Investigation of postweld heat treatment of quenched and tempered pressure vessel steels

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CHAPTER 5

DISCUSSION
5. DISCUSSION

Postweld heat treatment, which is a mandatory requirement in the manufacture of transportable pressure vessels, is a stress relieving process whereby residual stresses are reduced by heating to the PWHT temperature range and holding for a pre-set time. Residual stresses are reduced because the decrease in yield strength resulting from the stress relieving temperature causes localised yielding to occur under the internal stress field, and this effect, combined with creep strain due to thermally activated dislocation glide and climb, significantly reduces the level of residual elastic strain. The associated residual stresses are typically lower than 50% of the yield point at room temperature (Lochhead and Speirs, 1972).

The welding process used in this project was SAW with a single vee preparation welded from both sides with multiple weld runs. Figure 4.1 shows macrographs of the weld of 11 mm, 12 mm and 20 mm plates that were used for this research.

In Australia, stress-relieving requirements differ between transportable and non-transportable pressure vessels. In non-transportable pressure vessels PWHT is only required in samples over 15 mm in thickness, whereas in transportable pressure vessels PWHT is required for all plates. The philosophy behind this requirement is that transportable pressure vessels have a greater probability of high strain rate failure (high speed traffic accidents) and fatigue loading in transit. This chapter delves into these issues.

Chapter 5 is divided into four main sections, which discuss the results presented in Chapter 4 – Experimental Results. The four sections are:

5.1. The need for PWHT in achieving weldment properties.
5.2. The effect of multiple PWHT cycles on weldment properties.
5.3. The need for PWHT in reducing residual stresses.
5.4. The suitability of BIS80 as pressure vessel steel.
In these sections emphasis is placed on the correlation between structure and properties and the fundamental mechanisms underlying the observed mechanical behaviour. Some of these issues discussed in this light are:

- The structural factors responsible for degradation of impact toughness with increasing number of PWHT cycles.
- A comparison of the weldment zones to establish why failure occurs predominantly in the base plate in real-life situations.
- Factors accounting for scatter in impact energy values.
- The significance of PWHT cooling rate on impact toughness.
- The role of rolling direction and microstructural banding in the fracture of PM Charpy impact samples.
- Correlation of CTOD and impact toughness values for design purposes.

5.1 THE NEED FOR PWHT IN ACHIEVING WELDMENT PROPERTIES

Apart from the principal objective of PWHT to reduce residual stresses, there are other secondary reasons to conduct PWHT. Rosenbrock (1999) and Dumovic (1997) report that PWHT is also used to: allow hydrogen to diffuse/effuse out of the weld metal region, reduce distortion, promote stability during machining, and temper/control WM and HAZ microstructures.

A major objective of this research was to assess the need for PWHT in terms of reducing residual stresses and lowering hydrogen concentration. As a result, impact toughness, hardness, ductility (bends), and tensile properties were examined before and after 1 PWHT cycle. The microstructure before and after 1 PWHT cycle was also examined to determine the effect of PWHT on all of the weldment zones.
5.1.1 Microstructure and hardness

The PM microstructure of as-quenched BIS80PV and BIS80 is martensite with a hardness of 550 VHN (Bisalloy, 2000). After tempering, the hardness is typically reduced to 285 VHN. The end product, a steel pressure vessel base plate with a microstructure consisting of tempered martensite and mechanical properties that comply with AS3597-1993, is achieved by austenitising at 930-940°C, rapid water quenching, tempering at 610-630°C and still air-cooling. Figures 4.2 to 4.4 respectively show the end product microstructures of 11 mm BIS80PV, 12 mm BIS80 and 20 mm BIS80PV; and Figures 4.6 and 4.7 typify the original as-rolled microstructure (prior to quenching and tempering) for 12 mm and 20 mm plate. The 11 mm and 12 mm plates contained bands of pearlite that are thinner and longer than the 20 mm plate (compare Figures 4.6 to 4.7). Since, the as-rolled microstructure of the 11 mm BIS80PV plate is similar to the 12 mm plate, the banding present appears to be thickness dependant. This effect is expected because the plate thickness reduction ratio (RR) as a result of hot rolling will determine the spacing of the pearlite bands and their elongation in the rolling direction. Assuming a 200 mm thick cast strand, the RR is 10:1 (90% total rolling reduction) for 20 mm plate but 17:1 (94% total rolling reduction) for 12 mm plate.

The microstructure in Figure 4.6 was produced by austenitising at 950°C and annealing. The microstructural banding of pearlite arises because of the presence of Mn-rich bands with a lower Ar₃ temperature than the adjacent Mn-depleted regions. The segregation of Mn and other solute elements arises during the continuous casting of steel slabs and is difficult to remove by homogenisation during reheating and hot rolling. As ferrite forms selectively in the Mn-depleted bands, carbon is rejected into neighbouring austenite bands that ultimately transform to pearlite. Quenching and tempering prevents carbon partitioning, but the manganese segregation is still present even though it is not evident in a standard Nital etch. Microstructural banding is known to cause directional properties in steel plate and Australian Pressure Vessel Standards often require testing transverse to the rolling direction where lower mechanical properties are expected.
Optical microscopy revealed no evident change in the PM quenched and tempered microstructure with exposure to PWHT, as can be seen when comparing Figure 4.14 to Figure 4.15. Figure 4.15 is a photomicrograph of BIS80PV PM that has been postweld heat treated at 580°C for a holding time of 6 hours, and there is no significant change in the PM microstructure at the resolution of optical microscopy. There is no evidence of changes such as particle coarsening in the micrographs shown in Figures 4.14 and 4.15. The PM microstructure does not change because the PWHT temperature range for QT steels in the Australian Standards is between 540°C and 590°C, well below the A1 temperature, and lower than the tempering treatment temperature used for the quenched plate. For this family of steels, PWHT is carried out below the tempering temperature to eliminate further possible softening (Nicholas, 2002, p1).

The absence of a change in microstructure at the resolution of optical microscopy in the PM region of QT steels as a result of PWHT agrees with the findings O’Brien and Lumb (2000) who reported no significant changes in microstructure in a retired QT pressure vessel steel exposed to PWHT at 580°C.

Similarly, the WM and HAZ regions showed no obvious changes in microstructure after PWHT at 570°C. The as-solidified WM microstructures of the 11 mm, 12 mm and 20 mm plates are shown in Figures 4.8 to 4.10 and are dominated by fine acicular ferrite. The various zones of the HAZ (CGHAZ, FGHAZ, ICHAZ) also showed no change in microstructure at the resolution of optical microscopy after PWHT, Figures 4.11 to 4.13.

Hardness testing enabled the assessment of the effect of PWHT on hardness in various regions of the weldment. Figure 4.16, which is a plot of hardness versus the number of PWHT cycles, shows that PWHT had no significant effect on the hardness values of the PM of the 11 mm, 12 mm and 20 mm QT plates. The hardness values for the 11 mm, 12 mm and 20 mm PM plates, treated in a box furnace at 570°C with holding times of either 30 minutes (11 and 12 mm plate) or 50 minutes (20 mm plate), were between 275 VHN and 293 VHN. The hardness results for the PM agree with the findings of O’Brien and Lumb (2000), who reported no significant trends in hardness with an increase in the Holloman parameter.
The hardness properties of the WM and HAZ were compared to the PM, and the tempering effect of PWHT on the WM and HAZ regions was also analysed. The WM and PM regions before PWHT had similar hardness values (WM marginally under-matching), suggesting that the weld process and procedure used is suitable in obtaining matching properties between the WM and PM regions. In the as-welded condition, the hardness of the CGHAZ was significantly higher than the WM and PM regions. On the other hand, the FGHAZ and ICHAZ had hardness values lower than the corresponding PM and WM. The as-welded and/or as-received hardnesses of these regions are shown in the column graphs in Figures 4.19 to 4.21 and in the hardness traverses of the 11 mm, 12 mm and 20 mm plates in Figure 4.22. The CGHAZ is shown to have a hardness of approximately 320 VHN, whereas the FGHAZ has a hardness of 250 VHN for 11 mm and 20 mm BIS80PV and 225 VHN for 12 mm BIS80.

It has previously been reported that PWHT is required to temper the microstructure of the WM and CGHAZ in Cr-Mo steels (Bhadeshia, 2001, pp 626-640). Figures 4.19-4.21, which show the WM and CGHAZ hardnesses of all the plates before and after PWHT, support the findings of Bhadeshia (2001, p635) because significant WM and HAZ tempering occurs in all the welded samples after one PWHT cycle. This trend is supported by the hardness traverses shown in Figures 4.22 to 4.23, where a comparison of hardness values before and after 1 PWHT cycle in each weldment zone reveals the same effect. The softening that occurs as a result of PWHT in the WM region implies that the WM significantly under-matches the strength of the PM.

As a result of the findings from the hardness testing of the weldments before and after PWHT, the necessity for PWHT to temper the WM and HAZ to achieve adequate ductility needed to be established. Also low hardness values in the FGHAZ of the 12 mm BIS80 (244 VHN without PWHT and 221 VHN after 1 PWHT) draw attention to the tensile properties of the weldment. For these reasons, transverse guided bend and tensile tests of cross-weld specimens were used to determine the weldment ductility of all samples and the tensile strength of the 12 mm BIS80 weldment (discussed in Section 5.4).
5.1.2 Bend testing

Root bend testing was carried out in accordance with AS2205.3.1-1997 on all welded plates before and after one PWHT. Samples were bent 180° around a former with a diameter 6.7 times the thickness of the plate. The purpose of the bend tests was to test the ductility of the weld with and without PWHT. Bend test samples were ground flush and finished and machined to the required width of 30 mm for samples less than or equal to 20 mm in thickness. There was no evidence of cracking or tearing in any of the samples, as shown in Figures 4.27 and 4.28. Figure 4.27 shows a photograph of all the bend samples, and Figure 4.28 shows one edge of the test piece and the bend surface for the 20 mm BIS80PV sample without PWHT. The results indicate that there is no need for PWHT in order to improve weldment ductility. The absence of cracking also indicates that the hydrogen level without PWHT is lower than the critical amount required to induce hydrogen assisted cold cracking. The satisfactory ductility can be attributed to improvements in steel making over the last 50 years (lower carbon content and fewer impurities), ensuring a sufficiently ductile PM and heat affected zone. Weld processes and the quality of consumables (fluxes and wires) have also improved, leading to lower susceptibility of WM hydrogen cold cracking. SAW enables the production of consistent and relatively defect-free welds, and it is also a comparatively low hydrogen process in which the flux layer results in slower cooling. This promotes the escape of hydrogen into the environment during slow cooling. Moreover, the multiple weld runs used in the procedure also have the effect of tempering all previous weld runs, and effectively increases the time at elevated temperatures over which hydrogen effusion can occur from the weld zone.

5.1.3 Impact toughness and tensile properties

Impact testing was carried out in accordance with AS1544.2-1989. A striking energy of 325 J was used and the test temperature selected was –20°C as required by the Australian Pressure Vessel and QT Steel Standards (Australian Standard 4458, 1997; Australian Standard 3597, 1997). Five samples were used for each group of tests to improve the significance of the data.
Impact toughness is an important property of transportable pressure vessel steels. Australian Pressure Vessel (Australian Standard 1210, 1997) and QT Steel Standards (Australian Standard 3597, 1997) specify that the impact energy based on an average of three Charpy samples in the T-L orientation should be no less than 40 J at –20°C. Figures 4.32 to 4.34 show that 1 PWHT cycle leads to a decrease in the impact energy of the PM region. A similar trend is seen in the WM region (see Figure 4.46 and 4.47). Overall, the results indicate that the impact energy of the WM and PM regions are higher in the as-received or as-welded condition, and 1 PWHT cycle has an adverse, but still acceptable effect on impact energy. The reasons for this are discussed in more detail in Section 5.2.

Since the base plate or PM is subjected to cold forming in fabrication of the heads of transportable pressure vessels (Figure 2.9), impact toughness before and after PWHT was also measured for pre-strained PM. A plastic strain of 3.2% decreased the impact energy of 12 mm BIS80 (T-L) at –20°C from approximately 105 J to 80 J (Figure 4.36(b)). This decrease in impact energy is due to the significant increase in dislocation density as a result of cold working, which in turn increases strength and decreases ductility (Reed-Hill et al, 1992). PWHT at 545°C for 30 minutes marginally increased the impact energy of plastically strained 12 mm BIS80 (T-L) by approximately 5 J. This slight increase is due to the recovery process. Recovery is much more pronounced at higher treatment temperatures, but the PWHT of the QT steel was conducted well below the plate manufacturer’s tempering temperature to maintain the mechanical properties of the QT steel.

Moreover, tensile testing (cross-weld) was also carried out to evaluate weldment ductility before and after PWHT. Similar to impact energy, there was a significant decrease in the UTS of all samples (see Figure 4.59). However, the yield stress showed more variable behaviour: the 11 mm cross-weld sample was found to increase from 708 to 730 MPa with exposure to 1 PWHT cycle; whereas the yield strength of the 12 mm cross-weld weldment was found to decrease from 691 MPa to 614 MPa. After PWHT, the UTS and YS of 12 mm BIS80 cross-weldment sample (776 MPa and 614 MPa)
were not in compliance with AS3597-Structural and Pressure Vessel Steel – Quenched and Tempered Plate. This Standard requires the YS (or 0.2% proof stress) to be greater than 690 MPa and the UTS to range from 790 to 830 MPa. The percent elongation and reduction in area values were similar before and after PWHT (~50% reduction in area and ~35% elongation).

The failure location of all tensile samples was in the WM region. Even though the lowest hardness values were found in the FGHAZ, failure did not occur in this region of the cross-weld test-piece. This is likely to be because this region is relatively narrow and a ‘brazing effect’ operates in which this low strength volume is constrained by the higher strength regions surrounding it: the CGHAZ and the ICHAZ/PM (Honeycombe and Bhadeshia, 1995).

In summary, the results show that all plates exhibit adequate ductility (to satisfy Australian Standards) in the as-welded condition and after a single PWHT. However, in most cases, 1 PWHT cycle decreases the hardness, cross-weld tensile strength and impact toughness.
5.2 THE EFFECT OF MULTIPLE PWHT CYCLES ON WELDMENT PROPERTIES

Microscopy and mechanical testing were used to characterise the PM, HAZ and WM regions of submerged arc welded BIS80PV and BIS80 plate specimens with exposure to no or multiple PWHT cycles.

5.2.1 Microstructure and hardness

As mentioned previously, the as-manufactured microstructure of QT BIS80PV and BIS80 is tempered martensite with a hardness of 285 VHN. Optical microscopy revealed no evident change in the quenched and tempered microstructure with increased holding time (maximum of 16 hours), increased stress relieving temperature (540 to 620°C) or increasing number of PWHT cycles (up to 4). Figure 4.15 shows a micrograph of the PM region of BIS80PV after exposure to 6 hours holding time at a PWHT temperature of 580°C. There is no significant difference between this microstructure and that of Figure 4.14, which is of the PM of BIS80PV without PWHT. This finding agrees with O’Brien and Lumb (2000) who reported no significant changes in microstructure in a retired QT pressure vessel steel exposed to multiple heat treatments. There is no microstructural change at the resolution of optical microscopy for the number of PWHT cycles investigated because the PWHT temperature range for QT steels is below the tempering temperature of the base plate, as well as being below the A1 temperature for this type of low alloyed quenched and tempered steel. Long times of 580°C would be necessary to produce coarsening of carbides and recovery/recrystallisation of the ferrite, that is significant enough to be evident by optical microscopy.

Similar to the PM, the WM and HAZ regions revealed no changes in microstructure at the resolution of optical microscopy after 4 PWHT cycles at 570°C. The as-solidified WM microstructure shown in Figures 4.8-4.10 is typical of that observed for 0 to 4 PWHT cycles. The WM microstructure is fine acicular ferrite and is free of weld defects. The HAZ microstructures shown in Figures 4.11-4.13 are typical of samples
subjected to 0 to 4 PWHT cycles. The photomicrographs in these figures identify the CGHAZ, FGHAZ and ICHAZ. The ICHAZ shows a ‘necklace’ structure of darker etching islands decorating prior austenite grain boundaries with a matrix of well tempered martensite (Micrograph (d) in Figures 4.11-4.13), such a structure is often referred to as a “local brittle zone” (LBZ) because of a propensity for brittle intergranular failure. The islands result from partial austenitisation at grain boundaries during welding, forming high carbon pools that transform to hard constituents such as fine pearlite or bainite during cooling. Despite its potential for localised brittle fracture, this region did not appear to result in any marked adverse effect on Charpy impact energies, probably because it occupies only a small volume of the total HAZ.

Samples heat treated in the vacuum chamber of a dilatometer were used to study the effect of combinations of a wide range of PWHT temperatures and holding times on the hardness of BIS80PV PM. Figure 4.17(a), which combines the effects of temperature and time through the Holloman parameter, shows a decrease in hardness from approximately 295 VHN to 255 VHN with an increase in the Holloman parameter. Softening occurs because of carbide coarsening (Reed-Hill et al., 1992), and dislocation rearrangement and annihilation. The hardness results obtained using the dilatometer contradict the findings of O’Brien and Lumb (2000). They reported no trends in hardness with an increase in the Holloman parameter. However, trends are unclear in their data because of a modest holding time range and the use of only one treatment temperature. In this investigation, the hardness values for the 11 mm, 12 mm and 20 mm PM plates heat treated in a box furnace at 570°C for holding times of 0.5 to 3.3 hours, were between 280 VHN and 295 VHN with no clear trends with holding time (in agreement with O’Brien and Lumb (2000)). The nature of the HP equation shows that temperature has a more pronounced effect than time, and hence varying temperatures, as well as extending simulated PWHT times, has resulted in the outcome shown in Figure 4.17(a). As expected, the hardness does decrease with exposure to elevated temperatures due to structural coarsening and dislocation annihilation. Lochhead and Speirs (1972, p209) also reported and discussed a similar result.
Figure 17(b) shows that a slight secondary hardening effect occurred in the first hour of PWHT. Secondary hardening occurs due to precipitation of alloy carbides and, in this case, Cr, Mo and Ti are likely to form carbides that are more stable than Fe/Mn rich carbides, albeit with slower kinetics of formation. However, the effects observed in BIS80PV are relatively minor because of the low concentrations of strong carbide forming elements (Table 3.1).

The hardness values in all the weldment zones exposed to multiple PWHT cycles were also compared. The hardness values of the WM and PM regions before and after PWHT were in the range of 255 to 295 VHN (Figures 4.16 and 4.18). From these figures it is evident that the weld process and procedure used (without PWHT) was suitable for obtaining matching properties between these regions, even though the WM was slightly lower in hardness than the PM.

As mentioned previously, it is evident that one PWHT cycle softens the CGHAZ and WM, thus producing an under-matching WM compared to the PM (see Figures 4.19-4.21). It has been reported previously that PWHT tempers the microstructure of the WM and CGHAZ in Cr-Mo steels, reducing hardness and strength (Bhadeshia, 2001, p635). The structure of the CGHAZ without PWHT consists of lath martensite and bainite that is auto-tempered because of the maintenance of the interpass temperature, thermal spikes due to subsequent weld passes and relatively slow cooling after the completion of welding. As a result, the measured hardness of about 320 VHN is significantly lower than that of water quenched BIS80 or BIS80PV (~550 VHN). However, the hardness of the CGHAZ exceeds that of the PM after tempering (>285 VHN), and so it undergoes significant tempering during PWHT. This tempering process involves precipitate coarsening, subgrain formation and coarsening, and dislocation annihilation.

Figures 4.24-4.26 indicate that following the first PWHT cycle, further cycles do not significantly affect the hardness of the WM in the 12 mm and 20 mm plates. Hence the microstructure and hardness stabilised after multiple PWHT cycles. Moreover, the WM region would not be expected to soften significantly because of its low carbon content.
(0.06 wt%) and greater dependence, compared with the PM, on solid solution strengthening.

Additionally, after the initial PWHT, subsequent PWHT cycles do not appear to have a strong effect on the hardness of the sub-regions in the HAZ. For example, the change in hardness of the CGHAZ of 12 mm and 20 mm plates after 1 PWHT cycle is significantly higher than the change resulting from 1 to 2 post weld heat treatments (Figures 4.25 and 4.26).

### 5.2.2 Impact toughness

A major concern of this project was to investigate the effect of multiple PWHT cycles on the Charpy V-notch impact properties of the PM region of QT pressure vessel steel. The PM region is of paramount concern to the Australian Pressure Vessel industry because this is the zone where real-life reported failures in traffic collisions have generally occurred. Moreover, there is some evidence (O’Brien and Lumb, 2000) that cumulative PWHT cycles are detrimental to impact properties of QT steels used in pressure vessels. The WM and HAZ regions were also subjected to impact testing for comparison and to investigate why these regions perform better than their PM counterpart in the event of high velocity (high strain rate) collisions.

#### 5.2.2.1 Parent metal

Figures 4.29 and 4.30 show column graphs of impact energy (and lateral expansion) versus orientation of the test piece with respect to the rolling direction of the PM plate. Samples machined in the L-T or L-S orientation have higher impact energies and lateral expansion than samples machined in the T-L or T-S orientation. This result arises because samples aligned longitudinally have crack planes that are perpendicular to the rolling direction and the long axis of the compositional banding present in the plate. Therefore, a higher impact energy is required to cause failure. The outcome is a more ductile failure in the L-T and L-S orientations than in T-L or T-S orientations, with significantly higher impact energy and lateral expansion values.
In the T-L and T-S orientations the crack plane contains the rolling direction, and is a plane of weakness because of the microstructural banding and elongated inclusion particles. For example, Figure 4.5(a) shows a photomicrograph of an elongated MnS inclusion particle in the direction of rolling. These findings coincide with those of Boyer and Gall (1984, p232), who showed similar trends in results for rolled steel. The L-S and T-S orientations show slightly higher impact energy values than their respective L-T and T-L orientations because the notch in the former case runs parallel to the top of the plate and does not section the middle of the plate where banding and microstructural segregation are most prevalent.

Figures 4.37 and 4.38 respectively show low magnification images of Charpy V-notch fracture surfaces of as-received (no PWHT) plate of 12 mm BIS80 and 20 mm BIS80PV. Splitting is evident in most of these macrographs. It has been suggested that steels with a tendency to split or delaminate generally have lower impact energies than those that do not (Ryall and Williams, 1978; Bramfitt, 1977, p1263). BIS80PV (11 mm and 20 mm) has lower impact energies than BIS80 (12 mm) due to the higher contents of alloying elements such as Cr and Mn, which are known to promote splitting (DeArdo, 1977, p474; Bramfitt et al, 1977, p1263). For example, in the T-L orientation the 12 mm PM plate has an impact energy value of 104 J and the more highly alloyed 20 mm PM plate has an impact energy value of 60 J, and shows more pronounced splitting (Figure 4.38(a) compared with Figure 4.37(a)). DeArdo (1977) stated that splitting of the fracture surface of mechanical test specimens (tensile and impact) was found to occur when the levels of Cr in HSLA steels were ≥0.5%. However, the present results indicate that levels of less than 0.5%Cr can have an effect on splitting (Table 3.1).

It is demonstrated in Appendix F, that splitting or delamination in the Charpy fracture surface is the main reason for significant scatter in impact energy values. Splitting exerts a strong effect on the impact energy required to cause failure in high strain rate tests. It is a phenomenon that occurs in some steels and not others. Factors that have been reported to influence the initiation of splitting in Charpy V-notch samples are chemical composition (in particular, the presence of Mn and Cr), microstructural banding, centre-
line segregation, and testing in the upper-portion of the DBTT region. Regardless of the notch orientation, splitting results in scatter of impact energy values. Samples with identical notch orientation and exposed to the same conditions can show different impact energies depending on their capability to initiate splits. Results from work carried out on 12 mm and 20 mm BIS80PV samples show that splitting results in an increased value of impact energy when all other variables are kept constant. Figures F.1 and F.2 in Appendix F respectively support the significant effect splitting can have on impact energy.

Even though the increased incidence of splitting increases the impact energy of a Charpy sample exposed to the same conditions, it is associated with intergranular failure (see Figures F.3 and F.4 in Appendix F), which normally results in lower impact energies. Therefore, the impact energy values are expected to be lower than for steels that do not show splitting. However, for the same group of steels, increased splitting of the fracture surface was observed to result in an increase in the impact energy required to cause failure. It is proposed that this trend is a result of the increased fracture surface area, due to cracks propagating normal to the general crack plane, as well as the mechanism by which these splits initiate. It is evident in Figure F.4(b) in Appendix F that splitting initiates by microvoid formation and linkage at prior austenite grain boundaries in the Mn and Cr rich bands. In these regions there is a high concentration of carbides present at prior austenite grain boundaries. It is proposed that these particles can nucleate voids that link to produce intergranular fracture. The micro-plasticity associated with the fracture process is expected to contribute to the required energy of fracture.

The incidence of splitting was reported by Ryall and Williams (1978) to decrease in the lower portion of the DBTT curve. As the number of PWHT cycles was increased failure occurred closer to the lower portion of the DBTT and this typically resulted in less severe splitting. However, it was observed that severe splitting did occur after 4 PWHT cycles. It is proposed that other factors come into play, for example, the presence of elongated MnS inclusions can promote severe splitting, as can the number and width of Mn rich bands. Figures F.3 (a), F.3 (b) and F.5 (a) show splitting at wider Mn rich bands, and thus a high localised incidence of bands is likely to initiate more severe
splitting. In comparing the splitting characteristics of the 12 mm and 20 mm parent plate, it is evident that the 12 mm plate contains less splitting because of a relatively lower alloy content (less Cr and Mn).

Transportable pressure vessels are exposed to multiple PWHT cycles because PWHT is mandatory in the repair or manufacture of these vessels. Whenever weld repair is required, then PWHT must follow. The heat treatment must be of the entire vessel and PWHT holding time is dependent on the maximum plate thickness present in the walls of the pressure vessel. In Figures 4.31 to 4.35, which show a plot of impact energy at –20°C versus number of PWHT cycles, it is evident that the impact energy of the PM decreases as the number of PWHT cycles is increased. This trend is more pronounced in 11 mm and 20 mm BIS80PV samples exposed to slow cooling. After four PWHT cycles the 11 mm PM impact energy decreased from approximately 45 J to 27 J and the 20 mm plate impact energy decreased from approximately 60 J to 37 J. Similarly, when samples are cooled in still air (fast cooling) the PM also undergoes a decrease in impact energy (see Figures 4.31, 4.33 and 4.35). The decrease is less significant in fast cooled samples because there is less soaking time in the treatment temperature range of 540 to 590°C, and faster cooling suppresses additional precipitation as a result of decreasing solubility with falling temperature. A slowly cooled sample treated at 570°C has an extra time of 16.2 minutes in the PWHT range (≥540°C), due to heating and cooling, compared to a fast cooled sample, which has an extra time of 9 minutes. It should be noted that lower PWHT temperatures maintain the impact properties more effectively than higher PWHT temperatures. Hardness also shows a strong dependence on PWHT temperature (Figure 4.17(b)), with high temperatures producing a much steeper decrease in hardness with holding time than lower temperatures.

The superior toughness of 12 mm BIS80 is evident when comparing the graph in Figure 4.32 to those in Figures 4.31 and 4.34. After four PWHT cycles, the 12 mm PM has an impact energy of approximately 85 J and the 11 mm and 20 mm BIS80PV plates respectively have impact energies of approximately 35 J and 50 J. The higher impact energy of 12 mm plate is due to the lower alloy content that reduces the susceptibility to splitting (see Appendix F); and the lower carbon and alloy contents that significantly
lower the DBTT. The probability of splitting generally diminishes as the number of PWHT cycles is increased, due to testing closer to the lower shelf of the DBTT curve (Ryall and Williams, 1978). However, there are other factors that contribute to the scatter of impact toughness values, namely:

- Size and distribution of MnS and TiN particles (shown in the optical micrographs in Figure 4.5 and SEM fractographs in Figure 4.43 (b-c)), which play an influential role on the final impact energy value by creating fracture acceleration sites. Fracture acceleration sites lower the impact energy by promoting regions of easy (cleavage) fracture.
- Test completion time. The Australian Standard for impact testing states that the non-room temperature tests must be completed within 6 seconds of being removed from the temperature bath. A ‘fast’ six second test would result in a lower impact energy value than a ‘slow’ 6 second test.
- Variations in machining can influence impact energy. The most critical dimension is the radius of the notch, with sharper notches leading to lower impact energies than more well-rounded notches. The verification of sample dimensions is imperative for statistically valid data.

As the number of PWHT cycles increased, there was no apparent trend in the crystallinity or fibrosity of the fracture surface. This is due to the presence of splitting, which results in a significant scatter in the measurement of percent crystallinity. Lateral expansion, on the other hand, is a reliable measure of ductility (especially when comparing BIS80 to BIS80PV, or WM to PM), and in general a decrease is evident with an increasing number of PWHT cycles (see Appendix G).

SEM of the Charpy V-notch fracture surfaces was carried out to ascertain the mechanism by which increasing number of PWHT cycles resulted in decreasing impact energy. SEM fractographs presented in Chapter 4 were predominantly in the general fracture area, but for the 20 mm slowly cooled PM samples, exposed to 2 and 4 postweld heat treatments, the SEM fractographs are of the ductile shear lip region. The various regions of the Charpy V-notch fracture surface are shown schematically in Figure 3.20.
Figure 4.43 of the Charpy fracture surfaces of samples from the 20 mm plate demonstrates the mechanism by which impact energy decreases with increasing number of PWHT cycles. Figure 4.43(a) shows an image of a ductile fracture surface (dimples) in the as-welded condition and Figure 4.43(e) shows an image of a brittle fracture surface (cleavage) following four PWHT cycles. It is clear that the fracture appearance at −20°C progressively becomes more brittle as the number of PWHT cycles increases. It is implied therefore that PWHT raises the DBTT.

Honeycombe et al (1995) state that ductile fracture, in the form of void coalescence, initiates at fine second phase particles (most likely carbides in the present case). Figures 4.43(a-c) are indicative of void coalescence. The mechanism involves the nucleation, growth and linking of voids in the plastic zone ahead of the crack tip, thus propagating the fracture. In comparing Figure 4.43(a) to Figures 4.43(b-c) it is evident that the voids are smaller in diameter when there is no exposure to PWHT. Hence it can be deduced that very fine, dispersed second phase carbide particles exist throughout the matrix. As the second phase carbide particles coarsen, the voids increase in diameter and spacing and facilitate crack growth (Figures 4.43(b-c)). When the second phase carbide particles reach a certain size by a growth-coalescence mechanism their ability to initiate voids is impaired (Honeycombe et al, 1995) resulting in quasi-cleavage, a more brittle mode of cracking, at a lower impact energy (see Figures 4.43(d-e)). The precipitation of new carbides such as MoC may also occur and coarsen with increased number of PWHT cycles.

The effect of cumulative PWHT cycles on the Charpy fracture surface of the 11 mm fast and slow cooled PM samples (see Figure 4.41) is not as clear as 20 mm BIS80PV because all surfaces show quasi-cleavage fracture. In the fractograph of the 11 mm sample without PWHT, a quasi-cleavage brittle type fracture surface is evident (in comparison to the ductile fracture of the corresponding 20 mm sample). The reason for this difference could lie in subtle differences in chemical composition and the nature of the banding and segregation in the two different thicknesses of the same steel. The 11 mm BIS80PV plate contains approximately 40% more sulphur than the 20 mm
BIS80PV plate. Sulphur is a tramp element that combines with Mn to form MnS inclusions, which have a deleterious effect on impact toughness (Yeomans, 1994). The 11 mm base plate also contains longer bands and banding that is prevalent through the thickness, whereas banding in the 20 mm PM is more concentrated in the middle of the plate and the bands are shorter in length.

There was a significant decrease in impact energy after 2 PWHT cycles and then 4 PWHT cycles, but visual comparison is difficult because all of the surfaces are predominantly cleavage fracture. It is proposed that the coarsening of existing carbides continues, which leads to a lower impact energy failure due to shearing of larger particles. In Figure 4.41, the arrows point to ductile microvoid coalescence, which are ‘ligaments’ that join or link cleavage fractures at different levels. The difference between the fracture surfaces arising from the two cooling methods is a result of the rate at which particles coarsen, and this is reflected by their respective impact energies. For example, after 4 PWHT cycles, a slow cooled T-L sample has an impact energy value of ~30 J compared to ~35 J for a fast cooled T-L sample. This difference in impact energy is due to extra soaking time in the PWHT temperature range for carbide particle coarsening to occur.

Dunne (1999) in a report on the theoretical and experimental background of hydrogen assisted cold cracking in steel weldments, discussed the fracture modes of void coalescence, cleavage fracture and quasi-cleavage fracture. This analysis can be used to explain how coarser second phase particles promote cleavage fracture and result in lower impact energy.

Both transgranular fracture and intergranular fracture can occur by void coalescence. Fine second phase particles cause fine voids to initiate ahead of the crack tip, which propagates by linking or coalescing of these voids (Figure 5.1(a)). As the precipitates coarsen, the voids ahead of the crack tip increase in size, promoting fracture with less energy absorption. In cleavage fracture, voids do not form in the plastic zone ahead of the crack tip because cracking occurs through the particles or at the particle-matrix interface, as shown in Figure 5.1(b and c). These cracks propagate by cleavage under the
stress concentration zone surrounding the tip of the main crack. This type of fracture requires a lower amount of energy and has a smaller fracture area. The most common type of fracture occurs by quasi-cleavage in which a combination of void coalescence and cleavage occurs.

**Figure 5.1:** Schematic diagrams showing fracture ahead of the crack tip in the stress field generated by the crack (a) void formation and linkage at small particles, (b) particle cleavage and extension into matrix and (c) intergranular fracture and extension into matrix.
In the 12 mm BIS80 PM, the deleterious effect of PWHT cycles on the impact energy value is not as prominent as in the 11 mm and 20 mm BIS80PV series of samples, especially in the fast cooled (still air) condition. Figure 4.42 (a) shows a SEM fractograph of the as-received 12 mm PM. This fractograph is indicative of a relatively ductile fracture defined by the presence of fine microvoids. Upon exposure to 2 PWHT cycles (slow or fast cooled) there is a combination of void coalescence and cleavage fracture modes (quasi-cleavage) (see Figure 4.42 (b) and (d)). The voids after 2 PWHT cycles are also larger, implying the presence of larger second phase carbide particles, which have developed by coarsening.

Moreover, at 4 PWHT cycles there are even larger voids present in the fracture surface of the slow cooled sample (Figure 4.42(c)) and a greater amount of cleavage type fracture in the fast cooled sample (Figure 4.42 (e)). It is inferred that after 4 PWHT cycles the slow cooled sample should have a higher impact energy value than the fast cooled sample, consistent with the plots of impact energy versus PWHT for slow and fast cooled 12 mm BIS80 PM samples in Figures 4.32 and 4.33. The slow cooled T-L sample had an average impact energy value of 87 J and the fast cooled sample T-L had an impact energy value of 81 J after 4 PWHT cycles.

Figures 4.42(a, c and d) and Figure 4.43(b and c), which show large voids due to large MnS and TiN particles, lend support to the fact that void diameter is proportional to second phase particle size and spacing (whether they are carbides, nitrides or sulphides). In summary, as PWHT time increases there is an increase in the diameter of the voids due to the growth and coalescence of carbides.

Another result that supports the proposed mechanism for the effect of PWHT cycles on impact energy is shown in Figure 4.36(a). This figure shows that the DBTT (°C) decreases with an increase in the number of PWHT cycles. O’Brien and Lumb (2000), and Pimenta and Bastian (2000) have also reported an increase in the DBTT with an increase in the number of PWHT cycles. These findings support the progression toward a more brittle fracture in the PM region of QT steels with increasing exposure to PWHT.
5.2.2.2 Weld metal

Low carbon and high manganese contents are typical for the WM of QT steels (see Table 3.1). The carbon content is minimised to avoid weldability problems and manganese is used to counterbalance the loss in tensile properties associated with the low carbon content in the WM. Additionally, vanadium is added for increased strength, aluminium is added to avoid strain-ageing embrittlement by fixing nitrogen, niobium is absorbed from the parent steel (excess quantities can lead to problems of toughness), and boron in small concentrations is known to have a relatively strong influence on mechanical properties (Evans and Bailey, 2000, p355).

The WM region for the 11 mm, 12 mm and 20 mm plates showed greater notch impact toughness in the as-welded condition and after PWHT than the corresponding PM region. For example, Figure 4.56 shows that the impact energy of WM at -20°C in the 20 mm plate is 113 J compared to the corresponding PM impact energy in the T-L orientation, which is 60 J. This result reflects a weldment with WM under-matched in strength to the PM. The 12 mm BIS80 WM had the highest impact energy (134 J) of all welds due to dilution effects favourably influencing the impact toughness of the WM (see Figure 4.45). The specific reason for the superior weld metal impact toughness of the 12 mm WM is most likely due to lower levels of carbon and chromium than for the 11 mm and 20 mm WM. The WM carbon content of the 12 mm WM was 0.025% lower than the 11 mm sample and 0.005% lower than the 20 mm WM samples (Table 3.1). Table 3.1 also shows that the 12 mm WM contained 0.029% Cr, whereas the 11 mm and 20 mm BIS80PV WM contained 0.11% Cr and 0.087% Cr, respectively.

On the other hand, the BIS80 WM had higher concentrations of Si, Mo, Cu, S and N, all of which would be expected to adversely affect impact toughness. However, besides lower C content (0.09% compared with 0.115% in 11 mm BIS80PV WM), this WM also had lower Mn content (1.52% compared with 1.55%), lower Cr content (0.029% compared with 0.11%) and lower Ti content (0.003% versus 0.009%). It is evident that these elements have an overriding effect in determining the higher impact energies of the WM of 12 mm BIS80 plate.
Figure 4.48 shows low magnification images of the Charpy V-notch WM fracture surfaces of the WM of the 12 mm BIS80. There is no evidence of splitting in these samples, and the fractures are much more ductile in nature than the corresponding PM fracture surface shown in Figures 4.39 and 4.40. The lateral expansion values also confirm that the WM (Figure 4.45(b)) is more ductile than the PM in the T-L orientation (Figures 4.29(b), 4.30(b) and 4.31(b)).

Multiple PWHT cycles are not as detrimental to the WM as they are to the PM (see Figures 4.46, 4.47, 4.55 and 4.56). An initial decrease in impact energy in the WM after the first two PWHT cycles was observed, but then the impact energy increased in the third and fourth PWHT cycles for both the 12 mm and 20 mm plate. SEM was used to investigate the origin of this effect. It is important to note that the fast cooling PWHT method was used for all WM samples.

The SEM fractographs of the typical impact fracture surfaces of the WM region without PWHT reveal void coalescence, as respectively shown for the 11 mm, 12 mm and 20 mm weld plates in Figures 4.49, 4.50(a) and 4.51(a). In contrast to the PM, the ductile failure by the form of void coalescence in this case is initiated by non-metallic inclusions that provide nucleating sites for the voids (clearly shown in Figure 4.49(c)). Inclusion particles such as oxides play an integral role in weld metal fracture, more so in the as-welded condition where time for carbide precipitates to form is minimal. According to Evans and Bailey (1997, p414), nickel bearing weld metal shows carbide precipitation only after PWHT. The manganese, silicon and nickel levels in the welds investigated by Evans and Bailey (1997) were in the same range as the WM studied in this research project and welded by the same process (SAW).

After 2 PWHT cycles the fractographs of the 12 mm (see Figure 4.50(b)) and 20 mm (see Figure 4.51(b)) WM showed a significantly more brittle fracture surface with limited regions of microvoid coalescence. Similar to the PM, the microvoids present in this quasi-cleavage fracture are possibly due to the tearing fracture of ‘ligaments’ connecting cleavage facets at different levels. Furthermore, metastable Fe₃C or Fe₂₄C
precipitates that are expected to form after 1 PWHT cycle (Evans and Bailey, 1997, p414) coalesce and coarsen rapidly with 2 PWHT cycles, and hence override the role of inclusion particles by initiating cleavage fracture. Bailey and Evans (1997) have carried out extensive research on the effect of stress relief (580°C) on C-Mn weld metals (1997, p412). They concluded that stress relief (PWHT) after welding leads to the precipitation of Fe\textsubscript{3}C which can promote fracture by cleavage, consistent with the characteristics of the SEM fractographs of both the 12 mm and 20 mm weld metal after 2 postweld heat treatments. Although Fe\textsubscript{2.4}C or Fe\textsubscript{3}C particles are kinetically favoured in the welding and PWHT processes, they coarsen rapidly and eventually dissolve on tempering in favour of more stable alloy carbides, such as Cr\textsubscript{x}C\textsubscript{y} or Mo\textsubscript{x}C\textsubscript{y}.

After four PWHT cycles, the SEM fractographs (Figure 4.50(c) and 4.51(c)) show a higher proportion of void coalescence than for 2 PWHT cycles. A possible explanation is that the metastable Fe\textsubscript{3}C precipitates dissolve with precipitation of finer, more stable alloy carbides, that form with elements such as Cr, Ni, Nb and Mo. The propensity for cleavage fracture is reduced and both the alloy carbides and non-metallic inclusions favour void coalescence. Voids initiated by inclusion particles are evident in Figures 4.50(c) and 4.51(c). A major difference between the structures of the PM, (impact toughness decreasing further after 2 PWHT cycles) and WM (impact toughness increasing after 2 PWHT cycles) is that the well-tempered PM contained more stable carbides that formed before any PWHT began.

5.2.2.3 HAZ

The HAZ was impact tested to evaluate the performance of this region compared to the PM and WM regions. Samples were machined from the HAZ so that the orientation of the sample was in the T-S orientation, as shown schematically in Figure 3.8(b). The notch was positioned to initiate fracture in the most brittle section of the HAZ, that is, the CGHAZ. Corresponding PM samples (T-S and fast cooled) were tested for direct comparison with the HAZ. It was difficult to establishing a trend in Charpy impact energies with the number of PWHT cycles due to the fracture surface containing both HAZ and WM, as shown in Figure 4.52. The fracture surfaces of the 11 mm and 12 mm
samples contained less HAZ than the 20 mm plate due to the fact the 20 mm samples were extracted from the middle of the plate. Figure 4.53 shows the average areas of HAZ fracture surface for 11, 12 and 20 mm Charpy V-notch samples were 49%, 73% and 88%, respectively. Figure 4.54 shows low magnification images of the Charpy V-notch fracture surface, together with the portion of the sample that is HAZ or WM.

A comparison of the HAZ impact toughness was made against other regions of the weldment and results were dissimilar for the 12 mm and the 20 mm plates. In the 12 mm plate the HAZ impact energy was lower than the impact energies of both the WM and PM (see Figure 4.55). For the 20 mm plate the HAZ impact energy was between the WM and PM impact energies (see Figure 5.56). These differing results are probably due to geometric differences between the 12 and 20 mm welded plates. In the 12 mm plate the fracture initiates in the CGHAZ and the crack travels a significant distance in the CGHAZ (average 73% of total fracture area) before fracture proceeds into the WM. In the 20 mm HAZ, the fracture is initiated in the CGHAZ and travels through this region, as well as traversing the FGHAZ. The significantly more ductile FGHAZ results in an increase in the impact energy required to cause failure, hence promoting the impact energy of this region above the corresponding PM impact energy. The fracture path in the 20 mm HAZ either remained in the HAZ (FGHAZ or CGHAZ) or traversed some WM (an average of 12% of the total fracture area). The HAZ impact energies of the 12 mm series of samples was markedly lower than PM or WM (Figure 4.55) even though the fracture path included the impact tough WM region. It is inferred that the HAZ (particularly the CGHAZ) generally has lower impact toughness than the WM or PM regions.

After qualitatively establishing that the CGHAZ has the lowest impact energy of all weldment zones in QT steels, there are a number of possible reasons why failure in real-life transportable pressure vessels does not occur in this region.

- The difference in impact energy of the CGHAZ and the PM is small, especially as the number of PWHT cycles increases (see Figure 4.55).
- In the case of a real-life accident of a transportable pressure vessel, catastrophic failure is initiated at very high stress points. For example, in the case study shown in Figure
failure initiated where three folds met at one point in the PM region. Therefore, the site of impact determines the failure initiation site. The HAZ, due to its low volume percentage, has a low probability of being a location for failure.

- The final possibility is the ‘brazing effect’ or ‘two phase aggregate effect’ used to explain higher than expected tensile strengths of samples containing a thin transverse layer of weaker brazing metal (Honeycombe and Bhadeshia, 1995). This explanation can be extended to rationalise why failure does not occur in the CGHAZ even though it has the lowest impact energy of all the weldment zones. The ‘braze metal’ (CGHAZ) has a inferior impact toughness to the surrounding materials (WM and FGHAZ). However, when the CGHAZ is placed under an external load or pressure the reinforcement of this region is re-enforced by the constraints applied by tougher surrounding materials (Honeycombe and Bhadheshia, 1995). A similar argument can be used to explain why the ICHAZ that comprises a local brittle zone does not significantly compromise the toughness of the weldment (Section 5.2.1).

### 5.2.3 Tensile testing

Tensile testing was carried out to determine the yield and tensile properties of PM of the QT steels exposed to 0-4 PWHT cycles. There was no strong correlation between ultimate tensile test and the number of PWHT cycles for the 11, 12 and 20 mm PM plate. The room temperature UTS for the 11 mm sample increased marginally by 6 MPa after 1 PWHT cycle, and for the 12 mm BIS80 sample the UTS varied between 835 to 847 MPa for the PWHT cycle range (see Figure 4.57).

The room temperature YS, similarly, showed no significant trend for the PM region exposed to multiple PWHT cycles. The YS fluctuated from 798 to 836 MPa in the 12 mm samples and from 821 to 828 MPa in the 20 mm samples. The 11 mm samples showed a difference of 12 MPa after 1 PWHT cycle.

Other extensive work on the tensile properties of QT steels subjected to simulated postweld heat treatments was conducted by Lochhead and Speirs (1972, p189-219). They carried out similar (but not identical) work on a QT steel designed to ASTM A533
Grade B and reported a decrease in the YS (0.2% proof stress) and UTS with an increasing Holloman parameter. They graphed percentage change in YS or UTS, which is essentially a plot of sensitivity of the tensile properties across a PWHT temperature range. Lochhead and Speirs (1972) concluded that as the Holloman parameter increases, the tensile properties decrease in the PWHT temperature range of 610°C to 690°C. This conclusion is not consistent with the results of the present work for tensile properties as a function of the number of PWHT cycles at 570°C. The greater percentage change in tensile properties in the work carried out by Lochhead and Speirs (1972) is predominantly a result of the higher PWHT temperature range they investigated. Therefore is more influential in affecting mechanical properties than holding time. Gulvin et al (1967) also presented similar results to Lochhead and Speirs in which the percentage change in properties was plotted against the Holloman Parameter (see Figures 2.17-2.19).

An interesting point in Figure 2.17 is the positive percentage change (or increase) in the YS for some tests at a relatively low Holloman Parameter value of approximately 17. This suggests an increase in the YS at lower treatment temperatures, cumulative holding times or number of PWHT cycles. This is in agreement with the RT YS results shown in Figure 4.57, in which the more highly alloyed 11 mm and 20 mm BIS80PV samples show an increase in YS after 1 PWHT cycle. Moreover, the hardness results for the 11 mm plate steel in Figure 4.17(b) show a peak for short times in the temperature range 540–620°C. Since hardness correlates with YS it is inferred that transient strengthening can occur by secondary hardening during heat treatment in the PWHT range.

The lack of a trend in tensile properties of QT PM exposed to multiple PWHT cycles over the range studied, is consistent with the lack of a trend in percent elongation. The percent elongation for the 12 mm samples was 25% and for the 20 mm samples the percent elongation was 30%, irrespective of the number of PWHT cycles. The reduction in area for the 12 mm and 20 mm samples was approximately 55%, again irrespective of the number of PWHT cycles. The implication of these differences in % elongation is that BIS80PV steel has a higher work hardening exponent that promotes neck-resistant plastic flow, a higher uniform strain and a higher strain to failure. A higher density of
fine precipitate particles in QT BIS80PV is likely to homogenise deformation and promote strong work hardening. In contrast, the leaner BIS80 does not show the same work hardening capacity and the gap between YS and UTS is lower.

The stress ratio, defined as the ratio of YS to UTS, is a major concern for QT steels. The Australian Standard AS3597 specifies a maximum allowable stress ratio requirement of 0.93 at RT. It has already been noted that the 12 mm BIS80 PM plate does not comply with the stress ratio requirement (discussed in Section 5.4). Moreover, due to fluctuating values of YS and UTS with the number of PWHT cycles, an unacceptable stress ratio can result in plate that may have originally complied with the requirement. For example, the 11 mm BIS80PV plate initially had a stress ratio of 0.93, but after 1 PWHT cycle the stress ratio was 0.95. Hence it does not comply with the Australian Standard. However, tensile testing on the base plate is carried out after plate production and not after PWHT, and so this issue does not arise. Other cases where the stress ratio requirements were not met was for 1, 2 and 3 PWHT cycles for 20 mm BIS80PV. The stress ratio for all these samples was 0.94, but for 4 PWHT cycles the stress ratio returned to 0.93.

For a test temperature of –20°C, similar behaviour in the tensile properties was evident in the PM as the number of PWHT cycles was increased. There was no significant trend in the UTS and YS with the number of PWHT cycles (see Figure 4.58). At this temperature (which is closer to the lower shelf of the DBTT curve), the stress ratio requirements were met for all of the 11 mm and 20 mm BIS80PV samples (the stress ratio ranges from 0.92 to 0.93). This is attributed to an increase in UTS relative to YS, which remains similar to the room temperature value. It is inferred that, although dislocation motion can be initiated at about the same stress, work hardening is more effective because of reduced thermal activation leading to a higher fracture stress. There may have also been a geometric effect on the tensile test results at –20°C since a round cross-section was used as opposed to a rectangular cross-section at ambient temperature. Similar to the RT tensile tests, the stress ratio was greater than 0.93 for the 12 mm BIS80 plate at –20°C, but lower than the room temperature values. This observation is also consistent with the explanation given for the 20 mm samples.
5.2.4 Fatigue crack growth and CTOD fracture toughness

The resistance to fatigue crack growth is important in transportable pressure vessels because they are exposed to cyclic loading in transit. Fatigue crack growth data was collected on CTOD PM samples exposed to 0, 2 and 4 PWHT cycles (fast cooling). Figures 4.60(a) and Figure 4.61 show that as the number of PWHT cycles increases there is a slight increase in the fatigue crack growth rate (da/dN). Therefore the precipitation, growth and coalescence of carbides as a result of exposure to multiple PWHT cycles had a minor effect on the resistance to fatigue crack growth in the PM. Figures 4.60 (a and b) also show the higher resistance to fatigue crack growth of the 12 mm sample versus the 11 mm sample.

Additionally, CTOD fracture toughness testing was carried out to examine the effect of multiple PWHT cycles on CTOD (δ_m or u) and to establish a correlation between δ_m or u and Charpy V-notch impact toughness. All PM samples (11 mm, 12 mm and 20 mm) in the T-L orientation showed a decrease in δ_m or u as the number of PWHT cycles increased (see Figures 4.62, 4.63, 4.64 and 4.65). This is akin to the trend seen for the PM Charpy samples in the T-L orientation.

Similarly to the Charpy V-notch impact samples, SEM was used to examine and determine the mechanism by which PWHT leads to the decrease in δ_m or u. Figure 4.66 shows the fractographs for the 11 mm samples exposed to 0, 2 and 4 PWHT cycles. Figure 4.66(a), of 11 mm plate steel that has not been postweld heat treated, shows the dominance of void coalescence in the final crack region, which failed in a stable manner. For 2 PWHT cycles there was a growth in the size of the voids, as shown in the fractograph in Figure 4.66(b). This is indicative of growth and coalescence of second phase carbide particles that initiate ductile failure (Honeycombe and Bhadeshia, 1995, p228-249). After exposure to 4 multiple PWHT cycles even larger voids were present, with some regions of cleavage type fracture (see Figure 4.66(c)). Similar trends are shown in Figure 4.67, which consists of SEM fractographs of the CTOD samples of 12 mm BIS80 exposed to 0, 2 and 4 PWHT cycles.
The final crack of the CTOD samples of the 20 mm BIS80PV plate propagated in an unstable manner. This resulted in a significantly more brittle appearance in SEM fractographs, as shown in Figure 4.68. Figure 4.68(a) shows a predominantly brittle failure evident by the dominance of cleavage cracking. In this figure there is, however, some evidence of regions of coalescence of very small voids, possibly as a result of tearing between cleavage facets, as discussed previously. For 20 mm samples exposed to 2 and 4 PWHT cycles the fracture was still a predominantly quasi-cleavage fracture.

As the number of PWHT cycles was increased it is clear that there was a decrease in CTOD value and impact toughness, and an increase in the DBTT. Because of this common decrease in Charpy impact toughness and CTOD it was expected that a correlation could be found. The data in Figure 5.2 do indicate a general increase in impact energy with increasing CTOD value, but a strong correlation is not evident. This result is consistent with the lack of reports in the literature of a strong correlation between CTOD fracture toughness and impact toughness. The fact that there is no clear correlation between the two properties is expected to a degree because of the different nature of the two tests. The strain rates, sample dimensions, notch geometries and properties measured differ significantly.

However, there are correlations in the literature for the DBTT ($T_{27.1} (°C)$) with CTOD (mm) at –10°C, and the 0.1 or 0.2 mm CTOD transition temperature (°C) (Dolby, 1981). Therefore, the DBTT (°C) of 12 mm BIS80 PM and 20 mm BIS80PV PM was plotted against CTOD (mm) at –20°C (see Figures 5.3 and 5.44). Despite the limited number of data points, trends similar to those presented by Dolby (1981) were evident, that is, an increase in the DBTT (°C) correlated with a decrease in CTOD fracture toughness. The correlation appears to be material and thickness dependant, and further work is needed to establish a robust correlation. It can be concluded, however, that the current work is consistent with the expected trend, in that a decrease in DBTT (°C) results in a decrease in the CTOD value.
Figure 5.2: Impact toughness versus $\delta_m$ or $u$ for 11 mm, 12 mm and 20 mm PM samples in the T-L orientation (fast cooling) at –20$^\circ$C.

Figure 5.3: DBTT (°C) versus CTOD (mm) at –20°C for 12 mm BIS80 PM (T-L).
5.3 THE NEED FOR PWHT IN REDUCING RESIDUAL STRESSES

The research work has confirmed that PWHT (in particular multiple PWHT cycles) has a deleterious effect on properties that are vital to transportable pressure vessels. The impact toughness and fracture toughness are both compromised due to the precipitation, growth and coalescence of second phase carbide particles. This view is supported in part by the work of Evans and Bailey (2000, p355) and the research of Honeycombe and Bhadeshia (1995, p184). The latter researchers state that in low alloy QT steels the formation of new carbides occurs on heat treatment at 550°C. These carbides result from tempering or heat-treating between 500-600°C and the presence of carbide forming elements, such as Cr, Mo, V and Ti.

The perceived need for PWHT is mainly driven by the aim of reducing residual stresses. Residual stress measurements by the hole drilling method were carried out 100 mm from bandsaw cut edges along the weld centre-line in the final weld run (see Appendix A for weld procedure and Appendix I for stress measurement locations) for 12 mm BIS80 plate (before and after 1 PWHT cycle) and 20 mm BIS80PV plate (before
PWHT). The Standard recommends drilling of holes at least 50 mm apart. A residual stress measurement was also taken before PWHT in the base plate (PM) of 12 mm BIS80, 100 mm from the edge and from the centre of the weld.

The magnitudes of the residual stresses measured were relatively low in these test plates (see Table 4.1). The maximum principal stress measured was 243 MPa and this was in the 12 mm plate without PWHT, and the highest longitudinal stress was measured in the same plate (205 MPa). The weld centre-line is reported to be the region of the highest residual stresses (Gourd, 1991; ASM, 1983). In comparing the measured residual stresses to the yield stress of the 12 mm BIS80 plate (807 MPa) and 20 mm BIS80PV plate (823 MPa), it is evident that the maximum residual stresses measured are equivalent to only about 30% of the yield stress. Authors such as Lumb and Ambrose (2000, p1) state that PWHT reduces residual stresses to the range of 20-50% of the yield strength, but in the present work the residual stresses prior to PWHT were already in this range and hence relatively low. On this basis, there appears to be little need to reduce residual stresses by PWHT. However, it is unlikely that the residual stresses measured are truly representative of the entire test plate or those occurring in the construction of a transportable pressure vessel, even though the test plate welds were restrained. The magnitude of mid-thickness residual stresses can also differ from surface residual stresses, as shown by Suzuki et al (1978, pp87-112) in Figure 2.5.

However, it is unambiguous that PWHT effectively reduced the residual stresses measured close to the surface longitudinal and transverse to the direction of welding. In the 12 mm plate after PWHT, the residual stress in the transverse direction decreased from 205 MPa to 18 MPa, and residual stress longitudinal to the direction of welding decreased from 75 MPa to 37 MPa (see Table 4.1). The residual stresses are low in the elastic range at room temperature. This raises the question of the mechanism by which the residual stresses are reduced by PWHT. It is well documented (Gulvin et al, 1967; Lochhead and Speirs, 1972) that PWHT reduces residual stresses by the onset of plastic flow caused by reduction of yield stress at elevated temperatures, and dislocation glide and climb mechanisms play a role in the relaxation of these stresses (Nicholas, 1968). Figure 4.69, which shows a stress relaxation curve for 12 mm and 20 mm cross-weld
samples, supports this mechanism because at 570°C the YS is between 560 and 600 MPa, and after 1 hour of holding the initial stress of about 600 MPa relaxes to about 250 MPa. The strain present at the yield point at the PWHT temperature is elastic, but progressive replacement by creep strain occurs on holding. The residual stress corresponding to the residual elastic strain therefore decreases with holding time at temperature. Creep strain occurs by dislocation glide and climb at 570°C under the dynamically decaying external stress.

The residual stresses measured in all plates were compressive residual stresses (indicated by negative values in Table 4.1). The fact that no tensile residual stresses were encountered in the hole drilling strain gauge method does not imply their absence. Residual stresses are present without external forces, therefore the resultant force and the resultant moment produced by residual stress must effectively disappear (ASM Handbook Volume 6, 1983). Hence there are tensile residual stresses located somewhere in the welded plates, possibly in another section of the WM or the HAZ. Figure 2.4 is one example that shows that the HAZ can be under tensile residual stresses.

Compressive residual stresses in the WM region are not uncommon. Figure 2.5 shows that transverse and longitudinal residual stresses throughout a plate 165 mm in thickness can be both tensile and compressive. Two interesting points are shown in Figure 2.5. Firstly the residual stresses in the weld at the top and bottom of these plates are compressive in both the transverse and longitudinal directions, consistent with the residual stresses measured in the 12 mm and 20 mm welded plates tested in this research project. Secondly, the magnitudes of the compressive residual stresses in Figure 2.5 are much smaller in magnitude than in the near weld centre. Although the magnitudes of the maximum residual stresses in the weld plates are not known, the stress relaxation experiments (Figure 4.69) indicate that a stress close to the yield stress can be reduced to less than half within 1 hour holding at 570°C. In contrast, a stress of 270 MPa in the ‘elastic’ range was virtually unchanged (Figure 4.71) indicating that a stress of this level is not high enough to produce significant creep strain at this temperature. However, the stress relaxation experiments involve uniform tension and do not simulate the complex distribution of compressive and tensile stresses in the welded plate. The opportunity for
mutual cancellation of compressive and residual stress fields could increase the effectiveness of stress reduction in actual PWHT of welded plates. In summary, it can be deduced from the current residual stress experiments that stresses are substantially relieved by PWHT. Further detailed work on characterising the residual stresses in the test plates is currently being conducted at ANSTO using the neutron scattering technique, but this work is outside the scope of this thesis.

Compressive residual stresses are favourable because they retard or stop crack growth by causing crack closure (Gurney, 1979; Epselis, 1996). Therefore compressive residual stresses in the weld retard the growth of weld defects (which simulate cracks) and fatigue cracks. Resistance to fatigue crack growth near the surface would therefore be expected to be high. But as mentioned previously, residual stresses in a finite area cancel each other out, that is, the sum of compressive residual stresses equals the sum of tensile residual stresses in a member or component. Therefore the benefits of compressive residual stresses are counteracted by the tensile stresses in other regions of the weldment. In terms of tensile properties the TWI (Epselis, 1996, p8) reported that residual stresses, whether they are compressive or residual, have little effect on yielding because the small differences in elastic strain associated with residual stresses become insignificant compared to the plastic strain generated on yielding.

5.4 SUITABILITY OF BIS80 AS A PRESSURE VESSEL STEEL

BIS80 is a QT steel that has long been regarded as a potential candidate as 700PV grade pressure vessels steel due to its relatively high impact toughness, fatigue resistance and fracture toughness. It has a lower alloy content than BIS80PV and is austenitised at a slightly lower temperature (920°C) than conventional BIS80PV (930°C). It is also tempered at a slightly lower temperature (615°C) than conventional BIS80PV grade (630°C). BIS80, however, does not comply as a pressure vessel steel is due to the non-compliance of the stress ratio requirement, which is greater than 0.93.
The stress ratio requirement of the Australian Standards is based on the presumed importance of a significant gap between yielding and the UTS. A lower stress ratio (greater gap) is considered beneficial under conditions of internal pressure build up (slow strain rate conditions) in a pressure vessel beyond its design limits. Hence the true strength of the vessel will continue to increase until fracture occurs (work hardening) (Reed-Hill, 1992, p158). It is therefore believed that the higher work hardening capacity implicit in a lower stress ratio would allow more time to get away from the vessel after detection of a dangerous situation. However, with the strict guidelines on the manufacture of vessels to withstand conservative design pressures and the incorporation of safety relief valves required by the Australian Pressure Vessel Standards (AS4458-1997), the likelihood of a such a dangerous event must be low.

It can be argued that percent elongation (ductility) is equally, if not more significant than the stress ratio. The percent elongation in 12 mm BIS80 PM is 25% and this is well above the 18% minimum value required for 700PV pressure vessel steel outlined in AS3597-1997. A higher percent elongation suggests that any build up of internal pressure would be alleviated due to the bulging out of the vessel, thus reducing pressure by increasing volume (see Equations 4.3 and 4.4), and alerting onlookers to an over-pressure problem (‘yield before break’). The stress in the vessel wall, $\sigma$, is given by:

$$\sigma = \frac{PD_i}{2t}$$  \hspace{1cm} (Equation 4.3)

where $P$ is the internal pressure, $D_i$ is the internal diameter of the vessel and $t$ is the thickness of the plate (Faires, 1965, p34). In addition,

$$P \propto \frac{1}{\sqrt{V}}$$  \hspace{1cm} (Equation 4.4)

where $V$ is the volume of the vessel (Tipler, 1982).

The effect of external impact on transportable pressure vessels, especially at high strain rates (akin to a road collision), must also be considered. A lower stress ratio would result in increase in strength of the plate by work hardening until fracture occurs. The decrease in volume of the vessel due to the external impact would increase internal
pressure and result in higher stresses (see Equations 4.3 and 4.4). However, Figure 2.10(b), which is a photograph of the head of a vessel after external impact, shows that ultimate failure resulted from external impact and not the internal pressure build up arising from the decrease in volume. Hence the relevance of stress ratio, which is determined by a slow strain rate tensile test, is somewhat uncertain. Again, a key property in such a case is percent elongation (ductility), with a more ductile steel accommodating impact forces by plastic deformation rather than fracture and allowing safety relief valves to decrease the pressure, and hence the tensile stress on the plate. The superior fracture toughness and impact properties of BIS80 are therefore expected to be beneficial in resisting fast fracture in accidents involving high stresses.

However, BIS80 may only suitable as a pressure vessel steel if PWHT is deemed to be unnecessary by the statutory authorities. It has adequate ductility in the PM, WM and HAZ as shown by the bend, tensile and Charpy impact tests. These mechanical properties are superior to the corresponding properties of BIS80PV in all weldment zones. Further, the cross-weld yield strength and the tensile strength meet the minimum specified values without PWHT. However, this situation changes when BIS80 is exposed to single or multiple PWHT cycles. Due to the lower alloy content in BIS80 it is not able to maintain its strength (Figure 4.59) and hardness (Figure 4.25) in the WM and HAZ after PWHT.

There are inadequate levels of alloying elements such as Ni, Cr, Cu and Mn to form carbides that contribute to substantial secondary hardening. Hence, the tensile properties of cross-weld samples after PWHT are characterised by a YS and UTS that do not comply with AS3597-Structural and Pressure Vessel-Quenched and Tempered Plate. The YS decreases to 614 MPa (690 MPa is the minimum requirement) and the UTS decreases to 776 MPa (790 MPa is the minimum requirement) for 700PV grade steel (Figure 4.59). This strength deficit could be possibly overcome through the development of another weld consumable for this steel that produces a higher strength WM. However, increasing the WM strength may also compromise other mechanical properties such as the impact and fracture toughness of the weldment.
Finally, the stress ratio of BIS80 is in effect no different to BIS80PV in the presence of multiple PWHT cycles. The tensile properties (and other mechanical properties) of all these plates are quantified after plate manufacture. However, after fabrication and subsequent PWHT of the transportable pressure vessel, the properties of the WM only require qualification/quantification in the Australian Standards. Consequently, results presented in Figure 4.57 for multiple PWHT cycles show that the stress ratio can fluctuate in and out of compliance to the Australian Pressure Vessel Standards. This suggests that there are probably transportable pressure vessels on Australian roads that are not in compliance with the stress ratio requirement for the base plate (PM).

In summary, benchmarking against BIS80PV indicates that BIS80 may be suitable as a pressure vessel steel. It has a significantly higher resistance to fatigue crack growth, and higher impact and fracture toughness. The possibility of non-compliance of the stress ratio of BIS80PV after weld fabrication and subsequent PWHT cycles diminishes the strength of an argument to reject BIS80 as a pressure vessel steel on the basis of stress ratio. Moreover, the relevance of stress ratio is questionable in itself, since it is determined from low strain rate tensile tests and its importance to conditions of high strain rate impact loading is unclear.

In summary, the suitability of BIS80 as a pressure vessel grade may be justifiable if the PWHT requirement is removed from the Australian Pressure Vessel Standards. If however, PWHT remains as a mandatory requirement then a more appropriate weld consumable is required for welding fabrication of BIS80 to meet minimum cross-weld strength requirements and to justify its acceptance as a QT pressure vessel steel.