	0.7	0.3			
Hv ₁ ⁽⁴⁾	400	500	200			
microstructure of the MA constituent after the -196°C test	twinned martensite (TM)	twinned martensite (TM)	lath martensite	0	cleavage facets	decohesion at MA interfaces
microstructure of the MA constituent after the -10°C test	<ul style="list-style-type: none"> • larger or interpackets particles = TM • smaller ones = γ 	twinned martensite (TM)	thin austenite	75	ductile dimples	decohesion at MA interfaces + MA fracture

a)

b)

Figure 9: TEM aspect of the particles of MA constituent after a -196 °C impact tensile test in : a) steel B (Dt= 300s), b) steel E (Dt=100s).

primary austenite. Alternatively, and in agreement with its only 0.3 wt% carbon content, the austenite of steel E transforms into lath martensite.

³ $\Sigma\%$: reduction of area at fracture.

⁴ The Hv₁ microhardness of particles of retained austenite is deduced from its linear increase with carbon content [14].

Otherwise, the Mössbauer and TEM investigations with samples of steel A demonstrate that retained austenite does not transform only by a liquid nitrogen soaking. These results prove a mechanical origin for its martensitic transformation during the -196°C impact tensile tests.

In opposition with its completeness at -196°C , the martensitic transformation of retained austenite is only more or less partial during the -10°C impact tensile tests. These variations of stability have again a mechanical origin.

Indeed, and at both test temperatures, the destabilization of austenite arises from the stress concentrations at the particles interfaces. This influence is suggested by the SEM studies of longitudinal sections of the tensile test samples that show the existence of decohesions situated in the matrix and at the MA particles interfaces (Fig.10). Moreover, this stress concentration depends on both the strengths of the particles and of the matrix, and the morphology and localization of the MA particles. First, the stress concentration increases with the resistance of a particle to plastic deformation, and therefore with its size and carbon content [16]. This relationship is proved by the results obtained at -10°C with steels A and E that exhibit a like microstructure. The thin and soft particles of austenite in steel E remain untransformed contrary to the larger and harder austenitic domains of alloy A. On the other hand, the martensitic destabilization of austenite in steels A and E is enhanced by the diminution of the test temperature. This latter reduction decreases the matrix ductility, and thereby the stress relaxation in the softer matrix. Furthermore, the stress concentration is higher in close contact with elongated particles than with blocky ones. This phenomenon arises from both the enhancement of the notch effect of the particles and the greater anisotropy of plastic deformation in lath upper bainite. This explanation agrees with less frequent interfacial cavities in acicular ferrite than in lath upper bainite where they nucleate at the end of the particles elongated along the lath boundaries (Fig.10a). Moreover, these differences in stress concentration at the MA particles interfaces are also the causes for the more deleterious influence of retained austenite on the CGHAZ toughness in a ferritic side plates structure than in acicular ferrite (see Fig.6).

In addition, the MA particles located at the junctions of packets are the most stressed because of the accommodation of plastic deformation between the packets. The consequent high stresses on these interpacket particles in steel A results in their complete martensitic transformation during the -10°C tests. Furthermore, Table 3 indicates that the MA particles are broken in the neck of the samples because of the high local plastic deformation.

Finally, the kind of fracture surface is explained by the SEM study of secondary cracking on sections perpendicular to the main crack. At -196°C , the ferritic matrix cleaves because both of its low ductility and of the stress raising influence of the microcracks at the MA interfaces (see Fig.10b). Moreover, it is worth reminding that these interfacial defects arise themselves from the stress concentrations induced by the lower plasticity of the MA particles. In addition, a similar micromechanism of fracture exists in HT 80 low alloyed steels [16]. Alternatively at -10°C , the interfacial decohesions grow into dimples because of the higher matrix ductility.

Figure 10: SEM evidence of secondary cracking after impact tensile testing: a) steel A (-10°C), b) steel A (-196°C).

3.4. Optimum chemical composition

Figure 11 is a new version of figure 9 that takes the advantage of an explicit display of the general reduction of the volume fraction of retained austenite with the decrease of the silicon content of steels C through F. In accordance with the predominant effect of this second phase on toughness, figure 11 shows that the silicon content must be lower than 0.150 wt%. However, it must not be too low in order to ensure sufficient proof and tensile strengths [4, 8]. Otherwise, the Ti addition must not be overstoichiometric compared to the nitrogen content of TiN in order to ensure a uniform and fine TiN precipitation that allows a maximum of heterogeneous nucleation of acicular ferrite on these intragranular precipitates [17]. Moreover, this mechanism of nucleation will be the more efficient, the lower the niobium addition because this element limits the nucleation of ferrite. All these results were taken into account in order to manufacture an 0.09 wt% C, 1.5 wt% Mn, 0.47 wt% Ni, 0.003 wt% N and 0.095 wt% Si steel [4, 8]. Following the CGHAZ simulations used in this work, the new steel exhibits very good toughness. The CGHAZ transition temperature T_k is equal to -60°C and exhibits a minimum at -80°C for $Dt = 100\text{s}$. It is worth emphasizing that in accordance with the effect of silicon, this very good toughness corresponds with a low volume fraction of retained austenite that ranges between 1 and 2 % [4, 8]. Moreover, the minimum in T_k is observed with a 80 volume percent of acicular ferrite. These results indicate the advantage of a fine acicular ferritic structure with a small volume fraction of retained austenite for the CGHAZ toughness of 0.1 wt% C microalloyed steels. In addition, it is important to note that this beneficial effect of a low silicon content on toughness was further verified in both the intercritical and the coarse-grained heat-affected parts of real weld joints [4]. Moreover, a reduction of the silicon content leads also to an improvement of the cold-cracking resistance of the CGHAZ because of the lowering of its hardenability [4].

Figure 11: Effect of the silicon content on the volume fraction of retained austenite in alloys C through F.

4. CONCLUSIONS

This work affords a new proof of the necessity of the study of the microstructure in order to understand the CGHAZ toughness of 0.1 wt% carbon steels.

The reduction of the silicon content under 0.15 wt% is proved very suitable in order to decrease the volume fraction of retained austenite, and more especially at high welding energy. This microstructural effect of silicon arises from its hindering action on the formation of carbides that induces an increase of the carbon content, and thereby of the stability of retained austenite.

The particles of retained austenite are more or less completely transformed into martensite during an impact because of their high stiffness and of the strain accommodation that gives rise to interfacial stress concentrations. These stress concentrations bring about the formation of interfacial decohesions which promote the initiation and propagation of cracks, and thereby a decrease of the CGHAZ toughness with an increase of the volume fraction of retained austenite. This effect on the Charpy V transition temperature amounts to $+14$ and to $+8^{\circ}\text{C}$ per volume percent of retained austenite in ferritic side plates and acicular ferrite structures, respectively.

All these results conclude to the opportuneness of a silicon content under 0.15 wt% in order to improve the CGHAZ toughness of 0.1 wt% C microalloyed steels. The efficiency of this conclusion is demonstrated by the elaboration of a new steel with both a low volume fraction of retained austenite in its CGHAZ, and a CGHAZ Charpy V transition temperature always lower than -60°C.

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