Issue 2, February 2022

World Stainless Steel Duplex Seminar & Summit 2022

1 & 2 November 2022, Rotterdam

DUPLEX WORLD UPDATE

A technical event update for the global duplex community

Inside:

- **Duplex World Call for Papers**
- Improved 22% chromium duplex
- Sigma phase: a metallurgical challenge when welding duplex

Stainless Steel World

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Stainless Steel World

Duplex World Update is brought to you by the Stainless Steel World team

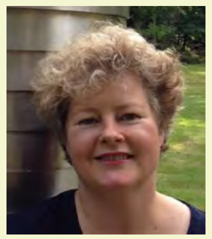
A platform for the global duplex community

Welcome to Duplex Update #2!

As the Duplex World team put the finishing touches to this technical update, there are only eight months to go before we meet again in Rotterdam on 1 & 2 November! Ok, it sounds far away, but the Steering Committee, led by Bruce Cowe from TotalEnergies, is busy evaluating abstracts that have been coming in response to our call for papers. There is still time to contribute; we've extended the deadline to 21 March so don't delay; send me your abstract today!

As travel restrictions ease around the globe, we look forward to welcoming our global duplex community back to Duplex World. Our virtual event in 2021 was a great success, but there's nothing like meeting face-to-face. Our event is renowned for networking, and often it's the people you meet in lunch breaks or during the fabulous networking dinner that prove to be the best business leads you'll make.

I hope you enjoy this second update; if you'd like to contribute to the next edition, don't hesitate to get in touch!



Best wishes,
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Organiser of the Duplex World Seminar
Tel: +31 575 585 298
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Speak at Duplex World 2022!

Don't miss the opportunity to present a paper or join a workshop discussion at Duplex World 2022, 1 & 2 November in Rotterdam, the Netherlands.

Send your abstract before *21 March* to j.mcintyre@kci-world.com

More information on the following pages!

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Duplex World Seminar & Summit 2022

1 & 2 November, 2022 • RDM Rotterdam, The Netherlands

Join the leading knowledge, networking and business event for the global duplex community!

Duplex World unites researchers, manufacturers, end-users and suppliers in an open and focused forum. Get ready to join the conversation!

Duplex World is the ideal platform to profile your company's expertise and products, network with industry peers, and share your knowledge with the international materials community. This dynamic and interactive event focuses strongly on personal interaction and a high-quality technical seminar program.

Exhibition of leading companies

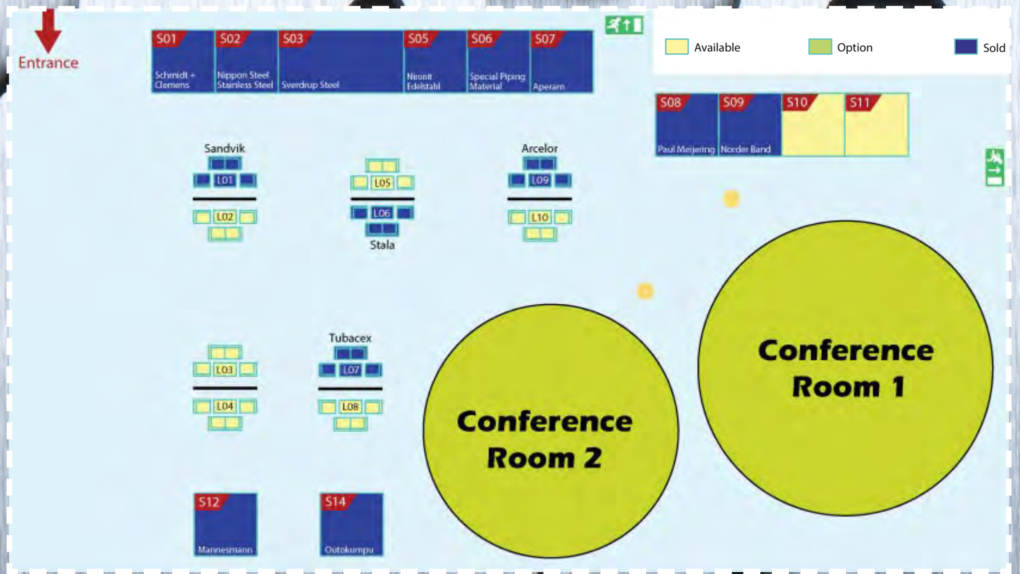
The RDM Rotterdam is a spacious location where conference rooms, exhibition stands, and networking lounges are all located on the same floor. It's ideal for facilitating the flow of visitors and delegates. Make use of this perfect opportunity to showcase your company, products and services and network with clients in a relaxed yet professional atmosphere. Choose between two exhibition formats:

Exhibition Stand Package 4,900 Euro

Exhibition Stands showcase your company's products and activities. Stand packages include stand building, basic furniture, plus a seminar ticket.

Networking Lounge Package 5,900 Euro

Networking Lounges provide an opportunity for companies to network directly with clients and peers in an informal setting ideal for private discussions. Lounges include a couch, chairs, table, back-drop with company logo, plus a seminar ticket.



To book your Exhibition Stand or Networking Lounge, go to www.stainless-steel-world-duplex.com or contact Mr Erik Schoppema
Email: e.schoppema@kci-world.com
Tel: +31-575-585-293

For information about the conference, contact Mrs Joanne McIntyre
Email: j.mcintyre@kci-world.com
Tel: +31-575-585-298

Showcase your company @ Duplex World 2022

Technical conference program

Duplex World seminar unites duplex researchers, manufacturers, end-users and suppliers in an open and focused forum. Network with the leading minds driving ahead innovation and excellence in materials science. The high-quality technical program is a dynamic and interactive event with a strong focus on personal interaction. Visit the website to view the advanced program from 15 May.

Why exhibit at Duplex World?

Multiple suppliers

Every participant at the event is involved in duplex stainless steel. From large scale manufacturers to medium-sized suppliers of duplex fittings, this is a niche event for customers to meet potential business contacts conveniently in one location.

Leading end-users

There is a strong end-user input into the conference and presence at the event.

Business networking

This is a highly targeted event; the network you build will benefit you for the rest of your career.

All aboard! Get ready for a fun-filled night of networking aboard the SS Rotterdam, a former cruise ship converted into a hotel and party venue. Our networking dinner and party on 1 November will bring together participants from Duplex World and our co-located event, Heat Exchanger World, for a fabulous night of food and fun!

In a world of rising prices, duplex appears more attractive than ever

As inflation once again takes hold, duplex stainless steel can be seen as an attractive alternative to higher-alloyed materials in many applications. This, together with advances in welding and fabrication, may explain why duplex is gaining the interest of a wider range of specifiers.

By James Chater

Unwelcome spectre

Those of us who are old enough to remember the 1970s are experiencing an uncomfortable sense of *déjà vu*. The oil shock of 1970 triggered high rates of inflation, which gradually tapered off in the 1990s. Now various factors, including low interest rates, post-Covid catching up, scarcity of certain raw materials and stretched supply chains, have caused the old spectre to return to the feast as an unwelcome visitor. The post-Covid recovery has led to a dramatic surge in pent up demand for such commodities as fuels (especially natural gas), land, wheat, steel rebar and copper, the prices of which have soared. This applies also to most of the key ingredients of stainless steel and CRAs: in 12 months, nickel rose from around 14,300 USD/T to 20,000 but has since fallen to around 18,000; molybdenum now stands at 41,846 USD/T, up from around 18,836 a year ago; titanium had also rebounded as the aerospace sector emerged from its Covid slump [1]. Prices of all steels and alloys have been rising, but it is notable that in the last two years lean duplex has become less expensive than certain austenitics (diagram).

Merits of duplex

No one knows if this is temporary or longer-lasting. In either case, it is likely to motivate specifiers to reassess the merits of duplex. With its carefully balanced austenitic and ferritic structures that ensure greater strength and toughness without sacrificing corrosion resistance, duplex is more sparing in its use of expensive alloying elements than nickel alloys or superaustenitics. Moreover, advances in ease of welding or fabrication have further increased its attractiveness. Experiments by Friedlander Contracting International Ortec Group show that it is possible to weld small-diameter and thin-walled superduplex pipes with orbital GTAW, without filler metal, using an orbital closed head welding machine. Posco can now produce welded duplex tubes for tube heat exchangers using the LBW welding process, with no loss of quality compared with the traditional seamless tubes, and with considerable savings of time and money.

Sandvik broke new ground when it made a WAAM-printed a impeller in superduplex.

Additive manufacturing

The main game-changer is likely to be additive manufacturing (3D printing). Sandvik and its part subsidiary BEAMIT can now 3D-print in superduplex Osprey® 2507 metal powder. In collaboration with Norwegian Eureka Pumps they have produced an impeller that is used in one of Equinor's offshore projects.

Norwegian scientists have investigated the possibility of using wire arc additive manufacturing (WAAM) and gained promising results, suggesting that AM and WAAM manufacture of large components in the process industries is not far off.

Meanwhile, in the construction industry, Takenaka Corp. and MX3D collaborated on a project to WAAM-print a structural connecter used in large structures. The hollow duplex stainless steel structure filled with concrete was found to be highly effective and economical for safety performance.

The Tramlink is a bi-directional tram made of duplex stainless steel. This tram runs in Erfurt, Germany. Photo: EVAG_Jacob Schröter.

Applications

Oil & gas

Oil & gas is still the most important market for duplex and superduplex grades. The last few months have seen a resurgence in demand and price, especially for natural gas, which should encourage the launch of new projects. On the other hand, environmental concerns may inhibit further projects or may lead to a preference for natural gas over oil.

Duplex is used in several applications: pumps, valves, heat exchanger tubes, pressure vessels and other process applications; also for structural components such as blast walls, where high strength is needed. Sandvik recently introduced SAF 2205™+ for use as bar in the oil & gas industry.

It is designed to withstand extreme corrosion and is certified for subsea requirements. Impure elements such as phosphorous and sulphur were eliminated to increase resistance to pitting and crevice corrosion.

Recent orders show duplex's versatility. Sverdrup Steel is supplying lean duplex, duplex and superduplex, along with 316L, for the HOD field in the North Sea. When delivering its API 610 VS4 vertical pumps and two API 610 OH2 horizontal pumps to the ADNOC Bu Haseer development, Amarinth faced two challenges: the very sour environment and restricted space. The pumps were made in superduplex and were split into two sections for reassembly on site. Sour environments are also driving the use of duplex in pipelines. One gas field in China had about 10% chloridion concentration in the water separated from gas, so grade 2205 welded pipe was applied.

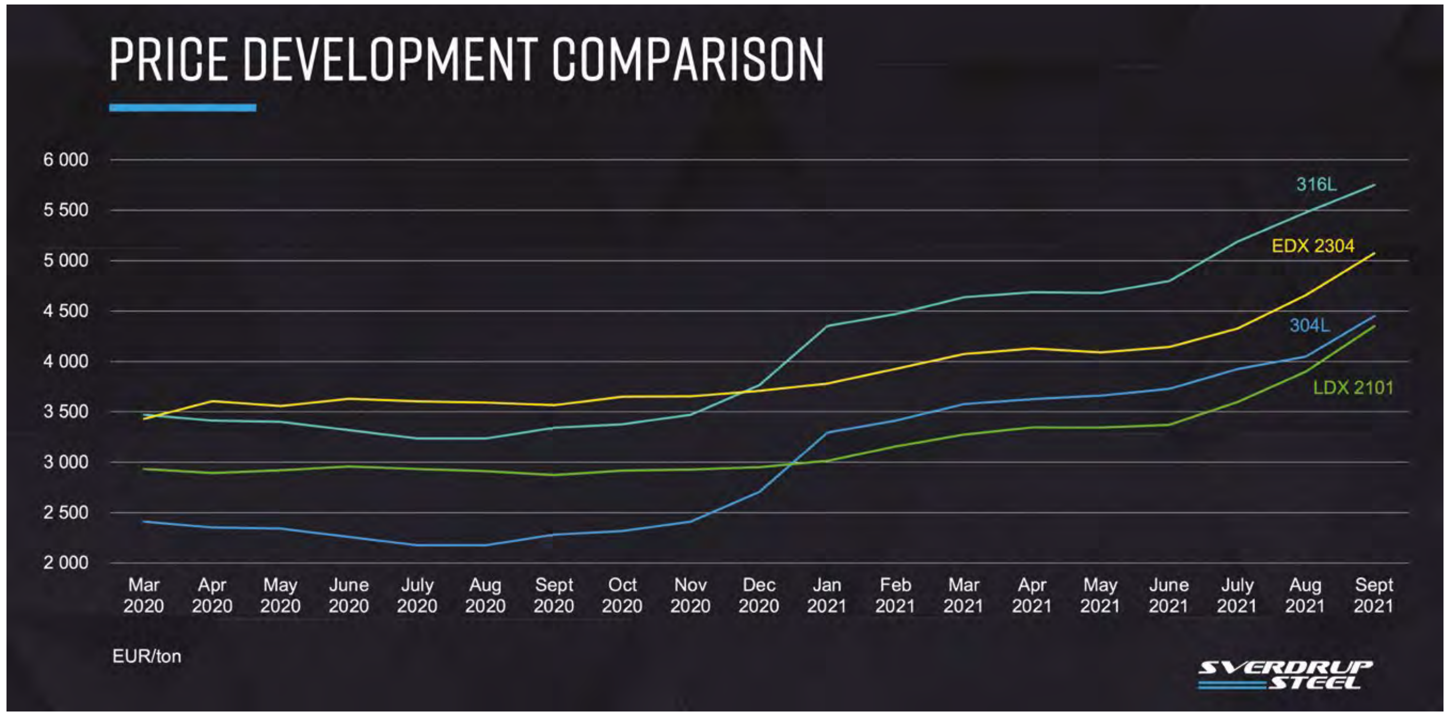
Other applications

Duplex is finding use in petrochemical, chemical, desalination and fertilizer plants, offshore wind turbines, geothermal power and water management. In the case of superduplex grades, high corrosion resistance is the chief criterion, for example in the use of Sandvik's SAF 2507® superduplex grade in a geothermal plant in Brazil. Other grades offer some corrosion resistance and afford a lighter structure, important in building and infrastructure projects.

Weigel+ medical and DOI ortho-innovativ made this prosthetic foot and knee system using superduplex fasteners from Bumax.

This the case in a new cyclists bridge in Sydney's Macquarie Park, which uses a double helix form similar to a pedestrian bridge, also made of duplex stainless steel, in Singapore. Another recent bridge made of duplex is the Pooley Road Bridge in England's Lake District. Midway Metals, Stirlings Performance Steels and Vulcan Stainless together supplied 170 tonnes of 2205 duplex, with most of the plate coming from Outokumpu. Because de-icing salt is used in winter, corrosion was a concern, so a duplex grade, Outokumpu's Forta LDX 2101, was selected.

Two relatively new applications of duplex are for transport and



Price movement of lead duplex and austenitic grades.

UK's first stainless steel road bridge was officially opened on Friday 23 October 2020 over the River Eamont at Pooley Bridge. It was designed by Knight Architects and uses duplex supplied by Outokumpu. Photo Courtesy of Outokumpu.

prosthetics. Bi-directional trams are a new kind of tram developed in order to avoid the complicated structures needed to allow uni-directional trams to reverse direction. They are in use in Amsterdam, Brussels, Erfurt, Gmunden and other European cities. Manufactured by Stadler, they consist of three car bodies made of duplex stainless steel, applied for corrosion resistance and weight reduction. In 2018 nine of these trams were ordered by the city of Lugano in northern Italy. In 2020 Milan followed suit, ordering 80 vehicles for its urban and interurban services.

Finally, duplex has entered the field of prosthetics. Bumax SDX 109 high-strength superduplex fasteners were found to be suitable for a prosthetic knee and foot system. The All Terrain Knee and Niagara Foot were designed by Weigel+ medical and DOI ortho-innovativ for those with an active lifestyle. The superduplex screws are resistant to corrosion in a marine environment and have high strength.

The WAAM-printed structural connector designed by MX3D and Takenaka, made in duplex stainless steel.

Conclusion

Duplex stainless is being applied over a broader range thanks to advances in fabrication, AM and welding. The current raw materials crunch may also focus end users' attention on the merits of this versatile class of stainless steel.

References

1. https://tradingeconomics.com/commodity/steel.

Duplex in India

India's ambitious infrastructure projects are likely to be a growing market for duplex stainless steel. The country's railway system has been undergoing an extensive upgrade. Part of this is the new Pamban Bridge, which will connect the town of Rameswaram on Pamban Island to the Mandapam railway station on the mainland and will supersede a structure built over a century ago. It will be of the vertical-lift kind. The location is known as the second most corrosive place on earth. Therefore RVNL, the engineers, specified duplex grade 2205 for the four spans and girders of the new bridge. The engineers have launched a tender for the first 120 tonnes of duplex, and Jindal has stated its intention to bid.

The demand for stainless steel in process industries is also increasing. Therefore in 2020 SAIL announced it now has the capability to produce superduplex SS 32205 or use in oil & gas, chemical processing, pulp & paper, food processing and biofuel plants.

Improved 22% chromium duplex stainless steel

Industeel recently developed a new controlled chemistry 2205 duplex stainless steel with enhanced weldability compared to the conventional 2205 material and improved low temperature toughness properties. This new optimized chemical composition promotes a stable 40% ferrite / 60% austenite phase balance in the base material. Industeel identifies the grade as Arctic 2205.

By John Grocki, Sandra Le Manchet, Anne Higelin, Industeel (ArcelorMittal group)

Better ferrite control keeps HAZ below 70%

The advantage of Arctic 2205 is that it presents an improved weldability compared to the standard 2205 duplex. It is well-known that duplex welding requires a strict control of the cooling rates to avoid either the formation of intermetallic phases or a destabilization of the ferrite / austenite phase balance. The most common standards used in the Oil & Gas and Chemical industries require a ferrite content in heat affected zone not higher than 70%.
Ten bead-on-plate coupons were welded using the SMAW process with varying heat inputs (0.55 to 4.7 kJ/mm) to evaluate the maximum ferrite content in the heat affected zone for given cooling rates. The ferrite content was measured according to the manual point count method described in ASTM E562. The results shown in Figure 1 reveal that the ferrite content in the heat affected zone is always below 70%, regardless of the heat input.

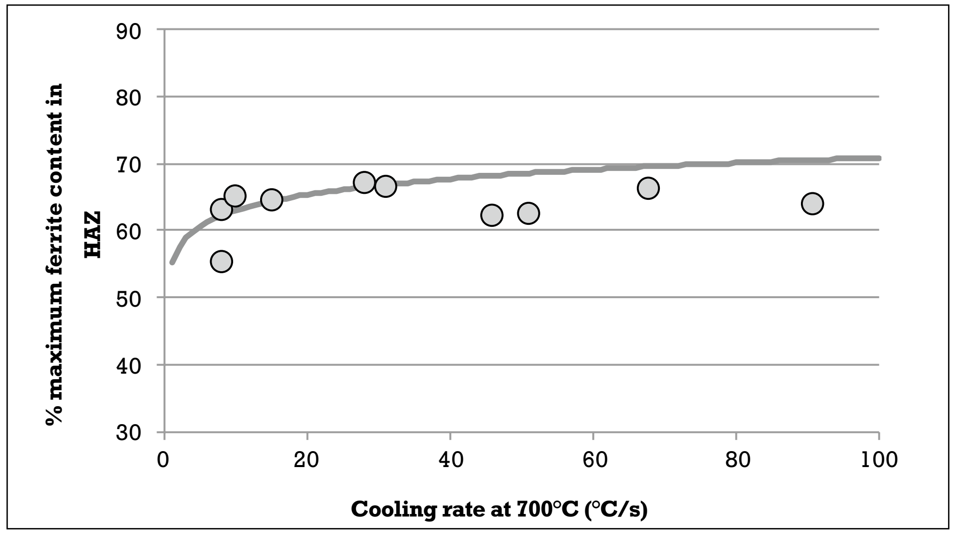


Figure 1. Ferrite content measured for Arctic 2205 as a function of the cooling rate.

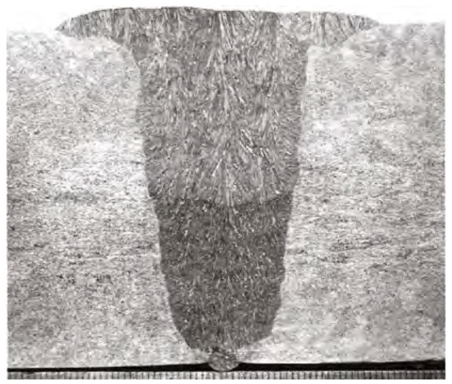


Figure 2. Macrograph of the Arctic 2205 sample welded by VRV (GTAW, SMAW, SAW) – 55 mm thick.

Welding assessment by fabricators

Industeel worked in partnership with several fabricators to assess the properties of welded Arctic 2205 samples.

Case study #1 – VRV
The VRV company performed welding trials on two materials: the standard 2205 and Arctic 2205. The filler material was 2209 and the gas mixture consisted in Ar+2%N2. The bevel design was U-shape 12° opening with a root gap of 3 mm, root face of 2 mm and radius of 8 mm. After the GTAW root passes, filling was performed by twenty passes with SMAW and the balance with SAW. The heat inputs were between 0,5 and 1,6 kJ/mm.
The microscopic examination in Figure 2 revealed that the microstructures are free from precipitates or intermetallic phases according to ASTM A923 method A.
Tensile specimens ruptured in the base metal at 750 MPa while bending tests did not lead to any crack initiation.
Figure 3 shows the results of the Charpy impact tests performed at -46°C in the heat affected zone and weld metal of the welded samples.
The toughness values in the heat affected zone are clearly better for the Arctic 2205 being almost twice those obtained for the standard 2205 material.

Figure 3. Absorbed energy and lateral expansion at -46 °C

Grade	Heat Affected Zone			Weld Metal		
	Top SAW	3/4 SMAW	Root GTAW (subsize samples)	Top SAW	3/4 SMAW	Root GTAW (subsize samples)
Standard 2205	63 J	84 J	28 J	68 J	82 J	31 J
Arctic 2205	122 J	116 J	43 J	74 J	80 J	41 J

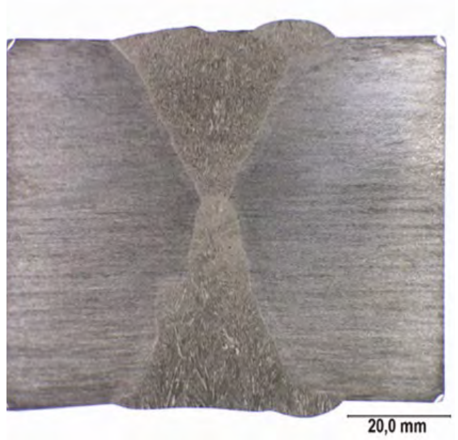


Figure 4. Macrograph of the Arctic 2205 sample welded by Enerfab (FCAW, SAW) – 55 mm thick.

Case study #2 – Enerfab
Enerfab performed welding trials on the Arctic 2205 with 2209 filler material using two welding processes: FCAW in the vertical position with Ar + 25%CO2 shielding gas on one side and SAW in the flat position on the other side. Welding heat inputs of the two processes were between 1,3 and 1,8 kJ/mm. The bevel design is a double vee opened at 60° with root gap of 2-3 mm. 17 FCAW passes were needed on the face side and 20 SAW passes on the root side.
The ferrite contents were measured according to ASTM E562 (50 points per grid) in four separate locations: as-welded or reheated weld metal areas (x500, twenty fields) and heat affected zones (x1000, thirty fields). The maximum ferrite content measured in heat affected zone is 63%.

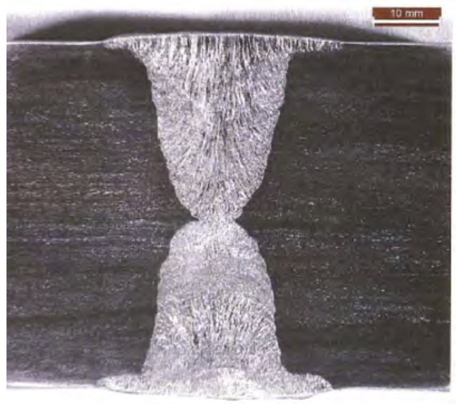


Figure 7. Macrograph of the Arctic 2205 sample welded by FBM Hudson (GTAW hot wire) – 55 mm thick.

Figure 5. Ferrite measurements in the weld metal and heat affected zone of Arctic 2205 welded using FCAW and SAW.

		Weld metal X500	HAZ X1000
FCAW	As-welded	42	63
	Reheated	37	53
SAW	As-welded	52	62
	Reheated	35	57

Figure 6. Absorbed energy and lateral expansion at -46 °C.

	Weld metal		Heat affected zone	
	Charpy test (J)	Lateral expansion (mm)	Charpy test (J)	Lateral expansion (mm)
Face side FCAW	58	0.77	108	1.18
¼ upper thickness FCAW	42	0.50	115	1.28
¼ lower thickness SAW	58	0.68	129	1.41
Root side SAW	55	0.68	107	1.24

The toughness values measured at -46 °C are above 100 J in the heat affected zone and around 50 J in weld metal (Figure 6).

Case study #3 – FBM Hudson
FBM Hudson Italiana performed welding trials on Arctic 2205 with 2209 filler material and Ar + 2%N2 shielding gas using an automatic GTAW Hot Wire process in the flat position. The welding heat input was 1,3 kJ/mm. 20 passes were performed on each side of the double U-shape 20° bevel.
The ferrite contents were measured according to ASTM E562 using a x500 magnification. The results reveal

Figure 8. Ferrite measurements in weld metal and heat affected zone of the Arctic 2205 welded with GTAW hot wire process.

	Weld metal	Heat affected zone
Upper side	38 %	51 %
¼ upper thickness	-	46 %
½ thickness	36 %	-
¾ lower thickness	-	45 %
Bottom side	35 %	46 %

Figure 9. Absorbed energy and lateral expansion at -101 °C.

	Weld metal		Heat affected zone	
	Charpy test (J)	Lateral expansion (mm)	Charpy test (J)	Lateral expansion (mm)
Upper side	188	1.7	146	1.6
½ thickness	196	1.7	-	-
¾ lower thickness	-	-	203	1.5
Bottom side	125	1.2	200	1.6

ferrite contents in the 45-50% range in the heat affected zone.

The toughness values are all above 100 J at -101 °C regardless of the test location.

Better toughness properties than standard 2205

The additional consequence of this balanced two-phase microstructure is the strong shift in the toughness transition curves towards lower temperatures compared to the standard 2205 duplex stainless steel (Figure 10). It may allow the use of this material down to at least -80°C while it is not generally recommended to use standard duplex stainless steels below -50°C.

Conclusion

Arctic 2205 duplex stainless steel was developed to provide enhanced weldability and improved toughness properties at low temperatures. The characterization of samples welded by several fabricators confirmed improved weldability through controlled ferrite contents, always below 70%, in heat affected zones and excellent Charpy impact test values at low temperature as well. This material presents the same corrosion and mechanical characteristics as the standard 2205 duplex material. The combination of these capabilities will help expand the use of the duplex material, particularly for heavy sections in applications requiring stringent low temperature toughness properties.

Arctic 2205 can be supplied as quarto-plate material up to 90 mm thick. The adjusted chemistry and processing parameters will result in a small premium in cost. However, due to the expanded applications in lower temperature environments, there may be an opportunity to transition to Arctic 2205 from traditionally more expensive super austenitic and Ni-based alloys. In addition, the increase in strength of duplex materials may allow thinner wall plates and pipes to achieve comparable design results versus thicker alternatives. That coupled with the improved weldability will keep the Arctic 2205 cost competitive in a variety of applications.

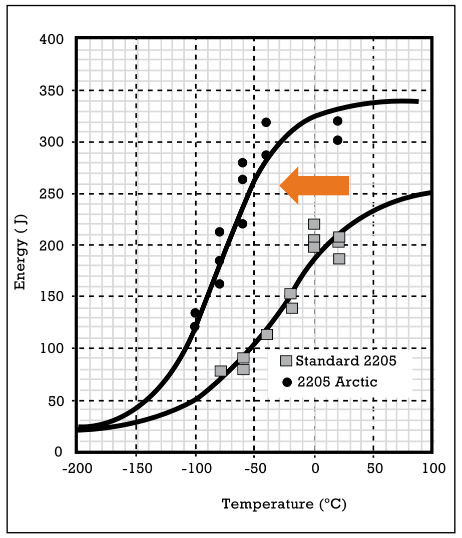


Figure 10. Toughness curves of Arctic 2205 and standard 2205 duplex stainless steel.

Acknowledgements

Industeel would like to acknowledge VRV, FBM Hudson and Enerfab who performed the welding trials on this new grade. Their welding expertise is greatly appreciated.

Sigma phase: a metallurgical challenge when welding duplex

While high strength was the focus of alloy development for many years, today a range of other properties are also essential[1]. Examining new types of failures and metallurgical aspects, especially during manufacturing processes requiring high temperatures such as welding and heat treatment, have led researchers to realise that high strength alone is not sufficient to meet the requirements for a reliable component. Instead, a range of material properties is needed, including toughness, stiffness, hardness, ductility, and corrosion[2, 3, 4].

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Duplex stainless steels (DDSs) consisting mainly of Cr, Ni, Mo and N, are widely used in the market. Duplex grades can be divided into three subgroups: lean duplex, standard duplex, and super duplex. The most common alloys for each of these three subgroups are 2304, 2205 and 2507 respectively. Since the early 1980s, DSS alloys have been commonly used in marine applications, petrochemical industries and oil refineries[5].

The microstructure of duplex is an almost 50:50 mix of austenite:ferrite at room temperature, although commercial alloys may have ratios of 40:60 (Figure 1). Duplexes possess a combination of high strength and corrosion resistance not readily attained in conventional single-phase austenitic or ferritic stainless steel. Compared to austenitic stainless steels, they have higher corrosion resistance, especially against pitting and stress corrosion cracking (SCC)[3, 6]. Compared to ferritic stainless duplexes offer improved formability, weldability and toughness. Although the welding metallurgy of these alloys is relatively well understood, heating DSS to high temperatures (450 to 1200°C) is challenging for end-users due to the formation of hard phases such as sigma (σ), chi (χ), intermetallic phases and varies chromium carbides[7] (Figure 2). These phases are often harmful, for example, enhancing embrittlement and reducing corrosion resistance. Besides the reduction in toughness, these also cause intergranular, pitting and stress corrosion cracking.

The heat created during welding of DSS may cause detrimental phases to appear. Various precautions may help prevent this:

- A filler material with 2-3% higher nickel content than the substrate, such as ER2307 and ER2209
- An inert gas environment can increase pitting resistance of the weldments[6]

Figure 1. Microstructures of a: 2304, b: 2205, c: 2507. Light is austenite γ, dark is ferrite α.

Heating duplexes to high temperatures (welding, hot working, heat treatment) causes σ phase precipitation. According to the eutectoid reaction - solid to another solid phase transformation, this change mainly occurs at the intersection of three grains and at the grain boundaries between ferrite and austenite - into a lamellar eutectoid structure of σ and secondary austenite γ: delta ferrite $\delta \rightarrow + \gamma$.

The formation of χ phase in DSS is often initially favoured. However, because χ phase may transform into χ phase following further ageing, the χ phase formation is favoured over longer ageing periods.

Based on the Time-Temperature-Transformation (TTT) diagram for DSS, there is a threshold time required to precipitate the intermetallic and hard phases. This time depends on the temperature and chemical composition of the alloy. In the worst-case scenario, the formation of σ phase, which takes place around 900-1100°C, takes around 2 minutes. Fast cooling by quenching in water or oil is therefore advisable. However, quenching often leads to distortion and cracking, particularly with thin components.

Solution annealing (heating to around 1050°C for the appropriate time) followed by air cooling can be an alternative for the quenching process for workpieces less than 10mm thick.

Figure 3 shows that the microstructural changes of 12mm thick super duplex

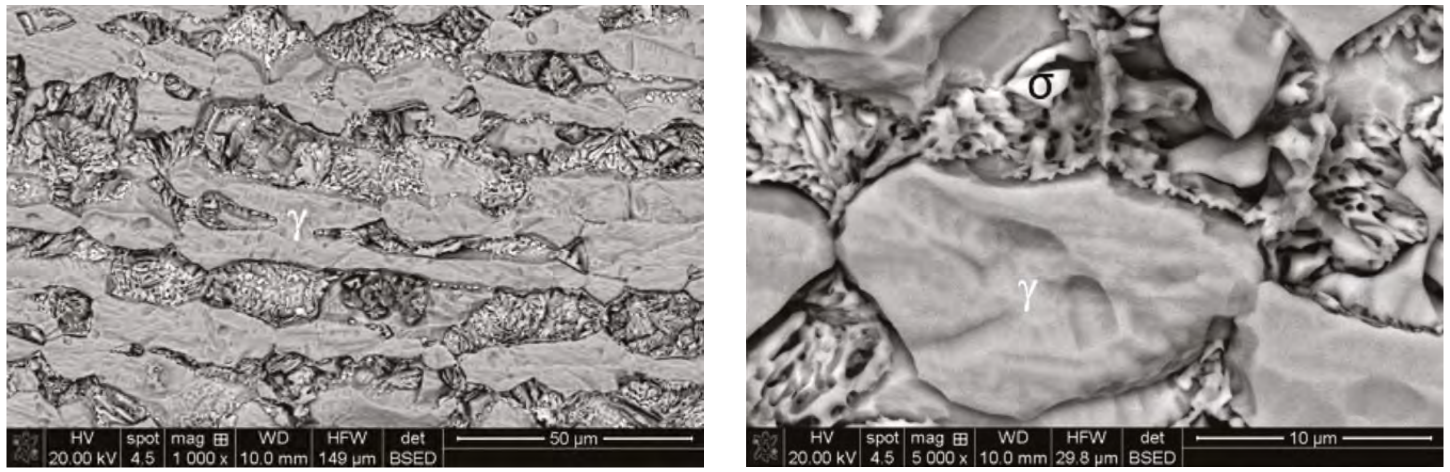

Figure 2. Sigma phase formation of 2205 alloy heated for 10 min at 900°C.

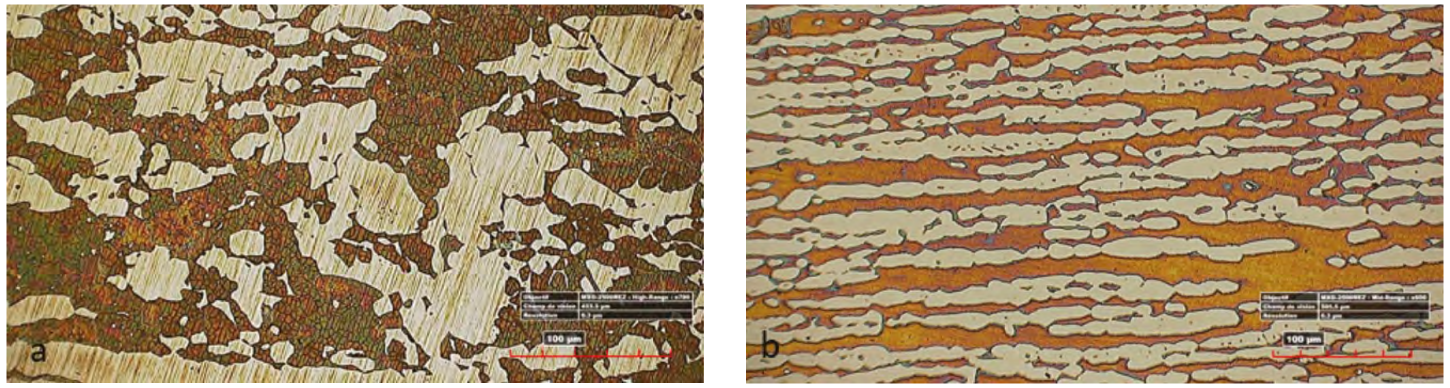

Figure 3. Microstructural recovery, a: heat-treated at 750°C for 1hr, b: heat-treated at 1050°C for 1hr (both air-cooled).

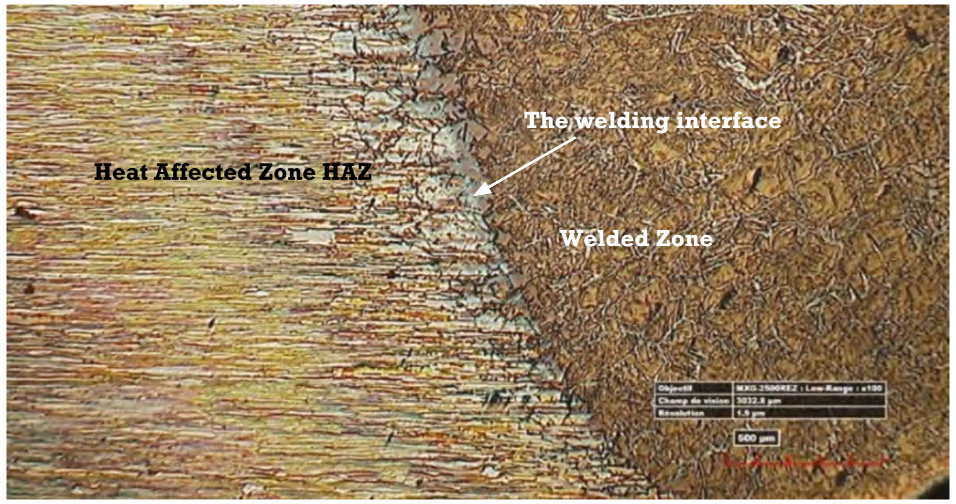

Figure 4. The interface between the welded zone and HAZ of TIG-welded 2205 alloy.

2507 can be recovered to the original microstructure through air-cooling after applying solution annealed treatment from 1100°C.
In the last few decades, numerous studies have investigated the negative impacts of welding on the mechanical properties of DSS, particularly on toughness and corrosion resistance. The modifications are always referred to as the metallurgical aspects that occur in the fusion zone and HAZ. However, because these two zones are relatively narrow and present intermixing (Figure 4), finding an accurate contribution of each zone on the whole alloy is difficult and always a point of discussion.
Figure 4 shows that in the welded zone, due to high temperature and subsequently fast cooling, most of the structure is ferrite content. However, the HAZ is a combination of both phases, γ and α; it can be σ too but this does not appear in low magnifications.

Investigation techniques

Different techniques and methods are available to investigate the metallurgical aspects of heating DSS alloys, including:

- Metallographic and chemical analysis
  Using electronic microscopes, SEM, TEM, EDX, x-ray diffraction and spectra. The metallographic images and chemical composition analysis of specific grains/phases help to recognise specific phases, e.g. σ (Figure 2). However, this is not sufficient to reveal all details about the effect of temperature on the microstructural changes.
- Thermal simulation techniques
  This method can help to characterise the subsequent changes of the microstructure at specific temperatures which have similar heating and cooling rates to the welding process. Applying the heat on samples at conditions similar to the welding situations, but below the melting point, i.e. for HAZ. as the equipment is not capable to record the temperatures of molten materials[4].
- Thermoanalytical Analysis
  Various thermoanalytical techniques are used to detect the metallurgical changes such as σ phase formation. These include magnetic force microscopy, equilibrium thermodynamic conditions, simultaneous high-temperature thermo-gravimetry, isothermal drop calorimetry, isothermal dilatometric, differential thermal analysis (DTA) and differential scanning calorimetry (DSC). These techniques measure the difference in the amount of heat required, enthalpy increments, dimensional variation, magnetic flux rate, etc of a sample and a reference material.

Summary

Heating, due to welding or heat treatments, above 500°C causes microstructural changes in DSS. Besides changing the 50:50 balance ratio of austenite: ferrite, hard phases such as sigma are formed at specific temperatures. Solution annealing, even by air cooling from 1050°C, can lead to recovery of the original microstructure. Different techniques, based on heat enthalpy, movement or atoms, dimensional changes, etc can be used to detect sigma phase in DSS alloys.

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DUPLEX WORLD UPDATE

The CSTR's after installation, before insulation and cladding was installed.

Continuously Stirred Tank Reactors (CSTR)

Continuously Stirred Tank Reactors (CSTR) are of particular interest for industrial biogas plants. A German fabricator has ventured into producing duplex stainless steel CSTR tanks; at 20 meters high these demand a steady hand and attention to detail for successful assembly.

Text & images by Stallkamp

A food manufacturer from northern Germany uses production waste in its own waste and sewage treatment plant in order to exploit the energetic waste in a biogas plant. This way, the company combines economic success with its ambitious ecological goals. The success of this endeavor meant that the expansion of the biogas plant had become a necessity. The decision was made in favour of two tall digesters made of high-quality stainless steel - designed, manufactured and assembled by Stallkamp from Dinklage, Germany.

Challenging construction

The two identical tanks have a diameter of 15 m and a height of almost 20 m, each having a capacity of 3,300 m³. The construction of the stainless steel digesters was carried out using a segmented design. For this purpose, individual shell plates with thickness of 3 to 6 mm were rolled to diameter. These were screwed together then sealed to form large rings at the construction site.
The first fully assembled ring was lifted with electrical lifting supports that were especially developed for this project. This was followed by the assembly of the next ring, which

DUPLEX WORLD UPDATE

The digester in operation, fully wrapped in insulation and cladding.

was again lifted by the 30 synchronously running lifting supports. When lifting the tank to assemble the final ring, more than 40 tons were weighing on the lifting supports, which lifted the weight evenly and safely. "We knew from multiple tests that the concept would work. Nevertheless, the calm and even lifting of the structure was a special moment," reports Erich Stallkamp (managing partner), who followed the construction progress daily.

Duplex the optimal material

In order to minimise corrosion in the anaerobic fermentation process, the plant operator consistently uses stainless steel for the new digesters - in its highest quality. All side panels as well as the 4 mm thick stainless steel bottom are made of duplex 1.4462 sheets. The material has particularly high yield strength as well as a very high corrosion resistance and is therefore particularly suitable for use in the biogas plant. The roof is also made of stainless steel: stable rafters supported by an inner and an outer ring form the basic structure. An inner and an outer stainless steel skin was then attached to this structure.
In the middle of the roof there is an opening for the central mixer. It constantly mixes and homogenises the fermentation mixture and thus ensures optimum process biology.

Three more under construction

For Stallkamp, the continuously stirred tank reactors are an important step, as they are the highest tanks of this type ever produced by the company.
"We are pleased with the successful completion of the project and showed that we can play along on this scale," stated Christoph Heseding (Managing Director) and Patrick Wienken (Head of the Technical Office Tank Construction).

Currently, three further CSTR for a mega biogas plant are under construction in Denmark. A total of 14 tanks are currently being built to treat up to 600,000 tonnes of biomass per year. Three of these digesters have a height of 20 m and a diameter of 23 m. Further information is available at www.stallkamp.de/en.

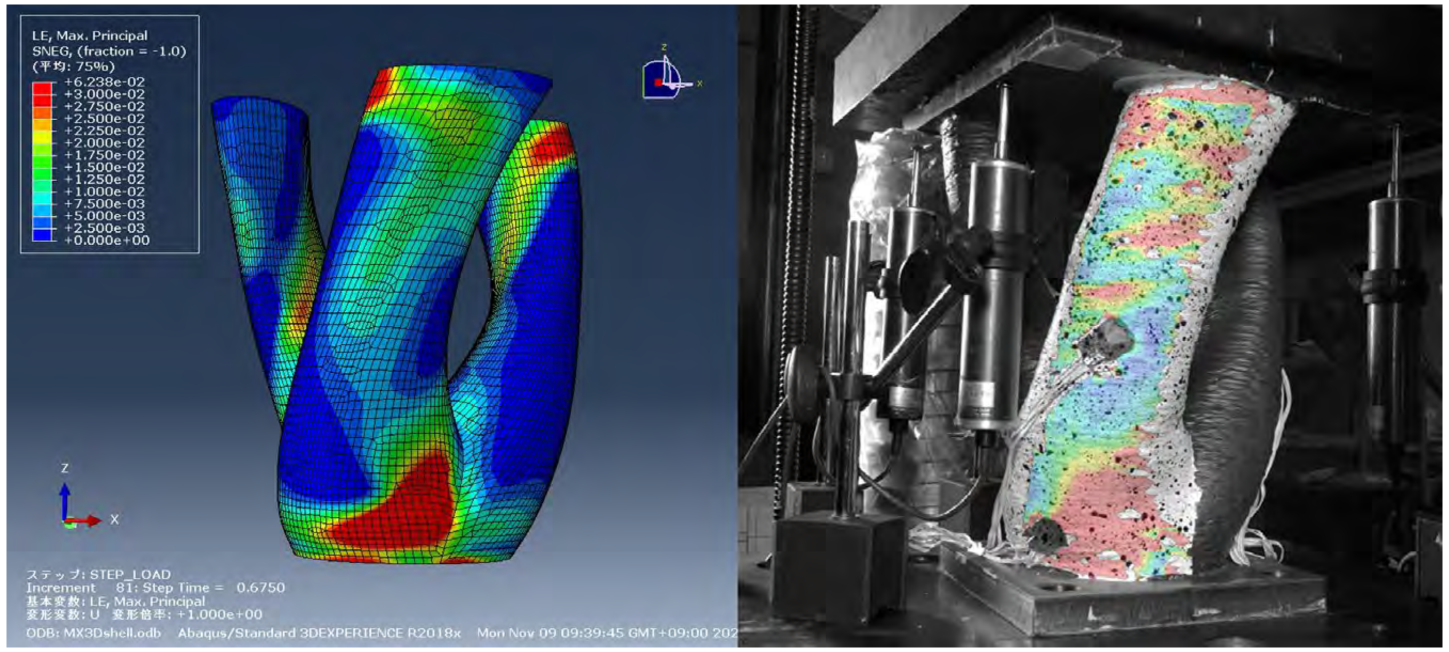
Positive results for 3D-printed duplex connectors

Large-scale 3D metal printing company MX3D has successfully produced a structural connector in duplex stainless steel, designed in collaboration with engineers from Takenaka, one of the largest architecture, engineering, and construction firms in Japan.

Text & images by MX3D/Takenaka

An artistic rendering of infrastructure possibilities of 3D-printed structural steel connectors.

This project demonstrated the progress in producing highly customized and engineered steel connectors using robotic 3D metal printing, i.e. Wire Arc Additive Manufacturing (WAAM). Destructive and non-destructive testing on a series of printed duplex steel connectors shows a strong and consistent performance of mechanical and material properties. The successful tests are a crucial step towards certification of the structural connector, which parties intend to use for innovative building projects.



Connector analysis predicted deformation vs actual deformation, Takenaka MX3D

Innovative infrastructure

In 2019, Takenaka Corp. and MX3D initiated a collaboration to explore the possibility of robotic WAAM technology to produce large customized steel connectors for the infrastructure industry. The project's goal is to automate both the design and production of complex connectors for large structures in the building industry. As the complexity of structures increases, skilled labour is scarce and build speed is key, so innovation is required. Following the successful production of the first connector prototype, the collaboration focused on mechanical and material properties testing, a crucial requirement for acceptance by the market.

Rigorous testing

Destructive testing on a series of printed structural connectors showed strong and constant mechanical and material properties, paving the way to certification. Tensile tests, fatigue tests, and buckling tests performed on the connectors showed convincing results. CT scans revealed the interior was dense and solid. Moreover, compressive failure tests confirm the clear effect of mortar filling on buckling resistance and ultimate strength. A 4mm-thick connector filled with mortar has approximately the same strength as an 8mm-thick connector (2.2 kN vs. 2.3 kN resp.), reducing the material required by 50% and making the freedom of form that 3D metal printing offers even more advantageous. The material properties and mechanical testing achieved desired specs and consistency, establishing the relevance of robotic 3D printing to the industry.

Duplex: the ideal material

The initial prototype series of connectors was printed in duplex stainless steel. The alloy is renowned for its good mechanical properties and excellent corrosion resistance, ideal for structural applications. The hollow steel structure filled with concrete is highly effective and economical for safety performance. The other steel bears bending and tensile forces and the inner concrete core delays or prevents local buckling of steel. Researchers from Takenaka Corp. used information from the physical testing of material specimens to generate a FE simulation model and assess the connectors' mechanical behaviour. These results were then tested against the real-life behaviour of the connectors undergoing compressive load. The results provided by the simulations were very close to the behaviour shown during the mechanical tests. Furthermore, tensile strength tests showed a highly consistent pattern, required for architects and engineers to include this technology in their tool kit. The full research will be published soon. Following the successful fabrication of the optimized and printed structural steel connectors and the strong test results, the partners involved intend to implement the bigger version(s) in an actual building project.

Opening up AM use in construction

Closing the digital design loop is key for the architecture, engineering and construction industry. The MX3D robotic WAAM technology used to produce these connectors allows for full digital control over the design, production, timeline and cost with logistic benefits and integration with BIM applications. MX3D's MetalXL now enables fabricators to quickly start 3D metal printing large-scale metal applications in their own facilities.
The strong mechanical and material results from the research project confirm the relevance of robotic 3D metal printing for architecture, engineering and construction and open up a new generation of structural connections for the AEC industry.

3D printed series of connectors in duplex stainless steel.

Duplex stainless steel quality - ASTM A923 vs ISO 17781

Two standards are available for users when they are considering applying supplementary testing to duplex stainless steels: ASTM A923 and ISO 17781. ASTM A923 was a strong first attempt to formalise supplementary testing, while ISO 17781 is a newer contribution.

By Roger Francis, RF Materials & Glenn Byrne, Rolled Alloys

History

Modern duplex stainless steels were developed in the 1970s and really came into their own in the 1990s and onwards. They quickly became a corrosion resistant alloy (CRA) of convenience in the oil and gas industry for seawater cooling/firewater systems and mildly sour process fluids.

Duplex stainless steels (DSS) are roughly 50/50 austenite and ferrite and it is necessary to have good control of the composition and the heat treatment to obtain satisfactory properties in both phases. Poor control of these can lead to the precipitation of third phases, such as nitrides, sigma, chi and alpha prime. These are all deleterious and can reduce both toughness and/or corrosion resistance. Similarly, poor control during welding of DSS can also result in the precipitation of third phases and poor properties of the joint.

ASTM 'product' specifications and ASME fabrication codes do not include microstructure, impact and corrosion tests that individually and collectively indicate the presence of deleterious phases in these steels or their welded joints. Since the mid 1980s, more sophisticated users have developed their own 'material' and 'fabrication' specifications that call up these tests as supplementary requirements to the product specifications and weld procedure qualification codes in order to assess the quality of the steel and the procedures used to weld it. This was mostly satisfactory in the 1990s when the major users, such as oil companies, and the major engineering design houses were writing testing specifications for these alloys and insisting on compliance. However, the use of DSS spread to other industries, such as chemical process, desalination, power, mineral processing etc. As the market for DSS expanded, more manufacturers around the world began offering duplex alloys as part of their portfolio. Similarly, more fabrication shops were offering to weld DSS too. A number of these new users either do not realise the importance of supplementary testing, or they consider it an unnecessary cost. So they relied on ASTM and ASME requirements alone and manufacturers and fabricators supplied accordingly.

The result has been that poor quality duplex has been supplied to some projects, either as parent metal or as fabrications, which have failed prematurely, usually by corrosion. There have been numerous reports in the literature of failures of both 22%Cr duplex and superduplex due to poor quality material. Some of the failures due to poor quality have cost millions of dollars.

There are two standards available to the user that can be considered and decided upon when trying to apply supplementary testing to DSS: ASTM A923 and ISO 17781. ASTM A923 was a strong first attempt to formalise supplementary testing and ISO 17781 is a much more recent contribution.

ASTM A923

ASTM A923, is designed to detect sigma phase in 22%Cr duplex and superduplex[1]. It does not address nitrides or alpha prime. (Later a second standard was written, ASTM 1084, for lean duplex). It quickly became apparent that ASTM A923 had some serious drawbacks. Test A is an etched microsection, but the sample is only etched in NaOH, which will not show nitrides. In our opinion, some of the comparative micrographs in A923 described as "possibly affected" are in fact definitely "affected" and the concept of using Method A alone as a "rapid screening test" is seriously flawed. This is because of how intermetallic phases form during conventional heat treatment, and how failures in heat treatment processes can cause localised precipitation of intermetallic phases rather than widespread formation. It is also the case that metallographic examination can be subjective, especially for welds, and it is influenced by sample preparation, etching and metallographer interpretation. For these reasons taking the results of microstructure checks, impact tests and corrosion tests as a collective is the best way forward.

Test method B is a Charpy impact toughness test, at minus 40°C. The standard is clear that the toughness acceptance criterion for each grade is only used to detect the presence of unacceptable third phases, rather than the minimum toughness to suit the actual application. We find that the Charpy impact toughness test had a rather low pass/fail criterion[2], which would not necessarily reject material with low levels of intermetallic particles in the microstructure. Indeed, when the standard was developed, Davidson[3] showed that 2205 plates meeting the 54J at minus 40°C acceptance level had already suffered a considerable loss in toughness and also some loss in corrosion resistance. The 54J acceptance criteria was applied because it was "regularly used for a wide range of process applications other than cryogenic applications"[3], and not because it was found to be related to some significant presence of intermetallic precipitates (IMP) in the microstructure. Our own work for superduplex stainless steels[2] showed that an acceptance level of 70J at minus 46°C was rather more discerning with respect to the presence of intermetallic phases. The problem is that low levels of acceptance such as in ASTM A923 Method B and other specifications, can allow materials that are predisposed to further precipitation of IMP to be fabricated by welding. In such cases they could suffer further and rapid IMP precipitation in the low temperature HAZ[4] and then be deployed in service. In such cases, if pre-existing rather than new weld procedure qualifications (performed on the susceptible material) are used it is unlikely that the problem will be discovered until it is too late.

Finally, the corrosion test in ferric chloride solution, at 40°C was judged by some to be at too low a temperature for wrought and cast superduplex in the solution treated and water quenched condition, such that it could also pass material that might contain low levels of intermetallic phase. This was rectified to some extent by the inclusion of Supplementary Requirement S1 which gave the option of corrosion testing at 50°C. Again our own work on superduplex in the solution treated and water quenched condition shows a test temperature of 50°C to be much more discerning in terms of detection of intermetallic particles than testing at 40°C[2]. However, other contributors to the standard argued that the 50°C test temperature should be discretionary, applied only to the "arduous" applications rather than the "commodity" application that would be covered by the 40°C test temperature. In saying this, it is our experience that these grades are always deployed in arduous applications, so we find the concept of a commodity application difficult to rationalise.

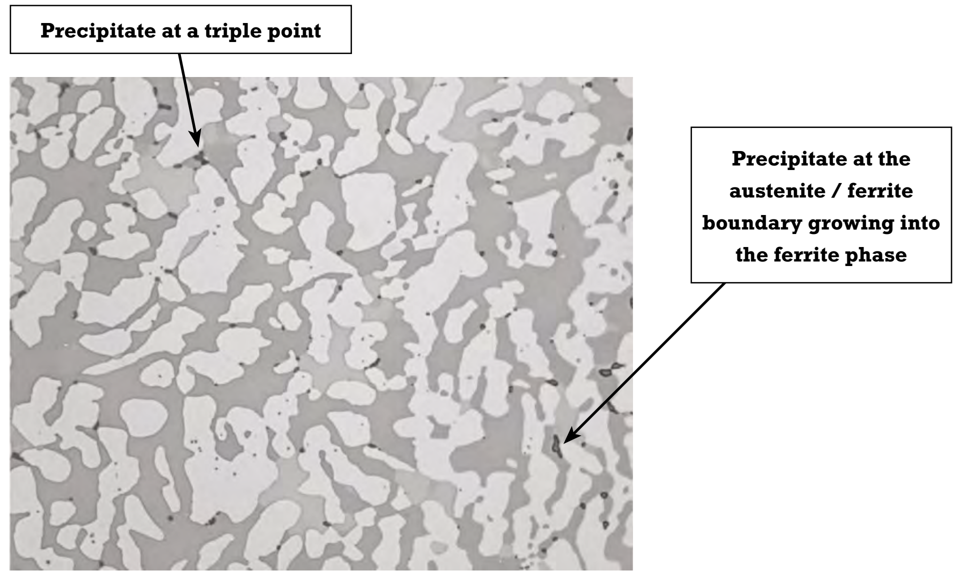


Figure 1. Showing typical locations of precipitates in a duplex stainless steel wrought microstructure.

ASTM A923 and welds

In addition, ASTM A923 does not address welds well; in particular, how to take samples, and this impacts on suitable pass/fail criteria. ASTM A923 allows testing of both machined flat, rectangular samples cut from welds, with polishing of all faces and also testing of samples with surfaces in the "as fabricated" condition and only cut edges polished. Both NORSOK[5] and TWI/ International Institute of Welding (IIW)[6] only require a cut out of the weld and polishing of cut faces, which means that both the as-manufactured and as-welded surfaces including the weld root run are tested. These areas are what a user wants to know about. The NORSOK requirement for a brief pickle prior to testing is supposed to remove test to test variability because it gave a distinct transition between the passive state and active pitting[7], but this might also remove some surface defects like poor pickling or areas of nitrogen loss in the root of welds where corrosion could initiate[2]. For this reason, the authors prefer to test the mill finished and as welded and cleaned condition (without pickling of the sample, unless pickling is going to be applied post welding).

These two test methods have different weight loss acceptance criteria too. ASTM 923 allows no more than 10mg/ decimeter^2/ day (10mdd), which equates to $1g/m^2$ in a 24-hour test while the TWI method has a maximum weight loss of $4g/m^2$. The lower weight loss in ASTM A923 is justified in terms of it being "discernable, measurable and not reliant on the subjectivity of visual determination of pitting"[3], but not in terms of presence of IMP's. When using ASTM A923 it is possible that all the as-manufactured faces have been removed by grinding and only the bulk metal is being tested. Whereas, when as-manufactured, or as-welded, surfaces are tested higher weight loss limits are appropriate[6]. It has been found that for weight losses exceeding $4g/m^2$ the rate of weight loss increases rapidly, indicating stable pitting. As such a weight loss of $4g/m^2$ and lower is considered acceptable. Further, the TWI method was developed on the basis of evaluating round robin testing of welds in a number of different laboratories.

As far as the authors are aware, no such testing has been done to justify the use of ASTM A923 Method C test for welds. Indeed, users have reported problems when trying to apply A923 corrosion test requirements as part of weld procedure qualification[8]. For this reason, the authors prefer the TWI method and its $4g/m^2$ weight loss limit.

ASTM A923 and its acceptance levels are based more on what a manufacturer would wish to supply rather than what an end user may need. When criticised because the test methods failed to detect problems associated with nitrides, ineffective pickling of parts, or nitrogen loss from the root runs of welds say, the custodians of the standard fall back on to the scope of the standard, arguing that it was dedicated only to the

About the authors

Roger Francis has been a corrosion engineer for 45 years, with 30 years spent largely on duplex stainless steels. He has published over 90 technical papers, many on corrosion of duplex stainless steels. He has written 6 books and co-edited two more. He is currently helping to write a guide to avoiding and solving corrosion problems for desalination plant engineers.

Glenn Byrne is a physical metallurgist with 35 years of experience in the metals industry. He has worked for multiple markets and industries around the world on the application, development and sales of duplex and super duplex in all product forms. He is Director of Technology & Projects at Rolled Alloys.

detection of intermetallic phases that cause significant loss in toughness or corrosion resistance, recognising that the test methods will not necessarily detect loss of toughness or corrosion resistance attributable to other causes. The standard has been modified over the years, but changes were slow to come about and did not always meet oil and gas companies' requirements. Hence, the oil industry users sat down to write a more robust standard based on their own requirements, under the auspices of ISO. This became ISO 17781[9], first issued in 2017.

What's in the ISO standard

The ISO standard addresses the quality of all grades of DSS, lean duplex, standard duplex, superduplex and hyper duplex, as well as welds of these alloys. It covers all major production routes, including wrought, cast and HIP. The document describes in detail how test samples should be taken, particularly for thicker section products, so that the tests represent the thickest material. The standard requires three different tests.

The first is a microsection and the standard says where and how it should be sectioned, and how it should be polished and etched. The most common etch is a two stage, in 10% oxalic acid and 20% to 40% NaOH or KOH. These etches will show nitrides, sigma and chi phases. This double etch must be specified, as a single NaOH etch is also an option. We find the double etch good for 22% and 25% Cr duplex grades. Outokumpu argue that the oxalic acid etch encourages transpassive attack, which exaggerates the apparent size of precipitates and causes "ditching" of grain boundaries, so they do not recommend it for etching of 2507. They recommend etch V2A, (50ml hydrochloric acid, 5ml nitric acid and 50ml water)[10].

The microsection is examined firstly at low magnification, scanning the whole area of the sample. Any areas that are then thought to be possibly affected are then examined at high magnification to confirm that they are not etching artefacts but are indeed third phases. This is usually done by considering the location of the particles. Intermetallics tend to precipitate at the austenite/ferrite grain boundaries and grow in to, and consume the ferrite phase. Locations like grain triple points and interdendrictic spacings in castings or welds are prime locations for precipitation. Figures 1 and 2 illustrate this.

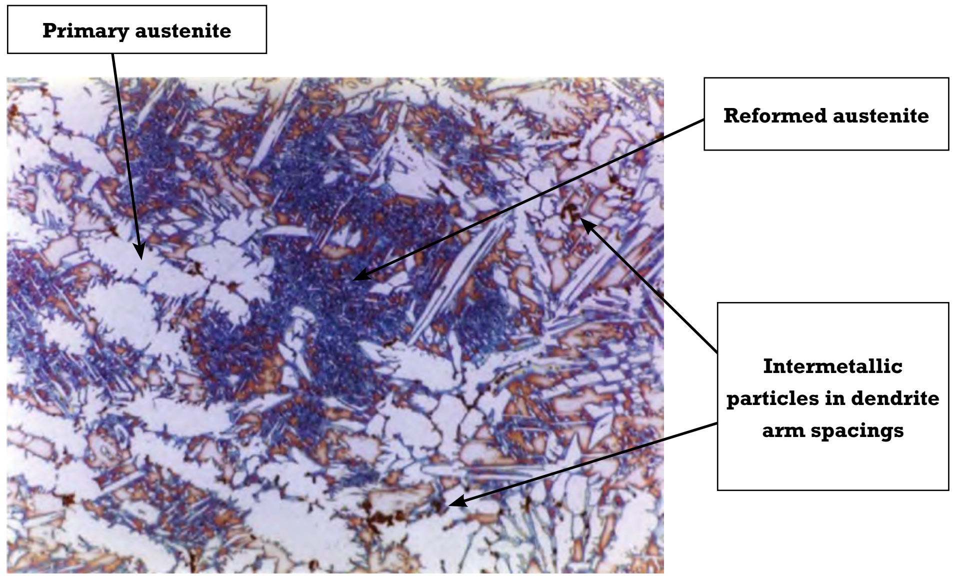


Figure 2. Showing typical location of precipitates in the root run of a duplex stainless steel weld.

The occasional third phase particle is not necessarily a fail, provided the material passes the other two tests. In these circumstances it is suggested that the microstructure of the corrosion and impact test samples be checked to ensure similitude between these and the original microstructure check sample. The microsection is also used to determine the phase balance, and the ferrite content must be in the range 35 to 60% for parent metal and 30 to 70% for welds in the as-welded condition.

The second test is a Charpy impact toughness test, with the test temperature specified for different duplex grades. The standard is also particular about where the samples are taken and their orientation. This is because the toughness varies significantly depending on whether it is orientated in a longitudinal or transverse direction with respect to the grain structure of the steel. For parent standard duplex and superduplex the test temperature is -46°C. The pass/fail criteria are specified for different product forms of the different grades, and some of these are split into two, with higher energies required for more demanding applications. As-welded joints have their own special requirements.

The third test is an ASTM G48 type corrosion test for higher alloys and an ASTM 1084 type for lean duplex alloys. The test temperature is specified for each grade and the pass/fail criteria include a maximum weight loss, in addition to having no visible pitting at x 20 magnification. This is because nitrides or ineffective acid pickling of the material can cause a high weight loss without showing any pitting. The standard also includes tests for as-welded welds with specific requirements on sample location and preparation, as well as different temperatures and pass/fail criteria for each grade. Table 1 summarises the test requirements

Table 1. Test requirements for some duplex stainless steel welds (taken from ISO 17781).

ALLOY		TEST		
		TOUGHNESS	CORROSION	MICROSECTION
Lean Duplex	Test	Charpy impact test at 20°C	ASTM A1084 for 24h+	Polished and double etched*
	Pass	45J Min average; 35J single minimum	No pitting at ·20; Wt. loss < 4g/m²	Ferrite 30 to 70%; No significant third phases
2205	Test	Charpy impact test at –46°C	ASTM G48 at 22°C for 24h	Polished and double etched*
	Pass	50J Min average; 40J single minimum	No pitting at ·20; Wt. loss < 4g/m²	Ferrite 30 to 70%; No significant third phases
Superduplex	Test	Charpy impact test at –46°C	ASTM G48 at 35°C for 24h	Polished and double etched*
	Pass	50J Min average; 40J single minimum	No pitting at ·20; Wt. loss < 4g/m²	Ferrite 30 to 70%; No significant third phases

** Double etching = electrolytic etching first in oxalic acid and then in NaOH.*

+ No temperature specified

for some duplex welds. The tests are the same for parent metal, but the test conditions and pass/fail criteria are higher, and they vary with the product form.

The importance of the tests

The tests in ISO 17781 were designed to prevent sub-standard material being supplied to the oil and gas industry. However, there have been failures of DSS due to poor quality in many other industries. The authors have seen failures in the desalination, mineral processing and chemical industries. The document is quite demanding in its testing requirements and pass/fail criteria, but this is based on the experiences of the oil and gas industry and what it takes to be sure that the alloy will not fail prematurely. Failures due to poor quality microstructures have cost tens of millions of dollars to rectify in some instances[11-13], so a little extra money spent up front on quality is felt to be justified.

Because of the experiences that drove this standard to be created, there is no reason for it not to be adopted by other industries when purchasing, or fabricating, duplex stainless steels.

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