Journal Pre-proof

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PII: S0921-5093(19)31505-9

DOI: <https://doi.org/10.1016/j.msea.2019.138719>

Reference: MSA 138719

To appear in: *Materials Science & Engineering A*

Received Date: 6 September 2019

Revised Date: 19 November 2019

Accepted Date: 20 November 2019

Please cite this article as: H. Tervo, A. Kaijalainen, S. Pallaspuuro, S. Anttila, S. Mehtonen, D. Porter, J. Kömi, Low-temperature toughness properties of 500 MPa offshore steels and their simulated coarse-grained heat-affected zones, *Materials Science & Engineering A* (2019), doi: <https://doi.org/10.1016/j.msea.2019.138719>.

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Low-temperature toughness properties of 500 MPa offshore steels and their simulated coarse-grained heat-affected zones

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Keywords: CGHAZ, fracture toughness, impact toughness, inclusions, microstructure

ABSTRACT

Offshore steels for cold climate conditions require not only relatively high strength to improve the cost-efficiency of the structures, but also excellent toughness at low temperatures to guarantee the safety of the structures in harsh environments. The most challenging locations to fulfil both requirements are in the welded joints of these steels because the weld thermal cycles cause irreversible changes to the steel microstructure. Titanium and niobium are often used in these steels to form nanoscale precipitates and reduce the austenite grain growth during the thermal cycle. However, there is a risk that in addition to the nanoscale precipitates formed from the solid state, microscale coarse inclusions are also formed from the melt. These inclusions are hard and brittle and thus likely to deteriorate mechanical properties such as fracture toughness and impact toughness. In this study, two experimental 500 MPa offshore steels with different carbon contents and with differing inclusion structures were studied to find out whether microscale titanium-based nitrides significantly deteriorate the impact and fracture toughness of the coarse-grained heat-affected zone (CGHAZ) of these steels at low temperatures. A Gleeble 3800 was used to simulate the CGHAZ with two different cooling rates, the fracture toughness was determined by three-point bend testing, and the impact toughness was determined by Charpy V-notch testing. The study was limited to single-pass welding scenarios. Inclusions were characterized using FESEM-EDS. It was found that both impact and fracture toughness of the steel with coarse titanium-based nitrides were lower and the scatter higher than in the steel with a higher quantity of calcium-based inclusions and without the coarse nitrides. Fractographic examination showed that the failure in the samples with the lowest toughness was initiated by the coarse titanium-niobium nitrides. Thus, to avoid CGHAZ brittleness under

cold conditions, it is necessary to mitigate the formation of coarse nitrides by careful control of steelmaking process and continuous casting to avoid the segregation of titanium, niobium and nitrogen.

1 INTRODUCTION

Offshore steels are typically used in applications such as oil drilling platforms, where service conditions are harsh and safety of the structures is of extreme importance. The opening of new oil fields in cold climates increases the demand for steels that can withstand the harsh conditions. Offshore steels are typically manufactured by thermomechanically controlled hot rolling process (TMCP) that leads to fine-grained microstructure. This way good strength and toughness in the base plate are obtained, while the low content of carbon and other alloying elements provides good weldability. This publication is concerned with offshore steel with a minimum specified yield strength of 500 MPa. Such a strength class has been used, for example, in the Valhall oil drilling platform in Norway. The good combination of strength, toughness and weldability means that TMCP steels in the 500 MPa yield strength class are also suitable for other structural applications.[1]

Offshore steels are typically welded using submerged arc welding (SAW) or flux-cored arc welding (FCAW). These welding methods are also specified for the welding of offshore steels by standards such as EN-10225 [2]. The welded joints are the most vulnerable sections in structures due to the weld thermal cycles that change the original microstructure of the steel that was engineered to have excellent material properties. One of the main phenomena in the coarse-grained heat-affected zone (CGHAZ) of the weld is the coarsening of the austenite grain size. The larger grain size itself impairs the toughness properties but, in addition to this, it makes the steel more prone to the detrimental effects of inclusions and other hard second-phases e.g. martensite-austenite constituents (M-A) [3,4]. Titanium and niobium are often used as microalloying elements to form nanoscale precipitates and to hinder the coarsening of the austenite grains. However, the steelmaking and continuous casting processes need to be carefully controlled in order to avoid the segregation of these elements and the formation of coarse microscale inclusions e.g. nitrides and carbides. If niobium content is high, NbC eutectic particles (inclusions) can form from the melt. Being so large, they do not dissolve during reheating prior to rolling. Brittle inclusions are known to deteriorate the toughness properties of steels. In the CGHAZs their impact is pronounced as the coarsened grains are less able to arrest inclusion initiated cracks than a fine-grained microstructure [3,5–9].

The effect of microstructure and TiN inclusions in various materials and HAZs has been studied extensively [2,4,5,7,9–16,]. Previous studies regarding the improvement of CGHAZ toughness include

e.g. the effect of TiN particles on the fracture toughness of simulated CGHAZs using two different peak temperatures and cooling rates in a non-specified structural steel [16], real welded samples of pipeline steels [17], the effect of cooling rate on the low temperature impact toughness of a high-strength offshore steel [10] and the effect of carbon content on the weldability of ultralow-carbon 570 MPa offshore steel on the CGHAZ impact toughness at -5 °C [11]. TiN inclusions are considered to be potential cleavage fracture initiators as they are hard and brittle, and strongly bonded to the steel matrix. As the hard inclusion deforms elastically in a plastically deforming matrix, the strong interfacial bond leads to the concentration of stresses and cracking in the inclusion after which the crack moves freely through the inclusion-matrix interface to the steel matrix [6,8]. Also, it has been shown that a coarse effective grain size together with coarse TiN inclusions significantly decreases impact and fracture toughness [12,16,18].

For demanding applications in cold climates, high toughness and the predictability of the fracture mechanical behaviour of the welded joints are essential. Because it is known that the weakest link of the structures can often be located to the CGHAZ, it is important to understand why and how different factors in the microstructure of the CGHAZ reduce the toughness. Therefore, in this study, the focus was on investigating the role of titanium-niobium nitrides and microstructures on the impact toughness and fracture toughness of the CGHAZ of two offshore steels with a specified minimum yield strength of 500 MPa.

Compared to the previous studies on the topic, e.g. [10,11,16,17], this study combines different CGHAZ microstructures obtained by varying alloying and cooling rates. Different inclusion structures are included in the study, and both fracture toughness and impact toughness are studied. Microstructures are characterized using up-to-date EBSD techniques. The emphasis of this study is on the low-temperature properties required by the strict standards for the steels used in offshore applications in cold climate conditions.

2 MATERIALS AND METHODS

2.1 Materials

Two experimental continuously cast microalloyed 500 MPa offshore steels (A and B) were studied. Steel A had a very low carbon content of 0.02 %, its strength provided by relatively high contents of Si, Mn, Cr and Mo. In Steel B, the carbon content was 0.07 %, while the content of other alloying elements was lower than in Steel A. The composition of both steels in wt.% is given in Table 1. The carbon equivalents of the studied steels, as expressed by the factor P_{cm} , were 0.156 for Steel A and 0.174 for Steel B, which should guarantee good weldability.

To study the effect of inclusions, two slabs from both Steels A and B with differing impurity, and thereby inclusion, contents were selected, and those samples were numbered as 1 and 2 (Table 2). From the centreline of continuously cast slabs, pieces of 180 x 80 x 55 mm³ were cut and hot rolled to the thickness of 9 mm using a laboratory rolling mill. The target finish rolling temperature was 820 °C, after which the steel was water cooled to 400 °C with a cooling rate of 30 °C/s followed by air cooling to room temperature with cooling rate of ≤ 0.3 °C/s.

Table 1. Alloying elements of Steel A and Steel B measured using an optical emission spectrometer.

	C (%)	Si (%)	Mn (%)	Al (%)	Nb (%)	Cr + Mo (%)	V (ppm)	Ti (ppm)	Ca (ppm)
Steel A	0.02	0.38	1.5	0.03	0.03	0.44	80	120	23
Steel B	0.07	0.30	1.3	0.03	0.03	0.07	100	130	25

Table 2. Impurities in Steel A and Steel B. The oxygen and nitrogen levels are based on combustion analysis, while sulphur level was analysed using an optical emission spectrometer.

	S (ppm)	O (ppm)	N (ppm)
Steel A1	24	17	56
Steel A2	14	8	45
Steel B1	16	17	54
Steel B2	12	12	43

Due to the the time-consuming procedure of determining and testing the HAZ of real welds, physical simulation of the HAZ provides an attractive alternative by enabling the reliable simulation, characterization and testing of different HAZ subzones, including the reheated regions occurring in multipass welds. By adjusting the thermal cycle parameters, it is possible to simulate various types of welding methods with varying heat inputs and cooling rates. HAZ physical simulation has been used successfully in this way for many years [19–22].

The CGHAZ was simulated using a Gleeble 3800 thermomechanical simulator. A peak temperature of 1350 °C with a holding time of 1 second was achieved with a heating rate of 100 °C/s. The free span was 20 mm. The cooling times from 800 °C to 500 °C ($t_{8/5}$) were chosen to be 6 s and 30 s to simulate flux cored arc welding (FCAW) and submerged arc welding (SAW), respectively, which are typical methods used in the welding of the studied steels. The Rykalin 2D cooling model [23] was used to derive the temperature - time cooling curves corresponding to the chosen values of $t_{8/5}$. The simulated CGHAZ region in this study would most accurately represent CGHAZ in single-pass welding scenarios or the non-reheated CGHAZ in multipass welding scenarios.

2.2 Mechanical testing

Tensile tests for the base material were performed at room temperature using 12 mm wide and 8 mm thick flat specimens with a gauge length of 75 mm cut parallel to the rolling direction (minimum 3 specimens/direction). The tensile testing samples were cut parallel to the rolling direction because the width of the laboratory rolled plates was insufficient for transverse specimens. From the tensile testing results, yield strength, ultimate tensile strength and elongation to fracture were determined. Hardness was measured using a Duramin-A300 (Struers) under a 100 N load (HV_{10}) in through-thickness direction from the RD-ND surface of the base material and from the centreline of the TD-ND surface and along the simulated CGHAZ. (Here, RD means rolling direction, ND the plate normal direction, and TD the transverse direction).

Standardised Charpy V-notch (CVN) impact toughness tests were carried out at temperatures ranging from -160 °C to +40 °C using sub-sized 5 x 10 x 55 mm³ specimens that were oriented transverse to the rolling direction [24]. Two specimens were tested in each temperature for the base materials. The impact toughness of CGHAZ was tested using from two to three specimens at each temperature. Exponential-fitted ductile-brittle transition temperature range with impact toughness transition temperatures (T_{35J/cm^2}) and upper shelf energies (USE) were determined from the results.

The European Standard EN10225 modified with NORSOK M-120 to match the harsh cold climate conditions specifies requirements for weldable structural steels to be used in the manufacturing of fixed offshore structures. According to this standard, it is required to perform fracture toughness tests preferably in accordance with EN ISO 12737 of certain heat-affected zones including the CGHAZ [2,25]. Thus, fracture toughness testing was performed using 5 mm thick single-edge notched bend (SENB)

specimens with an a/W ratio of 0.5 and with the pre-fatigued crack positioned at the centre of the simulated CGHAZ.

2.3 Microstructural characterization

The microstructure of nital etched specimens of the base material and simulated CGHAZ was characterized using a Zeiss Sigma field emission scanning electron microscope (FESEM) operated at 5 kV and a Keyence VK-X200 laser scanning confocal microscope (LSCM). Grain boundary misorientation distribution and average effective grain size were measured using the EDAX electron backscatter diffraction (EBSD) system on the FESEM with an acceleration voltage of 15 kV. For the base materials an area of $\sim 88 \times 88 \mu\text{m}$ and a step size of $0.2 \mu\text{m}$ were used, while for the simulated CGHAZ the area and the step size were $\sim 146 \times 146 \mu\text{m}$ and $0.3 \mu\text{m}$, respectively. From the original acquisitions three pixels were filtered and grains larger than $0.36 \mu\text{m}$ and $0.55 \mu\text{m}$ were used for the base material and simulated CGHAZ, respectively. Grain boundaries with misorientation greater than 15° were considered as effective grain boundaries from the point of view of cleavage crack propagation resistance. Equivalent circular diameter (ECD) was used to define the effective grain size. Fracture surfaces of the test specimens were studied using FESEM. To characterize the inclusion structure, energy dispersive spectrometer (EDS) analysis was performed at 15 kV and 3.5 nA on a Jeol JSM-7000F (FESEM-EDS). The data were acquired and analysed using Oxford INCA software. The working distance was 10 mm and each inclusion was measured for 1 s live time. The inclusions were analysed on cross-sections containing the rolling direction (RD) and the plate normal (i.e. thickness) direction (ND). The measured area covered the surface area from the top to the bottom surface. The acquired inclusion data included information about the number, size, location, shape and chemical composition of inclusions. The size of the inclusions was determined using their maximum length. The minimum inclusion size included in the results was $0.97 \mu\text{m}$.

3 RESULTS

3.1 Base material microstructures and mechanical properties

The typical microstructures as shown by EBSD with the corresponding grain boundary misorientation charts of the studied steels are presented in the Fig. 1. The microstructure in the Steel A consisted of mainly quasi-polygonal ferrite (Fig. 1a-b), while bainitic features were present in Steel B (Fig. 1c-d). As typical in ferritic microstructures, grain boundary misorientation charts in Fig. 1a-d consisted of all angles from 2° to 60°. However, the grain boundary misorientation distributions of especially Steels B1 and B2 (Fig. 1c-d) resemble that of a previous study [26] indicating the presence of granular bainite. Bainitic features were likely the result of the difference in the C-contents (0.02% in Steel A vs. 0.07% in Steel B) and alloying ($P_{cm} = 0.156$ in Steel A vs. 0.174 in Steel B) promoting hardenability in Steel B. These differences in the microstructure led to a smaller effective grain size in Steel B than in Steel A as presented in Fig. 2.

Figure 1. Microstructure as shown by EBSD with the corresponding grain boundary misorientation distribution in Steel A1 (a), Steel A2 (b), Steel B1 (c) and Steel B2 (d). Images are taken from the $\frac{1}{4}$ thickness. Red lines indicate the low-angle boundaries 2°-15°, while the blue lines indicate high-angle boundaries greater than 15°.

The EBSD-measurements revealed that there were slight differences in the grain sizes of the studied steels. Notably, the average grain size in Steel A1 was 2.4 μm and 2.1 μm in Steel A2, while in Steel B1 and B2 those were 1.9 μm and 1.8 μm , respectively. The 80 th percentile ECD in the cumulative grain area distribution followed a similar trend.

Figure 2. The effective grain size and the 80 th percentile ECD in the cumulative grain area distribution of the all the measured grains in the studied steels. Sizes are filtered and grains above 0.36 μm are used. The differences between A1 vs. B1 and B2 ($p < 0.05$), and A2 vs. B2 ($p < 0.05$) are statistically significant. (P values given as calculated using one-way ANOVA).

The hardness, the tensile testing results and the Charpy V-notch impact toughness testing results of the base materials are presented in Table 3. The yield and tensile strength were slightly below the nominal 500 MPa range, but reasonable given that the values were in the longitudinal direction. The hardness of Steel B with higher carbon content was slightly higher than that of Steel A. Transverse Charpy V-notch impact toughness testing results for the sub-size specimens showed that the upper shelf energy (USE) in each steel was generally well above 200 J/cm² and the ductile-brittle transition temperature was below -130 °C indicating an excellent level for offshore steels designed to serve in cold climate conditions.

Table 3. Mechanical properties of the base materials with 95 % confidence intervals for the means determined using the student-t method.

	Hardness (HV10)	Yield strength (MPa)	Ultimate tensile strength (MPa)	Elongation (%)	T _{35J/cm²} (°C)	USE (J/cm ²)
Steel A1	208 ± 2	470 ± 24	635 ± 27	23.4 ± 1.4	-146	239
Steel A2	206 ± 4	496 ± 24	621 ± 13	21.6 ± 2.8	-154	246
Steel B1	220 ± 4	483 ± 62	609 ± 12	20.5 ± 5.7	-132	208
Steel B2	225 ± 3	488 ± 28	605 ± 16	26.2 ± 8.0	-160	239

3.2 Inclusion structure

The size distribution of inclusions according the number of inclusions as well as the area fraction of inclusions in Steel A and Steel B are presented in Fig. 3. Steel A had generally a higher density of inclusions than Steel B. The difference was remarkable especially in medium size inclusions between 2.8-5.8 μm .

Figure 3. Number (a) and area fraction (b) of inclusions classified according to their size in the studied steels.

The number and area fraction of inclusions classified according to their chemical composition are presented in Fig. 4. The inclusion classes were defined based on the elementary composition of each inclusion measured using FESEM-EDS. The classes are presented as a few main classes and as combinations of them. The main classes were Ca (including calcium sulphides, calcium oxides and calcium aluminates), MnS (manganese sulphide) and TiN (including titanium and titanium-niobium nitrides). Because of the absence of carbon in the EDS analyses, the carbides were not included in the analysis. However, in reality, the nitride group also contained carbides i.e. carbonitrides. The class "Others" covers inclusions that did not fit into any of the main classes and also less frequent combinations of these main classes.

Figure 4. Number (a) and area fraction (b) of all inclusions as well as number of coarse inclusions (c) according to their chemical composition. Ca-inclusions represent CaS, CaO, CaO- Al_2O_3 and their combinations. TiN class represents titanium and titanium-niobium nitrides and carbonitrides. Other inclusions consist of miscellaneous inclusions e.g. MgO and of less common combinations of the above-mentioned inclusions.

In Steel A1, the TiN class was almost completely absent; the inclusions were almost solely Ca-based. Generally, these types of inclusions were spherical, relatively small and they were distributed evenly

within the steel matrix. In Steel A2, Ca-based inclusions were also prominent, but TiN either alone or mixed together with $\text{CaO-Al}_2\text{O}_3$ and CaS was present in a notable fraction. The observed nitrides were generally rectangular with sharp corners and they often located in the segregation bands as shown in Fig. 5. Steels B1 and B2 had an inclusion structure similar to that of Steel A2, but with a lower total number of inclusions. The majority of inclusions in Steels B1 and B2 were Ca-based but also the TiN type was present. Interestingly, both Steels A2 and B2 had a greater number of TiN than Steels A1 and B1, despite having about 10 ppm lower nitrogen content. A few MnS inclusions were detected in Steels A1 and B1, but mostly their formation was prevented by Ca additions that modified the sulphides to Ca-based (Mn,Ca)S and CaS.

Fig. 4. suggests also that the number density of inclusions was slightly higher in Steel A than in Steel B. Additionally, Steels A1 and B1 had a greater number of inclusions compared to their variants Steels A2 and B2 with decreased content of impurities. In steel A, this difference was clearly caused by the great number of (Mn,Ca)S in Steel A1, while in Steel B there were more Ca-based inclusions such as $\text{CaO-Al}_2\text{O}_3$ in Steel B1 compared to Steel B2. The majority of coarse inclusions ($> 4 \mu\text{m}$) were Ca-based in all the studied steels, except that in Steel A1 about half of the coarse inclusions were Ca-based, while the other half consisted of (Mn,Ca)S. Also, coarse TiN inclusion types were present in all steels except Steel A1.

The spatial distribution of inclusions belonging to the TiN class in the studied steels is presented in Fig. 5. The inclusions are grouped according to the sum $\text{Ti} + \text{Nb} + \text{N}$ being $> 90 \%$, $70-90 \%$ and $50-70 \%$ as measured in weight percent. The niobium content in these inclusions was typically between $3-10 \%$. When TiN nucleated on the surface of a pre-existing inclusion such as calcium aluminate or calcium sulphide, this resulted in a decreased fraction of $\text{Ti} + \text{Nb} + \text{N}$. The cases where $\text{Ti} + \text{Nb} + \text{N} < 50 \%$ are not shown. Especially in Steel A2 and to a lesser extend in Steel B1, a notable concentration of TiN inclusions lies at certain depths below the surface of the plate due to segregation of Ti, Nb and/or N.

Figure 5. The distribution of TiN inclusion class measured through-thickness on the RD-ND surface of Steel A1 (a), Steel A2 (b), Steel B1 (c) and Steel B2 (d). CL = centreline, S = surface.

3.3 Simulated CGHAZ

3.3.1 CGHAZ microstructure

Fig. 6. shows the simulated CGHAZ microstructures with $t_{8/5} = 6$ s and 30 s as revealed by LSCM, SEM and EBSD. Grain boundary misorientation charts corresponding to the presented microstructures are presented in Fig. 7. The steels were fully austenitized during the CGHAZ simulation. It was assumed that impurity contents would have not affected the simulated CGHAZ microstructure. Hence, only Steels A2 and B2 were characterized and taken as representative of both the variants of the steels.

The LSCM images in Figs. 6a, d, g and j show that the grains had coarsened, and the microstructure of Steel A consisted mostly of granular bainite with a small fraction of upper bainite with the slower cooling time $t_{8/5} = 30$ s, whereas with the faster cooling time $t_{8/5} = 6$ s, the majority of the microstructure consisted of upper bainite with some granular bainite. In Steel B with the cooling time $t_{8/5} = 30$ s, the microstructure consisted of both upper and granular bainite, while with $t_{8/5} = 6$ s, the microstructure was a mixture of lower bainite and martensite with a small fraction of upper bainite. Prior austenite grain size determinations were difficult to carry out because of the nucleation and growth of granular bainite on the grain boundaries. However, based on 6 s cooling time samples, the prior austenite grains were coarsened to a range of 77-112 μm , and at least to that with 30 s cooling time. There was a peak at about 45° in the grain boundary misorientation chart in the case of Steel A with both cooling times (Figs. 7a & b) and in case of Steel B with $t_{8/5} = 30$ s (Fig. 7d) indicating the presence of austenite [27].

The difference in the microstructures of Steel A and Steel B with short cooling time is the result of the higher carbon content in Steel B that increases the hardenability and helps the lath-like features to form during fast cooling. The higher hardenability explains the increased hardness in Steel B with $t_{8/5} = 6$ s as will be presented in Table 4. More details of the microstructures can be seen in the SEM images in Figs. 6 b), e), h) and k).

Figure 6. LSCM, SEM and EBSD figures of simulated CGHAZ of Steel A with $t_{8/5} = 6\text{s}$ (a, b, c) and 30s (d, e, f) as well as of Steel B with $t_{8/5} = 6\text{s}$ (g, h, i) and 30s (j, k, l). The images are from the TD-ND surface. Red lines in images c, f, i & l indicate the low-angle boundaries 2° - 15° , while the blue lines indicate high-angle boundaries greater than 15° .

Figure 7. Grain boundary misorientation charts measured with EBSD from the CGHAZ of Steel A with $t_{8/5} = 6$ s (a) and 30 s (b) as well as Steel B with $t_{8/5} = 6$ s (c) and 30 s (d).

The grain boundary misorientation distributions show that the CGHAZ of Steel B with $t_{8/5} = 6$ s (Fig. 7c) differed notably from the other variants. In fact, the grain boundary misorientation distribution resembles that of a fully martensitic steel as seen in an earlier study with the exception of a lower fraction of low-angle grain boundaries that are considered typical for a martensitic steel [9]. Thus, the microstructure was characterized as lower bainite that has a similar fraction of high-angle grain boundaries as a martensitic microstructure, but a low fraction of low-angle grain boundaries [28]. The grain boundary misorientations in Figs. 7a, b and d resemble that of the upper bainite as obtained in previous studies [26,28].

The EBSD data was used to determine the average effective grain size and the 80 th percentile in the cumulative grain area distribution of the simulated CGHAZs, see Fig. 8. The effective grain size and the cumulative 80% grain size were clearly smaller in Steel B with the cooling time of 6 s than in other simulated CGHAZ variants. This is due to the more martensitic-lower bainitic microstructure that provides a great number of high-angle boundaries inside the prior austenite grains. In other variants, a larger fraction of high-angle boundaries were actually prior austenite grain boundaries as can be seen in Figs. 6c, f & l, where the substructures inside the prior austenite grains consisted of lower angle boundaries.

Figure 8. The effective grain size and cumulative 80% grain size of the all the measured grains of the simulated CGHAZ in the studied steels. Sizes were filtered and the grains above 0.55 μm were included. Statistical significance (one-way ANOVA) between B 6 s vs. others ($p < 0.05$).

The hardness of the physically simulated CGHAZ of the test materials is presented in Table 4. The hardness was measured on the TD-ND surface on the middle section regarding the thickness and the values were calculated as an average along the line extending from the location of the thermocouple to 2 mm both sides. The position of the thermocouple experienced the highest temperature during the simulation and based on the hardness results, the real CGHAZ area extended a few millimetres either side of the thermocouple changing gradually into other heat-affected sub-zones and eventually to the base material. With $t_{8/5} = 6$ s the hardness increased slightly to about 240 HV in Steel A and even to about 280 HV in Steel B due to the change in the microstructure as was presented in Fig. 6. With $t_{8/5} = 30$ s the hardness did not differ remarkably from that of the base material, and there were not remarkable differences between the Steels A1 and A2 or the Steels B1 and B2. Tensile testing was not carried out for the simulated CGHAZ samples. However, hardness is generally recognised to be related to the tensile strength by a factor of 3.2. Thus, the tensile strength of the CGHAZ with $t_{8/5} = 6$ s was approximately 750 MPa and 900 MPa in Steel A and B, respectively, while with $t_{8/5} = 30$ s the tensile strength of both steels was close to that of the base materials at about 600-650 MPa.

Table 4. Hardness of the simulated CGHAZ. Values are averages of nine measurement points measured along a line extending ± 2 mm from the location of the thermocouple. 95 % confidence intervals for the means were determined using the student-t method. Using a one-way ANOVA test, statistical significance exists between A and B at 6 s ($p < 0.05$), and between A1 and A2 at 30 s ($p < 0.05$).

	Steel A1 6 s	Steel A2 6 s	Steel B1 6 s	Steel B2 6 s	Steel A1 30 s	Steel A2 30 s	Steel B1 30 s	Steel B2 30 s
Hardness (HV ₁₀)	239 $\pm$ 7	235 $\pm$ 4	280 $\pm$ 6	274 $\pm$ 9	214 $\pm$ 6	202 $\pm$ 6	210 $\pm$ 5	208 $\pm$ 2

3.3.2 CGHAZ impact toughness

The Charpy V-notch impact toughness results of the CGHAZ of the studied steels are presented in Fig. 9. The upper shelf energies were generally on a same level between 250 – 300 J/cm², i.e. even higher than in the base material (208 – 246 J/cm²). For the short cooling time ($t_{8/5} = 6$ seconds) the variants with a low number density of TiN type inclusions, i.e. A1 and B1, achieved excellent low transition temperatures (T_{35J/cm^2}) below -100 °C. More accurate estimation of the transition temperature for A1 and B1 was not possible due to the lack of test data at temperatures below -80 °C. The variants with a higher density of TiN type inclusions showed clearly higher transition temperatures at -67 °C and -77 °C for A2 and B2 respectively. On the other hand, with $t_{8/5} = 30$ s, the drop to the lower shelf was dramatic even for the cleaner variants A1 and B1. For Steel B (Fig. 9b), which was more brittle T_{35J/cm^2} was -52 °C (B1) and -41 °C (B2). Steel A2 with a high number density of TiN type inclusions had a mediocre T_{35J/cm^2} of -67 °C while for the cleaner A1 it was -85 °C. In general, the short $t_{8/5}$ of 6 seconds (resembling the welding cycle of FCAW) yielded good low-temperature toughness in both clean steel variants with a low number density of TiN type inclusions, but the differences between the two impurity levels narrowed with the long $t_{8/5}$, where none of the studied variants achieved excellent T_{35J/cm^2} .

Figure 9. Impact toughness of CGHAZ with $t_{8/5} = 6$ s (a) and $t_{8/5} = 30$ s (b) in the studied steels. Two or three specimens were tested in each temperature. Marks mean single test results and lines show the fitted transition curves. In Steels A1 and B1 with $t_{8/5} = 6$ s the lower shelf was not achieved in the measured temperatures. Thus, the approximation of T_{35J/cm^2} for these steels is < -100 °C.

3.3.3 CGHAZ fracture toughness

The fracture toughness test data is shown in Fig. 10 for all the material variants. Due to the fact that only few samples complied with the requirements of ASTM E1921, no fracture toughness reference temperatures could be defined. Along with impact toughness, also the best fracture toughness and with the lowest scatter was achieved in variants with a low number density of TiN type inclusions and a short cooling time ($t_{8/5} = 6$ seconds). The average value of $K_{Jc(1T)}$ at -40 °C is 215 MPa√m for the CGHAZ of

Steel A1 and 126 MPa $\sqrt{m}$ for Steel B1. The CGHAZ of Steel A was tough regardless of the presence of TiN type inclusions with $t_{8/5}$ of 30 seconds: A1 has $K_{Jc(1T)}(-40\text{ }^{\circ}\text{C}) = 193\text{ MPa}\sqrt{m}$ and for A2 $K_{Jc(1T)}(-40\text{ }^{\circ}\text{C}) = 177\text{ MPa}\sqrt{m}$, but these had substantial scatter of 80 MPa $\sqrt{m}$ and 118 MPa $\sqrt{m}$, respectively. The fracture toughness of the CGHAZ of Steel B was poor with a $t_{8/5}$ of 30 seconds. Fractographic investigations showed that the premature failures were caused by coarse TiN type inclusions (Fig. 12). The exceptions to this were with Steel A1, where the primary failure initiators were not found, and Steel B1 with $t_{8/5}$ of 6 seconds, where the suspected failure initiators were coarse carbides in the upper bainitic constituent (Fig. 6 h).

Figure 10. Fracture toughness test results for the two $t_{8/5}$: a) Steel A1 6 s, b) Steel A1 30 s, c) Steel A2 6 s, d) Steel A2 30 s, e) Steel B1 6 s, f) Steel B1 30 s, g) Steel B2 6 s, h) Steel B2 30 s.

4 DISCUSSION

4.1 Effect of chemical composition and heat input on the microstructures

The steels of this study had different carbon contents: 0.02 % (Steel A) and 0.07 % (Steel B). Overall, the base material microstructures consisted mainly of quasi-polygonal ferrite, but the higher carbon content of Steel B led to a somewhat more bainitic base material microstructure. Various CGHAZ microstructures were obtained as a result of the differences in the steel compositions and cooling rates. In all studied cases, as is typical for the CGHAZ [4,11,29,30], the increase in the prior austenite grain size leads also to an increase in the mean effective grain size of the transformed low-temperature microstructure and in the size of the coarsest grains in the grain size distribution. In this study, the prior austenite grain sizes after CGHAZ simulations were approximately 77-112 μm . However, as most variants contained granular bainite, the PAGSs could not be measured reliably due to indistinguishable boundaries of PAGs and granular bainite grains in the LSCM images. However, the coarsest grains (i.e. $d_{80\%}$ of the cumulative grain size distribution) increased from 7.7 μm to 40.9 μm and 44.4 μm in Steel A and from 6.3 μm to 8.7 μm and 33.5 μm in Steel B with $t_{8/5} = 6$ s and 30 s, respectively. Evidently, the more martensitic microstructure with $t_{8/5} = 6$ s in Steel B led only to a slight increase in the $d_{80\%}$.

In the low heat input CGHAZ simulations, the difference in the microstructure was more significant as Steel A consisted of upper and granular bainite, while the Steel B had a combination of martensite, lower bainite and a minute fraction of upper bainite. In the high heat input scenario, a mixture of upper and granular bainite was formed in both compositions. Hardness measurements confirmed that the CGHAZ of the Steel B with $t_{8/5}$ of 6 s had the most hardened microstructure with maximum hardness of about 280-290 HV. Correspondingly, Steel A with a lower hardenability (i.e. carbon content and P_{cm}) had a maximum measured hardness of only about 250 HV. However, the hardness of both steels with $t_{8/5}$ of 30 s did not remarkably differ from that of the base material. In addition, the formation of M-A constituents and carbides took place as is typical for the CGHAZ region [10,31]. Retained austenite and M-A constituents have been discovered previously in similar low-carbon content steels [4].

4.2 Toughness of the simulated CGHAZ

An increase in the effective grain size and the size of the coarsest grains occurred in the CGHAZ of all variants except Steel B with $t_{8/5} = 6$ s. Generally, an increase in the grain size leads to a decrease in toughness [30,31]. However, the effect of the grain size on the impact and fracture toughness was not straightforward. For example, excellent impact and fracture toughness was achieved in Steel A1 with both

cooling times, i.e. even though grain coarsening occurred. On the other hand, both impact and fracture toughness were lower in Steel A2, where the only difference to A1 was the abundance of TiN type inclusions. Additionally, the variant with the least increase in the grain size, Steel B with $t_{8/5} = 6$ s had very good impact toughness with $T_{35J/cm}^2 < -100$ °C only in variant B1 with a low number density of TiN type inclusions compared to B2, where $T_{35J/cm}^2$ increased to -77 °C.

In addition to grain coarsening and inclusions, the combined effects of various transformation products (upper, lower or granular bainite, and martensite) must be taken into account. Different morphologies of bainite have different mechanical properties [10,11,32]. In ultralow-carbon steels, such as Steel A, granular bainite has been reported to have better toughness characteristics than upper bainite. However, with higher carbon contents, such as in Steel B, the toughness of the granular bainite decreases due to formation of coarse M-A constituents [32]. In the current study, the microstructures of Steel A with both cooling times and Steel B with the cooling time of 30 s were mixtures of granular and upper bainite. These microstructures have a lower incidence of high-angle grain boundaries [28,33] that serve as obstacles to propagating cracks, thereby making these microstructures vulnerable to the brittle failure. Moreover, an increase in the average bainite packet size has been reported to decrease the cleavage fracture resistance in low-carbon bainitic steels [34]. Also, in the presence of certain types of inclusions, such as hard and brittle nitrides, or other second-phase particles, such as carbides and M-A constituents, the nucleation of cleavage cracks becomes more probable. In a previous study of Steel B by the present authors, it was shown that the coarse upper bainite seen with $t_{8/5} = 30$ s impairs the impact toughness compared to that obtained with the finer transformation products of lower bainite or martensite achieved with $t_{8/5} = 6$ s [35].

The improved fracture toughness in Steel A2 with $t_{8/5} = 30$ s compared to 6 s might be partly due to the different microstructures. There were slight differences in the effective grain size (3.5 μm vs. 5.7 μm) and $d_{80\%}$ (40.9 μm vs. 44.4 μm) with $t_{8/5} = 6$ s and 30 s, respectively. Finer features should favour the low heat input variant, but the difference was probably not sufficient to have an effect on the fracture toughness. However, with $t_{8/5} = 6$ s the microstructure was mainly harder upper bainite, whereas with $t_{8/5} = 30$ s an increased fraction of it was softer granular bainite. Besides that, in low-carbon Steel A1 without coarse TiN type inclusions even upper bainite showed relatively good fracture toughness. In Steel B, the differences in the grain size were bigger between the different cooling times, the effective grain size was 2.1 μm and 3.3 μm , while $d_{80\%}$ was 8.7 μm and 33.5 μm with $t_{8/5} = 6$ s and 30 s, respectively. Consequently, the best fracture toughness in Steel B was achieved in variant B1 with $t_{8/5} = 6$ s, where both grain size and number density of TiN class inclusions was reduced. However, the worst fracture

toughness of Steel B was encountered in variant B2 with $t_{8/5} = 6$ s, where especially $d_{80\%}$ was much smaller than in variants with $t_{8/5} = 30$ s, indicating that the decrease in the grain size alone is not sufficient to improve fracture toughness.

The most hardened microstructure was obtained in Steel B with $t_{8/5} = 6$ s where the microstructure was mainly a mixture of martensite and lower bainite. This type of microstructure provides an increase in hardness and strength, which also tends to decrease toughness by raising the flow stress and thereby the driving force for cleavage crack initiation and propagation. However, especially the impact toughness of this variant with a reduced number density of TiN class inclusions (Steel B1) was relatively good suggesting that the effect of increased strength and increase of prior austenite grain size was compensated to some degree by the introduction of lath-like features which reduced the effective grain size and $d_{80\%}$.

The coarsening of the prior austenite grains in welding can be reduced by introducing nanoscale precipitates such as titanium and niobium nitrides and carbonitrides into the steel [36,37]. These particles can pin the grain boundaries and prevent excessive grain growth during the thermal cycle. Although the precipitates were outside the scope of this work, it can be assumed that they were likely to be present in the studied steels, at least in Steels A2, B1 and B2, due to the presence of larger microscale nitrides that contained both titanium and niobium. In a previous study it was suggested that the addition of Nb reduced the high temperature stability of TiN-containing precipitates, thereby reducing the pinning effect and leading to larger austenite grains in the CGHAZ [38]. On the other hand, another study [39] concluded that the increase of Ti and a larger fraction of Ti compared to Nb in (Ti,Nb)N precipitates coarsened the particles and deteriorated the impact toughness of the CGHAZ.

As outlined in Fig. 11a, the best toughness results ($T_{35J/cm^2} < -100$ °C) were obtained when $t_{8/5}$ was 6 s and there were no or only a moderate number of TiN type inclusions such as Steels A1 and B1, respectively. Despite similar microstructures, the transition temperatures increased dramatically in the variants with a higher density of TiN type inclusions (Steels A2 and B2). The TiN type inclusions increased the transition temperatures of both Steels A and B also when $t_{8/5}$ was 30 s, but overall the toughness performance was weaker compared to results with $t_{8/5} = 6$ s. Additionally, with $t_{8/5} = 30$ s Steel A had a lower T_{35J/cm^2} than Steel B with or without TiN type inclusions, indicating that the microstructure that results from the lower carbon content as in Steel A improved the impact toughness in that case.

Fig. 11b shows the dependency between inclusion number density and upper shelf energy (USE) in the studied steels. The highest absorbed energy was obtained in Steel A2 where the total number density of

inclusions was lower than in the Steel A1. The difference compared to T_{35J/cm^2} is that it was better in Steel A1 due to the lack of TiN class inclusions, even if its total number density of inclusions was higher than in Steel A2. This indicates that the USE goes well in line with the total number density of inclusions regardless of their type. Additionally, in Steel B, the USE was generally lower than in Steel A, although the number density of inclusions was also lower than in Steel A. This can be explained as a result of slightly higher strength in Steel B compared to Steel A. It is generally observed that as strength increases USE drops, presumably due to easier microvoid coalescence.

Figure 11. Effect of number density of TiN type inclusions on transition temperature T_{35J/cm^2} of the CGHAZ (a) and the effect of number density of all inclusions on the upper shelf energy of the CGHAZ (b). CVN testing in case of simulated CGHAZ was not performed below -80 °C. However, predicted T_{35J/cm^2} transition temperatures for 5 mm sub-size specimens of some variants were below -100°C.

The fracture toughness results were analogous to those of the impact toughness. That is, Steel A1 had superior fracture toughness to that of the Steel A2 in the low heat input scenario. Furthermore, Steel A2 contained a greater number of coarse TiN type inclusions, while in Steel A1, the inclusions were mostly Ca-modified oxides and sulphides. Surprisingly, Steel A1 had actually more inclusions, more coarse inclusions and a larger overall area fraction of inclusions than that of Steel A2. However, the detrimental effect on the fracture toughness was dependent on the type of the inclusions. Presumably, the small and spherical Ca-modified oxides and sulphides were less harmful than the hard, brittle and sharp-edged nitrides.

Overall, the fracture surfaces of both Steels A1 and A2 were similar with the investigated specimens which had $K_{Jc(1T)}$ below 150 $MPa\sqrt{m}$. In these cases, the primary cleavage crack initiation sites that originated the early failures (Fig. 10) could be located. The crucial difference between the materials was that in Steel A2 the coarse TiN type inclusions were found at these sites whereas no clear initiators were found in Steel A1. In Steel A1, the causes of failure are presumably interfaces of M-A constituents or large upper bainite carbides. Fig. 12 shows an example of how a compact TiN cluster caused a brittle failure in Steel A2. Taking into account the frequent coarse TiN type inclusions observed at the initiation

sites and on the fracture surfaces of SENB specimens, it was rather obvious that these were the reason for the impaired toughness in Steel A2.

The fracture surfaces of Steels B1 and B2 with $t_{8/5} = 6$ s differed from other investigated specimens due to the more hardened microstructure. The finer effective grain size was observed directly from the fracture surfaces as having smaller cleavage facets. As a consequence, cleavage fracture initiators were more challenging to find than in the other variants, and in the case of Steel B1 with only a moderate number density of TiN class inclusions, they could not be identified at all. It is likely that in that case the fracture was initiated by coarse carbides of upper bainite in otherwise mainly lower bainitic or martensitic microstructure. With $t_{8/5} = 30$ s, TiN type inclusions were identified as the fracture initiators also in Steel B1, indicating that not even a moderate number density of these inclusions can be tolerated when the effective grain size increases.

Figure 12. Initiation of a brittle failure in Steel A2 ($t_{8/5} = 30$ s.) caused by a compact TiN cluster.

4.4 Measures for improving the impact toughness of the CGHAZ

Table 5 summarizes the microstructural factors, mechanical properties and other features of the simulated CGHAZ of the studied steels. It was evident that Steel A1 with $t_{8/5} = 6$ s had superior impact toughness and fracture toughness. This variant did not have coarse nitrides and the microstructure was a mixture of upper bainite and granular bainite with a larger fraction of upper bainite. The same Steel A1 with $t_{8/5} = 30$ s had a slightly greater effective grain size and $d_{80\%}$ compared to the microstructure resulting from the shorter cooling time and this led to a slight decrease in the impact and fracture toughness. Steel B1 with $t_{8/5} = 6$ s also had excellent impact toughness and relatively good fracture toughness due to a reduced effective grain size and $d_{80\%}$ compared to other variants, and a reduced number density of TiN class inclusions compared to B2. Impact toughness and fracture toughness followed generally the same trend in the studied steels. However, Steel B2 with $t_{8/5} = 6$ s had relatively good impact toughness compared to variants with $t_{8/5} = 30$ s, but the lowest fracture toughness among the studied steels, which emphasises the role of the brittle fracture initiators. This can be a reason for the reduced fracture toughness in Steel B2 with $t_{8/5} = 6$ s since this variant had an abundant number density of TiN class inclusions.

Based on earlier physical simulations, it has been suggested that the impact toughness of CGHAZ region in high strength steels should not be lower than 50 J at -50 °C for a full size specimen (approx. 25 J at -70 °C for 5 mm specimen [40]) in order to guarantee the safety of the structures in service conditions [31]. This criterion was clearly achieved with $t_{8/5} = 6$ s in the CGHAZs of Steels A1 and B1, which did not have any or only a moderate amount of TiN type inclusions, while in Steels A2 and B2 the inclusions increased the transition temperatures of the CGHAZs to close to or above the 25 J at -70 °C limit. Therefore, preventing the excessive formation of these inclusions appears to be the key to ensure sufficient impact toughness in the CGHAZ region of the studied 500 MPa offshore steels when using low heat-input welding methods such as FCAW.

With $t_{8/5} = 30$ s the 25 J at -70 °C requirement was only achieved in Steel A1 indicating that lowering the carbon content alone is not enough to improve the impact toughness in CGHAZ of the studied 500 MPa offshore steels when using high heat-input welding methods such as SAW.

It was shown that upper and granular bainitic microstructures with carbon content of 0.02% and lower bainitic microstructures with carbon content of 0.07% are beneficial in the CGHAZ region of the studied 500 MPa offshore steels when it comes to the impact and fracture toughness in low temperatures. However, in practical offshore multipass welds, various heat-affected sub-zones can initiate cracking and promote crack propagation. Thus, as the current study was limited to CGHAZ, further research is needed to examine the influence of multiple heat cycles and other heat-affected sub-zones.

Table 5. Summary of the key material performance measures and the resulting toughness values.

Sufficient toughness values are indicated with bold type, and the arrows show the effect of the factor: ↑ increases toughness and ↓ lowers toughness.

		Microstructure / (Hardness HV ₁₀)	D _{eff} (μm) / D _{80%} (μm)	TiN class (pcs mm ⁻²)	Failure initiator	T _{35J/cm²} (°C)	K _{Jc(1T)} @ -40 °C (MPa√m)
Steel A 6 s	A1	UB-GB / 235-239	3.5 / 40.9	0 (↑)	Undefined	< -100	215 ± 8
	A2			6.7	TiN class	-67	119 ± 29
Steel A 30 s	A1	GB-UB / 202-214	5.7 / 44.4 (↓)	0 (↑)	Undefined	-85	193 ± 80
	A2			6.7	TiN class	-67	177 ± 118
Steel B 6s	B1	M-LB-UB / 274-280	2.1 / 8.7 (↑)	0.8	Potential coarse UB carbides	< -100	126 ± 16
	B2			3.5	TiN class	-77	71 ± 23
Steel B 30 s	B1	GB-UB / 208-210	3.3 / 33.5	0.8	TiN class	-52	107 ± 23
	B2			3.5	TiN class	-41	106 ± 24

5 CONCLUSIONS

Two experimental 500 MPa offshore steels with different compositions were studied. Samples were taken from two different slabs of the steels giving different impurity levels and thus different inclusion structures. The focus of the study was on the low-temperature impact toughness and fracture toughness of physically simulated CGHAZs using cooling times of 6 s and 30 s from 800 °C to 500 °C in order to simulate the two different welding methods commonly used for welding offshore steels, i.e. FCAW and SAW. This also resulted in different transformed microstructures in the CGHAZs of the both steels, thereby enabling the study of the effect of the microstructure together with inclusions. The main difference in the inclusion structures of the studied steels regarding the impact toughness and fracture toughness was the presence or absence of coarse titanium-niobium nitrides. The following conclusions can be drawn:

- The difference in the carbon content caused different microstructures to form after the CGHAZ simulation. This was most significant with the cooling time of 6 s from 800 °C to 500 °C where the carbon content of 0.02 % resulted in predominantly upper bainite while the carbon content of 0.07 % caused the microstructure to change to lower bainite with a higher hardness. With the cooling time of 30 s from 800 °C to 500 °C, the simulated CGHAZ microstructure consisted a mixture of granular and upper bainite with both carbon contents.
- Fractographic examination showed that brittle fracture was initiated by coarse titanium-niobium nitrides in most of the cases. Therefore, it is evident that coarse TiN type inclusions should be avoided in order to ensure the best possible toughness in harsh service conditions.

- Where primary failure initiators could not be identified, they were expected to be coarse carbides in an upper bainitic matrix. This indicates that with high heat-input welding, lowering the carbon content may be necessary to achieve optimal transformed microstructures.
- With low heat-input welding methods, fracture toughness and impact toughness are improved mainly by the absence of coarse titanium-niobium nitrides. The resulting fine-grained and potentially lath-like microstructures are beneficial for toughness properties.
- Charpy V-notch upper shelf energy in CGHAZ is dependent on strength. However, in identical microstructures with the same strength it was shown that USE decreases when the total number density of inclusions increases.

ACKNOWLEDGEMENT

The authors are grateful to Business Finland for financing this work as a part of the research projects DIMECC SIMP – System Integrated Metals Processing and FLEX – Flexible and Adaptive Operations in Metal Production. Dr. Pallaspuro thanks the Academy of Finland (#311934) for funding.

DATA AVAILABILITY

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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