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Effect of welding on microstructure and mechanical response of X100Q bainitic steel through nanoindentation, tensile, cyclic plasticity and fatigue characterisation

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ABSTRACT
This paper presents an experimental characterisation of fatigue at welded connections for the next-generation high-strength low-alloy offshore riser steel, X100Q. An instrumented girth weld is conducted with a parallel programme of physical-thermal simulation (Gleeble) to develop heat affected zone (HAZ) test specimens. X100Q is shown to exhibit superior fatigue performance to the current state of the art offshore riser steel, X80. Significant differences are demonstrated between the parent material and simulated HAZ in terms of hardness, monotonic strength and cyclic plasticity response, which can be related to the observed microstructural transformations: the refined grain and bainitic block size in the fine-grained HAZ are shown to give a harder and stronger response than parent material, whereas the coarsened bainitic lath structure in the intercritical HAZ gives a softer and weaker response. The simulated HAZ materials exhibit superior fatigue performance to the parent material and weld metal. A significant reduction in life is shown for cross-weld specimens, indicating susceptibility to failure due to HAZ softening for matched or over-matched X100Q welds.

1. Introduction

Flexible marine pipe is currently the most common solution for offshore risers; however, its viability in deep- and ultra-deepwater is limited due to its high cost and low-pressure capacity. Due to these limitations, currently, the deepest installed flexible riser is at a depth of 1900 m, with an internal diameter of 190 mm \cite{1}. Steel catenary risers (SCRs), are an attractive alternative, which can withstand the pressure loads associated with extreme water depth and large riser diameters, typically require less capital expenditure, and offer a greater service life. The uptake of higher-strength line pipe steels for SCRs facilitates access to ever-increasing sea depths, such as the Shell Stones field at a depth of 2926 m \cite{2}, by allowing for the specification of a lower wall thickness pipe, thus reducing the buoyancy requirements due to self-weight, as well as allowing for increased efficiency in welding and installation.

Welding-induced degradation of the refined microstructure required to obtain high strength and toughness is a key challenge in the use of next-generation high strength steels for SCRs. A reduction of mechanical performance in the HAZ combined with the stress concentration associated with the weld geometry (\cite{3-5}) create the potential for fatigue hot-spots in the weld-affected zone. The microstructure and mechanical performance in the HAZ may be improved to a large extent through close control of the welding process, post weld heat treatment and the addition of alloying elements \cite{6}. However, the use of lean alloy steels and high throughput welding processes are necessary to maximise the efficiency of offshore welding during pipe laying.

Welding trials are required to accumulate the necessary body of knowledge and experience, with respect to the suitability of welding processes, parameters, and consumables, to facilitate the uptake of next-generation materials. In a recent review on welding of high-strength line pipe steels, Sharma et al. \cite{7} identified a gap in the literature relating to published work on API 5L X100 \cite{8}. The majority of X100 welding trials to date have focussed on large diameter seam welded pipe sections typical of onshore gas pipelines. For example, Thewlis \cite{9} and Hudson \cite{10} conducted a series of trials and found the tensile and impact strength of the weld to be highly sensitive to both the weld metal and...
composition and the welding process parameters. The most comprehensive fatigue test programme published to date on welded X100 is presented in the European FATHOMS report [5], where a series of unbacked girth welding trials using flux-cored arc welding (FCAW) and laser hybrid gas metal arc welding (GMAW) processes were conducted, using pipe sections and welding positions typical of offshore installations. The laser hybrid GMAW process was shown to deliver significantly improved welding productivity and a superior weld profile to the FCAW process. During the fatigue test programme, the weld root was shown to be the critical location for fatigue failure, which is in agreement with the findings of Buitrago and co-workers [11] and Maddox et al. [12] for the fatigue of full-scale SCR girth welds.

The small size and continuously graded nature of the HAZ presents a challenge for mechanical characterisation via conventional test methods. Hardness measurements are an effective indicator of the variation in mechanical properties across the weld and may be used to determine local elastic and viscoplastic properties [13], however, the manufacture of tensile and fatigue test specimens is required to ascertain failure behaviour. It is possible to manufacture miniature test specimens from the HAZ [14], but there are issues associated with the influence of microstructural inhomogeneity and the size effect. Physical-thermal simulation is an experimental technique which has been developed to overcome these difficulties by allowing for the creation of homogeneous simulated HAZ specimens via controlled heating and cooling cycles [15]. Ringinen and co-workers [16] examined the influence of cooling rate on the microstructure, hardness and toughness of X100 HAZ using Gleeble physical-thermal simulation. Guiying et al. [17] also used this technique to characterise the effect of peak temperature on the strength and toughness of X100 HAZ. HAZ specimens created using a peak temperature of 950 °C, corresponding to fine-grained HAZ (FGHAZ), were shown to give the lowest yield and tensile strength, whereas impact

Table 1
The chemical composition of the as-received X100Q (wt. %).

| C  | Si  | Mn  | P   | S   | Al  | B  |
|----|-----|-----|-----|-----|-----|----|
| 0.14 | 0.21 | 1.17 | 0.008 | 0.001 | 0.084 | 0.0025 |
| Cr | Cu  | Mo  | N   | Nb  | Ni  | Ti |
| 0.32 | 0.01 | 0.3  | 0.004 | 0.027 | 0.02  | 0.005 |

Table 2
The chemical composition of Böhler NiCrMo 2.5-IG filler wire [25] (wt. %).

| C  | Si  | Mn  | Cr | Ni | Mo |
|----|-----|-----|----|----|----|
| 0.08 | 0.6  | 1.4 | 0.3 | 2.5 | 0.4 |

Fig. 1. (a) Schematic of the X100Q pipe section highlighting the thermocouple array location with (b) detail views showing the pipe bevel dimensions and thermocouple (TC) array and (c) images of the instrumented pipe ring (Dimensions in mm).
strength was lowest for specimens created using a peak temperature of 750 °C, corresponding to intercritical HAZ (ICHAZ).

The variation of monotonic and cyclic response in the HAZ for materials with hierarchical microstructures, such as bainitic X100, can be related to the effect of microstructural transformations on the key strengthening mechanisms: dislocation density and obstacles to slip [18]. For example, high-angle boundaries (HABs), comprising of prior austenite grain boundaries (PAGBs), packet boundaries and block boundaries, and low angle boundaries (LAB), compromising of bainitic lath boundaries, contribute to strength following inverse-square relationships ([19–22]). However, under cyclic plastic deformation, the strengthening effects of dislocation density and LABs typically diminish for bainitic steels as a result of microstructural degradation ([23,24]).

The motivation of the present work is the development of process-structure-property relationships which may be implemented via computational modelling to predict the mechanical and fatigue performance of metallic materials with complex thermal histories, such as the weld-affected zone. The experimental methodology and results from a

Fig. 2. The specimen designs used for (a) tensile and fatigue tests conducted at Fraunhofer IWM and (b) fatigue tests conducted at NUI Galway.

Fig. 3. A schematic highlighting the locations from which WM and CW fatigue specimens were manufactured from the X100Q girth weld, with weld metal and cross-weld section cuts shown inset.
representative full-scale SCR girth weld on an X100Q pipe section and a parallel programme of (Gleeble) physical thermal-simulation of HAZ are presented. The key microstructural zones of the girth weld and simulated HAZ are characterised using microscopy and nanoindentation. The constitutive and fatigue performance of X100Q PM, weld metal (WM) and simulated HAZ are characterised for the first time through a programme of tensile and strain-controlled fatigue tests. The fatigue performance of the weld is investigated via a programme of force-controlled fatigue tests conducted on both PM and cross-weld (CW) specimens.

2. Methodology

2.1. Girth welding

The quenched and tempered X100Q steel was supplied by Eisenbau Krämer (Kreuztal, Germany) in the form of a 406 mm outer diameter seam welded pipe section with a wall thickness of 25 mm. The chemical composition of the steel is shown in Table 1. In preparation for girth welding, two 250 mm long pipe rings were cut from the as-received pipe and a compound bevel was machined on each pipe ring using a lathe. Böhler NiCrMo 2.5-1G 1.2 mm filler wire [25] was used for welding. The reported chemical composition of the filler wire is shown in Table 2.

The temperature history in the vicinity of the weld zone was recorded using six type K thermocouple probes. The thermocouple probes were inserted via an interference fit into blind holes on the inner surface of the pipe. The holes were drilled to three unique positions, in terms of through-thickness depth and distance from the bevel, as shown in Fig. 1(a and b), such that the path for thermal conduction to any probe was not impeded and the variation of temperature history through the wall thickness could be captured. Three high-temperature strain gauges, with built-in temperature compensation, were used to measure the axial and hoop strains in the vicinity of the girth weld. The strain gauges were bonded to the inner surface of the pipe, adjacent to the thermocouple array and 5 mm from the pipe bevel, using high-temperature ceramic cement. Images of the instrumented pipe ring are shown in Fig. 1(c).

Prior to welding, the bevel and adjacent surfaces of the pipe rings were ground and degreased to remove any contamination from the weld zone. The pipe rings were then tack welded together using tungsten inert gas welding, to ensure correct alignment and a root gap of 4 mm. Welding was conducted at Glenfield Engineering (Kilmallock, Ireland) using a Rotoweld 3.0 automated welding station [26].

2.2. Physical-thermal simulation of HAZ

Physical-thermal simulation was conducted using a Gleeble 3150 thermomechanical simulator at Fraunhofer IWM (Freiburg, Germany) to manufacture homogeneous specimens of HAZ representative material for mechanical characterisation. Thermal cycles consistent with four distinct regions within the HAZ were applied to cylindrical specimens, 120 mm in length and 10 mm in diameter. Two thermal cycles with a peak temperature above $A_c_3$ of 950 °C, corresponding to FGHAZ, were selected based on the work of Guiying et al. [17], who found that the yield and tensile strength of X100 simulated HAZ was lower for this peak temperature. Cooling rates of 10 °C/s and 30 °C/s, spanning the favourable field of welding conditions determined for the girth weld, were used for the simulated FGHAZ thermal cycles. Two thermal cycles representative of the ICHAZ were also chosen due to the reduction of

| Material     | Strain range (%) | No. of tests |
|--------------|------------------|--------------|
| PM           | 1.2              | 2            |
|              | 1.0              | 4            |
|              | 0.8              | 1            |
|              | 0.7              | 1            |
|              | 0.6              | 1            |
| WM           | 1.2              | 1            |
|              | 1.0              | 1            |
|              | 0.8              | 1            |
|              | 0.7              | 1            |
|              | 0.6              | 1            |
| (5 and 10 °C/s) ICHAZ | 1.2 | 1 |
|              | 1.0              | 1            |
|              | 0.8              | 1            |
| 10 °C/s FGHAZ | 1.2              | 1            |
|              | 1.0              | 4            |
|              | 0.8              | 2            |
|              | 0.7              | 1            |
|              | 0.6              | 1            |
| 30 °C/s FGHAZ | 1.2              | 1            |
|              | 1.0              | 5            |
|              | 0.8              | 1            |
|              | 0.7              | 1            |
|              | 0.6              | 1            |

| Material     | Stress range (MPa) | No. of tests |
|--------------|--------------------|--------------|
| PM           | 1200               | 1            |
|              | 1150               | 1            |
|              | 1100               | 1            |
|              | 1020               | 1            |
| CW           | 1100               | 1            |
|              | 900                | 1            |

Table 3

The strain-controlled fatigue test programme for X100Q PM, WM and simulated HAZ. Fully reversed loading was used for all tests.

Table 4

The force-controlled fatigue test programme for X100Q PM and CW. Fully reversed loading was used for all tests.
hardness shown in this region of the girth weld, with cooling rates of 5 °C/s and 10 °C/s based on the experimentally observed values.

For all thermal cycles, the specimens were resistance heated at a rate of 100 °C/s to the peak temperature, which was maintained for half a second before the specimens were actively cooled to 110 °C, at which point they were allowed to passively cool. The simulated FGHAZ specimens were cooled using air blasting and by conduction through the water-cooled copper grips. Ambient air cooling and conduction through the grips were sufficient to achieve the required cooling rates for the simulated ICHAZ specimens.

2.3. Microstructural analysis

Optical microscopy was performed on specimens which were extracted from the PM, simulated FGHAZ and girth weld, then etched using Vilella’s reagent to expose the microstructural features. The sample preparation technique is described in detail by Devaney [27]. The grain sizes of the PM, 10 °C/s and 30 °C/s simulated FGHAZ were determined by using binary images generated from optical micrographs using ImageJ processing software [28] in a MATLAB grain size measurement tool developed by Lehto et al. ([29,30]). The average grain size, \( d \), was determined for each microstructure following the linear intercept length method [31].

For materials with an inhomogeneous grain structure, the relationship between the average grain size and mechanical response may not adhere closely to well-known relationships, such as the Hall-Petch equation ([19,20]). In particular, larger grains are associated with a reduced strength level [32], due to the length and number of slip bands within the grain. Therefore, a small number of large grains can substantially influence the mechanical response of a material. To account for this effect a rule of mixtures approach was also implemented, where the contribution of each grain to the average grain size is considered to be proportional to the volume of the grain. Thus, the volume-weighted average grain size, \( d_v \), is defined as:

\[
d_v = \frac{1}{V_T} \sum_{i=1}^{n} V_i d_i
\]

where \( V_T \) is the total volume of material and \( V_i \) is the volume of grains corresponding to the grain size \( d_i \). To determine the volume-weighted average grain size, the point sampled intercept length method described by Gunderson et al. ([33,34]) is used in a manner similar to the linear intercept length method. However, the measurements taken from random points within the microstructure, and the orientation in which the intercept length measurement is taken from each point is also selected at random. Consequently, the grain sizes are measured in proportion to their surface area fraction. Further detail on the

Fig. 5. (a) The thermal histories obtained from thermocouples TC2, TC4 and TC6 (as labelled in Fig. 1) and (b) the axial strain history recorded at the inner surface of the pipe section during welding.
volume-weighted average grain size methodology is given by Lehto et al. [29].

Scanning electron microscopy (SEM) and backscatter electron microscopy (BSE) analysis were conducted at NUI Galway using a Hitachi S-4700 SEM with energy-dispersive X-ray spectroscopy (EDX). Electron backscatter diffraction (EBSD) scans of X100Q PM, 10 °C/s and 30 °C/s simulated FGHAZ were taken at the University of Limerick using a Hitachi SU-70 SEM. The scans were conducted at a tilt angle of 70° using a step size of 0.4 μm over an area of 120 μm by 120 μm. The scan results were processed in MATLAB using the MTEX toolbox [35], where non-indexed pixels were removed by assigning the orientation of the surrounding pixels and microstructural boundaries were plotted based on misorientation angle. Boundaries with a misorientation between 2° and 15° were determined to be LABs and boundaries with a misorientation above 15° were determined to be HABs [36].

2.4. Nanoindentation testing

Nanoindentation testing was conducted to characterise hardness in the PM, simulated FGHAZ and CW (girth weld) samples using a Keysight G200 nanoindenter with a Berkovich diamond tip. A convergence study on the effects of loading rate and maximum load on the indentation hardness of X100Q PM was conducted to determine suitable loading conditions. The measured hardness was found to reduce, and the consistency of hardness measurements increase with increasing maximum load. These trends can be attributed to the increase of indentation size with load, and therefore the reduced influence of size effect [37] and individual material phases ([38,39]), leading to a bulk response. An appropriate maximum load of 850 mN was determined, with a hold time of 10 s and a loading rate of 6.7 mN/s. Following the recommendations of Shirazi [40], all indentations were conducted with a spacing of 150 μm, greater than thirty times the indentation depth.

2.5. Tensile testing

X100Q PM, simulated ICHAZ and FGHAZ specimens machined from the longitudinal direction of the pipe section were used in the tensile test programme. The specimens were tested under strain control, at a strain rate of 0.01%/s, using a knife-edge extensometer. A non-standard tensile specimen design, following the ASTM E606 standard [41] as shown in Fig. 2(a), was used to ensure that the reduced section, was entirely contained within the calibrated thermally transformed region representing the simulated HAZ. This was key to ensuring that the measured response related to the simulated HAZ material and to avoid failures outside this region as obtained in an initial test programme ([27, 42]) (results not shown here for brevity).

Strain was measured using a knife-edge extensometer fixed to the specimen gauge length by elastic bands and control was achieved using a closed-loop feedback system. For strain-controlled tests, fully reversed loading was applied using a sinusoidal waveform at an average strain rate of 0.2%/s. Force-controlled fatigue testing was used to characterise X100Q CW specimens, due to the inhomogeneous strain distribution which was expected as a result of the multi-material gauge length. Force-controlled testing was conducted following the ASTM E466 [43] standard, using fully reversed loading with a sinusoidal waveform at 1 Hz. For comparative purposes, a series of force-controlled tests were also conducted on X100Q PM using the same test conditions.

The strain- and force-controlled fatigue test programmes are shown in Table 3 and Table 4, respectively.

3. Results

3.1. Girth welding

The weld root was completed using a single surface tension transfer pass, with the subsequent eleven fill passes and two cap passes deposited using spray-transfer GMAW. Further details of the welding process are given elsewhere by Devaney et al. ([27,44]). An image of the completed girth weld is shown in Fig. 4(a); the weld pass sequence that was followed is shown in Fig. 4(b) and an etched cross-section of the completed weld is shown in Fig. 4(c).

The thermal and axial strain histories recorded adjacent to the weld are shown in Fig. 5(a) and (b), respectively. The axial strain on the internal surface of the pipe section is tensile as the welding torch approaches the strain gauge location, due to thermal expansion of the pipe, and becomes compressive as the torch reaches the opposite side of the pipe. Damage to strain gauges during the welding process prevented measurement of hoop strain.
3.2. Physical-thermal simulation of HAZ

An image of a FGHAZ physical-thermal simulation in progress is shown in Fig. 6(a) and typical examples of the experimentally measured thermal cycles for both the simulated FGHAZ and ICHAZ specimens are shown in Fig. 6(b).

3.3. Microstructural analysis

A sample optical micrograph of X100Q PM and the associated binary image, which was generated for the purposes of grain size measurement,

| Material     | \( d \) (\( \mu \text{m} \)) | \( \Delta d/d \) | \( d_v \) (\( \mu \text{m} \)) |
|--------------|-------------------------------|------------------|------------------------|
| PM           | 18.96                         | 3.23             | 28.3                   |
| 10 °C/s FGHAZ | 13.6                          | 2.5              | 18.7                   |
| 30 °C/s FGHAZ | 8.1                           | 2.5              | 11.1                   |

Fig. 7. (a) An optical micrograph of X100Q PM, (b) the optimised binary image used for grain size measurements created using ImageJ and (c) the measured grain size distribution for X100Q PM, with a normal probability density function fitted.

Fig. 8. SEM micrographs of X100Q PM with a grain boundary highlighted and (b) a close-up view of the area surrounding the grain with microstructural features labelled.
are shown in Fig. 7 (a and b), respectively. The measured grain size distribution for X100Q PM is shown in Fig. 7 (c) with a fitted normal probability density function.

The average grain size, relative grain size dispersion, $\Delta d/d$, and volume-weighted average grain size values determined for the PM, 10 °C/s and 30 °C/s simulated FGHAZ are shown in Table 5. SEM micrographs of X100Q PM, highlighting a grain boundary, regions of bainite, granular bainite and retained austenite (RA), are shown in Fig. 8.

The EBSD orientation maps obtained for X100Q PM, 10 °C/s and 30 °C/s simulated FGHAZ are shown in Fig. 9. The LABs are represented by white lines and the HABs are represented by black lines. The microstructural texture of the PM and simulated FGHAZ materials were also compared by plotting the orientations of 1000 randomly selected sample points from the orientation maps on the IPFs, also shown in Fig. 9.
The distributions of bainitic block size determined for X100Q PM and simulated FGHAZ using the circular diameter method [45], are shown in Fig. 10.

3.4. Nanoindentation testing

A labelled image of the CW sample which was used for microstructural and hardness characterisation of the girth weld is shown in Fig. 11 (a). Nanoindentation characterisation of the girth weld was conducted in the outlined area, which spans the PM, HAZ and WM. The indentation hardness values obtained from a single trace through the CW sample, with the corresponding weld regions labelled, are shown in Fig. 11 (b). The weld regions were identified based on the microstructural morphology observed at each indent. There is a consistent level of hardness in the PM, which then decreases to its lowest value in the ICHAZ, before increasing through the FGHAZ and coarse-grained HAZ (CGHAZ), where there is a sudden reduction at the fusion zone in the WM.

Fig. 12 shows the indentation hardness map of the girth weld sample region outlined in Fig. 11(a), with optical micrographs of the primary microstructural regions. The trends in hardness variation with microstructure, shown in Fig. 11(b), are consistent for all traces in the hardness map. The microstructure in the softest region of the girth weld sample corresponds to ICHAZ, where the hardness drops to a low of 3 GPa on average. The effect of weld geometry on the hardness within the characterised region can be seen in the hardness map, where the soft ICHAZ region follows the bevel angle of the girth weld.

The optical micrographs show defined grain boundaries in the PM, with a microstructure consisting of lath bainite and granular bainite with small amounts of polygonal ferrite. The grain size in the ICHAZ is similar to the PM, but the grain boundaries are less defined and there is a significantly reduced lath microstructure, with martensite-retained austenite islands and spheroidised carbides visible, indicating partial transformation. There is a significant reduction of grain size in the FGHAZ due to the occurrence of complete transformation and recrystallisation during welding, with defined grain boundaries visible and a microstructure consisting of granular bainite with some lath bainite and polygonal ferrite. The grain size in the CGHAZ is significantly greater relative to the PM, with defined grain boundaries and a microstructure consisting primarily of lath bainite.

The microstructural morphologies of the simulated FGHAZ materials, shown in Fig. 13, are similar to the FGHAZ of the girth weld. The simulated FGHAZ materials have a refined grain size with defined grain boundaries and microstructures consisting of granular bainite, with some lath bainite and polygonal ferrite. The level of grain refinement in the 30 °C/s simulated FGHAZ microstructure is increased relative to the 10 °C/s simulated FGHAZ.

Typical load-displacement curves obtained from indentations in the PM, girth weld ICHAZ, WM, 10 °C/s and 30 °C/s simulated FGHAZ are shown in Fig. 14. The mean values of indentation hardness for the materials are presented in Table 6. The differences in hardness are consistent with deviations in displacement at maximum load for each material.

3.5. Tensile testing

The tensile engineering stress-strain responses obtained for X100Q PM and simulated HAZ materials are shown in Fig. 15. As expected, the higher cooling rate simulated HAZ materials give a stronger response in each case. The measured tensile properties for each material are shown in Table 7. The WM tensile properties were obtained from the first quarter cycle of the strain-controlled fatigue tests.

3.6. Fatigue testing

The stress-strain responses and stress amplitude evolutions observed for X100Q PM, WM, 5 °C/s ICHAZ and 30 °C/s FGHAZ during the strain-controlled fatigue test programme are shown in Fig. 16 to Fig. 19. Continuous yielding behaviour is shown in the first quarter cycle for the PM and simulated HAZ materials, in contrast to the WM, which exhibits a yield plateau.

Comparisons of the first and half-life cycle stress-strain responses obtained for a strain range of 1.2% are shown in Fig. 20. Consistent with

Fig. 10. The measured block size distributions in X100Q PM, 10 °C/s and 30 °C/s simulated FGHAZ.

Fig. 11. (a) An etched cross-section of the girth weld, showing the area used for nanoindentation testing and (b) the indentation hardness values obtained from a sample trace across the girth weld, with the identified weld regions labelled.
the tensile performance discussed above, simulated FGHAZ exhibits the highest stress range in the first cycle, with the higher cooling rate material giving a slightly stronger response. At the half-life cycle, the stress range of simulated FGHAZ is significantly greater than the PM, WM and simulated ICHAZ, which exhibit an essentially converged saturated stress-strain response as a result of cyclic softening.

Comparisons between the stress amplitude evolutions and cyclic softening behaviours of the materials for the same strain range are shown in Fig. 21. Cyclic softening behaviour is exhibited by all materials, with the majority of softening occurring during the early cycles, which is followed by a period of stabilised secondary softening until the cycles preceding failure, where softening accelerates.

The strain-life and plastic strain-life (Coffin-Manson) type relationships observed during strain-controlled testing are shown in Fig. 22. Simulated FGHAZ displayed superior fatigue performance to PM, WM and simulated ICHAZ, with 10 °C/s FGHAZ giving the most consistently high fatigue performance. Simulated ICHAZ exhibits superior fatigue performance to PM and WM. The cyclic stress-strain power law constants for X100Q PM, WM and simulated HAZ, which were fitted to the half-life response for each material, are shown in Table 8 and the identified Coffin-Manson fatigue constants are shown in Table 9.

During force-controlled fatigue testing of X100Q PM, a small level of strain ratcheting occurs in the early cycles, and the plastic strain range increases due to cyclic softening; however, an essentially stabilised value is achieved relatively quickly until the cycles preceding failure, where the tensile strain amplitude increases significantly due to the influence of crack propagation. The stress amplitude-life relationships obtained for X100Q PM and CW during force-controlled fatigue testing...
are shown in Fig. 23. The authors are not aware of a comparable fatigue characterisation of X100Q; therefore, a comparison is presented against results obtained by Jung et al. [46] for X80. The fatigue life of X100Q is on average over twice that of X80 for a given stress amplitude. Examination of the post-test fatigue specimens revealed the influence of inclusions and voids within the material on fatigue crack growth. Fatigue cracks were found to propagate along paths with a higher density of voids and inclusions, typically of type Ca–O or Al–O. Cross-sections of post-test PM and simulated FGHAZ specimens are shown in Fig. 24, with detailed SEM micrographs of the voids and inclusions within the PM fatigue crack which were identified using EDX.

4. Discussion

The tensile axial strain recorded at the weld root exceeds the yield strain during welding, as shown in Fig. 5. However, the residual strain is compressive due to the repeated uneven shrinkage resultant from the welding process. These findings are typical of a multipass girth weld and qualitatively in agreement with those shown for X100 girth welds in the FATHOMS report [5] and those reported by Maddox and co-workers [12] for girth welds conducted on X52, X65 and X80 pipe sections with thickness greater than 15 mm.

The microstructural transformation to simulated FGHAZ is apparent through the pronounced reduction in grain and block size, and the equiaxed appearance of the FGHAZ microstructures compared to the PM, shown in Fig. 9. Although the mode value of block size is essentially the same amongst the microstructures, there is a greater variance in the distribution of block size in the PM, as shown in Fig. 10, which is indicative of the inhomogeneous and elongated block microstructure. There are no clear texture effects in the PM or simulated FGHAZ microstructures due to the pipe manufacturing or physical-thermal simulation processes. The random sample points in Fig. 9 are more uniformly distributed for the simulated FGHAZ than the PM, although this difference may be attributed to the increased sample size, in terms of number of bainitic blocks, for the simulated FGHAZ EBSD scans.

The girth weld ICHAZ softening, shown here through nanoindentation testing, is in agreement with trends shown in the literature for welded X100 [47] and similar lean high-strength low-alloy (HSLA) steels, as discussed by Pisarski et al. [48]. This softening phenomenon is attributed to the partial transformation of the bainitic lath microstructure to form regions of ferrite, see Fig. 12, as shown for similar quenched and tempered steels in the same strength class [49]. The hardness of the simulated FGHAZ is significantly increased relative to the PM. However, the hardness of the simulated FGHAZ microstructure is also significantly higher than the FGHAZ of the girth weld, shown in Figs. 11 and 12. This difference in hardness (between simulated FGHAZ and girth weld FGHAZ) is attributed to the tempering effect of subsequent weld passes in the girth weld, which was not represented during the physical-thermal simulation process. The significance of this tempering effect may be investigated in future work via bead on plate welding trials.

The yield strength of the PM is greater than the simulated ICHAZ or FGHAZ, as shown in Fig. 15 and Table 7, which is attributed to the strengthening effect of LABs in the refined bainitic lath microstructure, which has been shown to follow an inverse square relationship [21, 15].
Fig. 16. Strain-controlled fatigue results for X100Q PM showing: (a) first cycle stress-strain responses, (b) half-life cycle stress-strain responses, (c) evolutions of stress amplitude and (d) superposition of the half-life stress-plastic strain responses.
Fig. 17. Strain-controlled fatigue results for X100Q WM showing (a) first cycle stress-strain responses, (b) half-life cycle stress-strain responses, (c) evolutions of stress amplitude and (d) superposition of the half-life stress-plastic strain responses.
Fig. 18. Strain-controlled fatigue results for 5 °C/s simulated ICHAZ showing (a) first cycle stress-strain responses, (b) half-life cycle stress-strain responses, (c) evolutions of stress amplitude and (d) superposition of the half-life stress-plastic strain responses.
The PM lath width is estimated to be in the order of 0.3 μm based on analysis of the EBSD orientation maps [51], in agreement with transmission electron microscopy analyses conducted by Wang et al. and Duan and co-workers ([52,53]). Physically-based constitutive modelling presented by the authors elsewhere ([27,54]), indicates that the strengthening effect of lath LAB structures in the 10 °C/s simulated FGHAZ is reduced by 43% compared to the PM. However, the refined grain and block HAB structures in the simulated FGHAZ are predicted to provide a 42% greater contribution to strength than in the PM, and a significantly increased level of strain hardening. For the 5 °C/s simulated ICHAZ the contribution of LAB strengthening is minimal due to transformation-induced coarsening and degradation of the lath structure. Transformation-induced coarsening in the simulated ICHAZ is predicted to influence grain and block HAB strengthening to a lesser extent, with a reduction of 17% compared to the PM. The values of elongation at failure obtained for the PM and simulated HAZ microstructures using the short tensile specimen design are higher than would typically be expected. This is due to the significant proportion of the gauge length which undergoes necking prior to failure. However, the yield strength, tensile strength and uniform elastic and plastic elongation are unaffected by this phenomenon.

The cyclic-plastic responses of the different weld zones can be explained by reference to their microstructural characteristics. The PM and simulated FGHAZ microstructures exhibit non-Masing behaviour, see Figs. 16 and 19, which is indicative of coarsening of the bainitic lath microstructure via dislocation motion to form stable dislocation cell structures [55]. This phenomenon results in a dependence of saturated softening stress on strain range. Conversely, the WM and simulated ICHAZ conform to Masing behaviour and exhibit no dependence between saturated softening stress and strain range, as shown in Figs. 17 and 18. The extent of cyclic softening is greatest in the PM, as shown in Fig. 21, due to the reduction of strength associated with coarsening of the refined bainitic lath microstructure ([56–58]). This cyclic softening phenomenon is reduced in the simulated FGHAZ, due to the lower influence of LAB strengthening, and is almost absent in the WM and simulated ICHAZ microstructures due to their significantly reduced prevalence of lath structures. Indeed, it appears that softening reduces proportionally with LAB density within the microstructures. The simulated FGHAZ microstructures exhibit superior fatigue performance to the PM, WM and simulated ICHAZ, as shown in Fig. 22. This is attributed to the strengthening effect of the refined block structures and the reduced influence of LAB strengthening. As a result, the stabilised plastic strain amplitude for a given applied strain-range is significantly less in the simulated FGHAZ than in the PM, WM or simulated ICHAZ materials, as shown in Fig. 20. It is interesting to note that the plastic strain-life (Coffin-Manson) responses of the microstructures are much more similar than total strain-life responses, as shown in Fig. 22(b), particularly for higher strain amplitudes. Thus, the microstructure-induced differences in yield strength (e.g. HAB, LAB densities) and cyclic softening (LAB density) behaviour are predominant factors for fatigue performance of the weld-affected zone. A similar correlation between low cycle fatigue performance and grain size has been shown for CoCr alloy by Sweeney et al. [59].

The detrimental effect of inclusions and voids on the fatigue
The performance of the tested materials is apparent from the regularity at which they are observed in the cracks of post-test fatigue specimens, as shown in Fig. 24. The primary inclusions which have been identified are regular shaped and of type Al–O and Ca–O, which are typically by-products of the deoxygenation process during steel manufacture. No significant difference has been noted in the regularity of inclusions in the PM and simulated HAZ materials, which is expected due to their melting temperature above 2000 °C. These inclusions act as sites for crack nucleation or propagation under fatigue loading. The detrimental effect of inclusions on fatigue performance may be estimated using a modified fatigue limit, which accounts for inclusion diameter and hardness [60]. However, geometrically complex inclusions, which are associated with casting or thermomechanical processing, have been shown by O’Hara and co-workers [61] to negatively impact fatigue life to a greater extent.

The influence of the two primary welding process conditions, peak temperature and cooling rate, on microstructure, mechanical and fatigue performance have been investigated in a range typical for an SCR welding process via physical-thermal simulation. Within this range of conditions, peak temperature is shown to have the greatest effect on microstructure, mechanical and fatigue performance, contributing to a reduction of up to 60% in grain size and a reduction of up to 46% in yield strength. In contrast, an increased cooling rate (from 10 to 30 °C/s) is shown to contribute to a reduction in grain size of 40% and an increase in yield strength of just 3% for the simulated FGHAZ and microstructures. Similar behaviour is shown for the simulated ICHAZ microstructures, where an increased cooling rate (from 5 to 10 °C/s) results in an
increase of 2% in yield strength and 4% in tensile strength, in agreement with the trends shown by Ringinen et al. [16] and Guiying et al. for X100.

The superior fatigue performance of X100Q compared to the current state of the art offshore riser steel, X80, can be seen from the force-controlled fatigue test results. However, the fatigue performance of X100Q CW is significantly reduced from that of the PM, due to inhomogeneous gauge length containing PM, WM and HAZ, where failure was found to occur. The reduced fatigue performance of the X100Q CW specimens is attributed to strain localisation due to the presence of a softened ICHAZ, a phenomenon shown by Zhang and co-workers [49] for quenched and tempered HSLA steels in the same strength class and by Adeeb et al. [62] via digital image correlation on X100 CW tensile specimens. This indicates susceptibility to tensile and fatigue failure in the ICHAZ for matched or over-matched X100Q welds, due to this strain localisation and the resultant inhomogeneous material softening.

5. Conclusions

An experimental programme has been conducted following a process-structure-property methodology to investigate the microstructure, monotonic and fatigue performance of welded X100Q. This work represents the first constitutive and fatigue characterisation of X100Q parent material, weld metal and simulated HAZ microstructures. The key conclusions are:

- A significant variation in hardness is shown across the girth weld, with a reduction of 17% exhibited in the intercritical HAZ for welded X100Q, consistent with findings for other HSLA steels.
- For the welding conditions investigated via physical-thermal simulation, peak temperature has a greater influence than cooling rate on the microstructure, mechanical and fatigue performance of the HAZ.
- The parent material exhibits the highest yield strength due to its refined bainitic lath microstructure; however, the refined bainitic block microstructure in the fully transformed simulated fine-grained HAZ give the highest tensile strength. The yield strength of the partially transformed simulated intercritical HAZ microstructures is reduced by almost 50% compared to the parent material. The higher cooling rate simulated HAZ microstructures give a stronger response in each case.
- Non-Masing cyclic softening behaviour is displayed by the parent material and fine-grained HAZ microstructures, which is attributed to lath coarsening and dislocation cell formation. The simulated intercritical HAZ conforms to Masing behaviour and exhibits the lowest levels of cyclic strength and cyclic softening, due to coarse

| Material | \( K' \) (MPa) | \( n' \) |
|----------|----------------|----------|
| PM       | 835            | 0.054    |
| WM       | 952            | 0.092    |
| 5 °C/s ICHAZ | 2116        | 0.24     |
| 10 °C/s ICHAZ | 1796        | 0.21     |
| 10 °C/s FGHAZ | 1442        | 0.087    |
| 30 °C/s FGHAZ | 1848        | 0.12     |

Table 9

The identified Coffin-Manson constants for X100Q PM, WM and simulated HAZ.

| Material | \( \varepsilon_f \) | \( c \) |
|----------|-------------------|--------|
| PM       | 0.55              | 0.65   |
| WM       | 0.18              | 0.53   |
| 5 °C/s ICHAZ | 0.20        | 0.47   |
| 10 °C/s ICHAZ | 1.29        | 0.67   |
| 10 °C/s FGHAZ | 0.55        | 0.62   |
| 30 °C/s FGHAZ | 0.39        | 0.60   |

Fig. 22. The (a) strain amplitude-life and (b) Coffin-Manson relationships observed for X100Q PM, WM and simulated HAZ specimens during strain-controlled fatigue testing.

Fig. 23. The stress amplitude-life relationships obtained for X100Q PM and CW during force-controlled fatigue testing and stress amplitude-life relationship for X80 from Ref. [46].
bainitic block size and the low prevalence of bainitic lath microstructure.

- The stress-controlled fatigue life of X100Q is shown to be generally twice as long as that of X80.
- The simulated fine-grained HAZ displays superior fatigue performance to the parent material, weld metal and simulated intercritical HAZ due to its higher stabilised cyclic strength, and therefore lower cyclic plastic strain amplitude, for a given test. However, the plastic strain-life (Coffin-Manson) relationships of the microstructures are similar, indicating that microstructure-induced differences in yield strength (related to HAB and LAB density) and cyclic softening behaviour (related to LAB density) are the predominant factors in the variation of fatigue performance among the microstructures.
- Although the fatigue performance of the simulated HAZ specimens surpasses that of the parent material and weld metal, the detrimental effect of the material inhomogeneity associated with a weld is highlighted by the reduced performance of cross-weld specimens.

Data availability statement

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.

CRediT authorship contribution statement

Ronan J. Devaney: Conceptualization, Investigation, Methodology, Formal analysis, Writing - original draft. Padraic E. O’Donoghue: Supervision, Writing - review & editing. Sean B. Leen: Funding acquisition, Project administration, Conceptualization, Supervision, Writing - review & editing.

Declaration of competing interest

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.

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