

MECHANICAL PROPERTIES AND CORROSION RESISTANCE OF DUPLEX STAINLESS STEEL FORGINGS WITH LARGE WALL THICKNESSES

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Duplex stainless steels are widely used in chemical process, oil and gas industry. One of the main applications are disconnect able mooring systems (SWIVEL connections) for offshore Floating Production Storage and Offloading units (FPSO). Due to the increasing demand for energy ever larger cast or forged parts are required in the offshore industry. It is a well known fact that in larger duplex stainless steel forgings precipitation of detrimental phases can take place, such as Sigma phase or nitrides. Impact strength at low temperature and corrosion resistance will be influenced by these precipitations. Therefore mechanical properties and corrosion resistance of ring rolled products with large wall thicknesses are the main objective of this report. Hence, mechanical properties including impact strength were determined as a function of specimen test location depth in a part that failed during acceptance testing. Additionally two test rings with identical wall thickness and different forging ratios were investigated in the same manner. Corrosion resistance tests were performed according to ASTM G48 and in some cases Critical Pitting Temperature (CPT) was established.

The microstructure was examined via Optical Microscopy as well as Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). A Focused Ion Beam (FIB) device was used to micromachine TEM foils at evident sites in SEM specimens.

KEYWORDS: duplex stainless steel, ring rolling, large wall thickness, impact strength, precipitations, nitrides, sigma phase, mechanical properties, critical pitting temperature, focused ion beam

INTRODUCTION

Duplex stainless steels (DSS) provide very good corrosion and excellent stress corrosion resistance together with moderate mechanical strength compared to standard austenitic stainless steels [1-4].

These properties are mainly attributed to their twofold microstructure consisting of nearly 50% austenite and ferrite in most cases. Compared to ferritic stainless steels, DSS offer an improved resistance to intergranular corrosion. On the other hand, compared to standard austenitic grades DSS reveal better stress and crevice corrosion properties as

well as higher strength at lower costs due to their reduced nickel contents [2]. By virtue of their excellent features, DSS are widely used in oil and gas industry, as well as in chemical process industry [4,5]. However, despite their advantages, DSS are known for their complicated microstructural changes that can take place during forging and heat treatment. In a temperature regime reaching from 300 to 1000°C a more or less severe loss in toughness and corrosion resistance can be observed depending on the time spent at these temperatures [1,3,6,7]. This loss in properties is due to the precipitation of detrimental intermetallic phases or carbides and nitrides from the metastable ferritic phase. As all processes dominated by diffusion, these reactions are mainly temperature and time dependent and therefore also dependent on the wall thickness, controlling the local cooling rates [2,6]. Other factors influencing the properties of a forging are the chemical composition and possible segregations in the starting ingot and of course the forging schedule determining the microstructural fineness and distribution [4]. Nowadays the demand for energy is increasing worldwide and thus ever larger parts are required by

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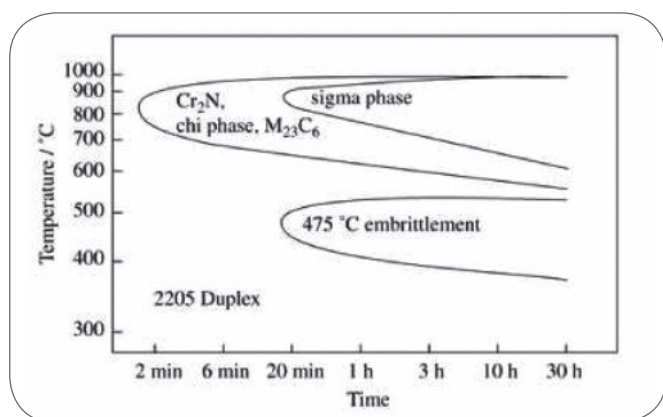
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oil and gas industry [5,6]. For example large forgings and rolled rings are used in offshore disconnect able mooring systems (so-called SWIVEL connections). The SWIVEL connection is a turnable device which makes it possible to convey oil and gas from a deep sea oil field trough a buoy while the Ship-like Floating Production Storage and Offloading unit (FPSO) connected with the buoy is free to rotate to get the most stable position in rough waters and under windy conditions. Therefore the material of choice has to bear high mechanical loads, while being exposed to corrosive environments and high pressures. Good resistance to stress corrosion and crevice corrosion as well as high mechanical strength are essential requirements for these applications [5]. This paper is concerned with the mechanical properties and corrosion behaviour of large wall thickness parts made from duplex stainless steel grade SA/A182-F51 (UNS31803/32205, DIN Nr. 1.4462). A close look was given at the nature of rejected or re-heat-treated parts and it was revealed that almost all problems came along with heavy weight and large wall thicknesses. Therefore it was decided to manufacture test rings with similar dimensions and different forging ratios. These test rings and a rejected part were investigated in detail including mechanical testing over the whole wall thickness. Optical Microscopy and Scanning or Transmission Electron Microscopy (SEM, TEM) were used to examine the microstructure in different test locations. In order to investigate precipitations at ferrite grain boundaries or ferrite/austenite phase boundaries, a Focused Ion Beam (FIB) device was applied to micromachine TEM foils at the respective sites. Critical Pitting Temperature CPT was determined according to ASTM G 48 test method E [8]. The results of the investigation were used to optimise the production processes for DSS in order to provide large wall thickness rings, flanges and forgings with adequate overall properties for customer's demands.

PHASE STABILITY OF DUPLEX STAINLESS STEELS

As can be seen in Fig. 1 the microstructure of DSS can undergo a variety of changes during cooling from high temperatures or during forging. The reactions can be divided into an



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Fig. 1

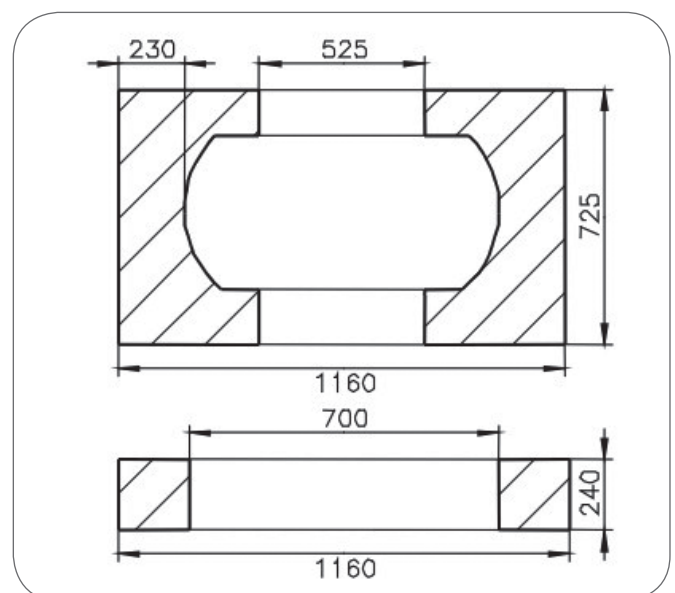
Schematic Time-Temperature-Transition diagram of SA/A182-F51 [1]

Diagramma schematico Tempo-Temperatura-Formazione di fasi per l'acciaio SA/A182-F51 [1]

upper and a lower embrittlement regime. At temperatures between 650 and 1000°C the precipitation of Cr_2N , $M_{23}C_6$, Chi- and Sigma phase can take place [3,7]. Especially the carbide or nitride precipitation can start in less than 2 minutes at 750 to 900°C. The precipitation of Sigma phase which is reported to have an severe influence on impact strength and corrosion properties will take at least 20 minutes at 850 to 900°C. In the lower temperature regime the decomposition of ferrite to Cr-enriched and Cr-low ferrite, the so called 475°C embrittlement, is often reported [7]. A very good overview of the possible precipitation reactions in DSS is given in Nilsson's review paper [3]. One has to be aware that the precipitation reactions in the upper regime can be drastically enforced by hot working at these temperatures [3,4]. Therefore the hot working temperature of duplex stainless steel shall be restricted to above 950°C. However, temperatures in excess of 1200°C can lead to severe distortion of semi-finished parts and should also be avoided. To avoid any precipitation, the cooling from forging and heat treatment temperature has to be as fast as possible. Since the quenching efficiency of core regions of large forgings is mainly controlled by the heat conductivity of the material, precipitation is likely to take place in locations far from surface [6].

PRODUCTION OF TEST FORGINGS AND EXPERIMENTAL TECHNIQUES

The nature of rejected or re-heat-treated parts was analysed. It was revealed that all affected parts were rejected on base of unacceptable low impact strength at low temperatures. All of these parts were tested at -40 to -50°C. Minimum impact strength was 45 J average and 35 J single value as often required in specifications and data sheets based on NORSOK standards. In summary, one can assume that as a rule heavy parts with large wall thicknesses and low forging ratios are likely to fail during acceptance testing.



▲
Fig. 2

Dimensions of rejected ring and test forgings.
Dimensioni dell'anello forgiato scartato e dei coupon di prova.

These findings were anticipated since diffusion controlled precipitation reactions in DSS are likely to be enhanced with decreasing cooling rates during quenching as it is the case with increasing wall thickness and weight. Also low forging ratios will result in coarse microstructures, therefore provoking low impact strength values.

To learn more about the properties of large wall-thickness DSS parts one rejected ring and two trial forgings were investigated. The rejected ring had a profiled cross section given in Fig. 2, weighing 4500 kg in premachined condition. The two test pieces were forged to approximately the same dimensions as far as wall thickness is concerned but had a square cross section (Fig. 2). A schematic sketch of ring rolling in general and ring rolling of contoured parts is given in Fig. 3.

The rejected part and test ring A were manufactured starting from preformed material (melt A) with 650 mm diameter providing a high overall forging ratio. The second test ring B was made from an ingot casting (melt B) having a diameter of 500 mm. The chemical composition of both melts is given in Tab. 1. As a first result the compositions do not differ much from each other.

The schematic forging schedule of all parts is given in Fig. 3. The overall forging ratio for part A (melt A) is by factor 2 higher than for part B (melt B). All forgings were heated to 1220°C in gas-fired furnaces. Hot forming was performed at 1200 to 950°C. The heat treatment given to all parts was solution annealing at 1070°C with 5 h soaking time controlled by thermocouples directly attached to the parts. In

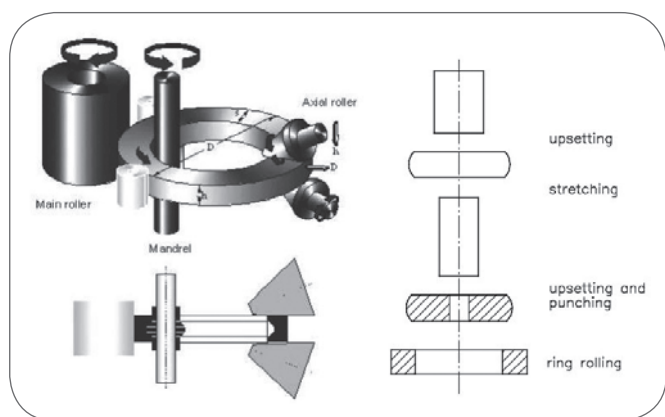


Fig. 3 Schematic sketch of ring rolling process (left upper part) and profile ring rolling (left lower part) and schematic sketch of forging process (right part).

Disegno schematico del processo di fabbricazione (parte sinistra superiore) e profilatura (parte sinistra inferiore) degli anelli, con rappresentazione schematica (parte destra) delle fasi del processo di forgiatura.

melt	C	Si	Mn	P	S	Cr	Ni	Mo	Cu	N
A	0,024	0,59	1,2	0,019	0,0008	22,73	5,68	3,15	0,06	0,175
B	0,019	0,55	1,31	0,022	0,001	22,6	5,58	3,18	0,15	0,16

Tab. 1 The chemical compositions of the steels. Composizione chimica degli acciai.

case of the rejected part a re-heat-treatment was done at 1080°C with 8 h soaking time. The time necessary to reach the given solution annealing temperature was 4 to 5 h except for the first heat treatment of the rejected part where 20 h were required for heating up. This was due to heavy weight and present furnace performance. Each part was subjected to impact strength and tensile testing in 7 depth levels from sub-surface to 110 mm using Charpy V-notch and tensile specimen according to ASTM A 370.

In addition to the above mentioned tests two further simulation heat treatments were performed and subsequently evaluated by means of impact strength testing. First a solution annealing at 1070°C/30 min was given to 15 mm thick plates machined at different distances to surface of the rejected part.

This was to determine the influence of wall thickness on toughness. Secondly an isothermal aging treatment was conducted at 475°C with 15 mm thick sections of part A and a known impact strength of 300 J in order to evaluate the effect of low temperature embrittlement.

Microstructure was revealed using Murakami's Reagent which darkens the ferrite and leaves the austenite phase nearly unaffected and bright. In cases Optical Microscopy revealed imperfections, Scanning Electron Microscopy (SEM) measurements were performed additionally. In two cases a Focused Ion Beam (FIB) device attached to the SEM was used to micromachine thin foils perpendicular to the specimen's surface at interesting sites. These foils were investigated in Transmission Electron Microscope (TEM) to identify the nature of the precipitated phases.

Critical pitting temperature was determined according to ASTM G 48 method E subjecting specimen to a ferric chloride test solution at different temperatures for 24 h with temperature steps of 5°C each. CPT specimens were investigated by means of mass loss and by identification of pittings via low-magnification microscopy. A mass loss greater than 4 g/m² and the incidence of pittings were set as a criterion for CPT.

EXPERIMENTAL RESULTS AND DISCUSSION

Mechanical testing

The yield strength (Rp0,2), ultimate tensile strength (Rm) as well as the elongation at fracture (A5) and reduction of area (Z) of the rejected ring are given in Fig. 4. The respec-

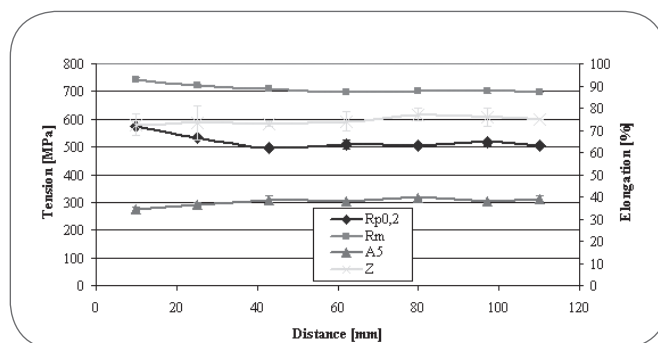


Fig. 4 Tensile Properties of rejected part as a function of distance to surface. Proprietà tensili delle parti scartate in funzione della distanza dalla superficie.

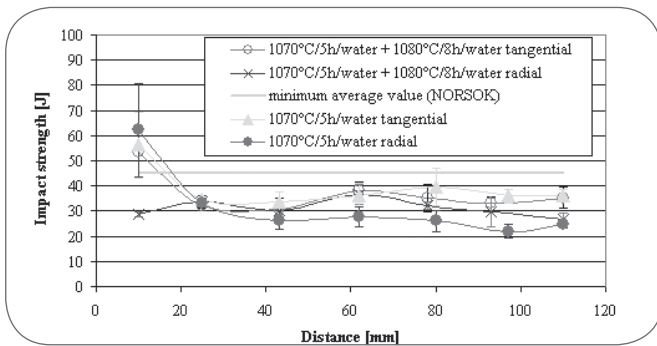


Fig. 5 Impact strength at -46°C as a function of distance to surface - rejected part.

Resilienza a -46°C in funzione della distanza dalla superficie - parte scartata.

tive values comfortably meet the required properties. No or little change in strength or elongation can be observed as a function of distance to surface. The slightly increased yield strength and tensile strength near the surface can be attributed to the higher degree of hot working compared to the core region.

The -46°C impact strength properties of the rejected part are plotted against distance to surface in Fig. 5. The values are given for the first ($1070^{\circ}\text{C}/5\text{h}/\text{water}$ quenching) and second heat treatment ($1080^{\circ}\text{C}/8\text{h}/\text{water}$ quenching) respectively. As can clearly be seen, the impact strength values are low, only surpassing the minimum requirement indicated by the red line in the surface-near test location. Also the additional heat treatment at 1080°C with 8 h holding time did not alter the impact strength values under consideration of experimental scattering. This indicates that a simple re-heat-treatment may not be sufficient to dissolve all detrimental phases. Specimens from this rejected part were given a detailed microstructural analysis to identify the reasons for the poor impact properties. More details will be given in the respective section.

The additional solution treatment of 15 mm thick plates machined with a distance of 10, 45 and 110 mm to surface resulted in impact strength values between 100 and 240 J. This is well above the requirements and the values obtained from the ring at the same depth level. On the contrary the re-heat-treatment of the ring did not induce any improvement in impact strength (Fig. 5). The cooling rate in the heavy part might have been too low to circumvent the reprecipitation at possibly undissolved particles acting as nuclei. In the 15 mm thick plates this reprecipitation could have been suppressed by the high cooling rates.

The impact strength values of the two test forgings are presented as a function of distance to surface in Fig. 6. Both rings show very high impact strengths well above the minimum values for locations between 10 and 25 mm distance to surface. However, part B has significantly lower impact strength compared to part A. As distance to surface increases the impact strength decreases drastically and drops slightly below the minimum value at a depth of 80 mm for part B. Part A reveals values between 50 and 120 J from 45 to 110 mm distance to surface. These values are well above the minimum and part A would pass the stringent impact requirements even in the core region. As mentioned in the section above, melt A is preforged material,

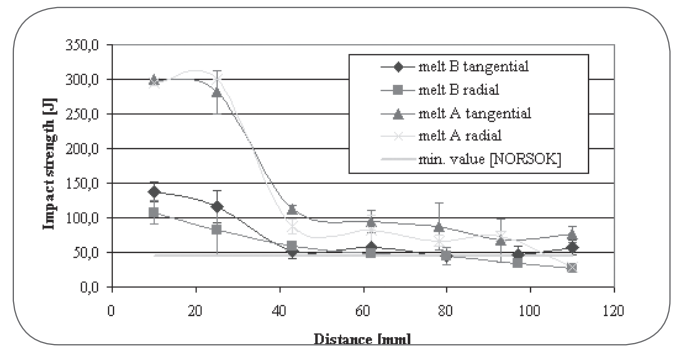


Fig. 6 Impact strength at -46°C as function of distance to surface - trial forgings A and B.

Resilienza a -46°C in funzione della distanza dalla superficie - pezzi di prova forgiati A e B.

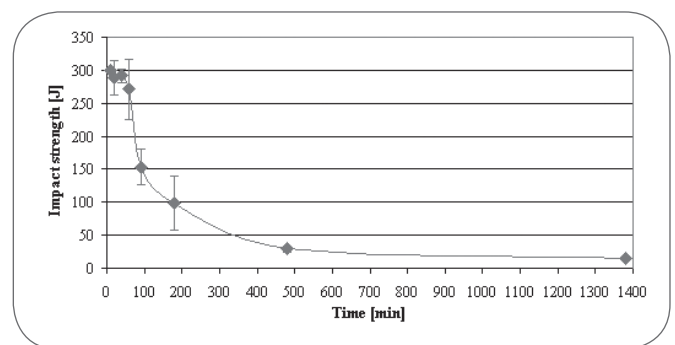


Fig. 7 Impact strength at -46°C as function of isothermal aging time at 475°C .

Resilienza a -46°C in funzione del tempo di invecchiamento isoteramico a 475°C .

melt B represents cast material.

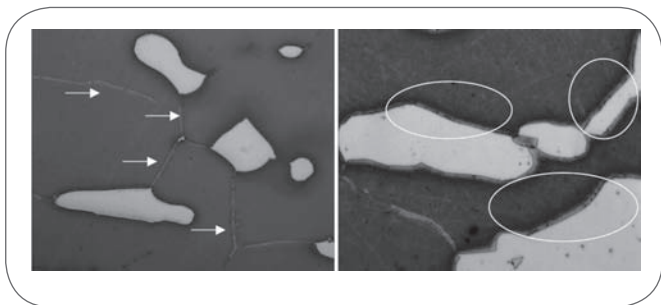
Therefore the overall forging ratio from cast ingot to final product differs much between the two test forgings, whereas differences in chemical compositions are only marginal. Hence the above findings indicate that the overall forging ratio has a strong impact on low temperature toughness.

As one would assume, the tensile properties of the two test forgings A and B are again nearly independent from distance to surface. Therefore their delineation is set aside.

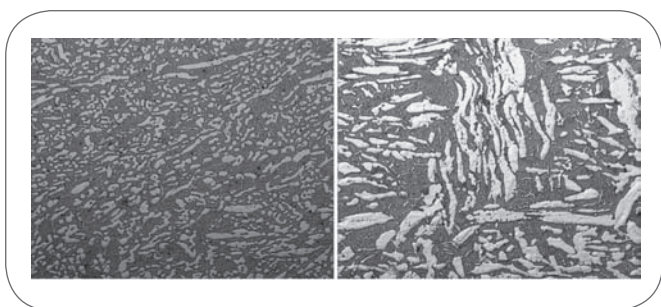
The effect of isothermal aging at 475°C is displayed in Fig. 7. As can clearly be seen, impact strength at -46°C is affected after 1 h. The original impact energy values are reduced to one half after 1,5 h and to a third after 3 h. Thus the 475°C embrittlement is likely to influence the impact strength of very large duplex forgings, limiting the maximum wall thickness.

Microstructural Investigation

The above mentioned results in mechanical properties needed to be evaluated in terms of microstructural aspects. Therefore the broken Charpy specimens of the rejected part and the two trial forgings A and B were grinded and subsequently electropolished. For Optical Microscopy the microstructure was etched using Murakami's Reagent. The typical microstructure of the rejected part is given in

▲
Fig. 8

Microstructure at 10 mm and 110 mm distance from surface (Murakami's R eagent, 1000x).
Microstruttura alla distanza di 10 mm e di 110 mm dalla superficie (reagente Murakami, 1000x).

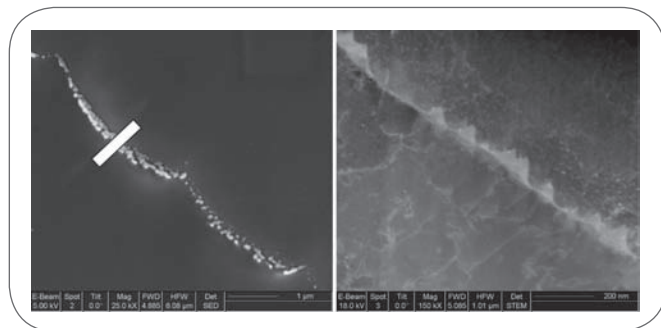
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Fig. 9

Typical microstructure of part A (left) and part B (right) respectively (Murakami's Reagent, 50x).
Microstruttura tipica rispettivamente della parte A (sinistra) e B (destra) (reagente Murakami, 50x).

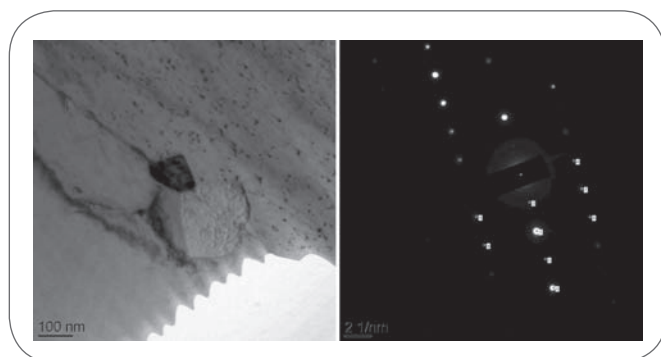
Fig. 8. All over the wall thickness of this part precipitations are visible on the ferrite grain boundaries (as indicated by arrows in Fig. 8). On the ferrite/austenite phase boundaries some particles can be observed in locations distant to surface (indicated by circles in Fig. 8), whereas the respective boundaries in the outer test locations are smooth (left part of Fig. 8) and seem to be free from precipitates visible in optical microscope.

The microstructures of test ring A and B were totally free from any precipitates visible in optical microscope. As can be seen in Fig. 9 test forging B having a lower overall forging ratio shows a coarser and inhomogeneous microstructure, which is likely to affect impact strength adversely.

Specimens from the rejected part were also investigated using SEM and TEM. In both cases the FIB technique was applied to TEM preparation at the respective boundary as indicated by the white bar in Fig. 10. The ferrite/austenite phase boundary in SEM-image (left picture) and the respective TEM micrograph (right picture) perpendicular to the boundary and to surface are displayed in Fig. 10. One can clearly see a coral-like precipitation character in the SEM image and triangle-shaped particles in TEM micrograph. Note the high magnification of both images and the low size of the precipitates having a diameter of less than 100 nm. Therefore, high quality Selected Area Diffraction Patterns (SADP) could not be obtained. However, Electron Dispersive X-ray (EDX) analysis revealed high Cr and Mo

▲
Fig. 10

SEM image (SE, 25000x) of ferrite/austenite phase boundary and bright field TEM micrograph of ferrite/austenite phase boundary with precipitates.
Immagine SEM (SE, 25000x) dalla linea di transizione tra le fasi ferrite/austenite e micrografia TEM in campo chiaro di tale linea, che evidenzia i precipitati.

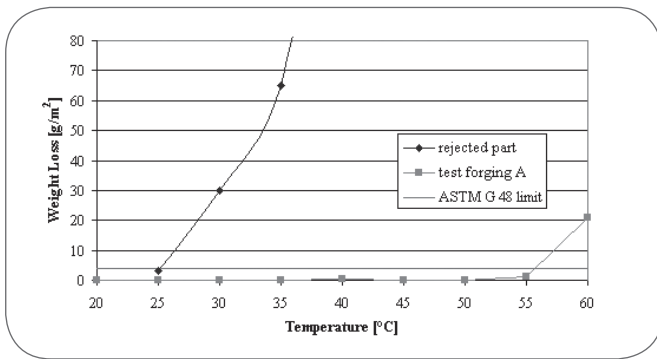
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Fig. 11

TEM micrograph and SADP of Cr₂N precipitates at ferrite grain boundary (zone axis [1-10]).
Caratterizzazione di precipitati di Cr₂N al bordo del grano di ferrite, tramite TEM e SADP (asse di zona [1-10]).

content in the precipitates. Iron was depleted in respect to the matrix but well present in the particles. These EDX findings and the phase morphology as well as the precipitation sites indicate that the respective particles are likely to be Sigma phase or one of its precursors [1,3]. The precipitates at the phase boundary appear in high density with an almost uninterrupted character.

In contrast to that, the particles observed at the ferrite grain boundaries were few in number and were interrupted by long precipitation-free distances. Some of these particles had sizes up to 200 nm in diameter and it was possible to obtain SADP of sufficient quality to analyse their structure. In Fig. 11 the SADP of the large particle in the left picture can clearly be identified as Cr₂N by selected zone axis [1-10]. EDX proved high Cr content and moderate Mo content in the particles, whereas Fe was nearly totally depleted. Therefore one can assume that the particles precipitated at the ferrite grain boundaries are Cr₂N.

Hence, it was proved that the low impact strength of the rejected part can be attributed to precipitation of Cr₂N and presumably Sigma phase particles. Cr₂N can be found in the entire wall thickness of the part, whereas Sigma phase can only be found in regions distant to surface. As impact



▲
Fig. 12

Weight Loss [g/m²] as a function of Temperature in CPT-Test according to ASTM G 48 method E.

Perdita in peso [g/m²] in funzione della temperatura in una prova CPT secondo il metodo E del ASTM G 48.

strength was higher in the outer and Sigma-free specimens of the part, it can be assumed that Cr₂N precipitates are less detrimental in regard to impact strength.

Corrosion Testing

The results of the CPT tests according to ASTM G 48 method E are displayed in Fig. 12 for the rejected part and test forging A. CPT is reached if weight loss is greater than 4 g/m² or if pittings can be observed by low-magnification optical microscopy. Both specimen sets were taken not deeper than 15 mm below the surface of the parts as deeper regions would not get in contact with corrosive media.

In case of the rejected part ASTM G 48 corrosion test at 25°C would have been passed, however high weight loss indicated low corrosion resistance. Massive pittings and very high weight loss can be observed at temperatures above 25 °C. On the other hand specimens from test part A have very low weight loss and no signs of pittings up to 55°C which is amongst the highest CPT values ever reported for UNS 31803 [7].

Hence the low impact strength of the rejected part and the precipitation of Cr₂N also indicated low corrosion resistance even though the basic requirements of ASTM G 48 test were met and the part would have been fit for purpose. The optimised processing routine used for test forging A resulted in very high corrosion resistance also indicated by very high impact strength values at -46°C and the total absence of precipitations.

SUMMARY AND CONCLUSIONS

The condition of rejected parts was analysed by comparison of impact strength test results. It proved that almost all parts having relatively low impact strength values at -40 to -50°C were heavy in weight in combination with large wall thickness and low forging ratios.

The mechanical properties of a rejected part and two test forgings of similar heat treatment dimensions were investigated as a function of distance to surface. Tensile properties revealed little or no influence of distance to surface, whereas impact strength was strongly dependent on specimen location depth. The impact strength values of the

rejected part were low and only met the requirements in the outer sample location. This could be due to the long time required to reach temperature in the first solution annealing step, resulting in a long period spent at critical temperatures. An additional heat treatment of the rejected part could not alter the low impact strength values, whereas solution annealing of 15 mm thick plates machined from this ring resulted in moderate impact strength. Therefore it can be assumed that the additional heat treatment was unable to dissolve all precipitates and that the low cooling rate in the heavy ring resulted in reprecipitation at undissolved particles acting as nuclei. In contrast cooling rate in thin plates is high enough to circumvent this effect. Test forgings A and B with different forging ratios revealed much higher impact strength values, especially part A with the highest forging ratio met the stringent requirements even in the core region of the ring. Therefore it can be concluded that forging ratio has a strong impact on the properties of DSS. In aging treatments at 475°C it was shown that after relatively short aging times of 90 min the impact strength was drastically reduced. Hence, forgings with very large wall thickness might suffer from 475°C embrittlement.

The low impact strength of the rejected part was due to the precipitation of Cr₂N at ferrite grain boundaries all over the wall thickness. Investigated in TEM these particles were few in number and separated by a large intermediate particle-free space. In regions far away from surface presumably Sigma phase or one of its precursors could be observed at the ferrite/austenite phase boundaries.

These particles formed a nearly continuous layer at the respective phase boundaries. Their influence on impact strength was much stronger compared to the Cr₂N. The microstructures of the two test forgings were essentially free from any precipitates visible in optical microscope up to 1000x magnification.

Corrosion resistance of the forgings was determined by means of ASTM G 48 tests at different temperatures. All parts even those with low impact values and visible Cr₂N-precipitates passed the test at 25°C which means they were fit for purpose. Test forging A had a CPT of 55 °C, being one of the highest CPT ever reported for SA/A182-F51 DSS. However, this was indicated by the excellent low-temperature impact strength values and the precipitation-free microstructure of the test forging.

The following statements can be given as conclusion:

- parts having low impact values just above 45 J at -46 °C and having Cr₂N-precipitates at the ferrite grain boundaries can nevertheless pass ASTM G 48 corrosion tests at 25 °C
- precipitates on ferrite grain boundaries like Cr₂N affect impact strength but Sigma phase or its precursors being precipitated at the ferrite/austenite phase boundaries have a much stronger influence on toughness
- forging ratio and forging temperature have a vital influence on impact strength of forgings and rolled rings with large wall thickness; overall forging ratio should be high and all forging operations should be performed well above 1000°C in case of SA/A182-F51 DSS
- heat treatment conditions are critical: quench facilities should be as effective as possible to ensure high cooling rates; handling time between furnace and quench facility should be short to avoid preprecipitation stages; parts have to be machined close to final dimensions before solution annealing
- if high forging ratios and strict temperature control

are applied, precipitation free microstructures, very high impact strength and excellent corrosion properties can be achieved in DSS even in the core region of forgings with large wall thicknesses

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ABSTRACT

PROPRIETÀ MECCANICHE E RESISTENZA ALLA CORROSIONE DI PRODOTTI FORGIATI CON PARETI DI GROSSE DIMENSIONI IN ACCIAIO INOSSIDABILE DUPLEX

Parole chiave: acciaio, precipitazione, forgiatura, prove meccaniche

Gli acciai inossidabili duplex sono largamente impiegati nell'industria chimica, petrolifera e del gas. Una delle principali applicazioni riguarda i sistemi di ormeggio scollegabili (connessioni girevoli SWIVEL) utilizzati sulle piattaforme off-shore di produzione, stoccaggio e trasbordo (Floating Production Storage and Offloading - FPSO). A causa della sempre crescente domanda di energia nel settore off-shore sono necessarie parti forgiate o colate sempre più grandi. E' noto che nei prodotti forgiati di grosse dimensioni in acciaio inossidabile duplex può verificarsi la precipitazione di fasi potenzialmente pericolose, ad esempio fase sigma o nitruri. Queste precipitazioni

hanno una influenza sulla resilienza alle basse temperature e sulla resistenza alla corrosione. Quindi le proprietà meccaniche e di resistenza alla corrosione di forgiati ad anello con pareti di grosso spessore sono l'obiettivo principale di questa relazione. Pertanto, le proprietà meccaniche, tra cui la resilienza, sono state determinate in funzione dell'affondamento del provino nello spessore del materiale in prova, che proveniva da un pezzo scartato ai test di accettazione. Inoltre sono stati esaminati nello stesso modo due anelli di prova con identico spessore di parete ma con diversi rapporti di forgiatura. L'accertamento della resistenza alla corrosione è stato eseguito secondo i requisiti ASTM G48 e, in alcuni casi si è individuata una Temperatura Critica di Pitting (CPT).

La microstruttura è stata esaminata mediante microscopia ottica e microscopia elettronica, a scansione (SEM) e a trasmissione (TEM). Un sistema a fascio ionico focalizzato (Focused Ion Beam - FIB) è stato utilizzato per ottenere lamine sottili, da esaminare al TEM, in punti rivelatisi significativi nell'osservazione al SEM.