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Sigma Phase Effect on Pitting Corrosion of Super Duplex Stainless Steel UNS S32750

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Abstract

The welding process on Super Duplex Stainless Steel material can cause decreased corrosion resistance of Super Duplex Stainless Steel material. This process occurs a lot in the heat treatment process piping and pipeline. This decrease in corrosion resistance occurs due to the formation of sigma phase precipitation due to heat treatment of the metal. This research will determine the relationship between the ratio of heat input cold pass: the ratio of heat input root pass to the percentage of sigma phase and the relationship between the percentage of sigma phase to the weight loss value. accompanied by the formation of pitting corrosion on UNS S32750 Super Duplex Steel. Test Coupon (TC) specimens obtained from as-welded with the ratio of heat input cold pass : ratio of heat input root pass (TC-1=74%; TC-2=70%; TC-3=68%; TC- 4=61 %) which was then tested for microstructure. The percentage of sigma phase obtained is TC-1 = 13.96%, TC-2 = 8.33%, TC-3 = 8.33%, and TC-4 = 4.38%). The corrosion resistance test yielded weight loss values (TC-1 = 14.67 gr/m2, TC-2 = 11.71 gr/m2, TC-3 = 1.27 gr/m2, and TC-4 = 2.71 gr/m2). This study concludes that the formation of the sigma phase will be directly proportional to the value of the root pass heat input ratio: the cold pass heat input ratio. The higher the percentage of sigma phase, the higher the weight loss value in the corrosion test, which will cause pitting corrosion.

Keywords: Physical metallurgy, super duplex stainless steel, pitting corrosion , sigma phase

Introduction

Increasing drilling volume of oil and gas from well free offshore platform and deep-water wells, will increase fabrication several materials, among others manifold and spools (Amatsuka et al., 2023)(Arun et al., 2019). material Which used For make modern equipment This must distinguished with enhancement productivity in condition corrosion And pressure Which high , For ensure operation safe and free from problem (Olsson & Snis, 2007). Steel Duplex Stainless Steel (DSS) and Super duplex Stainless Steel (SDSS) is steel stand rust Which the more Lots used And applied infield This Because give characteristic mechanic which is good For application industry oil and gas Good For manifold or spools , high toughness and strength, properties capable weld (weldability) Which more Good compared to with ferritic stainless steel pure, also have high resilience against stress corrosion cracking and cost alloy which low (Olsson & Snis, 2007)(Ramkumar et al., 2014)(Monier, 2019) (Department, 2019).

Duplex stainless steel (22% Cr) and Super duplex Stainless Steel (25% Cr) has combination resilience corrosion and characteristic mechanic which excellent(Liu et al., 2022). Duplex Stainless Steel (DSS) and Super Duplex Stainless Steel (SDSS) is defined bystructure micro ferrite - austenite balanced Between both DSS and SDSS, classification which own resilience corrosion which far more good that is Super Duplex Stainless Steel (SDSS)with mark 25%Cr(Department, 2019). Presence fraction balanced ferrite -austenite phase volume andexists element alloy like Cr, Ni And Mo in content which more big give resilience which more good to corrosion pitting (pitting corrosion) and corrosion crevices _ corrosion) from most steel stand austenitic stainless steel (conventional austenitic stainless steel)(Chan & Tjong, 2014).

Literature review showing the weldability and hot working which is good on Super Duplex Stainless Steel (SDSS)(Ramkumar et al., 2014). However showing something challenge during welding, two problems main Possible arise because evolution or change from structure micro on moment cooling achievement phase from austenite to ferrite which is not balancedbetween ratio And precipitation in zone fusion (FZ) And zone Which caught hot (HAZ) on phase secondary, generally can lower resilience corrosion and toughness alloy (Monier, 2019)[9]. Attempts to balancing microstructure so that weldability remains awake that is with control heat inputs welding(Giorjão et al., 2019)(Hosseini et al., 2018).

Temperature is main parameter from the welding process especially in Heat Affected Zone (HAZ) area and weld metal. Wrong one control temperature is guard parameters of heat input and cooling rate when welding agar transformation ferrite-austenite the phase still balanced (Shin et al., 2012). Control temperature which bad including heat inputs Which tall can cause precipitation form intermetallic phase (Zhang et al., 2017). The intermetallic phase is something precipitation on the microstructure that occurs in variation temperature certain . A number of type intermetallic phase is among them is Sigma phase that occurs at a temperature of 600˚ – 1000ºC , Chi phase happen on temperature 700º – - 900ºC , Nitride occurs at a temperature of 700˚ – 900ºC ,Carbides occur at temperature 550º - – 650˚C , And prime alpha happen on 475 ˚C (Paulraj & Garg, 2016)

This study will made as base several cases that occurred in the field, that was SDSS material which done splicing with welding method, however experience failure's when tested on Destructive Test and Non-Destructive test (NDT) with method ultrasonic testing (UT). from testing This found a number of disabled weld like lacking of inter run fusion and crack on area weld metal. By this study, author want to prove linkages temperature parameters like heat input with appearance Sigma precipitation that causes the occurrence of pitting corrosion which becomes root problem main happen disabled weld.

Methodology

Inductive approach done in this research method. Method study done with learn literature and exists the facts that happened, the problem statement that later will compared to suitability and singularity between results study with theory as well as existing research _ previously so that can pulled A analysis and conclusion . Research Method can

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describe in flowchart in Figure 1.

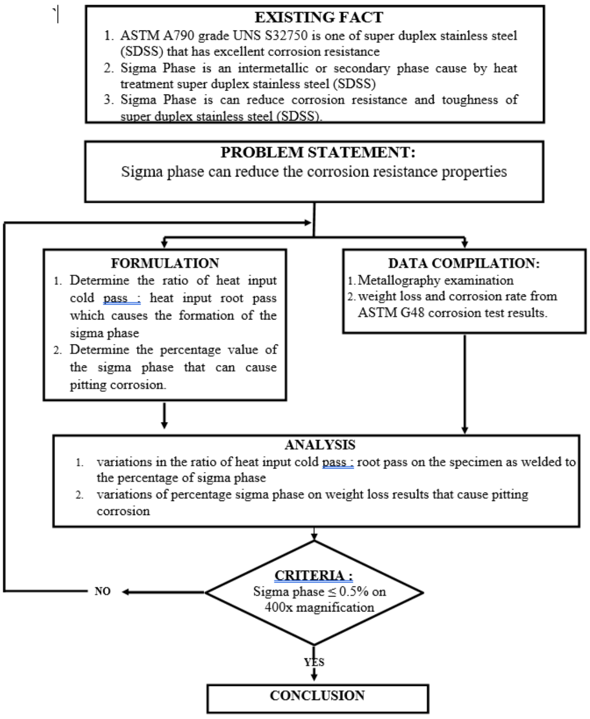

Figure 1. Research Methodology

Sample

Test coupon samples is made from welded specimen from SDSS(UNS S32750) alloy pipe material through Gas tungsten arc welding (GTAW) welding processed.

Details sample results welding showed on table 3.1 and figure 3.1.

Table 3.1 As Welded Specimen

No	*As Welded* Specimen Details	
1	**Material Type**	SDSS (pipe)
2	**Material Grade**	UNS S32750
3	**Diameter**	8"
4	**Thickness**	32.5 mm
5	**Joint Design**	Singe Vee Goove
6	**Filler Metal**	A5.9 ER2594

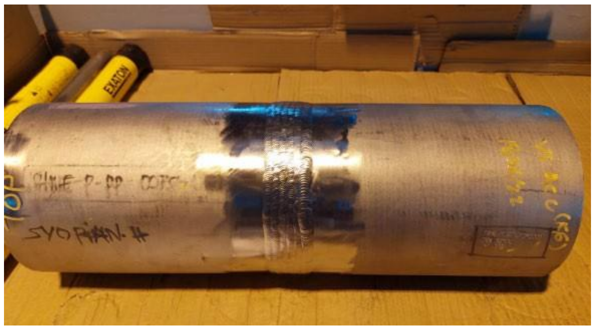

Figure 3.1 US welded specimen

Destructive Test (DT)

After NDT testing ,specimen US welded done cutting be a test coupon through machining process. 2 (two) samples will examined microstructure testing and corrosion test (G-48).

a. Corrosion testing

be prepared 4 test coupons Which Already finished done machining with each the dimensions of the test coupon are 25 mm x 50 mm. Following under This is steps testcorrosion G-48 with method A (ASTM, 2003):

i. Measurement dimensions test coupons.

ii. Pickling, in the pickling test coupon process dipped to in solution 20% HNO_3 + 5%HF with temperature of 60°C for 5 minutes . After the test coupon is washed and cleanedwith sodium hydroxide . After clean , tests coupon entered to in glass measure which Already filled solution propanol Then put it inUT cleaner during 5 minutes . So clean up Againwith sodium hydroxide .

iii. After pickling done , test coupons in passivation in in incubator during 1 x 24 O'clock

iv. weighing before loading. Test coupons Which Already in pickling And passivation Then done weighing test coupons And record mark the weight as much 5 time taking .

v. Immersion / loading. Pour it 100 gr $FeCl_3.6H_2O$ (ferric chloride hexahydrate) to in glass measuring Which Already filled with 900mlaquades Then put it test coupons to in glass measuring the And insert glass measuring theto in labtech waterbath with temperaturestandard 40°C ± 2 °C during 1x 24 O'clock.

Figure 3.2 Immersion / loading

1. Unloading. Appointment test coupon from in labtech waterbath Then take it out testcoupons from in glass measure .

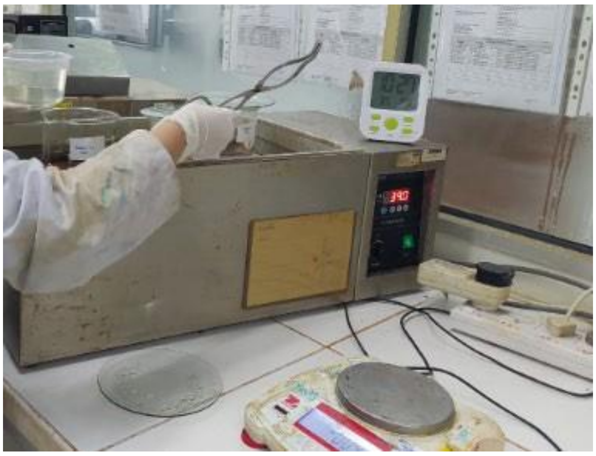

Figure 3.3 Unloading

2. Wash And clean up test coupons with sodium oxide.

Figure 3.4 Cleaning of Coupons Test

3. Dry the test coupon with a hair dryer so No There is Again fluid Which stick ontest coupons.

Figure 3.5 Drying of specimens

4. weighing after unloading. Test coupons already in the immersion Then weighed return and record value the weight Lots 5 time taking .

Figure 3.6 Process weighing (after testing)

5. Observation done with see visuals on the surface and weld root area test coupon use microscope with enlargement 20X.

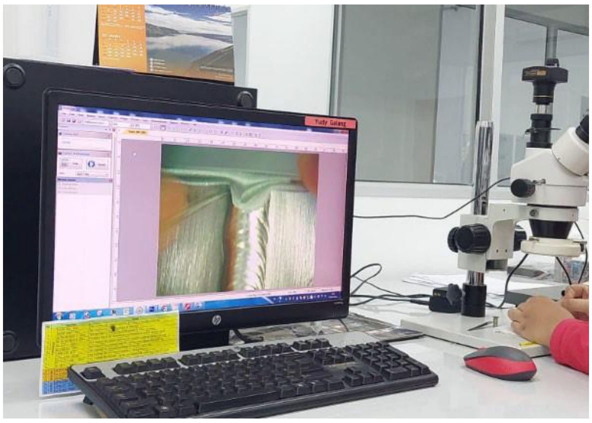

Figure 3.7 Observation on microscope enlargement 20X

6. Weight loss calculation.

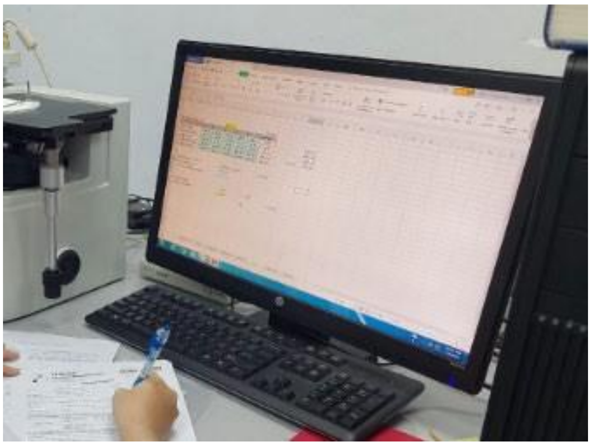

Figure 3.8 corrosion rates Calculation

b. Metallography examination

4 (four) test coupons samples which already finished done machining with each the dimensions of the test coupon are 25 mm x 50 mm. Following under This is steps test microstructure :

i. Polishing (sandpaper). Rubbing test coupons use sandpaper with order sandpaper from Which most rough until most smooth , namely grid 400, grid 600, grid 800, grid 1200, grids 2000
ii. Polishin (cloth). Rubbing test coupon use finished cloth _ sprayed diamonds paste until No There is Again scratches on appearance test coupons Which will in test.
iii. etching. Etching test coupon with 10% NaOH use electrolysis process until seen profile welds on test coupons.
iv. Wash and clean the test coupon using ethanol .
v. Observation . See structure micro on test coupons use microscope .
vi. Calculation phase ferrite. On process this , If visible sigma phase in the calculation process phase ferrite so stage furthermore is count return ferrite phase with the grid configuration use method square grids.

vii. Research Results and Analysis

Metallography test

Phase identification in the results of the appearance of the microstructure is determined based on previous literature that has been done by Moon [13]. The presentation value of the formation of the sigma phase is determined from the metallographic software.

1. Coupons test 1

On observation apparition structure micro with 400X magnification for the test coupon 1 shown in Figure 3.9 area of *the weld root* and in figure 3.10 on *HAZ root* found sigma phase with percentage 13.96%.

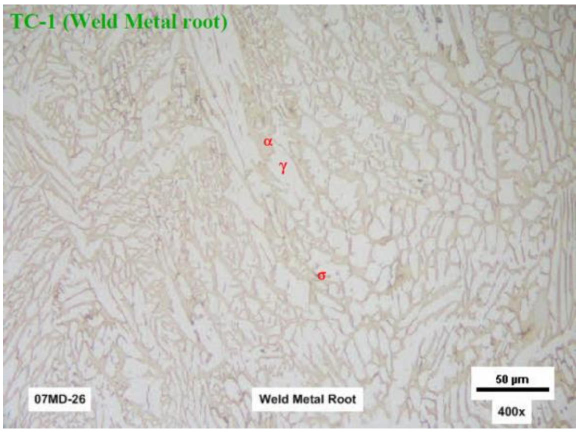

Figure 3.9 Microstructure of *weld root area*

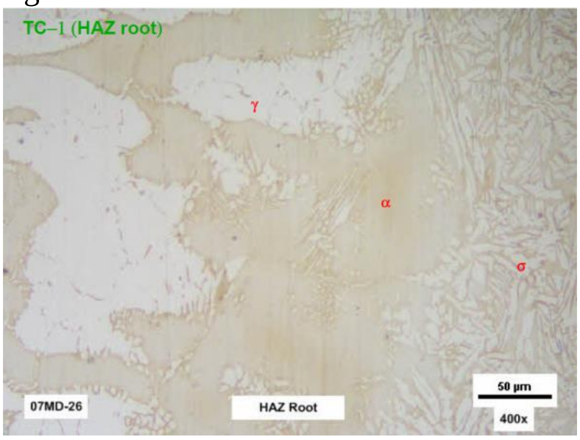

Figure 3.10 Microstructure of *HAZ area root*

2. Coupons test 2

On observation apparition structure micro with 400X magnification for the test coupon 2 shown in Figure 3.11area of *the weld root* and in figure 3.12 on *HAZ root* found sigma phase with percentage 8.33%

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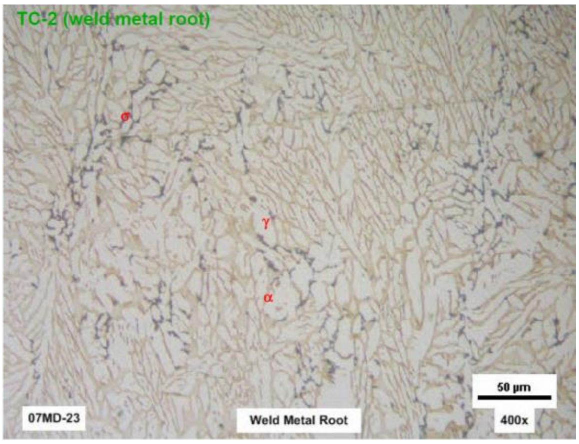

Figure 3.11 Microstructure of *weld root area*

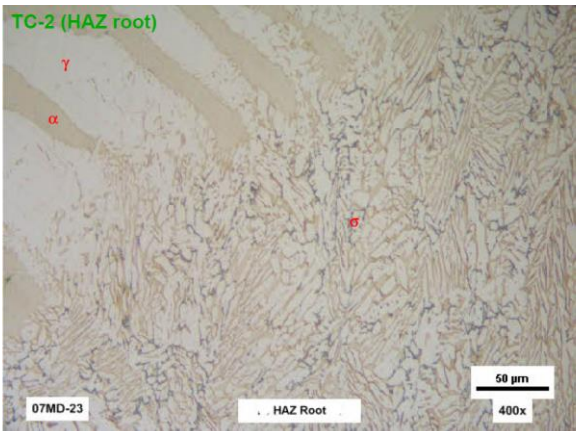

Figure 3.12 Microstructure of *HAZ area root*

3. Coupons test 3

On observation apparition structure micro with 400X magnification for the test coupon 3 shown in Figure 3.13area of *the weld root* and in figure 3.14 on *HAZ root* found sigma phase with percentage 8.33%.

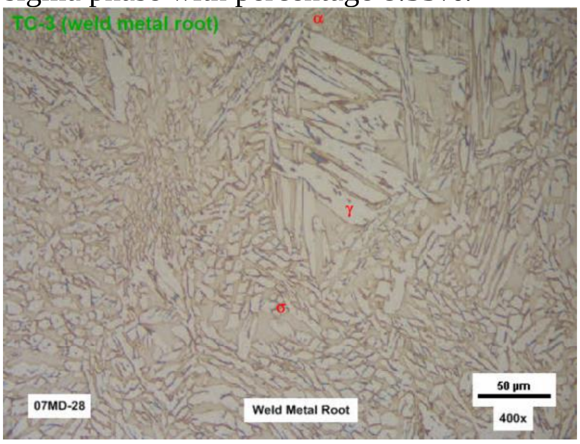

Figure 3.13 Microstructure of *weld root area*

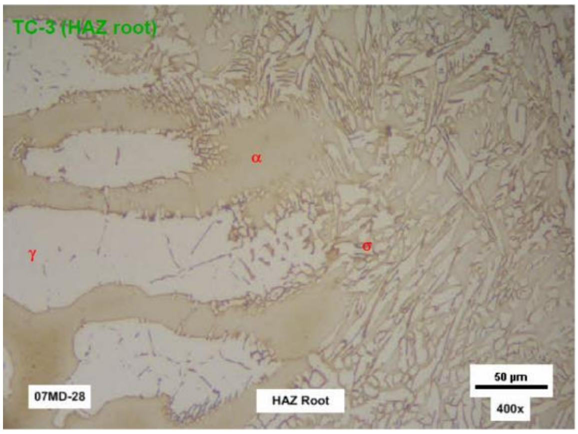

Figure 3.14 Microstructure and results interpretation in the *HAZ area root*

4. Coupons test 4

On observation apparition structure micro with 400X magnification for the test coupon 4 shown in Figure 3.15area of *the weld root* and in figure 3.16 on *HAZ root* found sigma phase with percentage 4.38%

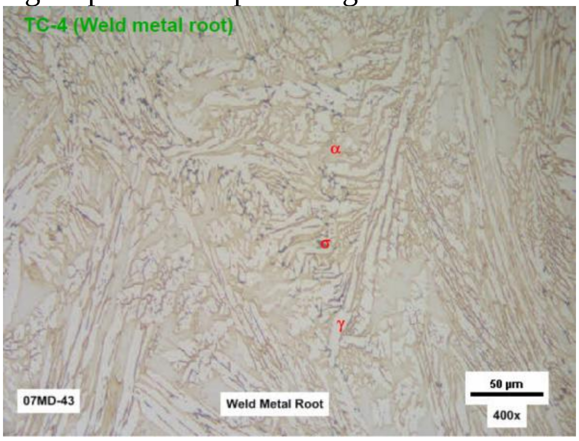

Figure 3.15 Microstructure and resultsinterpretation in the *weld root area*

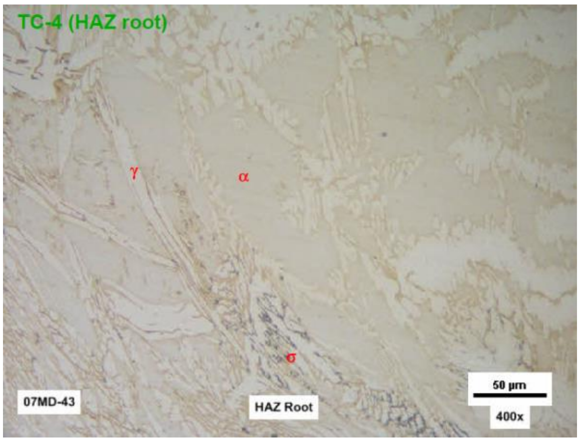

Figure 3.16 Microstructure and results interpretation in the *HAZ area root*

From the results apparition structure micro seen that Ratio heat input cold pass compared with heat input root pass ratio will be influence mark or amount from big sigma phase precipitation . this _ in accordance with references that have conducted by researchers previously for other super duplex materials (Hosseini et al., 2018)(Zucato et al., 2002)(Giorjão et al., 2019), where more large heat input will be enlarge amount from sigma phase precipitation .

Corrosion test

Corrosion test was carried out based on the ASTM G-48 procedure previously described, with the following results:

1. Coupons Test 1

On results observation before And after *immersion* on test coupons 1 generated there is *pitting* at 20x magnification . Can seen on table 3.2 as following .

Tabel 3.2 Hasil Observasi Uji Korosi Test Coupon 1

Data spesimen	Ukuran spesimen					Rata-rata
	1	2	3	4	5	
Berat sebelum tes (g)	408.025	408.025	408.025	408.024	408.024	408.025
Berat setelah tes (g)	407.891	407.892	407.891	407.891	407.891	407.891
Luas area (mm^2)	9090.346					
Kehilangan berat (g)	0.13					
Kehilangan berat (g/m^2)	14.67					

Ringkasan Uji Korosi *Pitting*	Observasi
Persiapan permukaan menggunakan larutan pickling 20% HNO3 + 5% HF pada temperatur 60˚C dalam waktu 5 menit dan diikuti/dilanjutkan dengan proses pasivasi diudara terbuka selama 24jam. Korosi pitting dilakukan pada area spesimen girth weld, mengacu pada/sesuai dengan specification ASTM G48 Method A. Sampel telah diuji selama 24 jam direndam dalam larutan FeCl3.6H2O (ferric chloride hexahydrate) mengacu pada / sesuai dengan ASTM G48 paragraf 6.1 pada temperatur konstan 40˚C ± 2˚C	Korosi pitting terdapat pada area tertentu (concerned area) diamati pada perbesaran 20x

Acceptance Criteria	*Result*
1. Tidak ada pitting dalam pembesaran 20x 2. Kehilangan berat/weight loss tidak boleh lebih dari 4.0 g/m2	*Failed*

2. Coupons Test 2

On results observation before And after *immersion* on test coupons 2 generated there is *pitting* at 20x magnification . Can seen on table 3.3 as following .

Tabel 3.3 Hasil Observasi Uji Korosi Test Coupon 2

Data spesimen	Ukuran spesimen					Rata-rata
	1	2	3	4	5	
Berat sebelum tes (g)	346.342	346.342	346.342	346.342	346.342	346.342
Berat setelah tes (g)	346.247	346.248	346.248	346.248	346.248	346.248
Luas area (mm^2)	8042.729					
Kehilangan berat (g)	0.09					
Kehilangan berat (g/m^2)	11.71					

Ringkasan Uji Korosi *Pitting*	Observasi
Persiapan permukaan menggunakan larutan pickling 20% HNO3 + 5% HF pada temperatur 60˚C dalam waktu 5 menit dan diikuti/dilanjutkan dengan proses pasivasi diudara terbuka selama 24jam. Korosi pitting dilakukan pada area spesimen girth weld, mengacu pada/sesuai dengan specification ASTM G48 Method A. Sampel telah diuji selama 24 jam direndam dalam larutan FeCl3.6H2O (ferric chloride hexahydrate) mengacu pada / sesuai dengan ASTM G48 paragraf 6.1 pada temperatur konstan 40˚C ± 2˚C	Korosi pitting terdapat pada area tertentu (concerned area) diamati pada perbesaran 20x

Acceptance Criteria	*Result*
1. Tidak ada pitting dalam pembesaran 20x 2. Kehilangan berat/weight loss tidak boleh lebih dari 4.0 g/m2	*Failed*

3. Coupons Test 3

On results observation before And after *immersion* on test coupons 3 generated there is *pitting* at 20x magnification . Can seen on table 3.4 as following .

Tabel 3.4 Hasil Observasi Uji Korosi Test Coupon 3

Data spesimen	Ukuran spesimen					Rata-rata
	1	2	3	4	5	
Berat sebelum tes (g)	364.665	364.665	364.664	364.664	364.664	364.664
Berat setelah tes (g)	364.654	364.654	364.654	364.653	364.654	364.654
Luas area (mm^2)	8308.421					
Kehilangan berat (g)	0.0106					
Kehilangan berat (g/m^2)	1.27					

Ringkasan Uji Korosi *Pitting*	Observasi
Persiapan permukaan menggunakan larutan pickling 20% HNO3 + 5% HF pada temperatur 60˚C dalam waktu 5 menit dan diikuti/dilanjutkan dengan proses pasivasi diudara terbuka selama 24jam. Korosi pitting dilakukan pada area spesimen girth weld, mengacu pada/sesuai dengan specification ASTM G48 Method A. Sampel telah diuji selama 24 jam direndam dalam larutan FeCl3.6H2O (ferric chloride hexahydrate) mengacu pada / sesuai dengan ASTM G48 paragraf 6.1 pada temperatur konstan 40˚C ± 2˚C	Korosi pitting terdapat pada area tertentu (concerned area) diamati pada perbesaran 20x

Acceptance Criteria	*Result*
1. Tidak ada pitting dalam pembesaran 20x 2. Kehilangan berat/weight loss tidak boleh lebih dari 4.0 g/m2	*Failed*

4. Coupons Test 4

On results observation before And after *immersion* on test coupons 4 generated there is *pitting* at 20x magnification . Can seen on table 3.5 as following .

In testing corrosion seen that lost heavy smallest happened on the TC 3 coupon test where mark heavy of 1.27 g/m ^{2} . this value can made reference in the process of selecting operating parameters welding Where recommended ratio _ between heat input cold pass: heat input root pass ratio of 68%. as explained by Rini Riastuti (2022) and Da Fonsesca et al (2017), conditions This can happen due to the heat input ratio kinetics the formation of the sigma phase will reduce (Bashari R Roszardi1), Rini Riastuti1), Wahyu Budiarto1), Nono Darsono2), 2022) (Da Fonseca et al., 2017). Amatsuka et al explain that formation clamp corrosion started with mechanism initiation that has connection with the ratio of the heat input is . (Amatsuka et al., 2023)

Tabel 3.5 Hasil Observasi Uji Korosi Test Coupon 4

Data spesimen	Ukuran spesimen					Rata-rata
	1	2	3	4	5	
Berat sebelum tes (g)	297.633	297.633	297.632	297.632	297.633	297.633
Berat setelah tes (g)	297.613	297.613	297.613	297.612	297.612	297.613
Luas area (mm²)	7393.102					
Kehilangan berat (g)	0.02					
Kehilangan berat (g/m²)	2.705					

Ringkasan Uji Korosi *Pitting*	Observasi
Persiapan permukaan menggunakan larutan pickling 20% HNO3 + 5% HF pada temperatur 60˚C dalam waktu 5 menit dan diikuti/dilanjutkan dengan proses pasivasi diudara terbuka selama 24jam. Korosi pitting dilakukan pada area spesimen girth weld, mengacu pada/sesuai dengan specification ASTM G48 Method A. Sampel telah diuji selama 24 jam direndam dalam larutan FeCl3.6H2O (ferric chloride hexahydrate) mengacu pada / sesuai dengan ASTM G48 paragraf 6.1 pada temperatur konstan 40˚C ± 2˚C	Korosi pitting terdapat pada area tertentu (concerned area) diamati pada perbesaran 20x

Acceptance Criteria	*Result*
1. Tidak ada pitting dalam pembesaran 20x 2. Kehilangan berat/weight loss tidak boleh lebih dari 4.0 g/m2	Failed

Influence ratio *heat input cold pass* is divided*heat input root pass* against percentage *sigma phase.*

Done test structure micro For know percentage *sigma phase* based on ratio of HI *cold pass* : HI *root pass*who have displayed in form chart on **Figure 4.1**

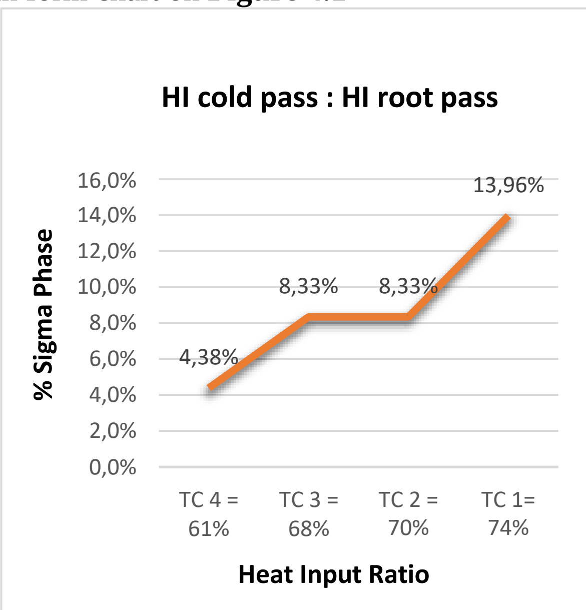

Figure 4.1 Comparison ratio *heat inputcold pass* : *heat input root pass* against percentage *sigma phase*

Found in results chart that *test coupons (TC)* with percentage *sigma phase* highest as big 13.96% got from comparison ratio HI *cold pass* : HI *root pass*with mark percentage the ratio is 74%. Second highest with results percentage *sigma phase*8.33% got from comparison ratio HI *cold pass* : HI *root pass* with mark percentage the ratio 70%. Third highest with results percentage *sigma phase* 8.33% got from comparison ratio HI *cold pass* :HI *root pass* with mark percentage the ratio 68%. For mark percentage *sigma phase* Lowest 4.38% got from comparison ratio HI *cold pass* : HI *root pass* with markpercentage the ratio is 61%.

From research previously carried out by D. Arun, et al (2019)(Koli et al., 2023) showed that ratio treatment hot will cause balance optimal phase of ratio achieved austenite and ferrite _ where the process parameters are adequate will influential to formation sigma phase (Arun et al., 2019)

Influence percentage *sigma phase* to mark *weight loss* from endurance test results corrosion (ASTM G-48).

Endurance test carried out corrosion based on ASTM G-48 (ASTM, 2003) from the same test *coupon* from test microstructure Which found *sigma phase*so comparison percentage *sigma phase* to mark *weight loss* is displayed in form graph in **Figure 4.2**

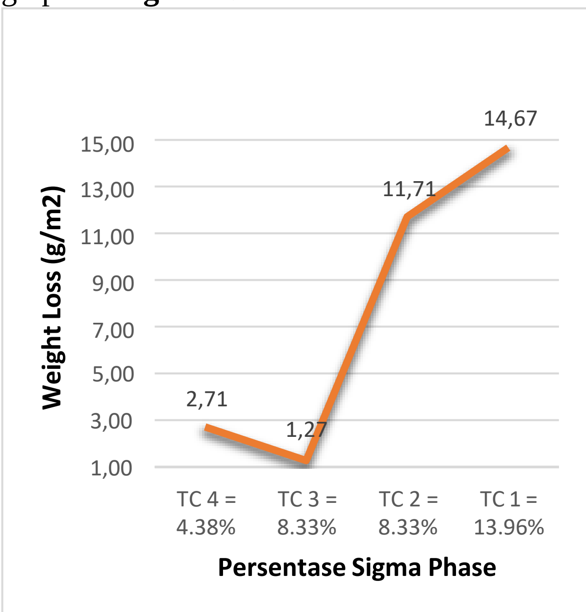

Figure 4.2 Comparison of sigma phase to weight loss value

Chart 4.2. showed that highest weight

loss as 14.67 g/m^2 with mark percentage *sigma phase* 13.96% which is also mark highest *sigma* phase compared to TC other . Highest second on TC2 have mark *weight loss* of 11.71 g/m^2 with mark percentage *sigmaphase* 8.33%. However on TC 3 have mark *weight loss* which Lowest as big 1.27 g/m^2 with mark percentage same *sigma phase* with TC2 ie 8.33% and TC4 has weight loss as 2.71 g/m^2 with mark percentage lowest *sigma phase* by 4.38%. as explained matter because kinetics establishment of sigma phase in ratio the reduce (Bashari R Roszardi1), Rini Riastuti1), Wahyu Budiarto1), Nono Darsono2), 2022)(Da Fonseca et al., 2017)(Reis et al., 2000). Possibility decline sigma phase formation as happened to the TC3 too, caused mechanism of the iterative process, where with the existence of a long process can also occur annealing process of the material in which the sigma phase is formed can solution treatment occurs, as explained by Wei Shen, et al (2021)(Shen et al., 2021)

Conclusion

1. Mark average HI or ratio comparison HI on cold pass must 75% more low compared with heat input on root passed . Because if pass or approach mark 75% so will potentially formation precipitation form sigma phase. this _ caused Because If mark heat inputs on cold pass from mark heat inputs on root pass will result root pass experience warmup return Which characteristic sudden And level possibility formation sigma phase will the more fast .
2. Cold pass HI ratio value : HI root ratio pass compared straight with mark percentage sigma phase. The more tall HI cold pass ratio : HI root pass ratio , so will the more tall mark percentage sigma phase.
3. The value of sigma phase not linier straight compared to corrosion loss. intermittent pitting that occurs in coupon so that weight loss becomes slower. However, it can be concluded that sigma phase will experience weight loss.
4. Every TC that experiences weight loss , then the TC Certain formed hole form pitting on site toe of root passed.
5. further research is needed for treatment resilience corrosion from Super duplex Stainless Steel UNS S32750, due to this sigma phase. preventative steps that can be taken done is use mechanism necessary post- treatment researched more continue .

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