Effects of different heat treatments on hardness of Grade 91 steel

Effekter av olika värmebehandlingar på hårdheten hos Grade 91 stål

Jonas Ohlsson
Abstract:

CCI Valve Technology AB is a company located in Säffle, Sweden, that manufactures and installs bypass valves. Due to requirements outside normal standards on the valve's hardness values, some measurements have had difficulties meeting such requirements. During this thesis work, tests were carried out to determine how to overcome the difficulties. The experiments focused on five different areas that may affect the components hardness, welding method, soaking temperature during post weld heat treatment, measuring procedure, component thickness and number of heat treatment cycles. The Grade 91 steel specimens that were examined consisted of five solid cylinders and three various pipes that were welded together by using shielded metal arc welding (SMAW) or gas tungsten arc welding (GTAW). Each pipe was sawed apart into three equal parts. All specimens were hardness tested and eight of the specimens' microstructure was studied with an optical microscope. The hardness measurement instruments used, LECO V-100-C2 and GE-MIC 10, are Vickers hardness testers, one stationary and the other one portable. The measuring results contain a vast number of different hardness measurement data. From the analyzed data, the conclusions were drawn that the most suitable soaking temperature during post weld heat treatment were 750° C, that the SMAW method creates a more stable hardness profile than the GTAW method, and that one heat treatment cycle is more beneficial than two or more.

Key words: Grade 91, Post weld heat treatment, Heat treatment cycles, Hardness.
Sammanfattning

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1. Introduction:

This thesis work was carried out at CCI Valve Technology AB in Säffle, Sweden, which is a global company that during the past 50 years have placed nearly 100 thousand valves into service. CCI Valve designs and manufactures valves for applications in pulp and paper plants, and in the power, oil and gas industry. CCI Valve purchases pipes, rods, outlets and valve housings. These components are machined, welded and heat-treated. Prior to delivery, the bypass valves are functionally tested and after approval, they are assembled with actuators. Recently, they have had difficulties with fulfilling the requirements on hardness outside standard requirements. Despite accurate post weld heat treatment, several valves show too high hardness values in the welds and too low hardness values in the base material when measured with a portable hardness tester.

1.1 Aim of thesis work

The aim of this thesis work is to discover why the valves fail to meet the outside standard requirements and how different parameters affect the material properties, so that CCI Valve can improve their products.

The main objectives of this thesis work are listed below:

- Examine how the number of heat treatment cycles affects the hardness of the material.
- Test how material properties are affected by the selection of welding method, pipe thickness and soaking temperature during post weld heat treatment.
- Examine how the value of hardness is affected by the operator or the measuring instrument.
1.2 Background of the material:

The material that CCI Valve use is called Grade 91, which is a modification of the steal alloy 9Cr-1 Mo. The alloy's chemical composition is presented in Table 1. Grade 91 was co-developed by the Oak Ridge National Laboratory and Combustion Engineering, who, in their study, demonstrated the alloy's good mechanical properties [1,2,3]. The standard 9Cr-1 Mo alloy was improved by addition of vanadium, niobium and nitrogen for precipitation strengthening the alloy additionally. The addition of these elements resulted in a ferritic alloy with greater creep strength than before.

In 1983, ASME boiler and pressure vessel code approved the Grade 91 steel and during the 1990's it was commonly used for upgrading traditional fossil plants and when manufacturing critical pressure part components for newer power plants [4]. However, some issues frequently appear when using this alloy, and one of them is the hardness.

Before the valve is installed, the hardness values should be in the range of 200-263 HV for components and 200-294 HV for welds [5]. However, the requirements outside normal standards implies that the hardness of the base material at field weld joint shall be a minimum of 210 HV after post weld heat treatment. And that the weld joints after post weld heat treatment shall be between 200-270 HV, with a spread no greater than 30 HV, from lowest to highest, at each of the locations.

Table 1 shows the Grade 91 steel's chemical composition. The elements marked with the letter A are not required to be controlled by ASME specifications, but the elements' values should be considered target levels. Elements marked with the letter D are Carbon and Nitrogen, the sum of these elements must exceed 0.12%. The minimum level of Chromium is marked with the letter C for the reason that, for piping the minimum amount of Chromium should be 8.5%. Composition values marked with the letter B is different from ASME.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Composition Pipe (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>0.08-0.12</td>
</tr>
<tr>
<td>Manganese</td>
<td>0.30-0.60</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.020 [max.]</td>
</tr>
<tr>
<td>Sulfur</td>
<td>0.010 [max.]</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.20 - 0.50</td>
</tr>
<tr>
<td>Chromium</td>
<td>8.00 - 9.50</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>0.85 -1.05</td>
</tr>
<tr>
<td>Vanadium</td>
<td>0.18 - 0.25</td>
</tr>
<tr>
<td>Columbium</td>
<td>0.06-0.10</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>0.035 - 0.070</td>
</tr>
<tr>
<td>Nickel</td>
<td>0.20 [max.]</td>
</tr>
<tr>
<td>Aluminum</td>
<td>0.020 [max.]</td>
</tr>
<tr>
<td>Titanium</td>
<td>0.01 [max]</td>
</tr>
<tr>
<td>Zirconium</td>
<td>0.01 [max]</td>
</tr>
<tr>
<td>Copper</td>
<td>0.25 [max.]</td>
</tr>
<tr>
<td>Arsenic</td>
<td>0.012 [max]</td>
</tr>
<tr>
<td>Tin</td>
<td>0.010 [max]</td>
</tr>
<tr>
<td>Antimony</td>
<td>0.003 [max]</td>
</tr>
<tr>
<td>Ni/Al ratio</td>
<td>4.0 minimum</td>
</tr>
</tbody>
</table>
1.3 Background on practices

1.3.1 Welding

When using the shielded metal arc process (SMAW), a minimum pre-heat temperature of 205° C shall be maintained for components that are highly restrained, for example components that have been forced together before welding. For a tube-to-tube butt weld, it is acceptable that the maintained pre-heat temperature reaches 150° C before welding, for both the shielded metal arc process and the gas tungsten process (GTAW) [5]. During the welding, the component shall not be subjected to temperatures above 350° C. If welding is interrupted, the pre-heat temperature must be maintained unless the weld is thicker than one third of the components thickness [5]. If so, the component shall be given a hydrogen bake, which involves heating the component above 300° C for one or two hours, depending on its thickness, followed by slow cooling. Once the hydrogen bake is finished, the component must be kept dry until the welding process is resumed.

There is no standard welding method for joining Grade 91 material; there are only recommended filler materials. However, the various welding methods differ a little from each other. The GTAW weld metal will not get as hard as the SMAW weld metal [6]. The heat affected zone (HAZ) and the base material may however not reach a higher hardness when using SMAW, compared to GTAW, as shown in Figure 1 [7]. The material examined and presented in Figure 1 is not Grade 91 steel, but a medium carbon steel (MCS).

![Hardness profile for SMAW and GTAW along medium carbon steel side](image)

Figure 1: Hardness profile for SMAW and GTAW along medium carbon steel side [7]. The distance shown on the X-axis is in mm.
1.3.2 Heat treatments

If the component is heat-treated directly after welding, its temperature must have dropped to at least 80° C before heat-treated. This must be done so that the austenite is completely transformed to martensite before heat treatment, otherwise martensite may form after the post weld heat treatment (PWHT) and that will result in a hard and brittle weld [8]. The post weld heat treatment performed at CCI Valve for Grade 91 involves heating the component at a rate of 40° C per hour until it reaches a temperature of 760° C (this soaking temperature can be varied between 740-780° C). The component is held at chosen soaking temperature for one or two hours (depending on its thickness) and is then cooled with a rate of 80° C per hour until it reaches 300° C, from which it can be air cooled to room temperature. A Common problem encountered with post weld heat treatments is that the different parts of a component can cool at different rates depending on their thickness, thicker components will cool with a lower rate, and this causes a variety of hardness in the product.

As previously stated, the soaking temperature can be varied between 740-780° C, but what happens to the material properties within that range? When tempering modified 9Cr–1Mo steel, or Grade 91 steel, at the temperatures 720° C, 740° C and 760° C for one hour, hardness measurements show that the material's hardness decreases faster in the beginning and then slower at the higher heat treatment temperatures, as the curve in Figure 2 shows [9]. The hardness decreases because of growing precipitates.

![Figure 2: Decreasing hardness due to heat treatments at various temperatures [9].](image-url)
1.3.3 Hardness measurements

Since the components are very expensive to produce a non-destructive hardness test is a good way to control their quality. The hardness testing method used in this report is the Vickers-method. The Vickers-method uses a diamond pyramid indenter. The indenter is pressed into the surface of the material with a preset load for a period of time. When the indenter is unloaded, the two diagonal distances between the corners of the indentation are measured and a mean value is calculated. With the load, F, and the average distance, d, known the hardness of the material can be calculated with Equation (1) [10].

\[
HV \approx \frac{1.8544 \times F}{d^2}
\]  

(1)

There are several guidelines to follow when measuring the hardness of a component, but the main one is to prepare the test surface to at least a 240-grit finish before using the GE MIC-10. The measuring instrument must be properly calibrated before measuring [5]. Pressure part components with a diameter smaller than 600 mm, shall be hardness tested at four locations around the circumference, with 90° between the locations. If the component’s diameter exceeds 600 mm, measurements shall be made at eight locations, with 45° between each location [5].
2. Method

2.1 Test 1

During Test 1, the specimens were measured using two different hardness testers, the stationary LECO V-100- C2 and the portable GE-MIC 10. The stationary hardness tester is shown in Figure 3 with one of the pucks placed on the V-shaped anvil. When the measurements were made, the machine was set to a pressure load of 20 kilopond. Ten measurements were obtained for each puck. After the heat treatments, the oxide layer was removed by polishing the pucks' surfaces, and then the same procedure was performed once again.

GE MIC-10, Portable hardness tester:
The measured values were obtained using the portable hardness tester GE MIC-10. Prior to the measurements, the instrument was calibrated against another metal ingot with a hardness of 225 HB. When calibrating the GE-MIC 10 a minimum of five readings are made on an ingot of the same material with a predetermined hardness, out of these five values an average is calculated. This average is then corrected so that the display shows the ingots predetermined value [11]. When measuring the ingot once more it can be determined whether the calibration is good enough, if not, the same procedure can be carried out once again, but with more readings. During the procedure, the instrument was held vertically against the surface of the metal puck, into which the indenter was pressed until a value shown on its display. This was done ten times for each puck. After the heat treatments, the oxide layer was removed by polishing the pucks' surfaces, and then the same procedure were performed once again, but with two different operators.

Figure 3: Stationary hardness tester, LECO V-100-C2, with puck placed on its V-shaped anvil.
Heat treatment:
The five specimens were placed in the middle of a furnace, shown in Figure 4, and three sensors were connected to specimen five, since specimen five is the last one to be removed from the furnace. Two of the sensors measured the specimen's temperature and the other sensor cancels the current cycle if the holding temperature is exceeded. The sensors also register the temperature in a heating diagram and these diagrams are attached in appendices. The furnace was programmed to increase in temperature at a rate of 40° C per hour, and when it had reached 760° C it would hold that temperature for two hours, followed by a cooling rate of 80° C per hour until the specimen reached 300° C. This represented one heat treatment cycle. When one cycle had passed specimen one was removed from the furnace. After every heat treatment cycle, another specimen was taken out until there was none left.

After the last hardness measurements, Puck 1 and Puck 5 were cut into smaller pieces. The pieces were then mounted into two separate Bakelite cylinders as shown in Figure 5. Thereafter the specimens' surfaces were polished to a 3µm finish, by using abrasive paper, fine grinding disks and DP-spray that contain polycrystalline diamonds. After every step of polishing, the specimens were cleaned in an ultrasonic bath. Lastly, the surfaces were etched.
After polishing the steel surfaces, the microstructure of the specimens were studied through an optical microscope, shown in Figure 6. Pictures were captured and analyzed.

Figure 6: Optical microscope connected to computer screen, used when studying the pucks' microstructure
2.2 Test 2

Six pipes were machined for this test, two with an outer diameter of 350 mm and a thickness of 25 mm, and four with an outer diameter of 50 mm and a thickness of 10 mm. The two larger pipes were welded together using SMAW, shielded metal arc welding. The four other pipes were welded together two by two, using SMAW and GTAW, gas tungsten arc welding. Before welding two pipes together, ceramic heating mats were connected to each pipe. In Figure 7, heating mats are connected to the large pipes before welding, and in Figure 8 the welding procedure for the two smaller pipes, using GTAW, is shown. When the parts had reached a temperature of approximately 225°C, the welding could begin. The pre-heating diagrams and the protocols taken during welding can be found in the appendices.

Figure 7: Heating mats strapped to the large pipe (later referred to as Pipe 1) with steel ribbons.

Figure 8: Welding two smaller pipes together with GTAW. These joined pipes will later be referred to as Pipe 2.
After welding, the pipes were sawed apart into three parts and each part's surface was finished according to EPRI's technical report, *Guidelines and Specifications for High-Reliability Fossil Power Plants*. Each part was hardness tested on its outer surface, with the stationary hardness tester, as illustrated in Figure 9. Some of the parts were harder to measure because of their uneven edges, but this was solved by using smaller triangular pieces and applying the 3-2-1 method for fixing their degrees of freedom.

Three parallel measurements were performed across the base material, the heat affected zones and the weld, where each measurement consisted of 15 indentations. The indentations are shown in Figure 10. Apart from those measurements, another one consisting of 10 indentations was carried out on the surface of the base material.

![Figure 9: Measuring the hardness of one part from the large pipe. The triangular piece was used to stabilize the object during measurements.](image)

![Figure 10: The 45 indentations made on one of the parts from a smaller pipe during three different measurements. The indentations were made approximately 2 mm apart on a 30 mm distance.](image)
The parts were then heat-treated in three different ways so that one part of Pipe 1, one part of Pipe 2 and one part of Pipe 3 went through the same heat treatment, and so on. The difference between the three heat treatments were only their soaking temperatures, 740° C during the first heat treatment, 750° C during the second one and 760° C for the third one. Their heating-and cooling rates were the same through all three. These heating diagrams can all be found in the appendices under the heading Post weld heat treatments for pipes.

Once the parts completed their heat treatment, their hardness was examined again, as close as possible to where previous measurements were made, without hitting earlier indentations. The parts from Pipe 1 and Pipe 2 were studied with the optical microscope as well. The parts were cut into smaller pieces and mounted into several Bakelite cylinders. As in the previous test, the parts were polished and etched before studying the various parts' microstructures.

The designations stated in Table 2 are how the parts will be referred to in the results.

<table>
<thead>
<tr>
<th>Treatments performed:</th>
<th>Designation:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Large pipe, SMAW, PWHT at 740° C</td>
<td>Pipe 1.1</td>
</tr>
<tr>
<td>Large pipe, SMAW, PWHT at 750° C</td>
<td>Pipe 1.2</td>
</tr>
<tr>
<td>Large pipe, SMAW, PWHT at 760° C</td>
<td>Pipe 1.3</td>
</tr>
<tr>
<td>Small pipe, GTAW, PWHT at 740° C</td>
<td>Pipe 2.1</td>
</tr>
<tr>
<td>Small pipe, GTAW, PWHT at 750° C</td>
<td>Pipe 2.2</td>
</tr>
<tr>
<td>Small pipe, GTAW, PWHT at 760° C</td>
<td>Pipe 2.3</td>
</tr>
<tr>
<td>Small pipe, SMAW, PWHT at 740° C</td>
<td>Pipe 3.1</td>
</tr>
<tr>
<td>Small pipe, SMAW, PWHT at 750° C</td>
<td>Pipe 3.2</td>
</tr>
<tr>
<td>Small pipe, SMAW, PWHT at 760° C</td>
<td>Pipe 3.3</td>
</tr>
</tbody>
</table>
3. Results

3.1 Test 1

3.1.1 Hardness by number of heat treatments

Figure 11 shows the various pucks' mean hardness. Each bar shows the hardness mean value calculated from ten readings on respective puck's surface. The hardness of each puck is measured before and after their individual heat treatment. Puck 1 has completed one heat treatment cycle, Puck 2 has completed two heat treatment cycles and so on. Studying Figure 11 shows that the decrease in hardness levels off after the first two heat treatments.

![Hardness before and after heat treatment](image)

Figure 11: Hardness before and after several heat treatments. The puck's name does also explain the number of heat treatment cycles it has completed.
Figure 12 shows the microstructure of Puck 1 and Puck 5 when studied with an optical microscope. The top two pictures show the microstructure of Puck 1 respective Puck 5 at a magnification of 20x, and the bottom two shows the microstructure of Puck 1 respective Puck 5 at a magnification of 100x. The big difference between Puck 1 and Puck 5 can be seen when comparing the 100x magnifications. The carbides in Puck 5 are bigger than the carbides seen in Puck 1. Larger carbides soften the material.

Figure 12: The microstructure of Puck 1 and Puck 5 at two different magnifications.
3.1.2 Hardness by measuring instrument

Figure 13 shows the differences between the mean hardness values from the measurements made by the various types of measuring instruments. The largest difference is more than 20 HV. The standard deviation for each testing method is shown as red bars in the figure. The lower the value, the more even measurements are made.

![Mean hardness values before heat treatment](image)

Figure 13: Mean hardness and standard deviation for the different measuring instruments.
3.1.3 Hardness by operator

Figure 14 illustrates how the obtained mean hardness values depend on the operator. Both operators have measured each puck with the GE-MIC 10 hardness tester. The difference between their results differs by almost 20 HV for each puck. The red bars show the standard deviation for the operators, but there is not a big difference between those values.

Figure 14: The mean hardness values and standard deviations of the various operators.
3.2 Test 2

3.2.1 Hardness by soaking temperature

Figure 15 shows the change in hardness after post weld heat-treating the various pipes at three different soaking temperatures. The blue lines show the hardness of Pipe 1, the red lines show the hardness of Pipe 2 and the green lines show the hardness of Pipe 3. The letter N on the X-axis represents the condition of the pipes prior to the post weld heat treatment.

Figure 15: Hardness of the pipes' base material before and after PWHT at different soaking temperatures. N shows the pipes' hardness before they were heat-treated.
The figures below, numbered from 16 to 24, show the hardness profile over the weld, the HAZ and the base material. Each point represents the mean value of three readings made on approximately the same distance from the weld center. Readings that seem unrealistic are not taken into count. The target area is restricted by the requirements outside normal standards, stated in the introduction.

The hardness profile of Pipe 1.1 is presented in Figure 16. Pipe 1.1 shows elevated hardness values in the weld and in the HAZ, these hardness values are not within the target area.

![Hardness profile for Pipe 1.1 before and after PWHT](image)

**Figure 16:** Hardness profile over the base material, HAZ and weld before and after PWHT at 740°C.
Figure 17 presents the hardness profile of Pipe 1.2. The hardness profile is stable and all hardness values are within the target area.

Figure 17: Hardness profile over the base material, HAZ and weld before and after PWHT at 750° C.

Figure 18 presents the hardness profile of Pipe 1.3. The hardness profile is less stable than the hardness profile presented in Figure 17, but all hardness values are within the target area.

Figure 18: Hardness profile over the base material, HAZ and weld before and after PWHT at 760° C.
Figure 19 shows the hardness profile of Pipe 2.1. This specimen has three elevated hardness values in the HAZ that do not stay within the target area.

**Hardness for Pipe 2.1 before and after PWHT**

Figure 19: Hardness profile over the base material, HAZ and weld before and after PWHT at 740° C.

Figure 20 shows the hardness profile of Pipe 2.2. This hardness profile is more stable than the hardness profile of Pipe 2.1, but one hardness value in the HAZ lies outside the target area.

**Hardness for Pipe 2.2 before and after PWHT**

Figure 20: Hardness profile over the base material, HAZ and weld before and after PWHT at 750° C.
Figure 21 shows the hardness profile of Pipe 2.3. The hardness profile is quite stable, but two hardness values, one in the HAZ and one in the base material, lies outside the target area.

![Hardness for Pipe 2.3 before and after PWHT](image)

Figure 21: Hardness profile over the base material, HAZ and weld before and after PWHT at 760° C.

Figure 22 shows the hardness profile of Pipe 3.1. The hardness profile is not that stable, but all of the hardness values lie within the target area.

![Hardness for Pipe 3.1 before and after PWHT](image)

Figure 22: Hardness profile over the base material, HAZ and weld before and after PWHT at 740° C.
Figure 23 presents the hardness profile of Pipe 3.2. The hardness profile is quite stable, and all of the hardness values lie within the target area.

![Hardness for Pipe 3.2 before and after PWHT](image)

Figure 23: Hardness profile over the base material, HAZ and weld before and after PWHT at 750° C.

Figure 24 presents the hardness profile of Pipe 3.3. The hardness profile is not that stable, and one of the hardness values in the base material lies outside the target area.

![Hardness for Pipe 3.3 before and after PWHT](image)

Figure 24: Hardness profile over the base material, HAZ and weld before and after PWHT at 760° C.
Figure 25 shows the microstructure of Pipe 1.1, Pipe 1.2 and Pipe 1.3 when studied with an optical microscope at two different magnifications. When comparing the different microstructures, it is easy to see that the carbides grow with increasing soaking temperature. Pipe 1.1 has very small carbides, in Pipe 1.2 the carbides have grown but they are still small and for Pipe 1.3 the carbides have grown big.

Figure 25: Microstructure of Pipe 1.1, Pipe 1.2 and Pipe 1.3 at two different magnifications.
Figure 26 shows the microstructure of Pipe 2.1, Pipe 2.2 and Pipe 2.3 when studied with an optical microscope at two different magnifications. There is not a huge difference between the three microstructures; the carbides are approximately the same size.
3.2.2 Hardness by welding method

The figures below show the hardness of the welds before and after PWHT. In all of these figures the green bar (Pipe 3) shows a higher value of hardness than the red bar (Pipe 2). The pipes represented by these two colors have the same dimensions, but are joined using different welding methods. The welding method resulting in a harder weld is, according to this data, SMAW.

**Figure 27:** Hardness in weld before and after PWHT at 740° C.

**Figure 28:** Hardness in weld before and after PWHT at 750° C.

**Figure 29:** Hardness in weld before and after PWHT at 760° C.
Figure 30 shows the microstructure in the weld of Pipe 1.1, Pipe 1.2 and Pipe 1.3 when studied with an optical microscope at two different magnifications. When comparing the different microstructures, it is easy to see that the carbides grow with increasing soaking temperature. Pipe 1.1 has very small carbides, in Pipe 1.2 the carbides have grown but they are still small and for Pipe 1.3 the carbides have grown big.

Figure 30: Microstructure in the weld of Pipe 1.1, Pipe 1.2 and Pipe 1.3 at two different magnifications.
Figure 3 shows the microstructure in the weld of Pipe 2.1, Pipe 2.2 and Pipe 2.3 when studied with an optical microscope at two different magnifications. The differences that can be seen between the various specimens are mainly the sizes of the carbides. But when studying Pipe 2.1, the shape of the grains also differs from the other two microstructures. The grains of Pipe 2.1 are round, but the grains of Pipe 2.2 and Pipe 2.3 are elongated.

Figure 3: Microstructure in the weld of Pipe 1.1, Pipe 1.2 and Pipe 1.3 at two different magnifications.
3.2.3 Hardness by thickness

The mean hardness of the base material affected by the thickness of the pipe can be seen when studying Figure 17. The differences between Pipe 1 (blue lines), Pipe 2 (red lines) and Pipe 3 (green lines) when subjected to the same heat treatments are also presented in Table 3 below.

Table 3: Mean hardness of the pipes’ base material. The highest hardness value is marked by green background filling and the lowest value is marked by red background filling.

<table>
<thead>
<tr>
<th>PWHT</th>
<th>Hardness of Pipe 1</th>
<th>Hardness of Pipe 2</th>
<th>Hardness of Pipe 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>740° C</td>
<td>227 HV</td>
<td>232 HV</td>
<td>237 HV</td>
</tr>
<tr>
<td>750° C</td>
<td>235 HV</td>
<td>236 HV</td>
<td>225 HV</td>
</tr>
<tr>
<td>760° C</td>
<td>223 HV</td>
<td>225 HV</td>
<td>220 HV</td>
</tr>
</tbody>
</table>
4. Discussion

4.1 Test 1

The first test was performed to evaluate the behavior of the material's hardness under the effect of numerous heat treatment cycles. When studying the results shown in Figure 11 the decrease in hardness grows with the number of heat treatments, the more heat treatments the softer the material gets. However, the difference between the mean hardness of Puck 1 and Puck 5 are only 1.5 HV. According to Figure 12, the growth of the material's carbides causes the decrease in hardness.

When comparing the different measuring instruments, the stationary hardness tester shows a much higher hardness of the material than the portable hardness tester. Even though both testers obtain mean values within the target area the total mean values for the two instruments, calculated by 50 readings, differs by almost 17 HV. The standard deviations for the stationary hardness tester (4.3 units) and the portable hardness tester (14.6 units) in Figure 13 show an even bigger difference.

However, the obtained hardness value may not depend on the measuring instruments but on the operator. When comparing the two results in Figure 14, the mean hardness values obtained by the various operators differ by almost 20 HV for each puck, but their standard deviations are rather similar. This indicates that something must have gone wrong during the calibration of the GE-MIC 10. As the calibration procedure is explained in the introduction, the accuracy of the calibration lies in the number of readings on the ingot's surface and the operators' effort during the procedure. It is the operator who decides when the GE-MIC 10 is calibrated and when it is accurate enough to start measuring. The accuracy may also be affected by how the hardness tester is held during the procedure, but in that case, the standard deviations would differ a lot more. The influence of the operator on the obtained hardness values should be reviewed.
Previous reports have shown that when heat treating Grade 91 material the hardness decreases with increasing soaking temperature (as shown in Figure 2), at least within a certain temperature range [9]. The soaking temperatures investigated in this report are in the range of 740-760°C. In Figure 15, the decrease in hardness is plotted against soaking temperature, but there are different results for the various pipes. Pipe 1 softens when heat-treated at 740°C and 760°C, but increases in hardness by 5 HV when heat-treated at 750°C. Pipe 2 and Pipe 3 however, decreases in hardness after every heat treatment. Although the curves are different, the trend shows that the hardness of the pucks decreases when they are heat treated at a higher soaking temperature. Studying Figure 25 and Figure 26, the growth of the material's carbides causes the decrease in hardness. Small precipitations increase the hardness of a material, but when the precipitations grow the hardness decreases, and this happens when post weld heat-treating the pipes at higher soaking temperatures.

However, the main reason for a PWHT is not to make the base material softer, that is only a side effect, the main reasons are to release residual stresses after welding and to eliminate susceptibility to stress corrosion cracking. Although, in this case one of the reasons is to decrease the hardness of the weld so that it satisfies the requirements outside normal standards. Throughout all of the different PWHT's the hardness of the weld meets the customer demands upper limit, and just like in the previous statement the hardness of the weld decreases with increasing soaking temperature. But how are the hardness affected by different welding methods?

When studying figures 27 to 29, the comparable pipes are Pipe 2 and Pipe 3, since the only parameter separating these two pipes is the type of weld. In each diagram the green bar (Pipe 3) shows a higher value than the red bar (Pipe 2). This means that, just like in Figure 1, the hardness of a shielded metal arc weld exceeds the hardness of the gas tungsten arc weld [7]. The GTAW process should therefore be more appropriate when welding these components, since CCI Valve have obtained too high values of hardness in the weld earlier. However, there are many parameters to take into account; the pipes joined with GTAW show a value of hardness in the HAZ that is not within the acceptable range. The figures numbered from 16 to 24 show the hardness profile over the weld, HAZ and the base material, and Pipe 2 (GTAW) does not meet the hardness demands of the HAZ after any of the PWHT's. That might be a coincidence, but when studying the specimens' hardness profiles it seems that SMAW generally is the best welding method. Although the shielded metal arc weld reach a higher hardness it does not affect the base material as much as the GTAW.

The effect the thickness of the pipe has on its hardness is something that is harder to evaluate from the obtained data. In Table 3, the mean hardness of the pipe's base material after the different PWHT's are presented. By looking at the results, there are very few conclusions that can be drawn since the difference between the various pipe's hardness only differ by 10 HV at most. There is not a considerable difference between them and neither is there an obvious trend. However, if any conclusions were to be drawn, it seems that the thinner pipes gain a harder base material than the thicker one. It is not a great difference, but it exists.
5. Conclusion

From the results obtained during this work the following conclusions can be made:

- When heat treating the Grade 91 material there is no significant benefit attained from putting it through several cycles, one heat treatment cycle is enough.

- Despite that the results obtained from the GE-MIC 10 are widely dispersed, the measurements seem to depend on the calibration of the measuring instrument and not on the measuring instrument itself. If the procedure of the measurement and the calibration could be standardized, so that every operator performed the calibration and the measurement the same way, the GE-MIC 10 would be an adequate measuring instrument.

- The welding method previously used at CCI Valve for larger pipes is SMAW, and according to the results the shielded metal arc weld gains a higher hardness but it affects the base material less than the gas tungsten arc welding method. A continued use of SMAW is recommended.

- When studying the different PWHT's a soaking temperature of 750°C is what generates the most stable hardness profile when measuring the hardness over the base material, HAZ and the weld. A PWHT at 750°C does not make the weld to hard nor the base material to soft.

- When heat treating components that vary in thickness the soaking temperature does not seem essential to the obtained hardness.
6. Acknowledgements

This thesis work has been completed thanks to the contribution from the following people:

Anders Gåård, mentor at Karlstad University, Sweden
Mattias Eng, quality assurance manager at CCI Valve Technology AB, Sweden
Johan Fremling, international welding engineer at CCI Valve Technology AB, Sweden
Nicklas Gustafsson, welding specialist at CCI Valve Technology AB, Sweden
Christer Burman, research engineer at Karlstad University, Sweden
Welders and other employees at CCI Valve Technology AB, Sweden

Thank you!
7. References


8. Appendices

8.1 Heat treatments for pucks
Comment
HÅRHDTEST DEL 1 AV 5.

Signature

Settings
Ramp rate 40 °C/h  Dwell temp 760 °C  Dwell time 2 h

Chart
Comment
HÄRDETSTEST DEL 2 AV 5

Signature

Settings
Ramp rate 40 °C/h  
Dwell-temp 760 °C
Dwell-time 2 h

Chart
Comment
HÅRDNÄSTTEST DEL 3 AV 5

Signature

Settings
Ramp rate 40 °C/h   Dwell-temp 760 °C   Dwell-time 2 h

Chart
Comment
HÅRDHETSTEST DEL 4 AV 5

Signature

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<th>Dwell-time</th>
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<td>760 °C</td>
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Comment
HÅRDHETSTEST DEL 5 AV 5

Signature

Settings
Ramp rate 40 °C/h  
Dwell-temp 760 °C  
Dwell-time 2 h

Chart
8.2 Pre-heat diagrams for pipes
InteliHeat

Date / Time
2014-04-24 11:02

Comment
SVETSPROV 2014 VO:308931
Rec 1

Signature

Settings
Ramp rate 0 °C/h  Dwell temp 0 °C  Dwell time 0 h

Chart

Nn. 4049

short circuit in heating mat

hydrogen bake
InteliHeat

Comment
SVETSPROV X10 50mm

Signature

Settings
Ramp rate: 0 °C/h
Dwell temp: 0 °C
Dwell time: 0/h

Chart

Nr. 4050

- Target
- Chan 1
- Chan 2
- Chan 3
- Chan 4
- Chan 5
- Chan 6
InteliHeat

Date / Time
2014-05-05 13:24

Comment
SVETSPROV X10 PINNE 50 mm

Signature

Settings
Ramp rate 0 °C/h  Dwell-temp 0 °C  Dwell-time 0 h

Chart

Nº 4081

[Graph showing temperature over time with various channels labeled: Target, Chan 1, Chan 2, Chan 3, Chan 4, Chan 5, Chan 6]
8.3 Welding protocols for pipes
**Protokoll för kvalificering av svetsprocedur (WPQR)**

**Svetsare / Welder:** Jan Sethehind

**Datum för svetsningen / Date of welding:** 2014-04-24

**Plats / Location:** Bas 9

**Produkt/Provningsstandard / Prod./test/Mat.**

**Förbands- & svetsförekom / Joint & weld type:** BW, stumsvets

**Metod för förberedning & rengöring / Method of preparation & cleaning:** Beatbetning, svärning

**Förmodad arbetstemperatur / Preheat temperature (°C):** 216 °C

**Särskild värmning eller tokning / Any special baking or drying:** Rotmejsling/rotslöt, detaljer / Details of back gouging/baking:

**Fogueradning & rengöring (metod):**

**Enkelt Färö, Båttyp (droppovergång) / Mode of metal transfer:**

**Hålsvetsning (metod) / Tack welding (method):** Pinne, SMAW

**Fogutformning / Joint design**

| 1 | SMAW OK 76.78 | ESAB | 2,5 | 25 | 0 | 225 | DC+ |
| 2 | SMAW OK 76.94 | ESAB | 4 | 4 | 170 | 225 | DC+ |
| 3 | SMAW - 11-11 | - | 4 | 165 | 225 | DC+ |
| 4 | SMAW - 11-11 | - | 4 | 165 | 225 | DC+ |

**Utmatning / Post-heating:** 300 °C, 1,5 h, långsam svalning → 80 °C

**Tillverkare / Manufacturer:** Jan Sethehind

**Datum / Date:** 2014-04-29

---

"* om så erfordras eller är tillämpligt / if required or applicable"
Protokoll för kvalificering av svetsprocedur (WPQR)

Svetsare / Welder: Christian Dahlberg
Enl. Figur / Acc. to figure: F-6238

Datum för svetsningen / Date of welding: 2014-04-30
Plats / Location: Bas 4

Produkt-/prövningsstandard / Prod./test. stan: 350

Materialpockel (mm) / Material thickness (mm): 45

Ytterdiameter rör (mm) / Outside pipe diameter (mm): 25

Forbands- & svetsart / Joint & weld type:

Method för fogberedning & rengöring / Method of preparation & cleaning:

Forhållande arbetstemperatur / Pre-heat temperature (°C): 238°C
Särskild värming eller torkning / Any special baking or drying:
Fogberedning & rengöring (mетод):
Enkelsträng Bagtyp (dopovergång) / Mode of metal transfer:
Haftsvetsning (metod) / Task welding (method):

Strängplanering / Welding sequences

Sträng / Run / Svets- / Elektrisk / Electrode / Diameter / Ström / Spänning / Strömtyp / Betoning / Designation / Fabrikat / Make / Diam (mm) / Current (A) / Voltage (V) / Polarity / Mode / Gas / Påträde / Träktäktighet / Anslutnings- / Längd / Stråkning-

5 SHAW OK 76,08 ESAB 4 167 24,7 DC+
6 11 167 24,7 DC+
7 11 167 25 DC+
8 11 167 DC+
9 11 167 DC+
10 11 262 0,76
11 167 239 0,99

Värmebehandling efter svetsning &/el. äldring / Post weld heat treatment &/or ageing:

(Tid, temperatur, metod, uppvärmnings- och svalningshastigheter)

Tillverkare / Manufacturer:
Namn / Name: Christian Dahlberg

Signatur / Signature: 2014-04-30
Datum / Date: 2014-04-30

Granskare (om annan än tillverk.) / Examiner (if other than manufact.)
Namn / Name: Jovias Ohlsson

Signatur / Signature: 2014-04-30
Datum / Date: 2014-04-30

* om så erfordras eller är tillämpligt / if required or applicable
Protokoll för kvalificering av svetsprocedur (WPQR)

Welding Procedure Qualification Record

Enligt / According to: SS-EN ISO 15614-1

- **Svetsare / Welder:** Christian Dahlberg
- **Datum för svetsningen / Date of welding:** 2014-04-30
- **Plats / Location:** Bas 4
- **Produkt- / provningsstandard / Prod./test. stanl:**

### Svetsläge / Welding position:
- PA, Flat (1G)

### Förbands-/svets typ / Joint & weld type:
- BW, slumslevets

### Method for preparation & cleaning:
- s-mätt el. a-mätt / Throat thickness (mm):

### Förhöjd arbetstemperatur / Pre-heat temperature (°C):
- 230°C

### Särskild värming eller torkning / Any special baking or drying:

### Fördämning & rengöring (metod):

### Enkelsträng Bågtyp / Mode of metal transfer:

### Häftsvetsning (metod) / Tack welding (method):

### Strängplanering / Welding sequences:

### Svetsmetod / Welding process:
- Spaw 0K76.92 ESAB 4 167 25 DC+

### Svetsyttemperatur / Post heat temperature (°C):
- 358 0.56
- 288 0.70
- 213 0.68
- 291 0.69
- 301 0.67
- 298 0.67
- 200 0.67

### Svetsbehandling efter svetsning &/el. åldring / Post weld heat treatment &/or ageing:
- (Tid, temperatur, metod, uppvärmnings- och svarningsfastigheter)

### Tillverkare / Manufacturer:
- **Namn / Name:** Christian Dahlberg
- **Signatur / Signature:** 2014-04-30

### Granskare (om annan än tillverk.) / Examiner (if other than manufacturer):
- **Namn / Name:** Jonas Ohlsson
- **Signatur / Signature:**
Protokoll för kvalificering av svetsprocedur (WPQR)

Svetsare / Welder: Christian Dahlberg
Datum för svetsningen / Date of welding: 2014-04-30
Plats / Location: Gas Y
Produkt-provningsstandard / Prod./test. stan: Ytterdiameter rör (mm) / Outside pipe diameter (mm): 350

Enl. Figure / Acc. te figure: F-6338
Grundmaterial beteckning / Parent material designation: A: B:
Materialtjocklek (mm) / Material thickness (mm): 25

Metr / method for preparation & cleaning: s-mått el. a-mått / Throat thickness (mm):
Forhåll arbetsressurs / Pre-heat temperature (°C): 230°C
Särskild värming eller torkning / Any special baking or drying: Melansträngstemperatur / Interpass temperature (°C): 220°C, 238°C

Rutmejsning/rotstöd, detaljer / Details of back gouging/backing:

Enkelsträng Bärgtyp (droppovergång) / Mode of metal transfer: Löt. nr. 351109
Hålsvektsättning (metod) / Tack welding (method):
Fogutförning / Joint design:

Strangplanering / Welding sequences:

Strang / Run / Svets- metode / welding process: 19 - 25
Svetsmetod / Welding method: SAW
Elektrodmaterial / Electrode material: OK 76.98 ESAB
Ströms / Current (A): 4
Spänning / Voltage (V): 167
Strömstyp / Polarity: DC+

Bärgtyp / Transfer mode:
Gas el. Pulver beteckning toppnot Gas or Flux
Gas-topnot (l/min) toppnot topnot
Gas flow
Trådmarknings- hastighet (mm/min) Wire feed
Utanöver- längd (mm) Stick out
Strömmang / Heat input:

Strånlängd (mm) el. Mätresultat inhalter:

Våteutdrivning / Post-heating:

Varmhållningstemperatur / Pre-heat maintenance temperature (°C):

Tillverkare / Manufacturer:
Namn / Name: Christian Dahlberg
Signatur / Signature: 2014-04-30
Datum / Date: 2014-04-30

Varmebehandling efter svetsning &/el. äldring / Post weld heat treatment &/or ageing:

Granskare (om annan än tillverkare) / Examiner (if other than manufacturer):
Namn / Name: Jonas Ohlsson
Signatur / Signature: 2014-04-30
Datum / Date: 2014-04-30
Protokoll för kvalificering av svetsprocedur (WPQR)

Welding Procedure Qualification Record

Enligt / According to: SS-EN ISO 15614-1

Svetsare / Welder: Mikael Gustafsson
Datum för svetsningen / Date of welding: 2014-04-30

Plats / Location: Bås 4

Produkt- / Provningsstandard / Prod./test stan: 350

Eln. Figure / Acc. to figure: A:
Grundmaterial beteckning / Parent material designation: F-6338
Materialjocklek (mm) / Material thickness (mm): 25
Ytterdiameter rör (mm) / Outside pipe diameter (mm): 350

Forbands- & svetsstyp / Joint & weld type: BW, Stunsves
Metod för fogberedning & rengöring / Method of preparation & cleaning: s-mätt ej a-mätt / Throat thickness (mm):
Förhöjd arbetsstemperatur / Pre-heat temperature (°C): 225°C
Sarskild värmning eller torkning / Any special baking or drying: Rotmejsling/roststöd, detaljer / Details of back gouging/backing:
Fogberedning & rengöring (metod):
Enkelstrång Bågtyp (droppovergång) / Mode of metal transfer *:
Häftsvesning (metod) / Tack welding (method):
Fogutformning / Joint design:

60°

Fogplanering / Welding sequences:

Svetsläge / Welding position: PA, Flat (IG)

Förrådsmässig temperature / Interpass temperature (°C):
221°C, 207°C, 227°C

Annan information / Other information *:
Lot. nr. 351109

Längd (mm) el. Heat (min.)
Stråk- energi (kJ/mm)
Length or speed
Heat input

Strål / Run
26
27
28
29
30

Svets- metod / welding process
Svets- metod / welding process
Svets- metod / welding process
Svets- metod / welding process
Svets- metod / welding process

Elektrodmaterial / Electrode material
Elektrodmaterial / Electrode material
Elektrodmaterial / Electrode material
Elektrodmaterial / Electrode material
Elektrodmaterial / Electrode material

Beteckning / Designation
Beteckning / Designation
Beteckning / Designation
Beteckning / Designation
Beteckning / Designation

Material / Make
Material / Make
Material / Make
Material / Make
Material / Make

Diam (mm)
Diam (mm)
Diam (mm)
Diam (mm)
Diam (mm)

Spanning / Voltage (V)
Spanning / Voltage (V)
Spanning / Voltage (V)
Spanning / Voltage (V)
Spanning / Voltage (V)

Ström / Current (A)
Ström / Current (A)
Ström / Current (A)
Ström / Current (A)
Ström / Current (A)

Störnings- typ / Potency
Störnings- typ / Potency
Störnings- typ / Potency
Störnings- typ / Potency
Störnings- typ / Potency

Bågtyp / Transfer- mode
Bågtyp / Transfer- mode
Bågtyp / Transfer- mode
Bågtyp / Transfer- mode
Bågtyp / Transfer- mode

Gas eller Pulver
Gas eller Pulver
Gas eller Pulver
Gas eller Pulver
Gas eller Pulver

beteckning
beteckning
beteckning
beteckning
beteckning

toppilot

toppilot

toppilot

toppilot

toppilot

Gasflow
Gasflow
Gasflow
Gasflow
Gasflow

Träkärlningst- hastighet
Träkärlningst- hastighet
Träkärlningst- hastighet
Träkärlningst- hastighet
Träkärlningst- hastighet

Wire feed
Wire feed
Wire feed
Wire feed
Wire feed

Stick ut
Stick ut
Stick ut
Stick ut
Stick ut

Stråk- energi* (kJ/mm)
Stråk- energi* (kJ/mm)
Stråk- energi* (kJ/mm)
Stråk- energi* (kJ/mm)
Stråk- energi* (kJ/mm)

257
248
154
258
347

0,76
0,79
1,07
0,76
0,57

Väteudrivning / Post-heating:
Värmebehandling efter svetsning &/el. åldring / Post weld heat treatment &/or aging:

Varmhällningstemperatur / Pre-heat maintenance temperature (°C):
(Tid, temperatur, metod, uppvärmnings- och svetsningshastigheterna)

Tillverkare / Manufacturer:
Namn / Name: Mikael Gustafsson

Gramskare (om annan an tillverk.) / Examiner (if other than manufacture.)
Namn / Name: Jonas Ohlsson

Datum / Date: 2014-04-30

ANM 2 Notera att ändring kan göras även av del av ISO 15614

* om så erforderas eller är tillämplig / if required or applicable

Mail WPQR, www.svets.se/toolbox - Svetskommissionen ©
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<td>Joms Ohlsson</td>
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Protokoll för kvalificering av svetsprocedur (WPQR)

Welding Procedure Qualification Record

Enligt / According to: SS-EN ISO 15614-1

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<th>Datum för svetsningen / Date of welding: 2014-05-05</th>
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<td>Förbands- &amp; svetsyp: BW, stuvsvets</td>
<td>Svetsläge / Welding position: PA, Flat (1G)</td>
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<tr>
<td>Metod för fogberedning &amp; rengöring / Method of preparation &amp; cleaning:</td>
<td></td>
<td>s-mätt o. a-mätt / Throat thickness (mm):</td>
</tr>
<tr>
<td>Förhöjd arbetstemperatur i pre-heat temperature (°C):</td>
<td>Melansträngstemperatur / Interpass temperature (°C):</td>
<td></td>
</tr>
<tr>
<td>Särskild värmdning eller torkning / Any special baking or drying:</td>
<td>Rotmejsling /rotdish, detaljer / Details of back gouging/backing:</td>
<td></td>
</tr>
<tr>
<td>Fogberedning &amp; rengörning (metod):</td>
<td>Annan information / Other information:</td>
<td></td>
</tr>
<tr>
<td>Enkelsträng Bågtyp (droppövergång) / Mode of metal transfer:</td>
<td>SB332421 2,5 mm</td>
<td></td>
</tr>
<tr>
<td>Häftsvetsning (metod) / Tack welding (method):</td>
<td>SB349092 3,2 mm</td>
<td></td>
</tr>
<tr>
<td>Fogutformning / Joint design</td>
<td>Strängplanering / Welding sequences</td>
<td></td>
</tr>
</tbody>
</table>

| Väteutdrivning / Post-heating: 300°C, 1,5 h, långsamt svalning → 80°C | Värmebehandling efter svetsning &/el. äldring / Post weld heat treatment &/or ageing: |
| Varmhallningsstemperatur i Pre-heat maintenance temperature (°C): | (Tid, temperatur, metod, uppvärmnings- och svalningshastigheter) |

| Tillverkare / Manufacturer: CCI Valve | Granskare (om annan än tillverkare) / Examiner (if other than manufact.) |
| Namn / Name: Jan Setterlind | Namn / Name: Jonas Olsson |
| Signatur / Signature: | Datum / Date: 2014-05-05 |
| Datum / Date: 2014-05-05 | Signatur /Signature: |

* om så erfordras eller är tillämpligt / if required or applicable

Mall WPQR, www.svets.se/toolbox - Svetskommissionen ©
8.4 Post weld heat treatments for pipes
InteliHeat

Date / Time
2014-05-14 10:56:57

Comment

Signature

Settings
Ramprate 40 °C/h  Dwell-temp 740 °C  Dwell-time 2 h

Chart

Nr: 13825 svetsprov 1
InteliHeat

Date / Time
2014-05-16 16:02:43

Comment

Signature

Settings
- Ramp rate: 40 °C/h
- Dwell-temperature: 760 °C
- Dwell-time: 2 h

Chart

Nr: 13831 svetsprov 3

- Target
- Channel 1
- Channel 2
- Channel 3
- Channel 4
- Channel 5
- Channel 6