Specifying and Securing Quality in Procurement, Manufacture and Fabrication of Duplex and Super Duplex Stainless Steel Parts for SWRO Applications.

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Abstract: The paper discusses the most recent developments in material specification, vendor and fabricator selection and qualification, manufacturing and fabrication procedures and processes, to optimise quality and performance of duplex and super duplex stainless steel in sea water service. This is as currently applied by other large volume industrial users of these products. Good practices that could also be applied by desalination contractors are identified and discussed.

1. Introduction.

Duplex and Super Duplex stainless steels find application in SWRO systems because of their combination of strength, corrosion resistance and relative ease of fabrication, ready availability and cost competitiveness. Super duplex stainless steels are particularly useful for seawater intake; high pressure feed and brine reject pipework and associated pumps and valves and energy recovery systems. Whereas, 22%Cr duplex stainless steels are not so resistant to corrosion in seawater and brine concentrates as 25% Cr super duplex grades, they do find application in second pass pipe work systems or indeed brackish water applications. The service performance / cost ratio these grades provide is attractive to the industry. However, performance can be significantly adversely affected by lack of awareness and control during manufacture [1-3] and fabrication [4-6] of these steels. It is apparent that product specifications like ASTM standards and fabrication codes like ASME IX alone are not sufficiently rigorous to be able to detect and filter out defective material in the supply chain. Other large scale industrial users, like the oil and gas industry, have realised this and have developed supplier and QA/QC strategies and supplemental specifications to minimise the risk in procurement of these grades. This paper details some of these developments and discusses their suitability for adoption by desalination contractors when they want to purchase duplex or super duplex stainless steels for their projects.

2. The Problem

Duplex and super duplex stainless steels can, if not properly heat treated or fabricated, precipitate a phase called sigma phase. This phase is rich in Chromium and Molybdenum which means that the material adjacent to the precipitate is denuded in both, usually to the extent that the property of stainlessness is lost and the metal becomes susceptible to corrosion attack. Sigma phase is also very brittle and can cause a serious loss in toughness of the affected product. The phase is formed when the steel spends too long in the temperature range 500°C to 1000°C [7]. It forms most rapidly at about 850 to 900 °C (Figure 1). The time spent by the material during any thermal cycle that takes the material through this temperature range is cumulative. The material retains the metallurgical results of its thermal history in terms of consuming the incubation period required for precipitation sigma phase. After it precipitates further time spent in this temperature range allows the precipitate to grow. This sets the
extent and degree to which the part is affected in terms of increasing the percentage of sigma phase precipitated and the development of its morphology and connectivity within the steel [8, 9]. This is the reason why these steels are supplied in the solution treated and water quenched condition. The idea is that the fast cooling during water quenching is not only too fast to allow sigma to form, but that it is sufficiently fast to allow subsequent thermal cycles during welding to be applied without these causing sigma to precipitate to detrimental levels in the welded joint or its heat affected zone. Sigma phase can form due to either poor heat treatment practice or due to poor welding practice. If sigma phase is detected in an item due to poor heat treatment it calls into question the integrity of the entire heat treatment batch and indeed the integrity of all items manufactured using the same production method and heat treatment processes. The only method of removing the sigma phase is by re-heat treatment of the affected parts to dissolve the phase, homogenise the chemistry and again rapidly cool by water quenching. Of course in these circumstances the full suite of testing must be applied once more to prove the integrity of the re-heat treatment process. If weld heat input or inter-pass temperatures are too high, or if set up, or welding technique is poor [10], then this too can cause sigma phase to form. Figure 2 shows the influence of weld heat input on corrosion resistance of welds. It is interesting to see how welding gasses can suppress sigma formation and enhance corrosion resistance [11], the use of Argon/Nitrogen shielding and Formier backing gasses has been found to be very effective in enhancing the corrosion resistance of weldments. If heat inputs are excessive, sigma phase is formed and the joint is susceptible to corrosion. In the case of fabricated spools re-heat treatment is impossible because of distortion in the furnace so the only remedial action is to cut out and re-weld. Fortunately, we find that the sigma phase formed in the steep temperature gradients experienced in welded joints is rather different in morphology to that formed isothermally in a heat treatment furnace. Because of this low level sigma phase precipitation tends to be more tolerable in welded joints than in primary products. Figure 3 shows tolerable sigma levels in welds in sea water service [8].

3. The Solutions To The Problem

3.1 Manufacturing Route / Manufacturer Qualification

The Norwegian oil and gas industry has led the way in the development of a manufacturing route qualifications system for suppliers of special grades of steel. They have written a standard known as NORSOK M650 [12]. This details the minimum requirements of manufacturers for consistent and reproducible manufacture of quality products in duplex and super duplex stainless steels and other alloys. It requires documentation of product form and manufacturing route specific processes and procedures backed by witnessed sampling and testing of product made. M650 considers a range of product forms castings, forgings, seamless and welded pipes and fittings. It focuses on heat treatment, furnace weight limits and loading patterns to allow free flow of hot air all around the parts during heating and equally free flow of coolant during quenching. Consistency of temperatures attained within the process zone of the furnace and proper measurement of temperature of the parts using contact and thermal mass thermocouples on parts located in the core of the furnace process zone and at its periphery. Requirements for water quenching are also detailed, transfer times, agitation of the parts, circulation and cooling of the coolant during quenching and allowable temperature increases of the quenchant during the process are all detailed. The route is proven by testing. Product located at the core and periphery of the furnace process zone is sampled. Samples are taken from the ends and mid length of long products and in the heaviest sections of forgings and castings, i.e. from areas that are believed to experience the lowest cooling rates during quenching and are therefore considered to be most susceptible to sigma phase.
formations were it to occur. The testing regime applied is also detailed in the standard. This includes metallurgical examination to look for sigma phase, corrosion and toughness testing (because sigma reduces corrosion resistance and toughness). Once the manufacturer has achieved satisfactory results and fully documented his processes and procedures in the form of a qualification test record (QTR), he is then considered to be qualified to supply. However, he cannot supply parts that are larger that the maximum size he has produced in qualification and he cannot go outside the production parameters he used during qualification. Also, when he manufactures parts to be certified as being made in accordance with his M650 qualification he must use the qualified manufacturing route from raw material supply through to using the same furnaces and quench tanks that he qualified with. This essentially freezes the manufacturing route that is known to deliver a quality product. The purchaser must ask for goods to be supplied in accordance with NORSOK M650 in his order otherwise he may not get it. Requesting M 650 supply does not usually come with a price premium as it tends to be very widely adopted. Unfortunately, the NORSOK system tends to be Eurocentric. Mills that do not hold NORSOK M 650 QTR’s may be very capable. Generally in such cases these mills often have adopted the elements of the M650 in their production processes. NORSOK M 650 is freely available for download from the internet and can be used by the buyer as a basis on which to audit his supply chain and home in on quality supply.

3.2 Use of a Material Specification Including Testing Requirements

ASTM product standards are often called up in purchase orders. These define the product clearly but they do not require microstructure checks, corrosion tests or toughness tests that can detect the presence of deleterious sigma phase. Hence, it is necessary to augment the ASTM standard with a material specification also. Rolled Alloys has its own Material Data Sheet (MDS) [13], for every product form. Table 1 compares the requirements of our MDS vs. the applicable product ASTM standards. You will note the absence in the ASTM standards of critical mechanical, microstructural, corrosion and NDE testing that is really needed to confirm quality. Our inventories of stock material are made to these standards to ensure that off the shelf items already comply with various industry product and material specification requirements and are made to the desired quality level and appropriate level of NDE. Many customers call up our MDS’s when purchasing as it means that they do not have to write their own material or product specifications. NORSOK does this by asking for testing to NORSOK MDS 630 [14]. This set of data sheets define the required level of testing and acceptance levels, along with levels of NDE that are also augment ASTM standards. These MDS’s are again a sound template for desalination contractors to use as a basis for their own requirements. However, there are further issues to be considered. NORSOK M 630 MDS’s do not give guidance on sample preparation and interpretation of microstructures and they also require samples for corrosion testing to be acid pickled prior to exposure. This enhances the corrosion resistance of the sample (increases the measured CPT) and reduces the weight loss result [15]. A more comprehensive document to specify and use for corrosion testing of base material would be ASTM A 923 [16], as this gives comparative microstructures for those examining the samples to use as a guide. Unfortunately, this standard does not cover welds and also the ferric chloride corrosion test temperature originally defined in ASTM A 923 was only 40°C, but recently a supplemental condition for “critical applications” has been added to the standard. This revision is for ferric chloride corrosion testing at 50°C. This is a more discerning test temperature for detection of sigma phase. We would recommend corrosion testing at 50°C as seawater service is a critical application. We would also recommend that the mill surface finish condition is tested also if this will see service (i.e. don’t grind the test surfaces of the corrosion sample). In this case we find acceptable weight
loss criteria of 4g/m² without visible pitting at x 20 magnification is quite discerning. This is in contrast to the A923 1 mdd (1g/m²) weight loss criterion applicable to machined finishes. These tests pick up production problems as shown in Figure 4. There are also issues with respect to the level of impact toughness that is acceptable to both NORSOK M 630 MDS’s and in ASTM A 923, both are too low. The authors company recommends 70J average at minus 50°C as a discerning test for the detection of sigma phase. Figure 5 shows the relationship between sigma phase formation, weight loss in ferric chloride and impact toughness [17]. It can be seen that high weight losses can be realised when toughness falls below 70 J at minus 50°C. Finally, welds are not included in the scope of ASTM A 923, but this will be resolved when a new ISO standard is released in the near future. This is ISO 17781[18], this document deals with welds also and will be more comprehensive than either NORSOK M630 or ASTM A 923 in its scope and detail. Sigma phase precipitation can be localised and patchy. For this reason the results of the three tests should be considered collectively. Any samples with less than expected performance should be investigated metallurgically to establish cause. To retest without any diagnosis of the initial problem is not acceptable. Material grade mix ups are also a problem because many of these grades look alike and the industry does not usually ask for parts to be colour coded. in these cases Positive Material Identification (PMI) is a valuable too to avoid costly alloy mix ups. Ferrite scope testing is also a useful tool to spot check for the presence of gross sigma phase in individual products because of its non destructive nature [19]. It is also possible to check every item. When sigma forms and grows it consumes the ferrite phase. As ferrite is magnetic and sigma is non-magnetic this changes the magnetic properties of the steel. This change can be calibrated against sigma content and it allows for a method for detection of the deleterious phase. However, the process does lack precision [20], and low ferrite scope readings can be caused by other factors like poor pickling of parts. So, in-situ metallography is necessary to fully diagnose the cause of low ferrite scope readings. Figure 6 illustrates the process.

3.3 Improved Specification and Control in Fabrication

Commonly weld procedures are qualified in accordance with ASME IX. This process ensures joints made to the qualified procedure are mechanically sound but there is no requirement for microstructure checks, corrosion testing or Charpy impact testing to confirm avoidance or lack of significance of any sigma phase that may have been formed during the welding process. It is necessary for the user to specify this separately in a fabrication specification. The requirements of ASME IX were developed on the basis of carbon steel and stainless steel fabrication practice. For example, a procedure qualified on a 5mm thick plate qualifies pipe joints between 1.5mm and 10mm thick. In the case of duplex and super duplex stainless steels developing cooling rates in the joint sufficiently fast to minimise sigma precipitation is fundamentally important and the cooling rates developed in 1.5mm thick joints are very much slower than those developed in a 5 mm thick joint. A qualification range of 0.8t to 2t where “t” is the thickness of the qualification test sample could be more appropriate for the range of products designed for 1000 psi applications typically. Equally, ASME IX does not consider pipe diameter an essential variable either. Smaller diameters tend to cool less quickly on welding so would be more prone to sigma phase precipitation. Establishing the qualification breaks for pipe diameters as an essential variable requires some consideration on a project by project basis as the range of diameters used may vary based on desalting capacity. Further, a butt weld qualification allows you to make fillet welds to the same procedure. But the heat input used in welding a 1.5” sch 40s, fully penetrating set on pipe branch, (as is commonly used in manifold connections) is significantly higher than that generated in a pipe butt weld. So, this joint geometry is more prone to sigma phase formation. Figure 7 shows pitting corrosion attack due to sigma formation in the weld metal in the root run and Figure 8 shows corrosion
attack in the Low Temperature Heat Affected Zone (LTHAZ) of a set on branch joint. Specially
developed procedures are needed to avoid this, or the use of pulled branches and butt weld

arrangements. ASME IX classifies alloys in groups that are assigned P numbers. Duplex stainless steels
are in Group P10. According to ASME a procedure qualified on a material within a P group can be used
when welding other grades within the same P group. Both 22% Cr and 25% Cr duplex grades ate listed
in material group P10. In the authors opinion it would be highly dubious practice to use a procedure
qualified on 22% Cr duplex to fabricate 25% Cr components because 25% Cr grades precipitate sigma
phase more readily than 22% Cr alloys do. As stated previously [15], acid pickling increases the
corrosion resistance in ferric chloride testing. Equally immersion of fabricated spools in
Nitric/Hydrofluoric acid mixes at 50 to 60°C can increase the critical pitting temperature of the welds by
10 to 15°C. So this is a rather beneficial process. In the of the view of the above it is recommended that
desalination contractors apply their own fabrication specification which deals with these issues and sets
requirements as a separate document to augment the requirements of ASME IX. Some guidance on this
is provided in Rolled Alloys MDS 12804/21[21].This MDS covers procedure and welder qualification
for ZERON 100 super duplex stainless steel. As a statement of good general practice in fabrication, BS
EN 1011 part 3 [22] is a useful document that can be specified as is NORSOK M601.

3.4 The Use of Specialist Package Suppliers and Distribution Companies

Quality begins with the steel making and primary products like billets, bars and plates. These set the
chemistry, cleanliness and microstructural range of the steel (if this is not correct it may not be possible
to further process these products or for the convertors or fabricators to realise the properties required in
the final product or welded structure). Primary products are then transformed by converters in to
seamless and welded pipes and fittings, forged fittings and flanges and other component parts of the
system. Converters not only change the shape of the product but they also apply the final heat treatment
process that sets the quality of the product. All of these individual product forms are made by separate
specialist mills. Procurement, qualification and vendor approval and supply chain management of the
number of vendors involved can be a costly and time consuming business. It is often the case that
desalination contractors do not have the necessary skill sets in house to properly make technical
assessments of mill manufacturing capability. There are a number of specialist suppliers who provide
the cost reducing service of mill assessment and approval. They understand the mill qualification system
and the metallurgy of these steels. They have gone through the qualification process such that they have
several qualified supply chain option for every product form they may need. This provides commercial
competitiveness, capacity, and delivery flexibility. Specialist packagers also coordinate the logistics of
supply of the complete bill of materials through the supply chain, dealing with final inspection,
compiling quality documentation and handling the logistics of shipping. Some provide contract managed
spool fabrication also. They have long established supply chain relationships and they essentially
minimise the risk in procurement of these grades in terms of both attainment of quality and realisation of
the due date for the purchaser. Guidance on procurement of duplex and super duplex stainless steels
from mills directly and though the use specialist supply companies are given in a document known as
EEMUA 218[23]. This is also a document prepared by the oil and gas industry to try to develop best
practice in procurement of these steels. Although not freely available it is a very detailed and helpful
guide for the buyer. Summary details along with industry experience that drove the need for EEMUA
218 are provided in the open literature by Howard et al [24].

4 Summary
Other industries with high volume usage of these steels, find that sourcing of these alloys requires careful vendor evaluation in terms of capability to supply, awareness of the potential problems they can encounter and the capability of the mill infrastructure to properly process the parts being made. Further, they find that ASTM standards lack key testing requirements that relate to the corrosion resistance of duplex stainless steel products. This requires a material specification to be used to augment ASTM standards that details the inspection and testing needed to realize quality parts. They apply a similar vendor strategy in terms of selecting fabricators. However, it is often complicated by the desire (or some times contractual requirement) to utilize local fabricators (i.e. located near to the construction site). This is often the case too for the desalination industry. Again international standards are not sufficiently robust in terms of dealing with the specifics associated with the metallurgy of duplex and super duplex stainless steels so fabrication specifications are required to augment international standards to achieve the desired quality of fabricated products. These should also include finishing processes like acid pickling by immersion and by pickling paste as these can have a very beneficial effect and provide robust performance especially in warm sea water applications. Vendor selection strategies, materials specification, testing, fabrication and finishing specification requirements have been discussed. There remain some points of contention surrounding appropriate corrosion test methods and acceptance levels. This is also the case with Charpy impact testing acceptance criteria, but practical recommendations have been made in the paper. These routines constitute best practice in procurement of these steels in terms of an optimized QA/QC process to drive quality through the supply chain. Specialist package suppliers with strong metallurgical expertise work to these routines across a number of industries and can supply value added and cost reducing quality management services in this respect to desalination industry contractors.

5 Conclusions

- A technical and quality audit of the manufacturer and qualification process has been adopted by other industries to improve quality in the supply chain. A similar system is recommended for the desalination industry. The NORSOK M 650 model is recommended. Specialist material suppliers have such systems in place and can provide this service to desalination contractors.
- ASTM standards do not include test regimes that confirm the corrosion resistance of the products supplied. A material specification fully detailing testing and inspection is required to do this. The additional testing would include
  - a microstructure check (using ASTM A 923 as a basis for wrought products)
  - Ferric chloride corrosion test (using 50°C test temperature with a 24 hour exposure without visible signs of pitting at x 20 magnification after mechanical probing and an acceptable weight loss of no higher than 4g/m² on samples with a mill finish condition if this will see service. This is essentially to ASTM A 923 method C but without machining the sample surfaces and with higher allowable weight loss results at the 50°C test temperature).
  - Charpy impact testing (with an acceptance level of 70J minimum average value tested at minus 50°C, again to ASTM A 923 but with a higher impact energy acceptance level).

These would be applied to each cast and heat treatment batch of steel product. The results of these tests should be considered collectively. Samples with less than expected performance should be subject to microstructure check to diagnose the problem and direct corrective action. Individual items can be quickly checked using both,
PMI testing and ferritiscope testing. Areas with low Feritiscope test results should be checked using in-situ metallography. Specialist package suppliers already have material specifications that customers can adopt or they can work to project or customer specific requirements. They can also provide PMI, ferritiscope and in-situ metallography facilities.

- International fabrication standards are also weak in certain areas when dealing with duplex stainless steels. Again a separate fabrication specification is required to augment the basic requirements of ASME IX. The fabrication specification should address
  - Corrosion test requirements on both procedure and welder qualifications
  - Range of qualification in terms of joint type, pipe diameter and thickness to which the procedure is applicable, especially when welding thicknesses less than that used in the qualification testing.
  - The extent to which prequalified procedures on alloys in the same P group can be applied
  - Detail requirements for acid pickling by immersion and by paste as required to enhance corrosion resistance
  - Welder qualification testing

- Again, some specialist package suppliers can assist fabrication sub contractors with both procedure and welder qualification and or supply directly fabricated, tested and pickled spools direct to the job site for mechanical assembly.

6 Acknowledgements

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7 References


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9 Figures

Figure 1. Temperature/Time Relationship for Precipitation of Deleterious Phases in Duplex Stainless Steels

Figure 2. Weld Heat Input and Weight Loss of Welds in Ferric Chloride Solution.
Figure 3: Tollerable Levels of Sigma Phase in Welds Tested in Ferric Chloride Solution at 35°C.

Figure 4: Appearance of the OD and ID of Ferric Chloride Corrosion Test Samples from 10” sch 40s Long Seam Welded Pipe After Testing.
Figure 5 Toughness/Corrosion Resistance Relationships of Various Super Duplex Stainless Steels, Heated in the Sigma Phase Precipitation Temperature Range

Figure 6. PMI and Ferritiscope Testing and Microstructure Examination by In –Situ Metallography
Figure 7. Corrosion Attack of the Weld Root Run and Associated Sigmatised Microstructure

Figure 8. Corrosion Attack at the LTHAZ due to Local Sigma Phase Formation