Solution annealing of thick walled large-diameter super duplex stainless steel pipes by induction heat treatment

The results of the simulated solution annealing, of a full scale heat treatment trial as well as the experience and test results of the subsequent production of 26 large diameter thick walled SDSS pipes are discussed in this paper.

Keywords: Solution annealing, super duplex, large-diameter SAW pipes, induction heat treatment, UNS S32750

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Abstract

Longitudinally submerged arc welded large diameter SDSS pipes require solution annealing to dissolve inter-metallic phases and to generate the desired austenite/ferrite ratio within the area of weld and HAZ. According to ASTM A 928 solution annealing shall be performed after welding within the temperature range of 1025 °C to 1125 °C followed by quenching using water or air.

It is also a well known fact that lower solution annealing temperatures cannot ensure a proper dissolving of inter-metallic phases. In contrast, rapid cooling from higher temperatures can lead to nitride precipitations.

Within the framework of an order, 26 SDSS (UNS S32750) 18” pipes with a wall thickness of 36.2 mm and lengths of 9,000 mm were produced. For this order the solution annealing was conducted by means of a continuous induction heat treatment facility. This process is characterized by a more rapid heating and a shorter holding time compared to conventional furnace heating and by the fact that the pipes are quenched only from the outside surface. Due to the characteristics of the induction solution annealing process of thick walled SDSS pipes, the metallurgical challenges are found in the limited holding time, the temperature gradient between outer and inner surface and in the different cooling speeds over the wall thickness.

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The actual heat cycle of the pipes was determined by a full-scale heat treatment trial using thermo couples and an infra-red camera. After the induction solution annealing the material was subjected to metallographic examinations including SEM and corrosion testing to qualify this heat treatment process for production. In addition, samples subjected to a simulated heat treatment were made in a lab furnace to support understanding of the kinetics of the induction solution annealing process.

1. Introduction

1.1. Pipe Production and Induction Heat Treatment

Following the JCO production route 26 SDSS pipes (UNS S32750) with dimensions OD 457 mm x WT 36.2 mm are formed by press bending and submerged arc welded, using similar filler material EN 25-9-4-N-L (AWS ER25594). The chemical composition for the base material and the weld deposit is given in Table 1.

Table 1: Chemical composition.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>weld deposit</td>
<td>0.01</td>
<td>0.7</td>
<td>0.3</td>
<td>0.02</td>
<td>&lt;0.01</td>
<td>26.8</td>
<td>3.5</td>
<td>0.1</td>
<td></td>
</tr>
<tr>
<td>base metal</td>
<td>0.02</td>
<td>0.5</td>
<td>0.8</td>
<td>0.03</td>
<td>&lt;0.01</td>
<td>26.6</td>
<td>3.4</td>
<td>0.2</td>
<td></td>
</tr>
</tbody>
</table>

The heat input has been kept relatively low at 1.5-1.7 kJ/mm, not only aiming to minimize undesirable phases but rather avoiding hot cracks and pores. A 1.6 MW induction heat treatment facility was chosen for the subsequent solution annealing. Because this process is the key process of the SDSS pipe production, the induction heat treatment is described in more detail.

The induction heating process is characterized by using a source of high frequency electricity to drive an alternating current through an induction coil. The passage of current through this induction coil generates a very intense and rapidly changing magnetic field in the space within the work coil. The pipe to be heated is fed in rotation by trapezoid rollers through the induction coil and its intense alternating magnetic field, which induces a high amperage current flow in the workpiece. The arrangement of the induction coil and the pipe can be thought of as an electrical transformer, in which the induction coil or work coil is like the primary where electrical energy is fed in and the pipe is like a single turn secondary that is short-circuited. Through this an eddy current is generated in a thin layer towards the surface of the pipe (skin-effect). The skin-effect increases the effective resistance of the metal to the passage of the large current and therefore it increases the induction heating effect caused by the current induced in the pipe. In turn the rotation of the pipe ensures a homogeneous temperature distribution in circumferential direction.

The utilized induction heat treatment facility at EEW is equipped with an IR camera which enables controlling the outer surface temperature which delivers the signal for the PID control element. Close to the coil exit a double loop water main is installed equipped with flat-stream nozzles spraying a high speed water jet ensuring high cooling rates by avoiding the Leidenfrost effect. Fig. 1 shows the induction coil exit in the view of the Infra-red camera.

1.2. Critical parameters of heat control

It is well known that the huge amount of alloying elements of duplex steels is linked to a complex transformation and precipitation behaviour. Therefore the history of heating and cooling is substantial on the mechanical and corrosive properties defining the applicability of this material. As a rule of thumb it can be stated that the precipitation of one Volume-percent α-phase reduces the toughness to a third of the original value. In addition, the diffusion of chromium and molybdenum into the α-phase decreases the corrosion resistance predominantly of the ferrite-matrix. It becomes obvious that finally all heat treatment parameters – minimum and maximum temperature, holding time and cooling speed – can be regarded as “critical” in attaining a proper solution annealing.

It is scientifically acknowledged that the formation of deleterious phases (sigma, chi and secondary austenite) in 2507 or S32750 takes place in the temperature range of 800-1000 °C [2,3,4,10,12]. Calliari et al. did not find precipitations aging at 800 °C. At 1000 °C he observed 0.5% chi and sigma at grain boundaries and inside the ferrite grains, after 5 minutes and after additional 10' he found the chi content slowly decreases while sigma amount was raised up to 4% [3]. Bonollo et al. found almost no α-phase precipitation below 780 °C and over 980 °C for holding times below 1 h [4]. Villalobos et al. could not observe any changes in the micro structure for ageing at 700 °C for 25 minutes but a formation of 8% α-phase after 30' at 800 °C [8]. The maximum velocity for sigma formation in S32750 is found to be in the temperature range of 850-900 °C [1,7,10]. At 850 °C the incubation time of α-phase precipitation is less than 1 minute so that about 5% α-phase precipitates at 800-900 °C after 5-6 minutes [10]. After 10-15' about 10% deleterious phases can be observed and almost 30% after 1 h exposure at 850-900 °C [7].

ASTM A 928 gives a minimum solution annealing temperature of 1025 °C but it is recommended that the 2507 SDSS shall be annealed at T > 1050 °C for a sufficient resolution of α-phase [2,3,12]. As well as for the holding time only few data are available for the critical cooling speed. For 2205 Duplex a holding time of 300 s at 1035 °C and 280 at 1050 °C with subsequent cooling by 0.3 K/s is regarded to be safe restoring the austenite/ferrite microstructure from initial 15% α-phase and to avoid undesired precipitations [12,14]. Critical cooling rates of 2.5 K/s [11] and 4 K/s [5] are elaborated for S329 by Pellizzari and Bennani et al. Calliari and Bonollo et al. state a critical cooling rate of 0.8-1.0 K/s for 2507 super duplex [3,4].

On the other hand, it has been shown that chromium nitrites precipitate in the ferritic phase when nitrogen alloyed duplex stainless steel is quenched with very high cooling rates from very high annealing temperatures [6,13]. The formation of CrN occurs when the solubility of nitrogen is exceeded and when the diffusion of the excessive nitrogen from the ferrite into the austenite is suppressed.

Summarizing the aforesaid, the following requirements can be specified for the solution annealing of UNS S32750:

1. Between 800 and 950 °C highest heating-up rates possible (tβ10 > 1 K/s) for suppressing additional α-phase formation during heating.
2. Minimum holding temperature of 1050 °C for 5 minutes holding time.
3. Maximum cooling speed from 1200-1000 °C of tα1<10 < 20 K/s.
4. Minimum cooling speed from 1000-500 °C of tα1<5 > 1 K/s.
2. Experimental Procedures

2.1. Preliminary heat treatment trials

The aim of the preliminary large scale heat treatment trials was to understand the heating sequence of a volume element, travelling through the induction coil and quenching unit and to find out, if and how the above described heat treatment requirements can be achieved. For this purpose one pipe has been equipped with thermo couples fixed at the inner pipe surface at a distance of 450 mm to the pipe end. This way, in addition to the outside temperature the temperature and cooling speed of the inner surface of the pipe wall was recorded. Provided sufficient electrical power and cooling capacity exist the pipe travel speed (\( V_p \)) through the heat treatment unit is the determining parameter due to the relatively low thermal conductivity, which is about 1/3 of carbon steels. Therefore, different feeding speeds \( V_p \)'s were tested. Fig. 2 show the graphs of the inside and outside heat cycles measured at the metering points. Fig. 2a exemplifies the temperature sequence for \( V_p = 2.5 \) mm/s, Fig. 2b for 1.8 mm/s.

Heating and cooling rates are little influenced by pipe travel speed but it determines the total heating time and the time of temperature equalization over wall thickness before quenching significantly. Accordingly the temperature gradient between inside and outside pipe surface for \( V_p = 2.5 \) mm/s was about 90 K and 50 K for \( V_p = 1.8 \) mm/s. Therefore the minimum required annealing temperature of 1050 °C was not reached at the inner pipe surface for trial A. In contrast, the slower travel speed of 1.8 mm/s exceeded 1050 °C over a period of 120 s with a maximum temperature of 1072 °C at the internal pipe surface and 1120 °C at the outer surface. Hence the heating and cooling speeds for both trials were within the desired range. The pipe feeding speed \( V_p = 1.8 \) mm/s was chosen for production.

The resulting heat treatment conditions for trial A and B are given in Table 2.

<table>
<thead>
<tr>
<th>Location</th>
<th>Interval</th>
<th>( \Delta T/\Delta t )</th>
<th>Trial A: ( V_p = 2.5 ) mm/s</th>
<th>Trial B: ( V_p = 1.8 ) mm/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outside</td>
<td>300-1090 °C</td>
<td>4.2 K/s</td>
<td>300-1120 °C</td>
<td>3.4 K/s</td>
</tr>
<tr>
<td></td>
<td>1090-950 °C</td>
<td>1.3 K/s</td>
<td>1120-950 °C</td>
<td>1.3 K/s</td>
</tr>
<tr>
<td></td>
<td>950-500 °C</td>
<td>20 K/s</td>
<td>950-500 °C</td>
<td>20 K/s</td>
</tr>
<tr>
<td>Inside</td>
<td>300-950 °C</td>
<td>3.2 K/s</td>
<td>300-1000 °C</td>
<td>2.2 K/s</td>
</tr>
<tr>
<td></td>
<td>950-1000 °C</td>
<td>1.0 K/s</td>
<td>1000-1070 °C</td>
<td>0.9 K/s</td>
</tr>
<tr>
<td></td>
<td>1000-950 °C</td>
<td>0.4 K/s</td>
<td>1070-950 °C</td>
<td>0.6 K/s</td>
</tr>
<tr>
<td></td>
<td>950-500 °C</td>
<td>4.2 K/s</td>
<td>950-500 °C</td>
<td>4.0 K/s</td>
</tr>
</tbody>
</table>

2.2. Laboratory trials and testing

2.2.1. Simulation of heat treatment conditions

In addition to the large scale heat treatment trials, the induction heating cycle was simulated in a laboratory furnace at the University Bochum. For this purpose, three samples of the welded joint (in the as-welded condition) was put into a pre-heated lab furnace at 1050 °C, 1100 °C and 1150 °C. The total annealing time including through-heating of the sample was 8 minutes. According to former trials the heating-up time of the samples was estimated to be 6-7 minutes so that the remaining holding time of 1-2 minutes was not longer than the corresponding one of the induction solution annealing during pipe production. After the dwell period, the samples were quenched in a water basin. The actual temperature of the samples during annealing was not controlled.

2.2.2. Metallographic examination

Four samples from a pipe in the as welded condition were cut. Three of them were simulated solution annealed and another sample from the pipe after induction solution annealing was cut for metallographic examination. After polishing the specimens were etched using V2A-pickel or beraha-2 solution for light and scanning electron microscopy. The ferrite content was determined according to the magnetic induction method. Work samples taken during pipe production were electrolytically etched and the ferrite content was determined according to ASTM E562.

2.2.3. Mechanical tests

For mechanical characterisation, tensile tests of the base metal 180° to the weld seam transverse to pipe axis and weld metal transverse to pipe axis as well as charpy V-notch impact tests at the positions base metal 90° to weld seam, 2 mm subsurface outside, mid thickness and 2 mm subsurface inside transverse to pipe axis and centre weld metal 2 mm subsurface outside, mid thickness and 2 mm subsurface inside at – 40°C were carried out. In addition, hardness tests HV10 were conducted on a weld joint macro-section.

2.2.4. Corrosion test

The ASTM G48 corrosion tests using ferric chloride solutions give a good correlation with the performance of CRA to pitting and crevice corrosion resistance in chloride-containing environments, such as natural seawater at ambient temperature. In turn, the corrosion behaviour is directly linked to the heating and cooling history of the material. Accommodating the different heating and cooling conditions over the wall thickness of the pipes during the induction solution annealing, a modified pitting corrosion test according to ASTM G48 method A was chosen. The aim was to prove the homogeneity of the corrosion resistance of the welded joint and the base metal over the wall thickness and pipe length. Therefore, samples from each pipe end were cut and the specimens were taken longitudinal and transverse and perpendicular to the pipe axis as well as at the outer and inner pipe surfaces.

3. Results

3.1. Metallographic findings

The ferrite content for each heat treatment condition is given in Table 3.

3.1.1. Initial microstructure before solution annealing

Light-microscopic pictures of the microstructure in the as welded condition are given in Fig. 3 and SEM micrographs in Fig. 4. No
peculiar findings are made in the weld metal. In contrast, the HAZ shows clearly marked-off grain boundaries in the ferrite and at some locations intermetallic phases can be seen (Fig. 3c). The SEM investigation confirms and completes the light-microscopic findings. No intermetallic phases could be found in the weld metal. The ferrite in the outer HAZ and base material shows some very fine facings on the inner grain boundaries composed of very small single particles (Fig. 4a and b). Due to amount and distribution an EDX-Analysis could not be made, but it is assumed that these precipitations are about $\chi$- or $\sigma$-phase.

After 8 minutes dwell period in the lab furnace at 1150 °C $\sigma$-phase is clearly detectable in the weld metal (Fig. 11a). Also the eutectoid phase was found (Fig. 11b). The fine precipitations within the HAZ have been coarsened (Fig. 12). Nitrides are clearly visible by SEM in the HAZ as well as in the base material (Fig. 13).

### Table 3: Ferrite content.

<table>
<thead>
<tr>
<th>method</th>
<th>magnetic induction</th>
<th>ASTM E562</th>
</tr>
</thead>
<tbody>
<tr>
<td>condition</td>
<td>as welded</td>
<td>solution annealed by lab furnace</td>
</tr>
<tr>
<td>base metal</td>
<td>53</td>
<td>51</td>
</tr>
<tr>
<td>weld deposit</td>
<td>44</td>
<td>43</td>
</tr>
<tr>
<td>HAZ</td>
<td>nd</td>
<td>nd</td>
</tr>
</tbody>
</table>

3.1.2. Microstructure after heat treatment

3.1.2.1. Simulated solution annealing

The annealing of 8 min. at 1050 °C furnace temperature did not deliver visible changes of the micro structure in comparison to the as-welded condition. The weld metal shows still no precipitations and the intra-ferrite precipitation in the HAZ and base metal have not been resolved but it seems that they have become a bit finer (Fig. 5 and 6). At 1100 °C (8 min.) annealing no precipitations were found in the weld metal by light microscopic investigation. The intra-ferrite grain boundary precipitations within HAZ and base metal are still not resolved (Fig. 7) but in addition, little etching pits are visible on the ferrite (Fig. 7 and 8b), which can be an indication for nitrides. In contrast the SEM pictures (Fig. 8) show fine precipitations at the intra-ferrite grain boundaries. The SEM investigations of the weld metal showed that a eutectoid phase had been formed (Fig. 9). An EDX analysis of the phase and the comparison with the surrounded matrix (Fig. 10) revealed an increased chromium and molybdenum content of this phase. Morphology and chemical composition indicated a $\sigma$-phase.
3.2. Mechanical test results

3.2.1 Tensile tests

Twenty tensile tests on 8 pipes from 3 heats after induction solution annealing were carried out. Figs. 17a to 17c show the mean values with the error-bars giving the minimum and maximum values. The ◦-sign indicates the values from a comparison sample which has been annealed at 1100 °C / 40 min. in a lab furnace.

All test results are well above the specified minimum values with a scatter range of about 10%.
3.2.3 Hardness testing

Six weld sections from six pipes were hardness tested after solution annealing by induction. Fig. 19 shows HV10 of base material, heat affected zone and weld metal 2 mm subsurface pipe outside and inside. Almost no hardness deviations between the different locations within the welded joint could be found and no difference between pipe outside and inside could be detected.

3.3. Pitting corrosion test

The results of the modified ASTM G48-A pitting corrosion test are summarized in Table 4. The exposure time was 24 h at the test temperature of 40 °C.

Table 4: Modified ASTM G48 method A pitting test corrosion results.

<table>
<thead>
<tr>
<th>pipe end</th>
<th>location</th>
<th>orientation</th>
<th>specimen size [mm]</th>
<th>surface [mm²]</th>
<th>weight 1 [g]</th>
<th>weight 2 [g]</th>
<th>loss [mg]</th>
<th>corro. [g/m²]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>weld metal</td>
<td>thru. thick. longitudinal</td>
<td>5.03 36.26 50.01</td>
<td>4494.60</td>
<td>70.7896</td>
<td>70.7887</td>
<td>0.9</td>
<td>0.2002</td>
</tr>
<tr>
<td>2</td>
<td>weld metal</td>
<td>thru. thick. longitudinal</td>
<td>5.06 37.88 50.01</td>
<td>4678.20</td>
<td>73.9474</td>
<td>73.9460</td>
<td>1.4</td>
<td>0.2993</td>
</tr>
<tr>
<td>1</td>
<td>weld joint</td>
<td>thru. thick. transverse</td>
<td>4.91 36.14 50.02</td>
<td>4461.54</td>
<td>68.7116</td>
<td>68.7104</td>
<td>1.2</td>
<td>0.2690</td>
</tr>
<tr>
<td>2</td>
<td>weld joint</td>
<td>thru. thick. transverse</td>
<td>5.05 38.19 49.98</td>
<td>4709.77</td>
<td>73.3366</td>
<td>73.3354</td>
<td>1.2</td>
<td>0.2549</td>
</tr>
<tr>
<td>1</td>
<td>weld joint</td>
<td>outside surface</td>
<td>5.06 25.03 50.02</td>
<td>3263.51</td>
<td>49.1853</td>
<td>49.1843</td>
<td>1.0</td>
<td>0.3064</td>
</tr>
<tr>
<td>2</td>
<td>weld joint</td>
<td>outside surface</td>
<td>5.02 25.01 50.05</td>
<td>3257.10</td>
<td>48.6229</td>
<td>48.6217</td>
<td>1.2</td>
<td>0.3684</td>
</tr>
<tr>
<td>1</td>
<td>weld joint</td>
<td>inside surface</td>
<td>5.04 24.98 50.05</td>
<td>3256.80</td>
<td>48.7554</td>
<td>48.7543</td>
<td>1.1</td>
<td>0.3378</td>
</tr>
<tr>
<td>2</td>
<td>weld joint</td>
<td>inside surface</td>
<td>5.04 25.03 50.05</td>
<td>3262.31</td>
<td>48.8844</td>
<td>48.8831</td>
<td>1.3</td>
<td>0.3985</td>
</tr>
<tr>
<td>1</td>
<td>base metal</td>
<td>thru. thick. longitudinal</td>
<td>5.05 37.95 50.09</td>
<td>4691.04</td>
<td>74.2176</td>
<td>74.2162</td>
<td>1.4</td>
<td>0.2984</td>
</tr>
<tr>
<td>2</td>
<td>base metal</td>
<td>thru. thick. longitudinal</td>
<td>5.05 37.55 50.09</td>
<td>4646.92</td>
<td>73.5885</td>
<td>73.5871</td>
<td>1.4</td>
<td>0.3013</td>
</tr>
<tr>
<td>1</td>
<td>base metal</td>
<td>thru. thick. transverse</td>
<td>5.00 25.01 50.04</td>
<td>3253.50</td>
<td>48.5126</td>
<td>48.5117</td>
<td>0.9</td>
<td>0.2766</td>
</tr>
<tr>
<td>2</td>
<td>base metal</td>
<td>thru. thick. transverse</td>
<td>5.03 24.98 50.02</td>
<td>3253.50</td>
<td>48.7625</td>
<td>48.7614</td>
<td>1.1</td>
<td>0.3381</td>
</tr>
</tbody>
</table>

The corrosion per square meter was determined at 0.2-0.4 g/m². Only few insular micro-pits of a size of less than 20 μm were found. No significant difference in the corrosion performance between the different specimen locations could be observed.

4. Discussion

The metallographic examination by light microscopy and SEM on samples in the as welded condition could not detect any intermetallic phases within the weld metal (Fig. 3a). In contrast, the HAZ and the base material showed etched ferrite grain boundaries with very fine precipitations which are assumed to be χ- or σ-phases (Fig. 3c and 4).

Laboratory heat treatment trials were carried out with the aim to simulate the heat cycle of the solution annealing by induction. The samples were put in a preheated lab furnace at 1050 ºC, 1100 ºC and 1150 ºC for 8 minutes followed by quenching in a water basin.

The metallographic investigations on the 1050 ºC annealed sample (Fig. 5-6) showed almost no microstructural changes in comparison to the as welded condition (Fig. 3-4). The small precipitations within the HAZ and the base metal have not redissolved but have become – subjectively perceived – finer.

Also the sample annealed at 1100 ºC still shows some fine precipitations at the ferrite grain boundaries (Fig. 8). In addition etching pits (Fig. 7) are found at the ferrite grains which indicate the precipitation of nitrides due to too rapid cooling from the holding temperature. Within the weld metal the SEM investigation reveals a coraline shaped eutectic phase (Fig. 9), which is most likely σ-phase identified by increased Chromium and Molybdenum content (Fig. 10).

The eutectoid phase and coraline shaped σ-phase within the weld deposit were found to be bigger in the sample which was simulated annealed at 1150 ºC and are visible even by light microscopy (Fig. 11). The initial fine precipitations at the HAZ and base material ferrite grain boundaries have become coarsened and block shaped. By SEM, nitrides are clearly visible within the HAZ and base metal (Fig. 13), which is an indication that the target temperature was reached and that the cooling rate down to approximately 1050 ºC was high enough to suppress the diffusion of nitrogen.

The weld metal of the samples taken from the induction solution annealed pipe was free of any precipitations (Fig. 14). Only slightly implied ferrite grain boundaries are visible within HAZ and base metal (Fig. 15a and 16a). The SEM investigations show that also stronger etched ferrite grain boundaries of the base metal are free of precipitations and that only within the HAZ some extremely small screenings are found (Fig. 15b). Therefore the effectiveness of the resolution of the intermetallic phases by induction annealing could be shown in a comparison to the as welded condition (Fig. 3c and 4a).

The difference in the metallographic findings between the lab furnace annealed samples and the sample which has been annealed by induction process is unexpected and needs further explanation. The approach is made by regarding the difference in heating and cooling.

According to previous temperature measurements using thermocouples fixed at the outside surface and in the centre of the sample, the heating rate between 800 ºC and 1000 ºC of the samples put into the preheated lab furnace was not more than T 9/10 = 0.4-0.8 K/s. The holding time at the target temperature is assumed to be not more than 60-90 s. Due to the Leidenfrost effect, the isolating vapour layer between water and sample surface, the cooling rate T 180/12 after the sample’s surface was probably less than 10 K/s and less than 0.3 K/s in the centre.
whereby the higher the quenching temperature the lower the cooling rate. With respect to the metallographic findings following derivatives can be made:

1. The residence time into the \( \sigma \)-phase existence area during heating with below 1 K/s is sufficient for \( \sigma \)-phase nucleation.
2. A holding time of 90 s is not sufficient for a complete resolution of the \( \sigma \)-phase nuclei precipitated during heating-up.
3. A cooling rate \( T_{\text{cool}} \) of the samples quenched in a water basin is not sufficient to suppress \( \sigma \)-phase growth.

In contrast the induction solution annealing parameters which have been elaborated by preliminary trials (Fig. 2, Table 2) are characterized by a more rapid heating exceeding 3 K/s, a holding time of about 120 s over 1050 °C with maximum temperatures between 1070 °C inside and 1120 °C outside, a relatively slow cooling rate down to 950 °C of 1 K/s and a rapid cooling below 950 °C of 4 K/s inside to 20 K/s at the pipes outside surface. With respect to the metallographic findings following derivatives can be made:

1. A heating rate of 3 K/s is sufficient to suppress \( \sigma \)-phase nucleation.
2. A holding time of 120 s is sufficient for the resolution of smaller amounts of \( \sigma \)-phase.
3. The cooling rate attained by a high speed water jet exceeding 4 K/s is sufficient to prevent any \( \sigma \)-phase precipitation.

Supporting the metallographic findings, the results from the mechanical testing are well within the specified range. The comparison with a control sample which has been conservatively solution annealed at 1100 °C for 40 minutes shows no significant deviations. The hardness values determined close to the outside and inside surface support the metallographic homogeneity over the wall thickness.

The pitting corrosion tests which have been executed at different position over the pipe wall thickness, length and perimeter showed a very good and homogeneous corrosion resistance at 0.2-0.4 g/m², whereas 20 g/m² is regarded to be sufficient. As well as the mechanical tests the corrosion tests support the metallographic findings and document the feasibility and effectiveness of the solution annealing of large diameter super duplex stainless steel pipes by an induction process.

In consideration of the above, the metallurgical advantage of the induction annealing process in comparison to a conventional process by bogie hearth furnace becomes clear. The rapid heating to above 1000 °C by more than 3 K/s ensures a very fast pass through the precipitation area between 800 °C and 1000 °C by crossing it at no more than 90 s. It is obvious that much less deleterious phases would form in this short period than during heating up in a bogie hearth furnace featuring a heating rate of about 50 K/h in this temperature field. In turn, \( \sigma \)-phases which have not been precipitated before need not be redissolved. The moderate cooling at about 1 K/s from maximum annealing temperature down to 950 °C prevents the formation of nitrites and the high cooling rate below 950 °C of more than 4 K/s ensures the suppression of any undesirable precipitations.

5. Conclusion
In this study the effectiveness of the solution annealing of large diameter thick-wall superduplex pipes by an induction heating and quenching process was investigated. Samples subjected to different heating cycles by lab furnace and samples taken from the production process have been compared and metallurgically investigated.

It could be shown that two minutes holding time between 1050 °C and 1070 °C is sufficient to recover the microstructure of submerged arc welded pipes. For this the absolute precondition is the rapid passing through the precipitation area between 800 °C and 950 °C during heating and cooling by not less than 2 K/s.

The metallurgical investigations as well as the mechanical and corrosion testing in this study prove that the induction solution annealing process with subsequent quenching by a high speed water jet is a suitable and efficient method for annealing large diameter thick-wall superduplex pipes.

6. References
[10] WANG XF, CHEN WQ, ZHENG HG: Influence of isothermal aging on sigma precipitation in superduplex stainless steel
[12] CHARLES J: Duplex Stainless Steels, a review after DSS ’07 held in Grado.

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