Materials and Design 59 (2014) 135–140

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Technical Report

Local mechanical properties of intercritically reheated coarse grained heat affected zone in low alloy steel

Veronica G. Haugen [a], Bjørn Rune Sørås Rogne [a], Odd M. Akselsen [a,b,*], Christian Thaulow [a], Erling Østby [b,1]

[a] The Norwegian University of Science and Technology, Dept. Engineering Design and Materials, Rich. Birkelands vei 2B, NO-7043 Trondheim, Norway
[b] SINTEF Materials and Chemistry, P.O. Box 4760 Sluppen, NO-7465 Trondheim, Norway

ARTICLE INFO

Article history:
Received 25 June 2013
Accepted 7 February 2014
Available online 24 February 2014

ABSTRACT

Steels applied in arctic climates are subjected to low temperature. Since they undergo ductile–brittle transition with falling temperature, their fracture toughness must be addressed, particularly after welding. To predict their behaviour requires knowledge on local properties. Thus, the present study concerns nanomechanical testing of typical microstructures present in the intercritically reheated coarse grained heat affected zone of a 490 MPa forging. Such microstructures were achieved by weld thermal simulation of samples with 11 mm × 11 mm cross section and 100 mm length, using peak temperature of 1350 °C in the first cycle and 780 °C in the second cycle. Both cycles used cooling time $\Delta t_{8/5}$ of 5 or 10 s. This caused formation of M–A phases along prior austenite grain boundaries and mixture of bainite/tempered martensite in the bulk. Nanomechanical testing was performed by compression of nanopillars prepared in grain boundary located M–A phases and in the bulk of the grains. The results achieved showed significant that the grain boundary phase possesses much higher strength than the grain bulk. It is also shown that there is large scatter in the stress–strain data, depending on the actual local microstructure being tested.

1. Introduction

In welding of low alloy structural steel, two potential local brittle zones are formed, namely the coarse grained heat affected zone (CGHAZ) and the intercritically reheated coarse grained HAZ (ICCGHAZ). In the latter case, embrittlement caused by the martensite–austenite (M–A) constituent is well known [1–6]. Low toughness is typically observed in multipass weld when coarse microstructures as martensite and upper bainite are subjected to an intercritical heat cycle with peak temperature of 770–820 °C, depending on the steel chemical composition and welding parameters. The M–A phase is formed because of the enhanced local hardenability in austenite due to carbon diffusion. It is proposed that the toughness degradation is linked to the low temperature phase transformation with an associated volume expansion setting up residual stresses and strains, with subsequent cleavage at the phase boundary [2], and the critical volume fraction of M–A seems to be around 0.06 [3]. The requirement that a certain strain level must be attained locally was also later proposed by Davis [4], and the debonding mechanism has been observed by many authors [5–7]. A recent study also reported cracking of the M–A phase [8]. It is also important to notice that the phase transformation is un-uniform and that the toughness is can be improved by welding conditions that provides higher uniformity [9].

Intercritically reheated coarse grained microstructures are typically tested by extensive impact and fracture toughness testing, which will bring forward some average of the properties of the microstructures present in the actual test sample. In order to obtain more local information on properties, nanoindentation has been utilized to achieve information on hardness levels on a nano-micro-scale [8–16], including effects of grain size [10] and phases [11] on hardness, temper softening [12], carbide spacing [13], strain rate effects [14], strain-induced transformation from austenite to martensite [15], and finally applied to spot welding [16]. Load–displacement curves may also be measured [17], and the fracture toughness of carbides in tool steels has been found through such experiments based on toughness calculation from hardness values [18]. Nanomechanics have also been employed in study of coatings [19,20] and composites [21].

More detailed information on the local properties of microstructures formed in arc welding is still missing, but the early work by Rogne [22] and Haugen [23] is very promising. Data representing local properties may be very useful to accomplish better understanding of the mechanisms involved in crack initiation and growth. Furthermore, such information would be helpful in

* Corresponding author at: SINTEF, P.O. Box 4760 Sluppen, 4765 Trondheim, Norway. Tel.: +47 9305 9450; fax: +47 7359 7043.
E-mail address: Odd.M.Akselsen@sintef.no (O.M. Akselsen).
1 Present address: Det norske Veritas (DNV), P.O. Box 300, NO-1322 Høvik, Norway.

http://dx.doi.org/10.1016/j.matdes.2014.02.010
0261-3069/

establishing more accurate models to predict the fracture behaviour and structural integrity of steel structures. Therefore, the present investigation was carried out with the objective to provide information on certain brittle microstructures occurring in low alloy steel in welding, here addressing the ICCGHAZ. The work was based on nanomechanical testing in terms of compression of very small pillars of 1 μm in diameter, as prepared by focused ion beam (FIB). The pillars were located in the grain bulk in different packets and at grain boundaries decorated by the massive M–A phase. It will be shown that important information can be found concerning properties of potential brittle microstructure constituents.

2. Materials and experimental procedure

2.1. Materials

For the present study, forged steel of F70 grade was selected. Its chemical composition is shown in Table 1. This steel possess massive M–A phase decorating the prior austenite grain boundaries [24] in the intercritically reheated HAZ. The base metal mechanical properties are reported elsewhere [25]. The axial strength varies between 500 and 510 MPa, depending on the thickness position.

2.2. Weld thermal simulation

Samples of 10 mm × 10 mm and 100 mm length were subjected to weld thermal simulation based on resistance heating with pre-programmed heat cycles. The specimens were cut in the axial direction of the forged ring. The weld simulation of intercritically reheated heat affected zone was performed as follows:

1st thermal cycle:	$T_p \sim 1350$ °C $\Delta t_{8/5} \sim 5$ and 10 s (cooling time between 800 and 500 °C)
2nd thermal cycle:	$T_p \sim 780$ °C $\Delta t_{6/4} \sim 6$ and 12 s (cooling time between 600 and 400 °C)

The heating rate was 150 °C/s for all samples. The temperature–time cycles were recorded by thermocouples (type K) spot welded on the specimen mid-length in the transverse direction. To ensure the same cooling rate as in the first cycle, the value of $\Delta t_{6/4}$ in the first cycle was used as pre-programmed value in the second cycle.

2.3. Nanomechanical testing

Samples for nanomechanical testing were cut from Charpy V notch specimens close to the fracture surface using thin cutting disc. Then, they were subjected to grinding, polishing and ultrasonic cleaning prior to electropolishing in sulphuric acid solution (53.5 ml H_2SO_4 per litre methanol) operating at 20 V for 15 s. Because martensite and carbides are etched with slower rate, height difference will appear in the surface, making it possible to separate phases in the scanning electron microscope (SEM). Since there is no established procedure available for making pillars for compressions testing, these had to be developed for each type of microstructure encountered, i.e., for grain boundary blocky M–A phase and grain interior bainite/martensite locations. This point is exemplified in Figs. 1 and 2, and is indexed "ductile" (grain interior) and "brittle" behaviour (grain boundary).

The first step in pillar fabrication is done with a high current ion beam, 9.3 nA. At this current, the Ga^+ ion indentation depth is large; so is also the amount of material sputtering. Thus, the geometry is rough and coarse. Refining the material removal is then done by milling with a lower beam current. The implementation depth is reduced, and smoother specimen surface is achieved. A total of 36 pillars were made. About 50% of the pillars were localized in the grain boundary in MA-particles, while the remaining pillars were placed within the grain (different packets). As the MA-particles in the grain boundary vary in size, from less than 1 μm up to about 4 μm, it is possible to locate the pillar within the particle, and thus evaluate the mechanical properties. The geometry of the pillar is crucial due to calculating stress and strain. The aim is to get a cylindrical shape, with a small tilt angle, and to maintain a flat top without a rounded edge. This is to ensure a homogeneous stress state, and to be able to calculate the interior stress field without the concern of varying diameter due to the height. The common ratio of height to diameter, h/d, is 3:1 to avoid buckling. If the ratio is relatively large, beyond 4:1, buckling may occur, thus the test results are meaningless as a measure of the fundamental compressive behaviour. Buckling occurs due to unavoidable small imperfections within the geometry and the specimen alignment with respect to the flat indenter tip. Conversely, if h/d is small, below 1.5:1, the diameter increases due to the Poisson effect. However, at the ends of the specimen, the friction retards this motion, resulting in a barrel shape. If the material is capable of large amount of deformation in compression, a low ratio may result in a situation where the behaviour of the pillar is dominated by the end effects, thus the test does not measure the fundamental behaviour. A compromising result is to have the ratio of 3:1 for more ductile pillars, and the ratio of about 2:1 for more brittle pillars. In this work, the ratio was around 3:1 for both types of samples. This resulted in severe buckling for almost 50% of the pillars in the 10 s sample, and was therefore excluded from the results. To be able to determine the pillar height, it is crucial that the bulk pillar is not located within a steep crater.

The nanoindentation experiments were carried out on with a flat indenter tip to ensure a homogenous loading situation. The diameter of the indenter is 10 μm. The specimen is loaded in compression instead of tension, but the elastic modulus and the yield strength is the same for both compression and tension. A nanoindenter was used for this purpose. The load/displacement results from the indentation process were transformed to stress/strain curves. The calculated pillar cross section is done using the average of the top and the mid-height diameters.

3. Results and discussion

3.1. ICCGHAZ microstructure

The metallurgical phenomena occurring in the ICCGHAZ is tempering of martensite and bainite matrix combined with formation of new M–A constituents along the prior austenite grain boundaries as well as inside the grains. The resulting microstructures are martensite and a mixture of martensite and upper bainite for the two different cooling rates, Fig. 3. The average hardness (HV_{10}) was 340 and 290 for the two cooling times of 5 and 10 s, respectively [24]. The size of the M–A islands is sufficiently large to produce nanopillars entirely within the phase, as indicated in Fig. 4.

Table 1
Chemical composition of F70 forged steel.

C	Si	Mn	Cr	Ni	Mo	Cu	P_{cm}[a]
0.07	0.25	0.60	0.79	0.85	0.19	1.19	0.234

[a] P_{cm} = C + Si/30 + (Mn + Cu + Cr)/20 + Ni/60 + Mo/15 + V/10 + 5B; all in wt%.