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Methods to Avoid Material Sensitization During Welding for Developing Corrosion Resistant Exhaust System

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***Abstract*—Manufacturing technology, as a process, revolves around material and material management. The choice of material based on its applications and the necessity of the performance plays as the major clause for manufacturing technology. Material selection itself has many constrains of which corrosion resistance, weld ability are critical in choice of material to suit needs. There are many factors to be considered during choice of material for welding like base material service temperature, compatibility to filler material, shielding gas, weld parameter compatibility - voltage, current and temperature, after weld treatment and cooling methodology etc. These parameters when not chosen to optimum suit, will lead to corrosion or material fracture. One such corrosion to be avoided is inter-granular corrosion near weld zone commonly termed as sensitization.**

Sensitization is the phenomenon during which carbides rich in chromium (M23C6 – 65% Cr) are formed along the grain boundaries during welding. Selection of base material, filler material, welding conditions and after weld treatment remains a prime clause to avoid this phenomenon. At conditions there will be restrictions to use materials that are prone to such corrosion and at such times after weld treatment are essential to avoid inter-granular corrosion (Sensitization). This paper explains the iterations carried out on 20 different welded samples prepared by result of DOE method. Experimentations are conducted to check the combinations that are susceptible to inter-granular corrosion near heat affected zones after welding. Paper also discusses about the after treatment methods that shall avoid sensitization.

I. Introduction

Sensitization is the loss of alloy integrity. It results from chromium depletion in the vicinity of carbides precipitated at grain boundaries. Sensitization occurs during welding or annealing after cold-working. Sensitization happens when SS Steel is heated at temperatures between 425C – 815C.

At this condition, carbides of chromium are formed near grain boundaries. The precipitation occurs because the carbides are insoluble at these temperatures. Sensitization promotes stress corrosion cracking failure in some stainless steels.

Sensitization often occurs in austenitic steels but however ferritic steels, Martensitic steels are also prone to this phenomenon.Any sensitized microstructure will undergo selective localized corrosion along grain boundaries leading to inter granular corrosion.

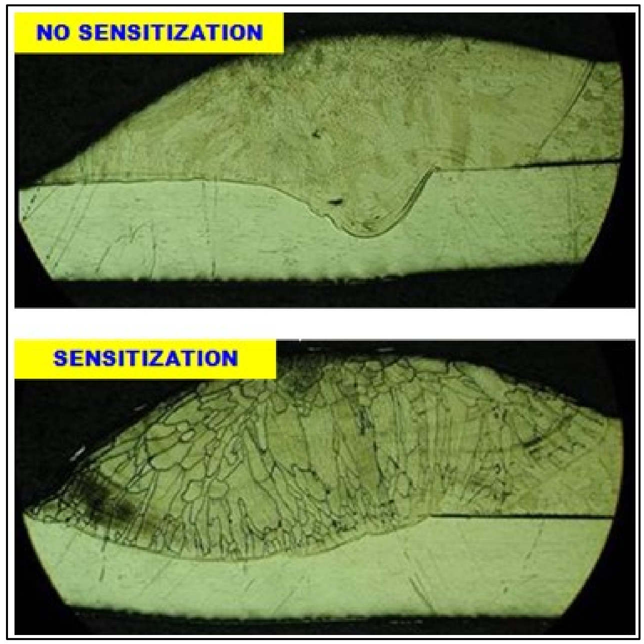

Figure 1: Intergranular Stress Corrosion

Sensitization is a phenomenon induces during welding and there are prevention and control measures to avoid or reduce this occurrence during manufacturing. To identify chromium depletion, there is a requirement of a specialized micro structural study of the welded material. Oxalic acid etch test is a rapid observation method of screening specimens for susceptibility to inter angular attack associated with chromium carbide participates. The test is used for acceptance but not for rejection of material.

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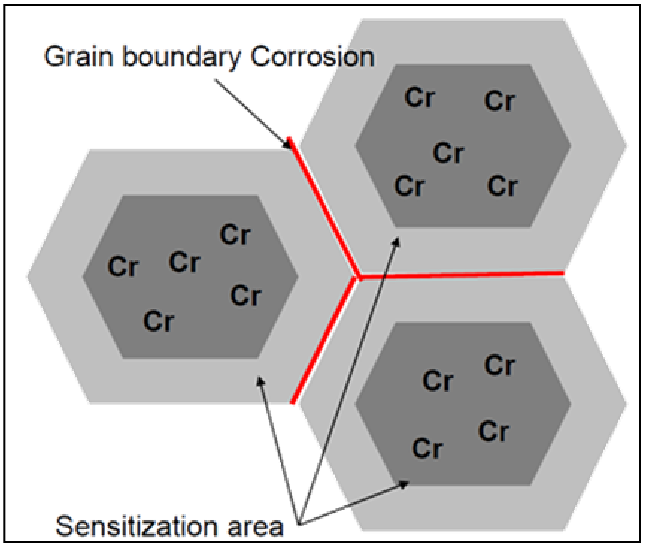

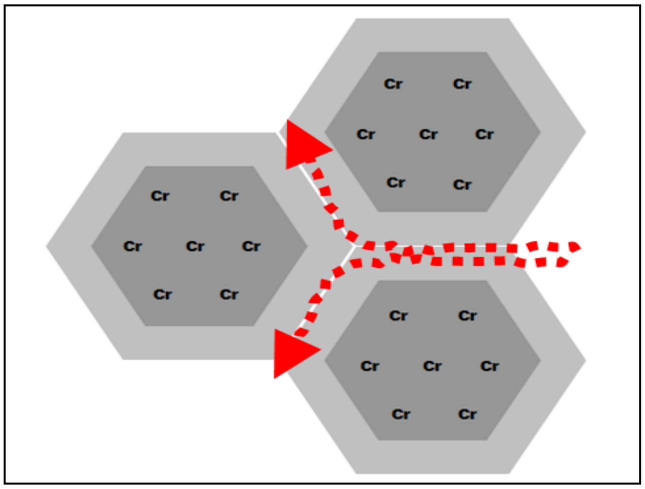

Figure 2: Stress cracking near HAZ

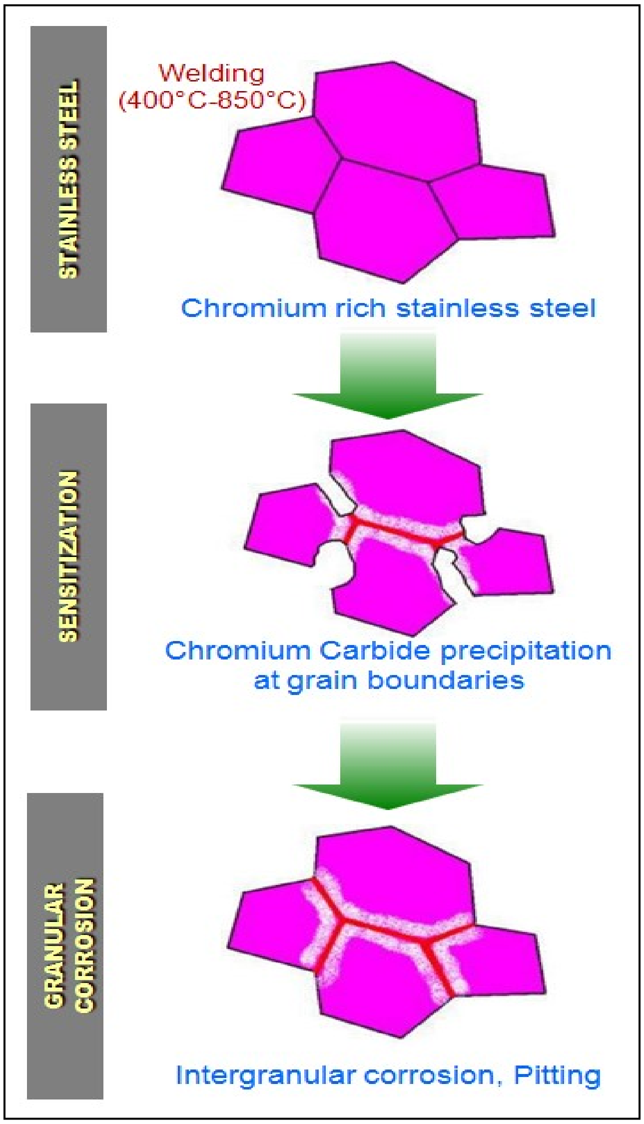

Figure 3: Stage wise propagation of granular corrosion

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Stainless Steels

Stainless Steels (SS): Defined as Iron-base alloy containing>10.5%Cr&<1.5%C.

Austenitic stainless steels typically have 16-26% chromium (Cr) and 8-22% nickel (Ni).

Ferritic stainless steels are iron-chromium alloys with body-centred cubic crystal structure having chromium content.

Martensitic stainless steels, which have chromium content between 12 and 18% with 0.15– 0.30% carbon.

Ferritic – austenitic (Duplex) stainless steels, which contain 18–25% chromium, 3–5% nickel and up to 3% molybdenum.

Martensitic-austenitic steels, which have 13–16% chromium, 5–6% nickel and 1–2% molybdenum.

Table 1
SS defined as Iron-base alloy containing > 10.5% Cr &< 1.5%C

Chemical Properties	Major families of Stainless Steel
Fe, <0.12% C	Austenitic Stainless Steel
16-18% Cr	Ferritic Stainless Steel
<0.75% Ni	Martensitic Stainless Steel
<1.0% Mn	Precipitation-hardening Stainless Steel
<1.0% Si	Duplex ferritic-austenitic Stainless Steel
<0.040% P	Austenitic Stainless Steel
<0.030% S	Ferritic Stainless Steel

Effects of Sensitization

1. Sensitization causes inter granular corrosion
2. Carbide precipitation removes Cr to <12% (Sometimes ~ 5%) – passive film is lost
3. M23C6 must not form, to prevent sensitization – use stabilized or L grades
4. N delays sensitization kinetics

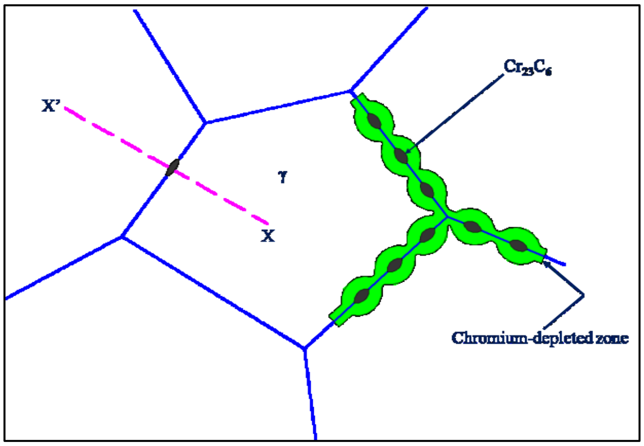

Figure 4: Intergranular corrosion observed @ temperatures between 400°C - 900°C

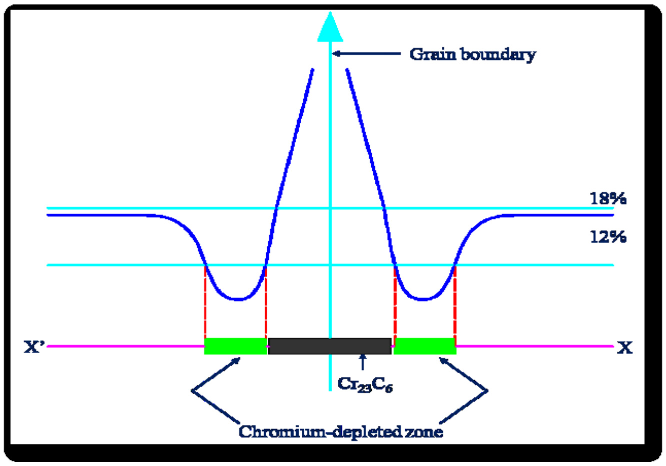

Figure 5: Chromium depletion due to sensitization @ temperatures between 400°C - 900°C

Austenitic Steel

- Austenitic stainless steels typically have 16-26% chromium (Cr) and 8-22% nickel (Ni).
- Type 304, which contains approximately 18%Cr and 10%Ni, is a commonly used alloy for welded fabrications and these alloys can be readily welded using any of the arc welding processes (TIG, MIG, MMA and SA).

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- They exhibit good toughness because they are non-hardenable on cooling, and there is no need for pre- or post-weld heat treatment.
- Chromium nickel alloys this is high temperature oxidation

Resistance

- High temperate strength: Carbon, nirogen, niobium and molybdenum.
- The Carbon levels of austenitic stainless steels are always relatively low, so strain-induced martensite is self-tempering and not brittle

Material Chemical Composition

- The chemical composition of the base metal and weld wires are compared.
- The 430LNb weld wire compositions compared with the SUS409, SUS441 and 308.

Ferritic Stainless Steel

- Ferritic stainless steels are iron-chromium alloys with body-centred cubic crystal structure having chromium content.
- Ferritic stainless steels have a Cr content of 11-28% with a carbon content below 0.1% .
- These steel exhibit good ductility, formability and moderately better yield strength, but the high temperature strength is somewhat poor

Conventional Availability of Steel

Table 2:
Classification of SS and its composition

Martensitic Stainless Steel	Martensitic stainless steels, which have chromium content between 12 and 18% with 0.15– 0.30% carbon
Ferritic - austenitic (Duplex) stainless steels	Ferritic-austenitic (Duplex) stainless steels, which contain 18–25% chromium, 3–5% nickel and up to 3% molybdenum
Martensitic - austenitic steels	Martensitic-austenitic steels, which have 13–16% chromium, 5–6% nickel and 1–2% molybdenum.

Choice of shielding gas:

Should use "$Ar+O_2$" instead of "$Ar+CO_2$"

Surface cleansing and prevention from oil & Dirt

Washing base metal before welding and cleanse it dirt & oil free before welding

Table 3:
Suitable conditions

PARAMETERS	CONDITION
Metal thickness (mm)	1.5
Filler wire diameter (mm)	1
Voltage (V)	30
Current (A)	225
Welding speed (cm/min)	208
Wire speed (cm/min)	965
Heat input (kJ/cm)	1.95
Shielding gas	EN 439-M3 (Ar+2% O2)
Back protection	EN 439-11(Pure argon)
Gas flow (L/min)	8

Root Cause - Precipitation of carbides and nitrides

Carbon is normally considered as an undesirable impurity in austenitic stainless steel . While it stabilizes the austenitie structure, it has a great thermodynamic affinity for chromium. Because of this affinity, chromium carbides, M23 C6, Form whenever carbon reaches levels of super saturation in austenite, and diffusion rates are sufficient for carbon and chromium to segregate into precipitates. The solubility of carbon in austenite is over 0.4% at solidification but decreases greatly with decreasing temperature. The solubility is given by;

$$\log\,(\mathrm{C\ ppm}) = 7771 - \frac{6272}{T\,(^{\circ}\mathrm{K})}$$

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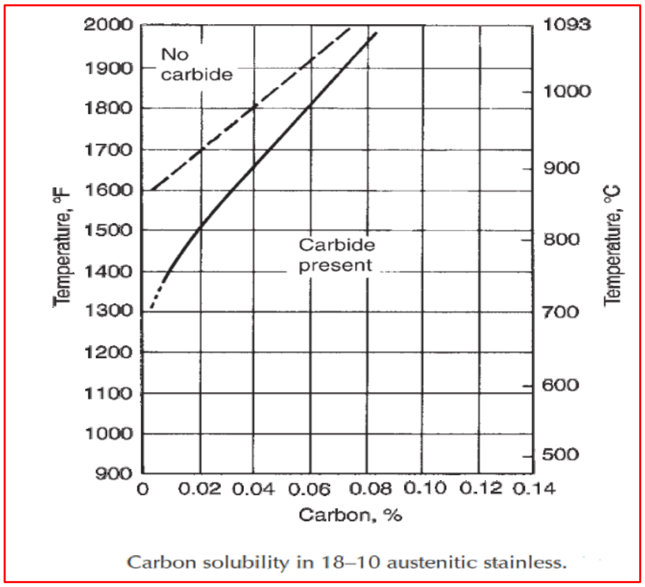

Figure 6: Equilibrium diagram for carbon in a basic 18%Cr 10% Ni alloy.

At room temperature, very little carbon is soluble in austenite; even the 0.03% of L grade is mostly in a supersaturated solution. The absence of carbides in austenitic stainless is due to the slow diffusion of carbon and the even slower diffusion of chromium in austenite. At a carbon level of 0.06%, which is found in most 304, super saturation increases exponentially, while diffusion decreases exponentially. These results in precipitation rates vary with temperatures. Grain boundary diffusion is much more rapid than bulk diffusion and grain boundaries provide excellent nucleation sites. This phenomenon induces precipitation along grain boundaries. Carbon diffuses several orders of magnitude more rapidly than Chromium essentially in situ, depleting the grain boundaries of chromium in solution.

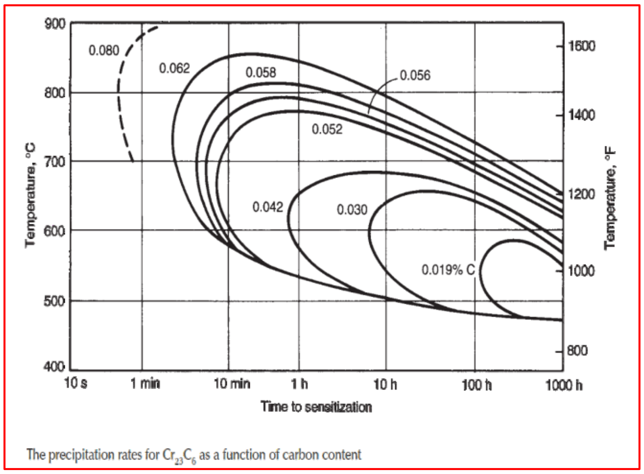

Figure 7: Precipitation rates for Cr23C6 as a function of carbon content.

It is understood that Sensitization is a phenomena which occurs mainly in austenitic stainless steel during fabrication which include welding.

Heat Affected Zone - HAZ can lose corrosion properties in ferritic steels and reason for that need not be sensitization. SMIL used mainly ferritic steels for its applications till now. However as a pro-active approach, SMIL has devised its experimentation to avoid sensitization.

The occurrence of sensitization is mostly due to the following combinations

- Material composition used for exhaust assembly
- Welding process used
- Filler wire/electrode used
- Thickness of the job
- Multi layer or single layer weld
- Cooling time(if any) between welding passes

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II. CONTROL MEASURES

Three primary ways to combat sensitization

1. Use a low carbon base and filler metal to reduce or eliminate carbon in the welding application.However, is not always practical as carbon is a vital alloying ingredient in some applications.
2. Minimize the time the weld and heat affected zone spend at temperatures conducive to sensitization. General consensus puts that range between 500°C - and 800°C.
3. Use filler metals with special alloying ingredients to prevent the formation of chromium carbides.For instance, titanium and niobium can be alloyed into the filler metal and help prevent reactions between chromium and carbon.

Manipulation of available options

The choice of base metal and Weld wire

Aim of ingredient: (Ti+Nb) / (C+N) ≥ 8

- Ti – Titanium (22)
- Nb –Niobium (41)
- C - Carbon (6)
- N – Nitrogen (7)

Note: Numbers indentify the Atomic Number

Base Material and Weld Wire (Filler) point of view

Addition of stabilizers such as niobium or titanium in base material and in filler material prevents sensitization

$$\text{Aim of ingredient:} \quad \frac{Ti+Nb}{C+N} \geqq 8$$

Process point of view

Carbon and Nitrogen presence induces and catalyses sensitization phenomenon. The surface of the base material should be cleansed before welding to remove carbon and nitrogen.

Parameter point of view

Usage of $Ar+O_2$ instead of $Ar+CO_2$ helps reduce granular corrosion

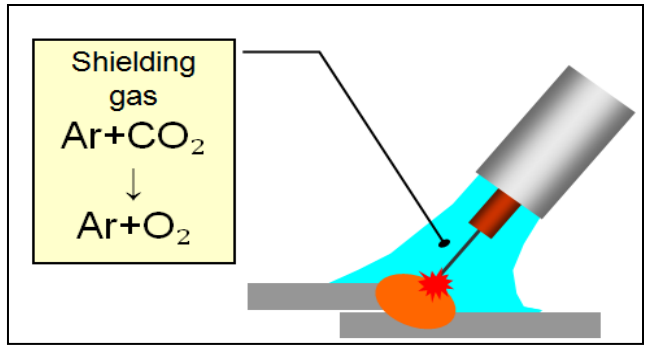

Figure 8: Weld process optimization

Sensitization can be prevented

Add strong carbide former (stabilizers) Ti, Nb to prevent M23C6 from forming.

Reheat steel to 950 - 1100°C to dissolve Cr23C6 & rapidly cool.

Reduction of carbon content : <0.03wt% or L grade steels

- Modern steel –making methods involving oxygen lancing
- @ 0.02% C complete immunity from inter granular corrosion in 18/8 steels.
- Reducing the time of exposure to the critical temperature range

Control of M23C6 kinetics

- Mo addition lengthens sensitization time
- Ni addition has an adverse effect
- Increasing Cr has beneficial effect

Use of strong carbide – forming elements

- Nb , Ti addition form trans granular carbides & scavenge C
- Less chances of Cr23C6.

Table 4:
Optimum Weld Parameters

Parameters	Trail 1	Trail 2	Trail 3
Voltage (V)	18 - 20	18 - 20	18 - 20
Current (A)	100 - 120	100 - 120	100 - 120
Shielding Gas	(Ar+5% O2)	(Ar+2 % O2)	(Ar+20 % O2)
Gas Flow (L/min)	10 - 15	10 - 15	10 - 15

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Table 5:
Base and Filler Material compositions

Items	BASE METAL		WELD WIRE	
	SUS 409	SUS 441	430 LNb	308LS i
C	.007	.014	.016	.017
Si	.5	.003	.44	.4
Mn	.2	.5	.28	.04
P	.021	.020	.029	.018
S	.004	.003	.002	.002
Ni	.1	.2	.25	.3
Cr	11.2	17.5	19.29	18.0
Nb	-	.55	.41	.32
Mo	.01	.02	.04	.04
Ti	.17	.15	.01	.01
N	.010	.018	.017	.017

Oxalic Etch Test

Oxalic acid etch test is a rapid method of screening specimens of certain stainless steel grades which are essentially free of susceptibility to inter angular attack associated with chromium carbide participates. The test is used for acceptance but not for rejection of material.

This test may be used in conjunction with other tests to provide a quick method for identifying specimens that are certain to be free of susceptibility to rapid intergranular attack. These specimens are identified by means of their etch structures.

Procedure

In an environment of 10% oxalic acid ($H_2C_2O_4$) an anodic current of 1A/cm^2 is applied for 1.5 minutes at room temperature to a polished sample. By microscopic analysis it is determined if the specimen needs to undergo further testing or if it is not sensitized. If the grain boundaries appear as ditches in the micrograph, it indicates that the sample needs further testing.

Extra low-carbon grades and stabilized grades are tested after sensitizing heat treatments at 1200 to 1250°F(650°C - 675°C), which is the range of maximum carbide precipitation. These sensitizing treatments must be applied prior to submitting the specimens to the oxalic acid etch test. The most commonly used sensitizing treatment is 1 hour at 1250°F (675°C)

Each practice specification contains a table showing which classifications of etch structures on a given stainless steel grade are equal to acceptable or non acceptable performance in that particular test. Specimens having acceptable etch structures need not be subjected to the hot acid test. Specimens having non acceptable etch structures must be tested in the specified hot acid solution.

The etch test is applicable only to those grades listed in the individual hot acid tests and classifies the specimens either as acceptable or as suspect.

Aim

To classify the Etch structures of Austenitic stainless steels using Oxalic acid Etch Test.

Apparatus Required

- Source of Direct Current- capable of supplying about 15 V and 20 A.
- Multimeter(to measure the flow of current)
- Variable Resistance (Note 1).
- Cathode- A cylindrical piece of stainless steel
- Large Clamp - To hold specimen to be etched.
- Metallurgical Microscope-For examination of etched microstructures at 250 to 500 diameters.
- Electrodes of the Etching Cell -The specimen to be etched is made the anode, and a stainless steel hollow rod as large as the specimen to be etched is made the cathode.
- Electrolyte -Oxalic acid, (H2C2O4·2H2O), reagent grade, 10 weight % solution.

Note 1-The variable resistance and the ammeter are placed in the circuit to measure and control the current on the specimen to be etched.

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Circuit Diagram

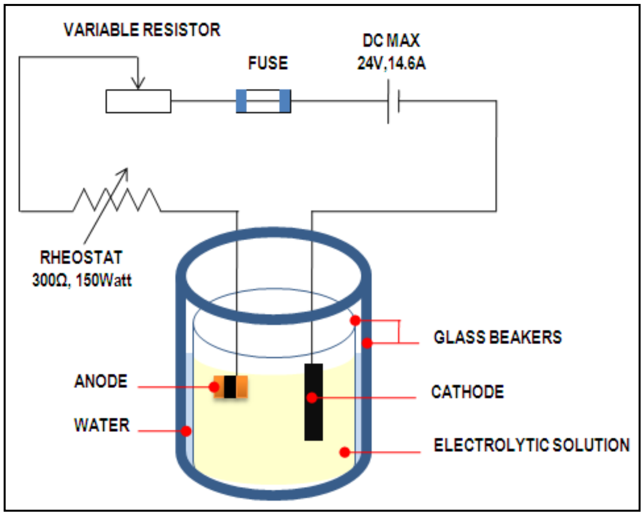

Figure 9: Sensitization experimental setup - circuit diagram

Principle: The experiment works on the principle of Ohm's Law.

Ohm's Law deals with the relationship between voltage and current in an ideal conductor. This relationship states that:

The potential difference (voltage) across an ideal conductor is proportional to the current through it. The constant of proportionality is called the "resistance", R.Ohm's Law is given by:

$$V = I R$$

Where; V is the potential difference between two points which include a resistance R and I is the current flowing through the resistance.

Test Practice

Table 6:
Test Specimen Preparation

Sample Description	Type of Welding	Filler Material
1.3 x 1.3 mm , SUS 304	MIG Welding	SUS 309

Table 7
Polishing

Sample Description	Polishing Time	Machines Used
1.3 x 1.3 mm , SUS 304	1 minute (Buff Polish) + 4 minute (220 Emery Paper)	Cutting & Polishing Machine

Observations

As per the Specification yellow-green film is should be formed on the cathode side. But here Yellow green film is obtained from Anode side.

Tested as per specification;

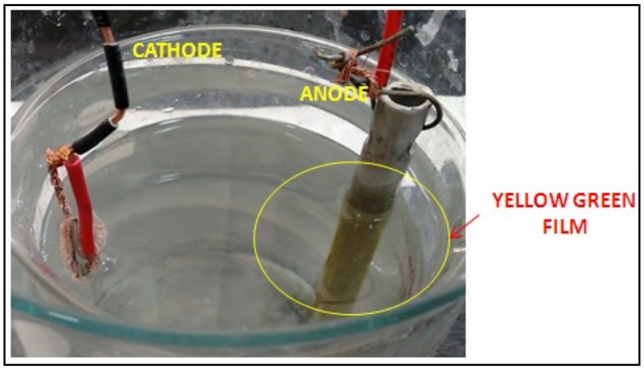

Figure 10: Yellow green formation – confirms passing of current through the setup

Table 8:
Rinsing

Sample Description	Rinsing Liquids	Rinsing Time
1.3 x 1.3 mm , SUS 304	Hot Water	3 min
	Acetone	3 min
	Nitric Acid	Just dip & take the specimen

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Microscopic Analysis - Defect Definition

- The oxalic acid etch test is used to identify intergranular corrosion
- Any sensitized microstructure will undergo selective localized corrosion along grain boundaries leading to inter granular corrosion

The occurrence of sensitization is mostly due to the following combinations;

- Material composition used for exhaust assembly
- Welding process , Filler wire/electrode used
- Thickness of the job , Multi layer or single layer weld
- Cooling time(if any) between welding passes
- After weld cooling time and methodology

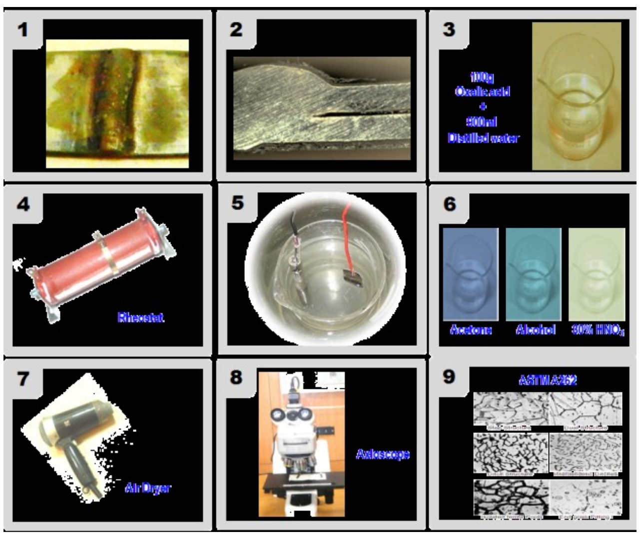

Figure 11: Experimentation step by step process

Legends

1. Base Metal Welding
2. Polishing
3. Etching Solution Preparation
4. Electrical Adjustment
5. Electrolytic Etching
6. Rinsing
7. Drying
8. Microscopic Analysis
9. Etch Surface Classification
10. Result and Summary

Experiment Definition

20 samples were tested upon on defined conditions. Iterated samples were checked with Etch structures Classification (ASTM A 262 – 02aε3) standard reference for sensitization to conclude on granular corrosion.

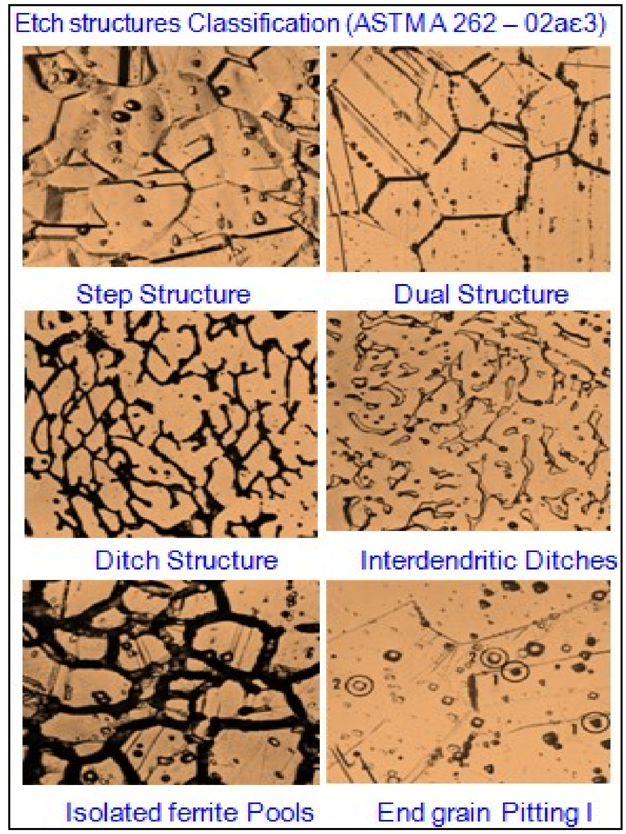

Figure 12: ASTM A 262 – 02aε3 standard reference for sensitization to conclude on granular corrosion

The etch structures obtained after conducting oxalic acid test on the 20 samples, are compared to the ASTM A 262 – $02a^{\varepsilon 3}$ etch structure to conclude on occurrence of sensitization. If the structures observed match with any 1 of the 6 samples, then it can be concluded that sensitization has occurred. If the observation does not match the structures of the ASTM standards, then we conclude that the sample is free from sensitization.

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Test Samples

Table 9:
20 sample base materials and filler materials with their respective after weld treatment followed for experimentation

S.No	Base 1	Base 2	Filler	Treatment
1	SUS 409L	SUS 409L	SUS 409	Atmospheric air cooled
2	SUS 409L	SUS 304	SUS 409	Atmospheric air cooled
3	SUS 304	SUS 409L	SUS 309	Atmospheric air cooled
4	SUS 304	SUS 304	SUS 309	Atmospheric air cooled
5	SUS 409L	SUS 409L	SUS 409	Hot air blown@141.6°C
6	SUS 409L	SUS 304	SUS 409	Hot air blown@141.6°C
7	SUS 304	SUS 409L	SUS 309	Hot air blown@141.6°C
8	SUS 304	SUS 304	SUS 309	Hot air blown@141.6°C
9	SUS 409L	SUS 409L	SUS 409	Water quenching
10	SUS 409L	SUS 304	SUS 409	Water quenching
11	SUS 304	SUS 409L	SUS 309	Water quenching
12	SUS 304	SUS 304	SUS 309	Water quenching
13	SUS 409L	SUS 409L	SUS 409	Oil dipped
14	SUS 409L	SUS 304	SUS 409	Oil dipped
15	SUS 304	SUS 409L	SUS 309	Oil dipped
16	SUS 304	SUS 304	SUS 309	Oil dipped
17	SUS 409L	SUS 409L	SUS 409	Heated to 1000°C
18	SUS 409L	SUS 304	SUS 409	Heated to 1000°C
19	SUS 304	SUS 409L	SUS 309	Heated to 1000°C
20	SUS 304	SUS 304	SUS 309	Heated to 1000°C

Experiment was conducted on all 20 samples and the after treatment as in above table was carried out. The samples were then studied under micro scope and the grains were compared to ASTM reference to confirm sensitization. Of all the samples sample no 12, 13, 15, 16, 17, 19 and 20 showed sensitization occurrence; marked in red in above table.

Sample preparation remains the initial step where 2 base metals are welded using a filler material (per table) and the sample near weld zone is cut to desired dimension. Area of the sample is calculated to regulate appropriate current to the sample while testing.

Table 10:
General settings and input parameters followed for all 20 samples

SAMPLE PREPARATION - APPLIES TO ALL			
Polishing	Buffing (1 min)	220 grit size emery paper (4 min)	
Anode	Sample to be etched (Welded)		
Cathode	Austenitic Steel Hollow rod		
Microscopic Analysis	500 X Observation		
Voltage	15V DC Supply		
Etching solution	100g Oxalic acid + 900ml Distilled water		
Yellow fumes found	Sample dipped in 30% HNO_3		
Air Dryer	Till the sample gets dried		
Hot Water	3 Minutes	Acetone	3 Minutes

The above said settings are common to all sample preparation and there are certain parameters that differ to suit the sample dimensions. Such settings are mentioned in tables above observations in the following samples.

Sample 12:

Table 11:
Input parameters for sample 12

SAMPLE 12			
Base 1	Base 2	Filler	
SUS 304	SUS 304	SUS 309	
Treatment		Water Quenching	
OXALIC ACID ETCHING			
Area	$1.60cm^2$	Current	1.75 A

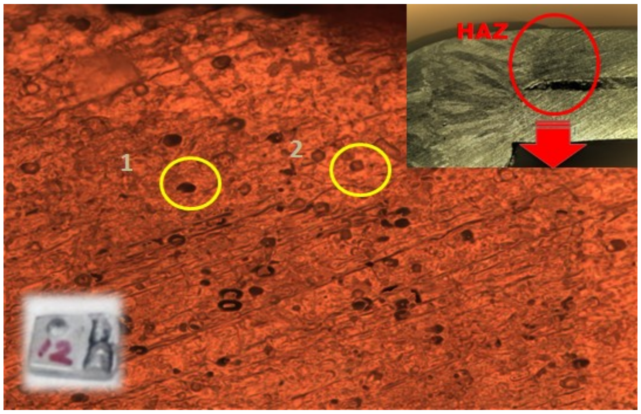

Figure 13: Sample 12 showing characteristics of sensitization

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The Etch structure obtained from the sample matches with the ASTM A 262 – 02a$^{\varepsilon3}$ etch structure (6). Hence the heat affected zone was found to be sensitized.

Sample 13:

Table 12:
Input parameters for sample 13

SAMPLE 13			
Base 1	**Base 2**	**Filler**	
SUS 309L	SUS 409L	SUS 409	
Treatment		Oil Dipped	
OXALIC ACID ETCHING			
Area	2.73cm^2	**Current**	3.2 A

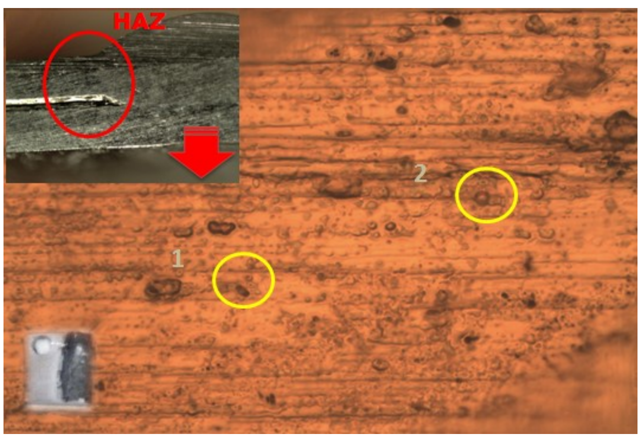

Figure 14: Sample 13 showing characteristics of sensitization

The Etch structure obtained from the sample matches with the ASTM A 262 – 02a$^{\varepsilon3}$ etch structure (6). Hence the heat affected zone was found to be sensitized.

Sample 15:

Table 13:
Input parameters for sample 15

SAMPLE 15			
Base 1	**Base 2**	**Filler**	
SUS 304	SUS 409L	SUS 309	
Treatment		Oil Dipped	
OXALIC ACID ETCHING			
Area	2.16cm^2	**Current**	1.75 A

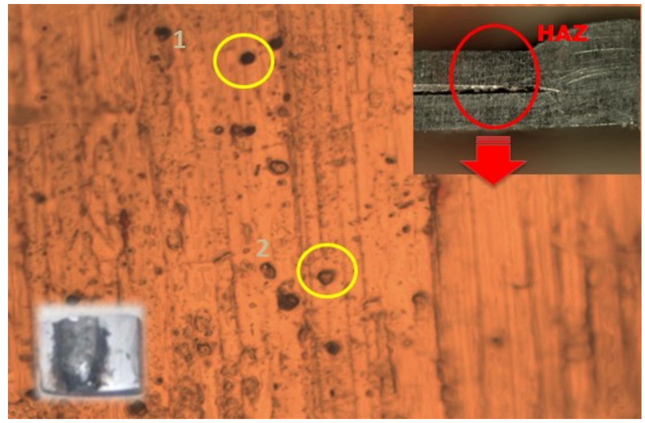

Figure 15: Sample 15 showing characteristics of sensitization

The Etch structure obtained from the sample matches with the ASTM A 262 – 02a$^{\varepsilon3}$ etch structure (6). Hence the heat affected zone was found to be sensitized.

Sample 16:

Table 14:
Input parameters for sample 16

SAMPLE 16			
Base 1	**Base 2**	**Filler**	
SUS 304	SUS 404	SUS 309	
Treatment		Oil Dipped	
OXALIC ACID ETCHING			
Area	1.75cm^2	**Current**	2.1 A

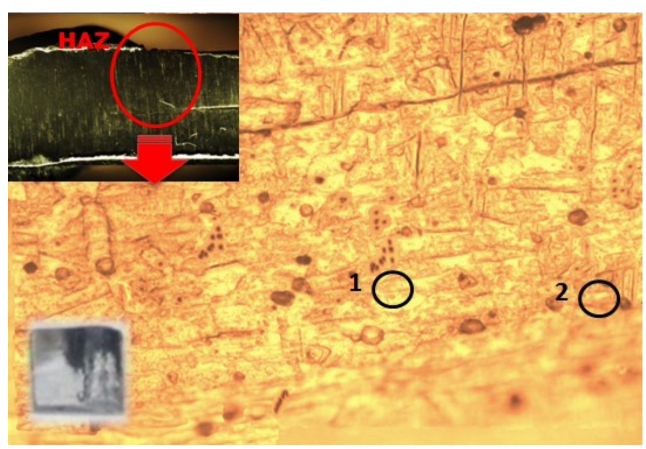

Figure 16: Sample 16 showing characteristics of sensitization

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The Etch structure obtained from the sample matches with the ASTM A 262 – $02a^{\varepsilon 3}$ etch structure (6). Hence the heat affected zone was found to be sensitized.

Sample 17:

Table 15
Input parameters for sample 17

SAMPLE 17			
Base 1	**Base 2**		**Filler**
SUS 309L	SUS 409L		SUS 409
Treatment	Heated to 1000°C using muffler furnace		
OXALIC ACID ETCHING			
Area	1.57cm^2	**Current**	2.31 A

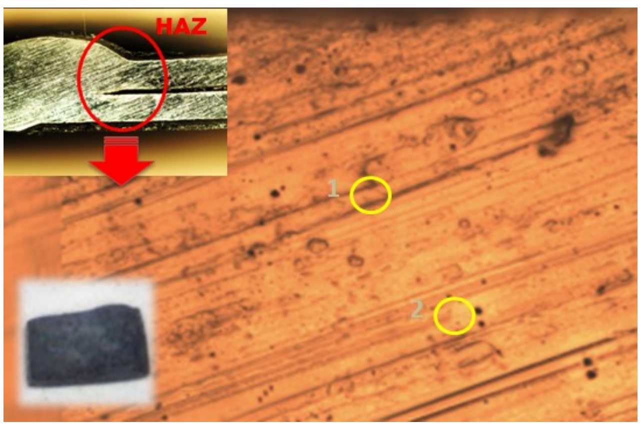

Figure 17: Sample 17 showing characteristics of sensitization

The Etch structure obtained from the sample matches with the ASTM A 262 – $02a^{\varepsilon 3}$ etch structure (6). Hence the heat affected zone was found to be sensitized.

Sample 19:

Table 16:
Input parameters for sample 19

SAMPLE 19			
Base 1	**Base 2**		**Filler**
SUS 304	SUS 409L		SUS 309
Treatment	Heated to 1000°C using muffler furnace		
OXALIC ACID ETCHING			
Area	2.34cm^2	**Current**	2.87 A

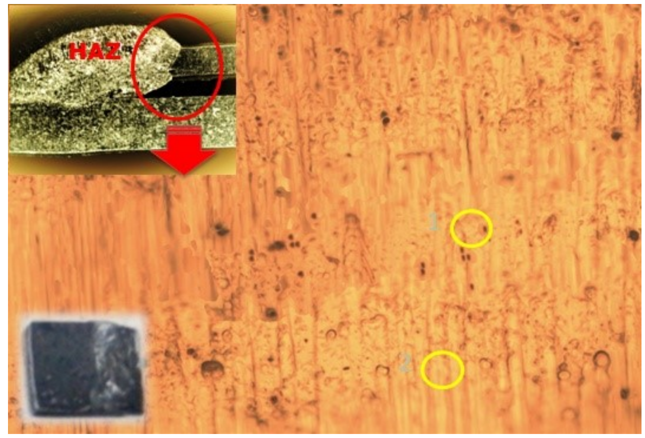

Figure 18: Sample 19 showing characteristics of sensitization

The Etch structure obtained from the sample matches with the ASTM A 262 – $02a^{\varepsilon 3}$ etch structure (6). Hence the heat affected zone was found to be sensitized.

Sample 20:

Table 17:
Input parameters for sample 20

SAMPLE 20			
Base 1	**Base 2**		**Filler**
SUS 304	SUS 304		SUS 309
Treatment	Heated to 1000°C using muffler furnace		
OXALIC ACID ETCHING			
Area	2.97cm^2	**Current**	3.01 A

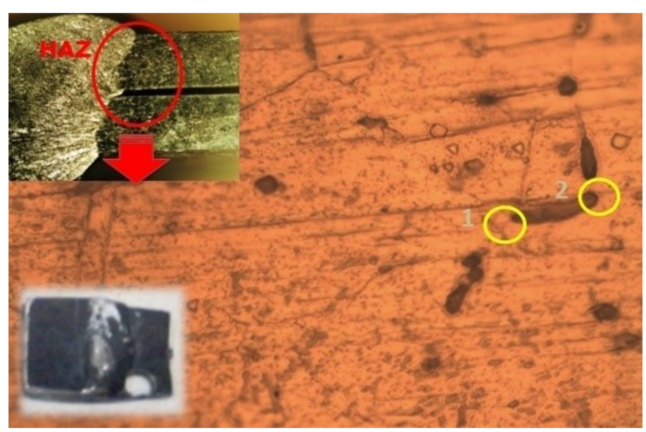

Figure 19: Sample 20 showing characteristics of sensitization

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The Etch structure obtained from the sample matches with the ASTM A 262 – 02a$^{\varepsilon 3}$ etch structure (6). Hence the heat affected zone was found to be sensitized.

III. Conclusion

After clearly studying the behavior of the 20 samples treated in different conditions, the followings conclusions are made;

- Sensitization will be avoided if the base material and the filler material's (Ti+ Nb)/(C+N) is > 8
- Sensitization can be avoided if the samples after welding are air dried
- Sensitization can be avoided if the samples are water quenched
- If the samples are dipped in oil after welding, sensitization phenomenon is most likely to happen
- If the samples are heated between 800F to 1650F, the austenitic steel undergoes changes and makes the sample susceptible to intergranularcorrosion. In this temperature range chromium carbides precipitates nearthe grain boundaries and sensitization phenomenon is most likely to happen

Listed are some lessons that were learnt during the course of the experimentation;

- Sample to be etched must be as small as possible since sample size is very important for etching (1 sqinch – as per ASTM A 262 – 02a$^{\varepsilon 3}$).
- Sample should be slightly polished up to emery papers 220.
- Yellow green formation indicates that resistance flows in the etching solution, there by chemical reactions take place.
- If no yellow green formation is observed, check the rheostat connection and vary for resistance flow.
- The weld area must free from spatters, burr & oil.

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