



Post weld heat treatment of welded low alloyed steel pipes

Use of AISI 4130 steel in hydrogen sulfide environment and its post weld heat treatment conditions.

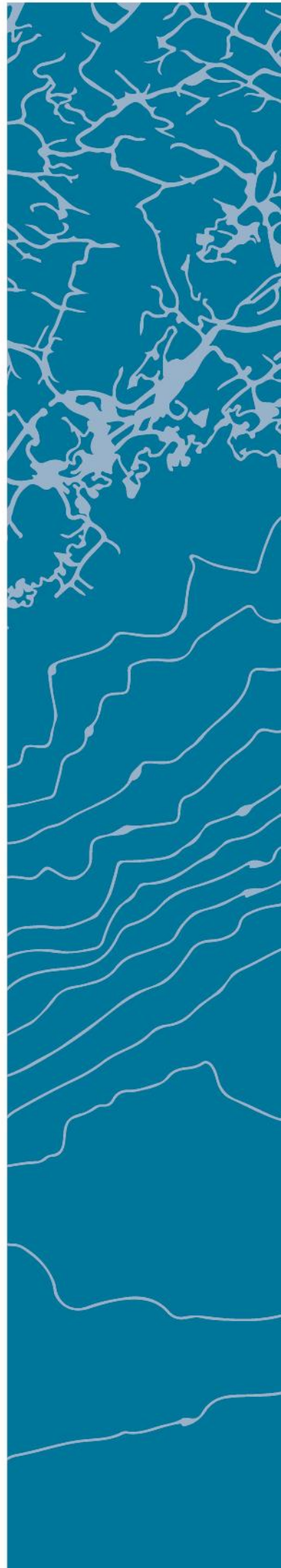
DMITRI RYBAKOV

SUPERVISOR

Dr. Dmitry Vysochinskiy

University of Agder (UIA), 2019

Faculty of Engineering and Science
Department of Engineering Sciences



Obligatorisk egenerklæring/gruppeerklæring

Den enkelte student er selv ansvarlig for å sette seg inn i hva som er lovlige hjelpemidler, retningslinjer for bruk av disse og regler om kildebruk. Erklæringen skal bevisstgjøre studentene på deres ansvar og hvilke konsekvenser fusk kan medføre. Manglende erklæring fritar ikke studentene fra sitt ansvar.

1.	Jeg/vi erklærer herved at min/vår besvarelse er mitt/vårt eget arbeid, og at jeg/vi ikke har brukt andre kilder eller har mottatt annen hjelp enn det som er nevnt i besvarelsen.	<input checked="" type="checkbox"/>
2.	Jeg/vi erklærer videre at denne besvarelsen: <ul style="list-style-type: none"> - ikke har vært brukt til annen eksamen ved annen avdeling/universitet/høgskole innenlands eller utenlands. - ikke refererer til andres arbeid uten at det er oppgitt. - ikke refererer til eget tidligere arbeid uten at det er oppgitt. - har alle referansene oppgitt i litteraturlisten. - ikke er en kopi, duplikat eller avskrift av andres arbeid eller besvarelse. 	<input checked="" type="checkbox"/>
3.	Jeg/vi er kjent med at brudd på ovennevnte er å betrakte som fusk og kan medføre annullering av eksamen og utestengelse fra universiteter og høgskoler i Norge, jf. Universitets- og høgskoleloven §§4-7 og 4-8 og Forskrift om eksamen §§ 31.	<input checked="" type="checkbox"/>
4.	Jeg/vi er kjent med at alle innleverte oppgaver kan bli plagiatkontrollert.	<input checked="" type="checkbox"/>
5.	Jeg/vi er kjent med at Universitetet i Agder vil behandle alle saker hvor det forligger mistanke om fusk etter høgskolens retningslinjer for behandling av saker om fusk.	<input checked="" type="checkbox"/>
6.	Jeg/vi har satt oss inn i regler og retningslinjer i bruk av kilder og referanser på biblioteket sine nettsider.	<input checked="" type="checkbox"/>

Publiseringsavtale

Fullmakt til elektronisk publisering av oppgaven

Forfatter(ne) har opphavsrett til oppgaven. Det betyr blant annet enerett til å gjøre verket tilgjengelig for allmennheten (Åndsverkloven. §2).

Alle oppgaver som fyller kriteriene vil bli registrert og publisert i Brage Aura og på UiA sine nettsider med forfatter(ne)s godkjenning.

Oppgaver som er unntatt offentlighet eller taushetsbelagt/konfidensiell vil ikke bli publisert.

Jeg/vi gir herved Universitetet i Agder en vederlagsfri rett til å

gjøre oppgaven tilgjengelig for elektronisk publisering:

JA NEI

Er oppgaven båndlagt (konfidensiell)?

JA NEI

(Båndleggingsavtale må fylles ut)

- Hvis ja:

Kan oppgaven publiseres når båndleggingsperioden er over?

JA NEI

Er oppgaven unntatt offentlighet?

JA NEI

(inneholder taushetsbelagt informasjon. Jfr. Offl. §13/Fvl. §13)

Preface

This work is prepared as a Master's thesis of the Master's Program in Civil and Constructional Engineering at the Faculty of Engineering and Science, University of Agder (UiA) in Grimstad, Norway. The framework for this master thesis was given by company MHWirth, that specializes in offshore drilling. This work is partly based on the Preliminary Project to this Master's Thesis that started in autumn 2018, but at the same time, it is independent enough to be read separately.

I want to give special thanks to my supervisor Dmitry Vysochinskiy and micrograph specialist Naureen Akhtar, for all their support and contribution to this work.

Would also like to thank MHWirth personnel: Geir Govertsen and Terje Naessen for taking the time to share their experiences and knowledge with me, and engineering company Nymo, who made this project possible by kindly providing us with material samples.

Author

Dmitri Rybakov
Kristiansand, May 2019

Summary

Based on the results from pre-project and work covered by this thesis, no better alternatives to AISI 4130 material was found, neither could it be safely concluded for removal of NACE MR0175/ISO 15156 requirements in any parts of high-pressure drilling fluid system. Since AISI 4130 has shown to be the optimal choice for the system, it was decided to look for improvements of welding procedure by optimization of post weld heat treatment for this steel.

Heat treatment is an expansive and time-consuming procedure that, if not done correctly, could weaken the material and jeopardize the safety of the system. In this thesis, with the help of theoretical research, experimental heat treatment and microscopy an optimal temperature of 650 °C with holding time of 150 minutes were proposed for PWHT of AISI 4130 steel pipes.

It is further suggested to look into increasing of preheating and interpass temperature to maximum allowed value of 250 °C, which is expected to reduce cooling rate, allowing for less brittle and tougher material structure.

This thesis includes preparation and examination of test pieces, including hardness test and microscopy. Thus giving a good foundation for the development of a welding procedure for AISI 4130 pipe.

Table of content

OBLIGATORISK EGENERKLÆRING/GRUPPEERKLÆRING	I
PUBLISERINGSAVTALE	II
PREFACE	III
SUMMARY	IV
TABLE OF CONTENT	V
LIST OF FIGURES	VIII
LIST OF TABLES	X
TERMINOLOGY AND ABBREVIATION	XI
1 INTRODUCTION	1
2 SOCIAL PERSPECTIVE	2
3 THEORY ON DRILLING FLUID	3
3.1 DRILLING FLUID (DF)	3
3.2 CIRCULATION OF DF	3
3.2.1 <i>Storage and Mixing of DF</i>	4
3.2.2 <i>Injection of DF</i>	4
3.2.3 <i>Treatment of DF</i>	5
3.2.4 <i>Pressure reducing system</i>	5
3.3 COMPOSITION OF DF	5
3.4 HYDROGEN SULFIDE DAMAGE	6
4 THEORY ON STEEL	7
4.1 WELDING	7
4.1.1 <i>Carbon equivalent</i>	8
4.2 PHASE DIAGRAM	10
4.3 EFFECT OF ALLOYING AGENTS	12
4.4 RESIDUAL STRESSES AND THERMAL EXPANSION	15
4.5 RECOVERY, RECRYSTALLIZATION AND GRAIN GROWTH	16
4.6 HEAT AFFECTED ZONE	19
4.7 MICROSTRUCTURES OF IRON-CARBON ALLOYS	20
4.7.1 <i>Pearlite</i>	20
4.7.2 <i>Martensite</i>	21
4.7.3 <i>Bainite</i>	24
4.7.4 <i>Spheroidite</i>	27
4.8 HEAT TREATMENT	27
4.8.1 <i>PWHT</i>	27
4.8.2 <i>Tempering</i>	28
4.8.3 <i>Normalizing</i>	30
4.8.4 <i>Annealing</i>	31
4.9 SCHAEFFLER DIAGRAM	32
4.10 TRANSFORMATION DIAGRAMS, TTT AND CCT	33
4.10.1 <i>Isothermal Transformation diagram</i>	33
4.10.2 <i>Continues Cooling transformation, CCT</i>	34
4.11 METALLOGRAPHIC ETCHING	35
4.12 SAE/AISI CARBON STEEL	36
5 RESEARCH QUESTION	37
6 CASE AND MATERIALS	38

6.1	MATERIAL AISI 4130.....	38
6.1.1	<i>Material Certificate</i>	38
6.1.2	<i>Heat treatment</i>	39
6.1.3	<i>Cooling transformation diagram</i>	40
6.1.4	<i>Material Data Sheet</i>	41
6.2	MANDATORY REQUIREMENTS.....	42
6.2.1	ASME B31.3	42
6.2.2	NACE MR0175	44
6.2.3	NORSOK M-601	45
6.2.4	ISO 14745	45
7	METHOD	47
7.1	WELDING.....	47
7.1.1	<i>Preparation</i>	47
7.1.2	<i>Preheating</i>	47
7.1.3	<i>SMAW Welding</i>	48
7.1.4	<i>Polishing</i>	48
7.2	CUTTING	49
7.3	POLISHING.....	49
7.4	ETCHING	51
7.5	SAMPLES.....	54
7.6	HARDNESS TESTING.....	55
7.7	HEAT TREATMENT	56
7.8	MICROSCOPY	57
8	SECONDARY TASKS	58
8.1	SPLIT OF SERVICES.....	58
8.1.1	<i>Partial pressure</i>	59
8.1.2	<i>Process values of H₂S</i>	60
8.1.3	<i>Conclusion on service split</i>	60
8.2	DUPLEX STEEL	62
8.2.1	<i>Weight reduction</i>	62
8.2.2	<i>Conclusion on duplex steel</i>	63
9	RESULTS	64
9.1	MICROSTRUCTURE	64
9.2	PREHEAT	66
9.3	PWHT	67
9.3.1	<i>Tempered martensite</i>	68
9.4	EDS ANALYSIS	71
10	DISCUSSION	73
10.1	HARDNESS TESTING	73
10.2	TEMPERING TEMPERATURE	74
10.3	BAINITE VS MARTENSITE	74
10.4	HOLDING TIME	75
11	CONCLUSION.....	76
12	SUGGESTIONS AND AFTERTHOUGHTS.....	77
13	BIBLIOGRAPHY	78
14	ATTACHMENTS.....	79
14.1	ATTACHMENT 1 – MATERIAL DATA SHEET	79
14.2	ATTACHMENT 2 – MATERIAL CERTIFICATE	1
14.3	ATTACHMENT 3 – 48.08 ELECTRODE.....	1
14.4	ATTACHMENT 4 – WPS FOR API 5L52 PIPE	2

14.5	ATTACHMENT 5 – HEAT INPUT DATA	3
14.6	ATTACHMENT 6 – HARDNESS MEASUREMENTS	4
14.7	ATTACHMENT 7 – ILLUSTRATIONS	6
14.8	ATTACHMENT 7 – HARDNESS MEASUREMENTS	7
14.9	ATTACHMENT 9 – MICROSCOPY	8
14.10	ATTACHMENT 10 – PIPING SPECIFICATION	9

List of Figures

FIGURE 1, PIPE BODY FAILURES VS. CAUSES.....	2
FIGURE 2: IRON-CARBON PHASE DIAGRAM.....	11
FIGURE 3, RESIDUAL STRESSES AND HARDNESS PROFILE OF A WELD.....	15
FIGURE 4, RECRYSTALLIZATION STEPS OF A METALLIC MATERIAL.....	16
FIGURE 5, INFLUENCE OF ANNEALING TEMPERATURE ON STRENGTH AND DUCTILITY OF BRASS.....	17
FIGURE 6, RECOVERY PROCESS.....	17
FIGURE 7, RECRYSTALLIZATION PROCESS.....	18
FIGURE 8, GRAIN GROWTH PROCESS.....	18
FIGURE 9, AREAS IN A WELDED MATERIAL.....	19
FIGURE 10, REGIONS IN HAZ & CORRESPONDING PHASE DIAGRAM.....	19
FIGURE 11, FORMATION OF PEARLITE FROM AUSTENITE.....	20
FIGURE 12, PERLITE STRUCTURE IN HYPO EUTECTOID STEEL.....	20
FIGURE 13, SCHEMATIC REPRESENTATION OF PERLITE IN STEEL.....	21
FIGURE 14, AUSTENITE TO MARTENSITE TRANSFORMATION.....	22
FIGURE 15, MARTENSITE STRUCTURE.....	22
FIGURE 16, MARTENSITE TEMPERING.....	23
FIGURE 17, TEMPERED MARTENSITE, HIGH RESOLUTION IMAGE.....	23
FIGURE 18, HARDNESS VS MATERIAL THICKNESS.....	24
FIGURE 19, ISOTHERMAL TRANSFORMATION DIAGRAM FOR AN EUTECTOID IRON-CARBON ALLOY.....	24
FIGURE 20, FORMATION OF PEARLITE AND BAINITE.....	25
FIGURE 21, BAINITE STRUCTURES, AT DIFFERENT FORMATION TEMPERATURES, 0,7% C STEEL.....	26
FIGURE 22, FATHERY BAINITE MICROSTRUCTURE.....	26
FIGURE 23, SPHEROIDITE IN CONTINUOUS PHASE OF A-FERRITE. 1000X.....	27
FIGURE 24, TEMPERING PROCESS.....	29
FIGURE 25, INFLUENCE OF MO CONTENT ON TEMPER SOFTENING (AFTER E.C. BAIN).....	29
FIGURE 26, NORMALIZING PROCESS.....	30
FIGURE 27, ANNEALING PROCESS OF HYPOEUTECTIROID STEEL.....	31
FIGURE 28, ANNEALING AND NORMALIZING PROCESS.....	31
FIGURE 29, SCHAEFFLER DIAGRAM.....	32
FIGURE 30, ISOTHERMAL TRANSFORMATION DIAGRAM FOR AN EUTECTOID IRON-CARBON ALLOY.....	33
FIGURE 31, CONTINUOUS COOLING DIAGRAM FOR AN EUTECTOID IRON-CARBON ALLOY.....	34
FIGURE 32, EXAMPLE OF ETCHING ART ON A METAL PLATE.....	35
FIGURE 33, RAW AISI 4130 PIPE SAMPLES FROM NYMO.....	38
FIGURE 34, TT DIAGRAM FOR AISI 4130 PIPE.....	39
FIGURE 35, WELDING PARAMETERS FOR AISI 4130.....	39
FIGURE 36, ISOTHERMAL AND CONTINUOUS COOLING DIAGRAM FOR SAE 4130 STEEL.....	40
FIGURE 37, CCT DIAGRAM FOR COOLING RATES OF AISI 4130 IN WATER AND IN STILL AIR.....	41
FIGURE 38, PWHT PARAMETERS FOR STEEL. ISO 14745.....	45
FIGURE 39, BUTT WELD END PREPARATION.....	47
FIGURE 40, PREHEATING WITH FLAME TORCH.....	47
FIGURE 41, PICTURES OF WELDING PROCESS. ROOT AND CAP WELDING STAGES.....	48
FIGURE 42, POLISHING OF WELDED SAMPLE.....	48
FIGURE 43, CUTTING OF SAMPLES.....	49
FIGURE 44, USE OF MILLING MAHINE FOR SURFACE CORRECTION AND SMOOTHING.....	49
FIGURE 45, GRINDING MACHINE AND POLISHED SAMPLES.....	50
FIGURE 46, ETCHING WITH 3% NITAL, TIME FROM 1 TO 6 IS 6 SECONDS.....	51
FIGURE 47, ETCHING WITH 50% HCL, TIME FROM 1 TO 6 IS 2 MINUTES.....	51
FIGURE 48, ETCHED SAMPLES.....	52
FIGURE 49, DIFFERENT ETCHING RESULTS.....	53
FIGURE 50, HARDNESS TESTING MACHINE.....	55
FIGURE 51, BUTT-WELD SURVEY METHOD FOR VICKERS HARDNESS MEASUREMENT, ISO 15156-2.....	55
FIGURE 52, CONTROLLED HEAT TREATMENT IN FURNACE.....	56
FIGURE 53, PWHT DIAGRAMS.....	56
FIGURE 54, PICTURE OF JEOL JSM-7200F MICROSCOPE AT UNIVERSITY OF AGDER.....	57
FIGURE 55, H2S PARTIAL PRESSURE = 0,3 KPA (0,05 PSI).....	59
FIGURE 56, TREATMENT OF DRILLING FLUID.....	61

FIGURE 57, SIMPLIFIED MUD SYSTEM MAP.....	61
FIGURE 58, WALL THICKNESS COMPARISON (UNS 31803, UNS S32760 AND AISI 4130).....	62
FIGURE 59, MICROSTRUCTURE OF AISI 4130 AT DIFFERENT HEAT TREATMENT STEPS.....	64
FIGURE 60, TRANSITION CARBIDES. SAMPLE 1.1, BASE MATERIAL, X100.000.....	65
FIGURE 61, MICROSCOPY OF AISI 4130 STEEL. SAMPLE 1.1, BASE MATERIAL, X2500.	65
FIGURE 62, MICROSCOPY OF AISI 4130 STEEL. SAMPLE 1.1, BASE MATERIAL, X5500.	66
FIGURE 63, TEMPERED BAINITE, AT X1200 ENLARGEMENT. SAMPLE 4.1, HAZ, X5500	66
FIGURE 64, HARDNESS MEASUREMENTS WITH AND WITHOUT PREHEATING TO 120°C.....	67
FIGURE 65, HARDNESS MEASUREMENTS OF HAZ.	67
FIGURE 66, RESULTS FROM HARDNESS TESTING AFTER PWHT AT 705 °C.....	68
FIGURE 67, EFFECT OF PWHT ON MARTENSITE IN HAZ	69
FIGURE 68, PARTICIPATION OF CARBIDES IN BASE MATERIAL OF AISI 4130.	70
FIGURE 69, EDS SPECTRUM OF AISI 4130.....	71
FIGURE 70, GRAPHITE FLAKES IN AISI 4130	72
FIGURE 71, MARTENSITE FORMATIONS IN HAZ OF SAMPLE 3.....	74
FIGURE 72, CARBON CONTENT VS HARDNESS OF MARTENSITE AND BAINITE	75
FIGURE 73, THE EFFECT OF HEAT INPUT ON COOLING RATES IN WELDS AS A FUNCTION OF PREHEATING TEMPERATURES	77

List of Tables

TABLE 1, PREHEAT TEMPERATURES, ASME B31.3	42
TABLE 2, POSTWELD HEAT TREATMENT, ASME B31.3	43
TABLE 3, ALTERNATE POSTWELD HEAT TREATMENT, ASME B31.3.....	43
TABLE 4, ACCEPTABLE HARDNESS VALUES FOR CARBON-MANGANESE STEEL, ISO 15156-2	44
TABLE 5, GRINDING PAPER GRID OVERVIEW	50
TABLE 6, TABLE OF SAMPLES.....	54
TABLE 7, COOLING RATE IN FURNACE.....	57
TABLE 8, EDS RESULTS.....	71

Terminology and abbreviation

SG	Specific Gravity
Sour service	Services with exposure to H ₂ S contaminated environment
GMAW	Gas metal arc welding
GTAW	Gas Tungsten arc welding
FCAW	Flux cored arc welding
SMAW	Shielded metal arc welding
SAW	Submerged arc welding
MAG	Metal active gas welding
MIG	Metal inert gas welding
TIG	Tungsten inert gas welding
LED	Lower electron detector
BED	Backscattered electron detector
BED-C	Reveals information about the composition
BED-S	Reveals both topographical and compositional information

1 Introduction

During the work on pre-project for this thesis, central parts of theory behind the deepwater drilling process was collected and formed a good theoretical base for further optimization steps for the high-pressure mud¹ system. During the pre-projects a possibility of reduce the wall thickness from schedule XXS to schedule 160 for 10.000 psi pipes was uncovered. That step will reduce the weight by 17% - 27% (depending on pipe dimension) and the amount of welding time. It is was decided to implement this reduction in next revision of the piping specification.

A good foundation was laid for further consideration of duplex steel as an alternative material for mud system and the possibility of avoiding NACE MR0175/ISO 15156 requirements. Answer on which is included in this thesis.

However, clarification of whether NACE MR0175/ISO 15156 requirements can be lifted for parts of the drilling fluid system and whether Duplex is a better material choice is relatively small parts of this work, and thereby not included in the main body of this thesis but clarification around those tasks was assigned the whole chapter 8.

Main task

The main task of this thesis is to discuss and propose optimal conditions for post weld heat treatment of welded AISI 4130 pipe that is used in hydrogen sulfide contaminated services. Secondary tasks were to look into Duplex steel as an alternative to AISI 4130 and possibility of avoiding the mandatory requirements of NACE MR0175 / ISO 15156 for hydrogen sulfide contaminated service (sour service) for parts of high-pressure drilling fluid system.

Collaborators

This work is done in cooperation and on behalf of MHWirth at faculty of engineering and science of Agder university as a thesis for master of "Civil and Construction Engineering". The task was defined by the company based on the needs of Piping and Layout department.

Notes to the rapport structure

For establishing of good overview and readability, we try not to mix the main and secondary tasks and separate the chapters accordingly. Since secondary tasks did not require a lot of effort, because the most of related work was done during the preliminary part for this thesis, those tasks were discussed and concluded on in just one chapter, chapter 8.

¹ Mud – drilling fluid

2 Social Perspective

Weld defects are one of the main causes for the failure of piping systems. Which are often caused by too high or insufficient heat input, fast cooling rates or wrong post weld heat treatment. In addition to health and safety issues, failure of high-pressure drilling fluid system can put a hold on the hole drilling operation forcing site workers out of work, delays the start of the production process and generates expansive repair costs. Every day of such a delay cause millions of dollars for the drilling company and thereby demanding the risk of poor welding joints brought to a minimum during the design phase.

Based on petroleum design and engineering company, Viking Engineering, 19 % of pipe body failures is the material deficiency, which is caused by a lack of fusion from welding, poor tempering, or quenching cracks.

In addition to material deficiency, poor welding could be a part of the combination, fatigue, corrosion or unknown reasons that altogether make up over 50% of failure causes.

Understanding the failure modes is an important part of a process that ensures the soundproof design and allows for as expected performance of the system, lowering thereby the in operation risks.

In this work, we are looking into material reaction on different heat effects and suggest the heat treatment conditions for the optimal performance and safety of welded joints.

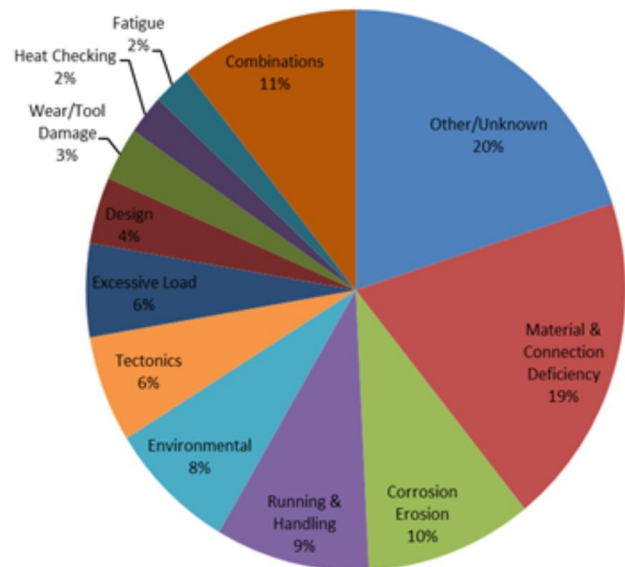


Figure 1, Pipe body failures vs. causes

Copied from www.vikingeng.net, Article: Common failures in onshore oil and gas wells.

3 Theory on Drilling Fluid

This theory chapter includes only the theory around drilling fluid that is considered important for a comprehensive understanding of this thesis. Readers that are not familiar with the drilling process are especially encouraged to absorb this theory chapter before further reading.

Main parts of this chapter are the theory behind circulation and composition of the drilling fluid and hydrogen sulfide damage mechanisms.

3.1 Drilling fluid (DF)

Drilling fluid (*further noted with abbreviation: DF*), also called Mud, is complex multicomponent viscous fluid that aids the drilling of wells and is one of the main and necessary components in deep drilling processes.

Main functions of drilling fluid are cooling and lubrication to prevent overheating of the drill bit, removal of cuttings from the well by carrying them up to the surface and providing of hydrostatic pressure to prevent blowouts.

There are three main categories of drilling fluid, used for deepwater drilling: Water Based Mud (WBM), Oil Based Mud (OBM) and Completion fluid.

Water-based mud uses water as its main component that is mixed up with clay and other chemicals. Because of danger that water can be absorbed by the formation, WBM is often only used in the top part of the well that is protected by a casing. Oil Based Mud is the most pollutant drilling fluid but also the safest one to use in deepwater drilling. It is commonly used in the parts of well that is depending on stable filter cake for a stable formation in the well [1]. Oil is the main component in addition to the water, clay and other chemicals. Completion fluid, as the name implies, is used after completion of the drilling process to maintain the hydrostatic pressure until the start of the production process. It can be both water and oil based, depending on the properties of the well, but water-based fluid is used in most cases.

In most cases all three drilling fluids are used in a drilling process:

- Stage 1. Drilling of the top hole – WBM*
- Stage 2. Drilling after casing - OBM*
- Stage 3. The time between drilling and production – Completion Fluid*

3.2 Circulation of DF

Design and layout of DF System may vary a lot depending on operations to be performed, operational philosophy and the actual layout of the drilling facility on the installation. The operators and oil companies may also have additional requirements that must be fulfilled, surpassing national and international rules and regulations. However, the main principle of the system remains the same.

Drilling fluid system is a heavy and complex which involves long stretches of pipe and many types of equipment. It can be split into five main parts:

- Storage part, where the drilling fluid and its components are stored
- Mixing part, where it gets mixed to achieve desirable properties
- Injection part of the system, that transfers the mud up to the top drive and into the well
- Treatment part, that handles and treats the used drilling fluid
- Pressure reducing part, that is not in use under normal operation but is absolutely vital part of the system in case of unexpected pressure spikes in the well.

3.2.1 Storage and Mixing of DF

The storage part of the DF system can be considered the first stage of the drilling fluid journey. That is where the base fluid mixture is stored after it was mixed and transported to the site in a cargo tanks. As the properties of the well formation changes with depth, different types of drill fluid and drill fluid additives must be used.

Properties of the fluid are altered by mixing in different additives like clay, weight materials, salts and polymers that is stored and added to the mixture in the mixing part of the system.

Base fluid and additives are stored under ambient pressure. Low pressure, carbon steel piping classes are used in both storage and mixture parts of the system. Following design parameters are generally applicable [1]:

Design pressure: 16 bar
Design temperature: -15 °C – 150 °C
Material: Carbon steel
Corrosion allowances: 3 mm.

3.2.2 Injection of DF

Mixed mud that is ready for injection is stored in so-called Active tank that is connected to a charging pump that transverse the mud to a high-pressure Mud pump. HP pump pumps the drilling fluid to a hight of around 50 meters up the derrick to the drilling machine and few kilometers down the drilling pipe inside the well [1].

Following parameters are generally applicable for the injection system of deepwater drilling [2]:

Design pressure: 517 bar
Design temperature: -29°C – 90 °C
Material: Low alloy steel. (usually AISI 4130 or 4140)
Corrosion allowances: 3 mm.

3.2.3 Treatment of DF

For maintaining control of the well it is important to control the composition of the mud. Drilling fluid that comes up from the well is highly polluted with drill cuttings and formation gasses that will have to be removed before it is pumped down to the well again.

The major gases found in wells are methane, carbon dioxide, nitrogen and hydrogen sulfide.

Following parameters are generally applicable for treatment system [2]:

Design pressure: 16 bar
Design temperature: -15 °C – 150 °C
Material: Carbon steel
Corrosion allowances: 3 mm.

3.2.4 Pressure reducing system

Some circumstances can lead to a situation when the pressure in the wellbore builds up above hydrostatic pressure of the drilling fluid column, that situation is called a kick, which can further lead to a blowout. Kicks are handled by a pressure reducing system called Choke and Kill.

Choke and Kill system (abbreviating: C&K) is an important part of offshore drilling installations. When pressure build-up above design pressure of normal operation, the BOP (blow out preventing) valve shuts down isolating the system from critical pressure, forcing the mud from the well inside the choke system that reduces the pressure to a required level and pumping it back into the well through the kill line. This process continues until the pressure in the well is stabilized and a normal operation can continue.

In extreme cases, when C&K system is not sufficient to handle the kick, there is an overboard line that is always present on all offshore installations. It sends the drilling fluid overboard and is only used as the last resort.

Design pressure: 690 or 1035 bar
Design temperature: -29 °C – 122 °C
Material: Low alloy steel. (usually AISI 4130 or 4140)
Corrosion allowances: 3 mm.

3.3 Composition of DF

The composition of drilling fluid is dependent on the formation, local regulations, depth of drilling and a range of other factors. Following are the main participants in mud for deepwater drilling:

Water - Fresh water is used in the water-based mud or as water phase in oil based

Oil - Oil is the main component of oil-based mud

Salts - For regulation of specific gravity (SG) and osmotic pressure)

Clay - For increased viscosity (usually Bentonite)

Polymers - Have a wide range of use (emulsifiers, thinner agents, amino acids for miscibility...)

Weighting agents - Minerals with high SG for the regulation of hydrostatic pressure (usually Baryte)

Alkalinity agents - For PH control of drilling fluid

When drilling fluid is circulated back to the surface, it is polluted with formation particles (cuttings) and gasses that was previously mentioned (mainly: methane, carbon dioxide, nitrogen and hydrogen sulfide).

3.4 Hydrogen sulfide damage

Hydrogen Sulfide can be a major problem during a drilling process. Exposure to H₂S-containing production fluids can lead to cracking and sudden failure of metallic piping components.

In 1940s and early 1950s in West Texas the oil and gas industry had its first challenges with gas fields containing H₂S (sour gas). Further failures were reported in Canada. To solve the problem, the international organization for corrosion control NACE, formed a committee to accumulate information and provide a solution to this hydrogen sulfide issue and has developed one of its most important documents: NACE MR0175.

Exact degree of expected drilling fluid contamination during drilling is unknown, in fact usually, it is not a problem, but since there is no certainty to it, the requirements in the NACE standard is mandatory to follow in all installations that can be subjected to H₂S contamination above certain levels

Cracking that can be caused by H₂S, including stress corrosion cracking, hydrogen-induced cracking and stepwise cracking, stress-oriented hydrogen-induced cracking, soft zone cracking, and galvanically induced hydrogen stress cracking [1], [3].

Hydrogen-induced cracking (HIC)

Planar cracking that occurs in carbon and low alloy steels when atomic hydrogen ions diffuse into impurity pockets of steel and then combines into molecular hydrogen at trap sites, expanding the pocket to a crack formation.

Hydrogen stress cracking (HSC) and Sulfide Stress Cracking (SSC)

Cracking that results from the presence of hydrogen in metal and tensile stress (residual and/or applied).

Stepwise cracking (SWC)

Cracking that connects hydrogen-induced cracks on adjacent planes in steel.

Stress corrosion cracking (SCC)

Cracking of metal involving anodic processes of localized corrosion and tensile stress (residual and/or applied). Does not involve hydrogen in its definition, but SCC can be accelerated in the presence of water and H₂S.

Stress-oriented hydrogen-induced cracking (SOHIC)

Staggering small cracks formed approximately perpendicular to the principal stress (residual or applied) resulting in a "ladder-like" crack array linking of Hydrogen induced cracks.

Soft-zone cracking (SZC)

Form of SSC that can occur when steel contains a local "soft zone" of low-yield-strength material. Does not involve hydrogen in its definition, but hydrogen embrittlement can accelerate cracking in "soft zones".

Galvanically induced hydrogen stress cracking (GHSC)

Cracking that results due to the presence of hydrogen in a metal induced in the cathode of a galvanic couple and tensile stress (residual and/or applied).

4 Theory on steel

The main objective of this theory chapter is to present the fundamentals of steel and welding. It includes the essentials of material science that are considered by the author as mandatory knowledge for understanding the reaction of steel on its exposure to heat.

4.1 Welding

Welding is the most effective way of joining metal pieces together, by heating them up to a melting point until they meld together. It can be done with or without filler material. The main task and challenge when defining a welding procedure are to create a procedure that gives a weld that is as strong as the surrounding metal.

Internationally approved welding processes are listed in ISO 4063 standard that covers almost 100 welding processes in addition to brazing, soldering, cutting and gouging. Choice of welding process depends on factors like production volume, accessibility, base metal type and condition, fabrication costs, production speed, and others.

Welding processes that are used today can be split into 5 main groups: Arc, Resistance, Gas, Pressure and Beam welding.

Arc welding	Short circuit welding, that creates an electric arc between two electrodes. Works with both AC and DC currents and produces a heat of up to 7000 °C inside the arc region. The welding area is usually protected by shielding gas, vapor or/and slag. This is a relatively cheap and well-known way of welding. Most common arc welding processes is GMAW (MAG/MIG), GTAW (TIG), FCAW, SMAW and SAW.
Resistance welding	Electrode on each side of the weld area generates heat by passing a strong electrical current through the working piece. The weld is created by heat from electrical resistance, combined with the time and force used to hold the materials together.
Gas welding	Simply explained it is a welding process that melts and joins metals by heating them up with a flame that is produced from fuel gasses and oxygen.
Pressure welding	Welds materials together by applying heat and mechanical pressure on the joining section. It is a fast process that can be performed in ambient temperature without the use of gasses and additives.
Beam welding	Generate a focused stream of high energy electrons in a vacuum. Giving a precise weld with a very small heat affected zone and high purity because of the vacuum environment.

Weldability

Weldability of a material describes its ability to be welded by a specific process and under specific conditions and depends mainly on the following factors: material composition and microstructure, choice of welding process and the welding environment.

Definition of weldability in ISO/TR 581 -2005 standard is as follows:

"A component consisting of metallic material is considered to be weldable by a given process when metallic continuity can be obtained by welding using a suitable welding procedure. At the same time, the welds shall comply with the requirements specified in regard to both their metallurgical and mechanical properties and their influence on the construction of which they form a part."

4.1.1 Carbon equivalent

Properties that are of the most importance when considering material weldability is the equivalent carbon content in the base and filler material. There is a range of carbon equivalent formulas out there, but the following types are most commonly used:

Carbon equivalent values (CE):

$$CE = C + \frac{Mn}{6} + \frac{Cr + Mo + V}{5} + \frac{Ni + Cu}{15}$$

This Carbon Equivalent formula was developed by John Dearden and Hugh O'Neill's in 1940 and further adopted by International Institute for Welding (IIW) in 1967 and somewhat simplified. It is mainly, but not exclusively, based on extensive experience data from carbon manganese type steels and is applicable to steels with a carbon equivalent (CE) in the range 0,30 % to 0,70 %.

The range of chemical composition of a material that can be used with this formula is specified in EN 1011:

- *Carbon* *0,05 to 0,25*
- *Silicon* *0,8 max.*
- *Manganese* *1,7 max.*
- *Chromium* *0,9 max.*
- *Copper* *1,0 max.*
- *Nickel* *2,5 max.*
- *Molybdenum* *0,75 max.*
- *Vanadium* *0,20 max*

Above mentioned formula (CE) is described as Method A in EN 1011 standard. While for low alloy high strength steels, Method B (CET) is preferred:

$$CET = C + \frac{Mn + Mo}{10} + \frac{Cr + Cu}{20} + \frac{Ni}{40}$$

The range of chemical composition of the material that can be used with this formula:

- Carbon 0,05 to 0,32
- Silicon 0,8 max.
- Manganese 0,5 to 1,9
- Chromium 1,5 max.
- Copper 0,7 max.
- Molybdenum 0,75 max.
- Niobium 0,06 max

Crack susceptibility factor (P_{cm}):

$$P_{cm} = C + \frac{Si}{30} + \frac{Mn}{20} + \frac{Cu}{20} + \frac{Ni}{60} + \frac{Cr}{20} + \frac{Mo}{15} + \frac{V}{10} + 5B$$

This formula for cracking parameter was developed in 1968 by Yoshinori Ito and Kiyoshi Bessyo who thought that Dearden and O'Neill used a testing method that was not precise and did not provide sufficient restraint to induce weld cracking. The formula was adopted by the Japanese Welding Engineering Society (JWES) in 1973 and is widely used for low carbon alloys, typically for steels with carbon mass fraction equal to or less than 0,12 %. It is also adapted by API 5L standard for use with PSL 2 pipes.

Carbon equivalent number (CEN):

$$CEN = C + A(C) * \left(\frac{SI}{24} + \frac{Mn}{6} + \frac{Cu}{15} + \frac{Ni}{60} + \frac{Cr + Mo + Nb + V}{5} + 5B \right)$$

The newest carbon equivalent formula, CEN, was proposed to evaluate the weldability of a wide variety of steels. It incorporates both CE from IIW and P_{cm} . The CEN is similar to the P_{cm} with carbon levels less than 0,17% and to CE for steels with high carbon content.

Yurioka [4] illustrated a good correlation between P_{cm} and CEN:

$$CEN = 2P_{cm} - 0.092 \quad (C \leq 0.17\%)$$

$$CEN = CE_{IIW} + 0.012 \quad (C \geq 0.17\%)$$

CEN applies well to both traditional steels (covered by CE) and low carbon low alloy steels (covered by P_{cm}) because of the hyperbolic tangent "tanh" term in the accommodation factor, A(C):

$$A(C) = 0,75 + 0,25 * \tanh\{20 * (C - 0,12)\}$$

4.2 Phase diagram

Material can take different phases, depending on temperature and composition. Material at different temperature stages can consist of single (homogenous) or mixed (heterogeneous) phases. Iron-Carbon system has liquid phase and three solid phases at atmospheric pressure¹. Those are denoted with Greek letters α , γ , δ . Residual carbon that cannot dissolve in allotropic² forms of iron, dissolves in another phase called Cementite³. See Figure 2 for phase diagram of Iron-Carbon alloy.

The first possible phase of iron is called delta phase (**δ -ferrite**) where the maximum concentration of carbon is 0.09 % at 1493 °C, but delta ferrite can exist at between temperature range 1392 °C and 1536 °C, depending on carbon concentration. It has BCC crystal structure like α -ferrite, but with a greater lattice constant⁴ because of the high temperature that increases the vibration of the atoms and thereby the distance between them.

The next phase is called gamma phase (**γ -austenite**), where the concentration of carbon can be as high as 2,06% carbon at 1147 °C, but austenite can exist at a temperature range between 727 °C and 1493 °C with lower carbon concentrations. This phase has FCC crystal structure.

At temperatures lower than 727 °C, the iron can only exist as alfa phase (**α -ferrite**). This phase cannot dissolve more than 0,02 carbon so the residual carbon will form cementite phase.

For steels with carbon concentration under 0,8 %, the nucleus of γ -ferrite will begin to appear and grow in between austenite cells as temperature falls. At temperature 727 °C, residual austenite will almost spontaneously turn into the layered structure of ferrite and cementite. This layered structure is called Pearlite.

Iron-Carbon composition with more than 2,06% carbon will form an additional material structure called ledeburite. Ledeburite is equal to perlite but it forms directly from the liquid phase. Alloy with more than 2,06% carbon are ranged under cast iron and are generally more hard and brittle. Cast Iron is not part of this paper and will not be discussed any further.

¹ Fourth phase, ϵ , with hexagonal close-packed (hcp) structure exists at pressure above 10 GPa.

² Allotropy is the property of some elements to exist in different forms. Diamond and graphite are two allotropes of carbon.

³ Cementite is iron carbide with formula Fe_3C and orthorhombic crystal structure that dissolves 6,67 % carbon.

⁴ Lattice constant refers to the physical dimension of unit cells in a crystal lattice.

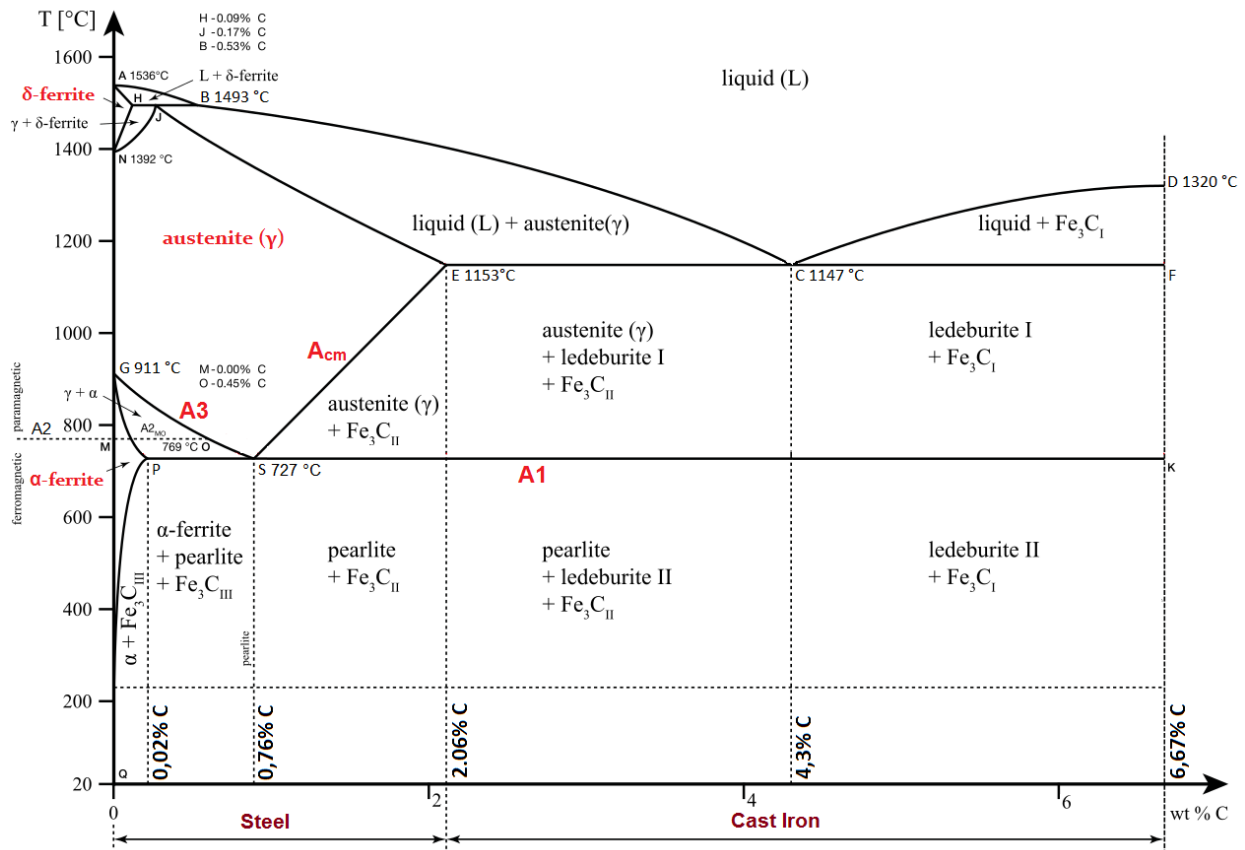


Figure 2: Iron-carbon phase diagram.

Adopted from Wikipedia.org, article: Steel. License: CC BY-SA 4.0

Critical temperatures:

As can be seen from the phase diagram (see Figure 2) the temperature boundaries between phases are marked with letters: A_1 , A_2 , A_3 , A_{cm} .

A_1 : Lower critical temperature.

It is the temperature of the austenite-to-pearlite eutectoid transformation. Below this temperature austenite does not exist. For steel, this temperature is 727 °C

A_2 : Magnetic transformation temperature.

Also called Curie temperature, is the temperature above which steel loses its permanent magnetic properties. For Iron, this temperature is 768 °C

A_3 : Hypoeutectoid critical temperature.

It is the temperature, below which ferrite starts to form from austenite in the hypoeutectoid alloys.

A_{cm} : Hypereutectoid critical temperature.

It is the temperature, below which cementite starts to form from austenite in the hypereutectoid alloys.

4.3 Effect of alloying agents

Steels of the simplest type consist of Iron and carbon for which Iron Carbon steel diagram is applicable. Although in practice pure steel is never used and alloying elements are always added to achieve the desired properties. In cases when more than just carbon is used as an alloyant, different phase diagram will apply, but the concept of equivalent carbon content that was described earlier can also be used to some extent. Carbon equivalent is mostly used to estimate hardness and thereby the weldability of steel. High hardness will significantly increase the risk of hydrogen cracking as it was described in pre-project for this thesis [1].

Alloy steel is steel that is alloyed with a variety of elements. The difference between low and high alloy steel are not clearly defined and varies from source to source, but usually, it lies somewhere around 5% of alloying elements. We can thereby classify alloy steels as follows:

- **Low alloy steels:** in which the sum of total Alloying elements is < 5%
- **High alloy steels:** in which the sum of total Alloying elements is > 5%

Iron

Steel is a mixture of several metals but mostly it consists of iron, where the content is no less than 97,9 % in pure carbon steel and no less than 50% for high alloy steel. Iron is much softer and weaker than steel, but because of its ability to take different allotropic forms, it can dissolve over 2 % of carbon in austenitic form. This ability allows for the creation of other materials like “steel” with improved material properties.

Carbon

This is the most important alloying element in steel, the presence of carbon in iron is necessary to make steel. Carbon is an essential element for the formation of carbides in steel that increases its tensile strength and hardness.

Alloying elements can be classified into categories but most of them are multiplayer and will participate in more than one category. Suggested categories:

- Substitutional Ferrite stabilizers
- Substitutional Austenite stabilizers
- Carbide formers
- Nonmetallic and Interstitial Alloys

Ferrite stabilizers are the element which promotes the formation of ferrite phase by increasing phase transformation temperature. (e.g. Cr, Mo, Ti, V and Al).

Austenite stabilizers are the element which stabilizes austenite by increasing the austenitic area in iron carbon diagram. It can be done by decreasing eutectoid composition and decreasing the eutectoid temperature with help of alloy elements like Mn and Ni, which are the most common austenite stabilizers.

Carbide formers are elements that form stable carbides and is composed of carbon and a less electronegative element (e.g. Cr, Mn, Mo, W, V, Ti, Zr and Nb). Fine particle carbides can either increase the strength by participation hardening¹ or by acting as an obstacle to the motion of dislocations. Thus increasing the hardness and wear resistance of steels.

It should be noted that the formation of carbides in martensitic steel will have an opposite effect than in steels that is in an equilibrium state and will reduce its hardness.

Nonmetallic alloys (e.g. Si, S P, H, C, N and O). Oxygen and Hydrogen are impurities in material and is unwanted in steel structures. The other elements have different tasks that will be briefly described further in this chapter. H, C, N and O can be arranged in a subgroup of interstitial alloying elements which have smaller atom size and normally takes unoccupied sites in the crystal structure.

Alloy elements

Nickel, Ni

Melting point 1453 °C

Nickel is a metal with face-centered cubic and therefore it is an austenite promoter. It is widely used for manufacturing of austenitic stainless steels. Nickel is also used in carbon steels to increase toughness and hardenability.

Manganese, Mn

Melting point 1221 °C

Manganese is another metal with face-centered cubic and thereby an effective austenite promoter. It assists in the deoxidation of the steel, prevents the formation of iron sulfide and inclusions, and promotes greater strength by increasing the hardenability of the steel. Most steels contain at least 0,3% manganese but it can be as high as 1,5 % in some carbon steels.

Silicon, Si

Melting point 1414 °C

Silicon is an effective deoxidizer and is often used in weld metal. It dissolves in iron and tends to strengthen it, but excessive amounts can also reduce the ductility.

Aluminum, Al

Melting point 658 °C

Aluminum is mainly used as a deoxidizer, but can also restrict austenite grain growth in reheated steels and is normally added as a grain refiner for improved toughness.

Copper, Cu

Melting point 1083 °C

Copper alloying is a means of securing atmospheric corrosion resistance when. It is effective when present in amounts of more than 0.30% in steels. It can dissolve in iron in an amount of up to 1%, extensive amount will form metal inclusions².

¹ Participation hardening is heat treatment technique that that is used to harden the material and increase its yield strength by producing fine particles of an impurity phase.

² An inclusion is a material that is trapped inside another material during its formation.

Nitrogen, N

Melting point -210 °C

Increasing N-content influences diffusion. It supports the strengthening and delayed precipitation of carbides and inter-metallic-phases during heat treatment. Although usually affords are made to eliminate hydrogen, oxygen and nitrogen from steel as it tends to promote brittleness.

Chromium, Cr

Melting point 1903 °C

Chromium reduces the critical rate of cooling necessary for martensite formation, thus increases hardenability, that gives improved susceptibility to hardening and tempering processes. Thermal and electrical conductivity is reduced in chromium steels, leading to reduced thermal expansion and smaller HAZ. The tensile strength of the steel increases by 80-100 N/mm² per 1% Cr.

To inhibit corrosion, at least 11% chromium is added to stainless steel so that a hard oxide layer can be formed on the metal surface.

Molybdenum, Mo

Melting point 2620 °C

Because of its high melting point, it increases the high-temperature strength, reduces the critical cooling rate and improves hardenability. Molybdenum reduces temper brittleness and promotes fine grain formation, thus favorably effects the weldability of steels.

Tungsten, W

Melting point 3410 °C

Another metal with a high melting point that increases the high-temperature strength and wear resistance of steel. It is therefore alloyed primarily to high speed and hot forming tool steels, as well as creep-resistant and ultra-hard steels.

Vanadium, V

Melting point 1890 °C

Vanadium forms stable nitrides and carbides, resulting in a significant increase in the strength of steel. It also refines grains giving fine and strong casting structure. Combination of its strength and ferrite promoting properties, thus shift of critical point to elevated temperatures makes it a perfect candidate for high-speed tool steels. Vanadium high-carbon steel alloys contain 0.15% to 0.25% vanadium, and high-speed tool steels (HSS) have a vanadium content of 1% to 5%.

Titanium, Ti

Melting point 1660 °C

Titanium has high affinity to oxygen, nitrogen, sulfur and carbon, thus making it a multilayer in alloyed steel with its deoxidizing, denitriding, Sulphur bonding and carbide forming properties.

As ferrite stabilizer, it is also used to reduce the grain size of austenite.

Niobium, Nb

Melting point 2468 °C

Niobium is very pronounced carbide former, thus usually alloyed in high temperature and chemical resistant steels. As melting point indicates, it is also pronounced ferrite stabilizer.

Zirconium, Zr

Melting point 1855 °C

Zirconium is a carbide former. It increases strength and limits grains sizes of steel. Very popular as an alloying element for deoxidation, as it leaves living little deoxidation products behind compared to other deoxidation agents. Produces restriction to the γ -phase.

Boron, B

Melting point 2300 °C

Boron is added to steel for only one reason: to enhance its hardenability. The optimal amount of Boron in solution with iron is always in between 0,001% and 0,005%. Adding too much will reduce the hardenability effect. Boron has a strong tendency to combine with oxygen and nitrogen to form B_2O_3 and BN. Therefore it is often added to deoxidized steel.

Phosphorus, P

Melting point 44 °C

Phosphorus is usually regarded as a steel parasite because of its low melting temperature and thus segregation on solidification of the melt. An attempt is made to keep the P content very low, with an upper limit of 0.03-0.05.

Sulfur, S

Melting point 115 °C

Another steel segregator that should be kept to a minimum in steels. Although Sulfur is sometimes added intentionally to steel for automatic machining in amount of up to 0,4% as an attempt to reduce the friction of the cutting tool.

4.4 Residual Stresses and thermal expansion

Residual stresses are stresses that persist in a solid material (that is free of external forces or temperature gradients) after the original cause of the stresses has been removed. The original cause of stresses can be split in two groups: cold deformation and thermal expansion.

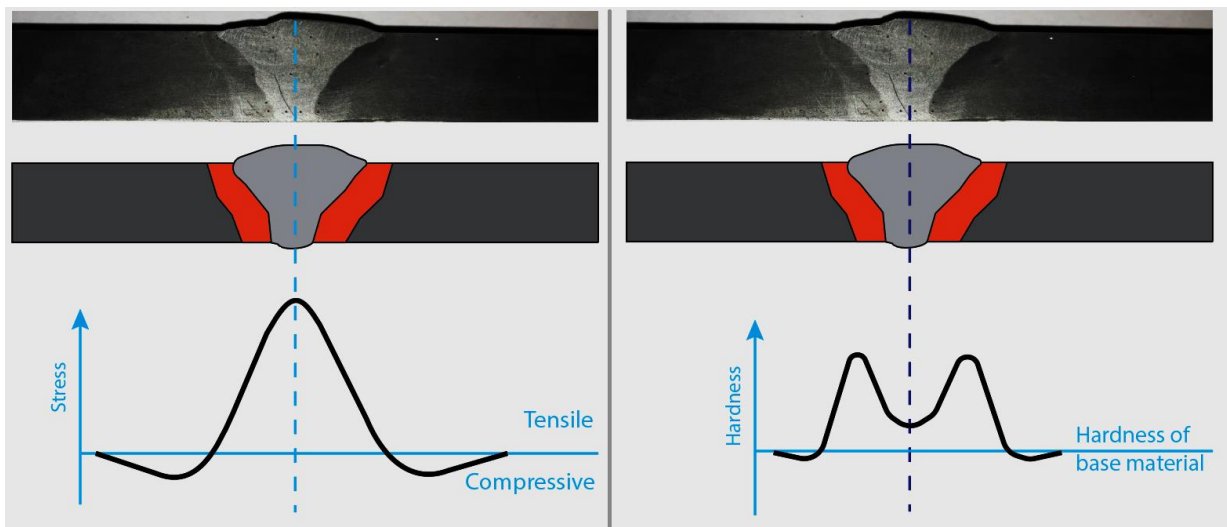


Figure 3, Residual stresses and Hardness profile of a weld

Made by author, License: CC BY-SA 3.0

In the case of welding, the residual stresses are the result of localized expansion during heating. After the weld is applied, both heat affected zones and the weld will start to cool and contract, leaving behind residual stresses and zones with increased hardness and brittleness, see Figure 3.

From Figure 3 it can be seen that hardness is mainly increased in HAZ of the base material, where the material was heated above its critical point and then relatively quickly cooled in room temperature. Hardness profile can be effectively altered by preheating the material or reducing the rate of cooling.

4.5 Recovery, recrystallization and grain growth

Residual stresses is a result of changes in microstructure that include changes in grain shapes as a result of plastic deformation, strain hardening, increased dislocation density and impurity mechanisms. Material strength and other properties like electrical conductivity and corrosion resistance can be modified as a result of stored internal strain energy. Material properties can revert back fully or partially by appropriate heat treatment or annealing.

Recovery of material can be described by two processes that take place at elevated temperatures: recovery and recrystallization. Continues heating after recrystallization can trigger extensive grain growth and weakening of the material.

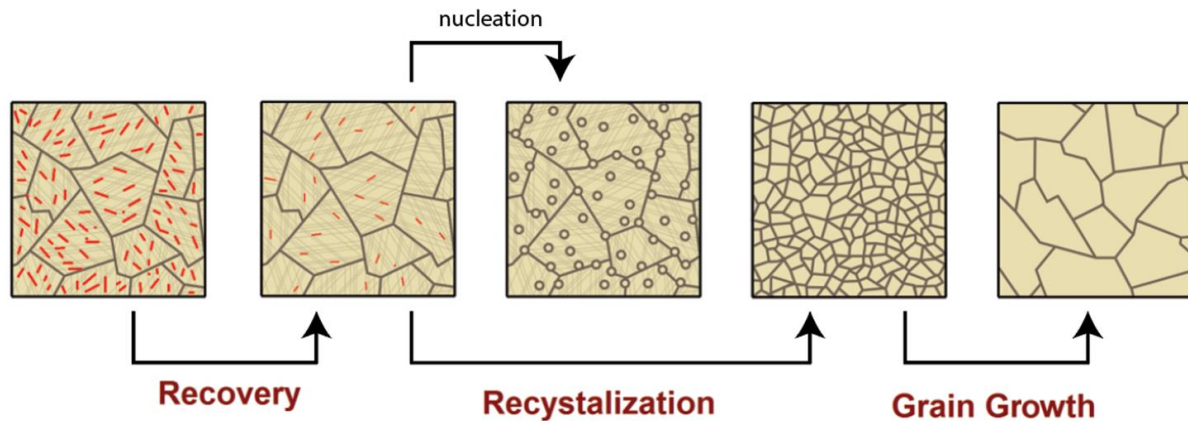


Figure 4, Recrystallization steps of a metallic material

Adapted and transformed from Wikipedia, work of Daniele Pugliesi. License: CC BY-SA 3.0

Recovery

The mechanism of recovery is mainly motion of vacancies in microstructure. This involves a migration of point defects to the grain boundaries or dislocations, and a combination of point defects. Recovery is the main mechanism that releases the internal stresses in welded materials without decreasing the strength that was acquired during material working and heat treatment.

As the material is heated during the recovery, the yield and tensile strength are reduced (see Figure 5), allowing for the stored internal strain energy to be relieved by a dislocation movement.

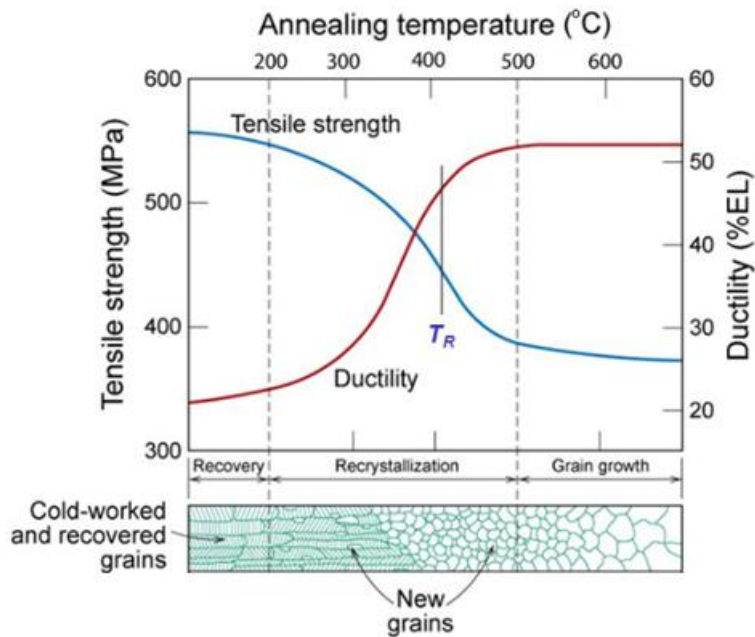


Figure 5, Influence of annealing temperature on strength and ductility of brass.

Adapted from Fig 7.22, *Material Science and engineering* by W. D. Callister and D. G. Rethwisch 8e.

It is important to note that although recovery significantly reduces the residual stresses in the material, it does not get rid of them all. Leaving some of the most persistent ones left, that can further trigger nucleation points at elevated temperatures.

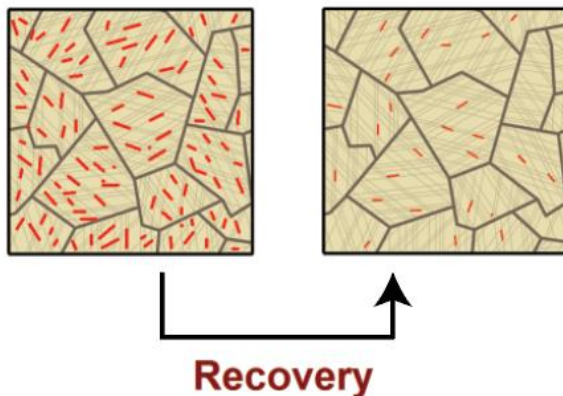


Figure 6, Recovery process

Adapted and transformed from Wikipedia, work of Daniele Pugliesi. License: CC BY-SA 3.0

Recrystallization

As mentioned above, even after the recovery is complete, the grains are still in relatively high strain energy state. In order to completely relieve the material structure from residual stresses, the material must be heated to the temperature of recrystallization¹, T_R .

An important thing to notice is that recrystallization temperature depends on many variables and is not a fixed temperature similar to melting temperature or critical temperature of alloys. The increased annealing time reduces the recrystallization temperature. Although the extent of a recrystallization

¹ Recrystallization temperature is the temperature at which a specific material is recrystallized within approximately one hour.

process is dependent on both time and temperature, nucleation will not be triggered under eutectic temperature unless there is sufficient strain energy in the material to trigger nuclei and to drive their growth. Thus the driving force to produce new strain-free grain structure is the difference in internal energy between the strained and unstrained material.

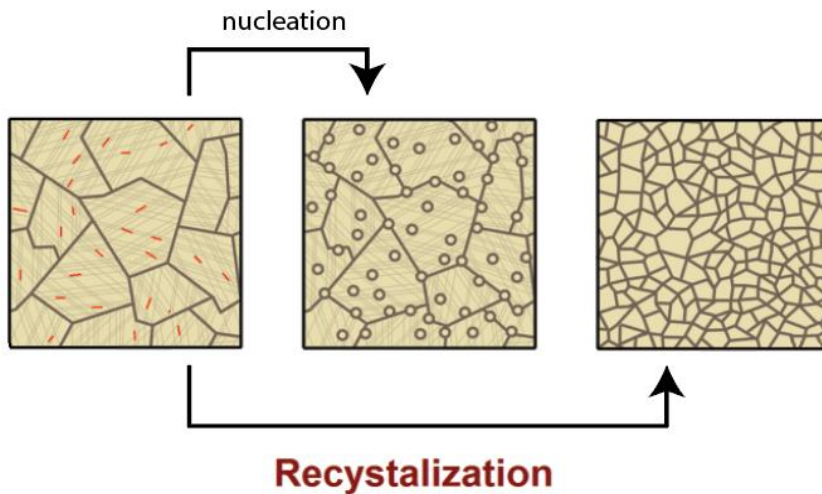


Figure 7, Recrystallization process

Adapted and transformed from Wikipedia, work of Daniele Pugliesi. License: CC BY-SA 3.0

Recrystallization is easier in pure metals than in alloys and occurs at lower temperatures, at approximately 30% of melting temperature, while it requires as much as 70 % of melting temperature for high alloyed steels. Thereby it can vary in the range of $0,3T_m - 0,7T_m$, depending on the amount of alloying elements in steel [5]. The recrystallization temperature of pure Iron is 450 °C, thus increasing with the amount of alloying elements.

Grain Growth

After recrystallization is complete, the structure will be completely free from internal stresses, but the strain-free grains will continue to grow at the expense of their neighbors if the material is kept at elevated temperature. Large grains will grow at the expense of small ones.

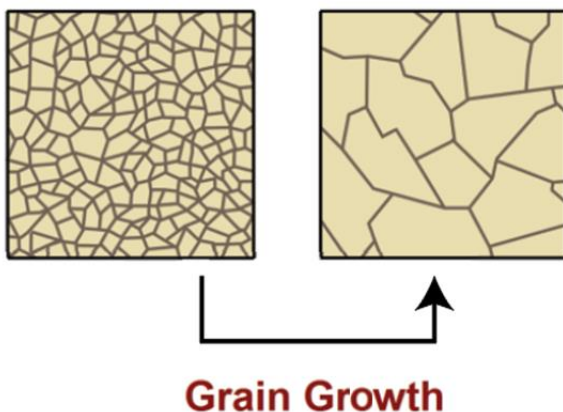


Figure 8, Grain growth process

Adapted and transformed from Wikipedia, work of Daniele Pugliesi. License: CC BY-SA 3.0

The driving force for grain growth is the energy that is associated with grain boundaries. As grains increase in size, the total boundary area decreases, leading to a reduction in the total energy of the

structure. An important fact is that grain growth does not require recovery and recrystallization prior to it.

4.6 Heat affected zone

The **heat-affected zone (HAZ)** is the area of the base material which is not melted but had its microstructure and properties altered by high heat input from the weld. A simplified area split of a welded joint is represented in Figure 9. Fusion zone is a small area of the melted pool that combines both base and filler materials.

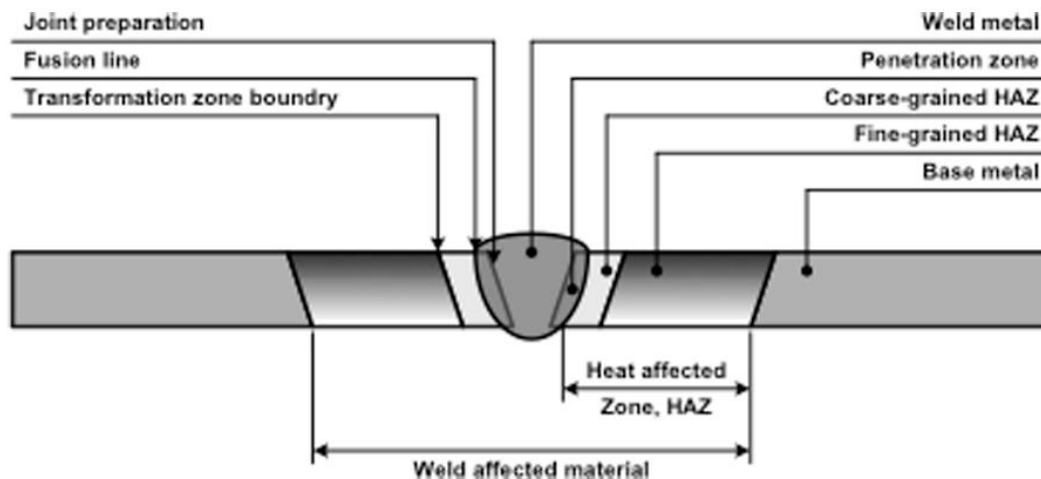


Figure 9, Areas in a welded material

Copied from <https://weldingproductivity.com>, work of Kirk Beaster, Article: Detrimental elements ©2019 Welding Productivity® Magazine Inc.

The amount and type of change in microstructure are dependent on the temperature, exposure time and cooling rate. The relation between temperature and various HAZ regions is represented in Figure 10.

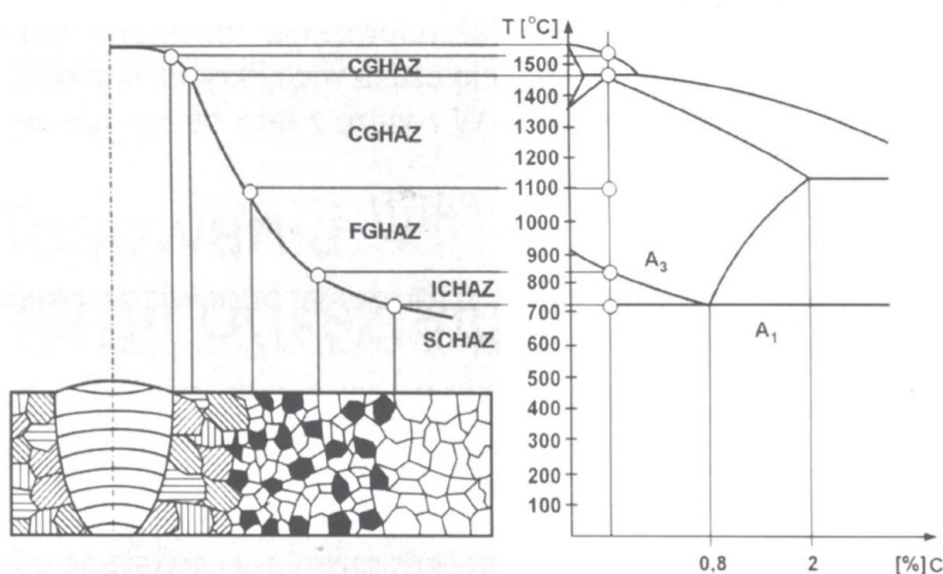


Figure 10, Regions in HAZ & corresponding phase diagram

Copied from <http://www.imim.pl>, work of Z. Śloderbach, Article: determination of ranges of components of heat affected zone including changes of structure.

CGHAZ (Coarse Grain HAZ) – HAZ with coarse grain structure, heated to a temperature ($1100\text{ °C} \leq T_{\max} \leq T_m$), where: T_m – steel melting point.

FGHAZ (Fine Grain HAZ) – HAZ with fine grain structure, heated to a temperature above A3 ($900\text{ °C} \leq T_{\max} \leq 1100\text{ °C}$),

ICHAZ (Inter critical HAZ) – HAZ heated to a temperature above A1 in the range A1 -A3 ($700\text{ °C} \leq T_{\max} \leq 900\text{ °C}$),

SCHAZ (Sub critical HAZ) – HAZ heated to a temperature below A1 in the range ($600\text{ °C} \leq T_{\max} \leq 700\text{ °C}$).

4.7 Microstructures of Iron-Carbon Alloys

There is a variety of possible microstructures of Iron steel that can be formed depending on heat treatment methods used during production or after weld. Those are: Pearlite, Bainite, Martensite and Spheroidite. All of them mentioned consists of main phases: Ferrite, Austenite and carbides (mainly Cementite).

4.7.1 Pearlite

Pearlite is a two-phase, layered structure composed of layers of ferrite (87.5 %) and cementite (12.5 %). It is formed during sufficient slow cooling from the austenite phase.

Transformation happens at so-called eutectoid temperature of steel, at 727 °C . As austenite cannot exist at lower temperatures, all austenite will be transformed to ferrite, but since ferrite cannot take all the carbon that was dissolved in austenite, it will be forced to form carbides with iron, called cementite. Leading to the layered structure of ferrite and cementite, see Figure 11. The layered structure of pearlite steels makes it one of the strongest commercial bulk materials on earth.

Figure 12 illustrates a pearlite structure under a microscope. The material on the picture is hypo eutectoid, thus with a carbon content of less than 0,76%

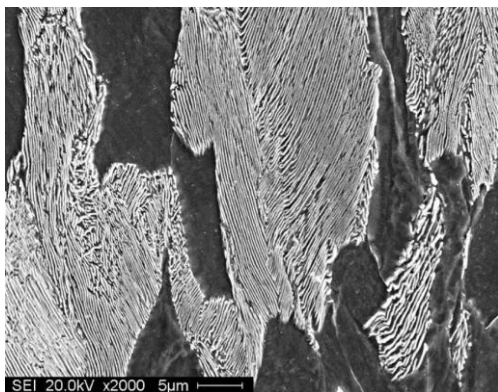


Figure 12, Pearlite structure in hypo eutectoid steel

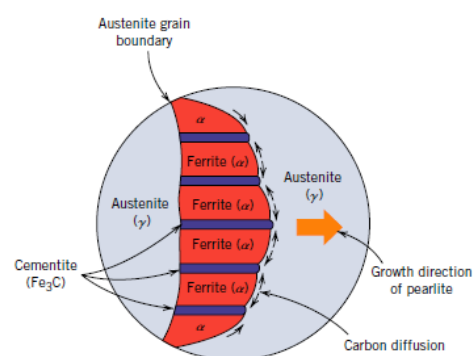


Figure 11, Formation of pearlite from austenite

Adapted from Fig 9.28 Material Science and engineering by W. D. Callister and D. G. Rethwisch 8e.

Copied from Wikipedia.org. Article: Pearlite. License: Public domain

There are three types of steels: Hypoeutectoid, eutectoid and hypereutectoid. The difference is the amount of carbon present in the alloy. It is so that steel cannot hold more than 0,76% carbon, without being forced to form iron carbides prior to the eutectoid temperature of 726 °C during cooling. Less than 0,76 % carbon will trigger the formation of ferrite along grain boundaries of austenite prior to the eutectoid temperature of 726 °C during cooling. That makes steel with 0.76 % carbon the iron-carbon composition that can transform entirely into pearlite, see Figure 13.

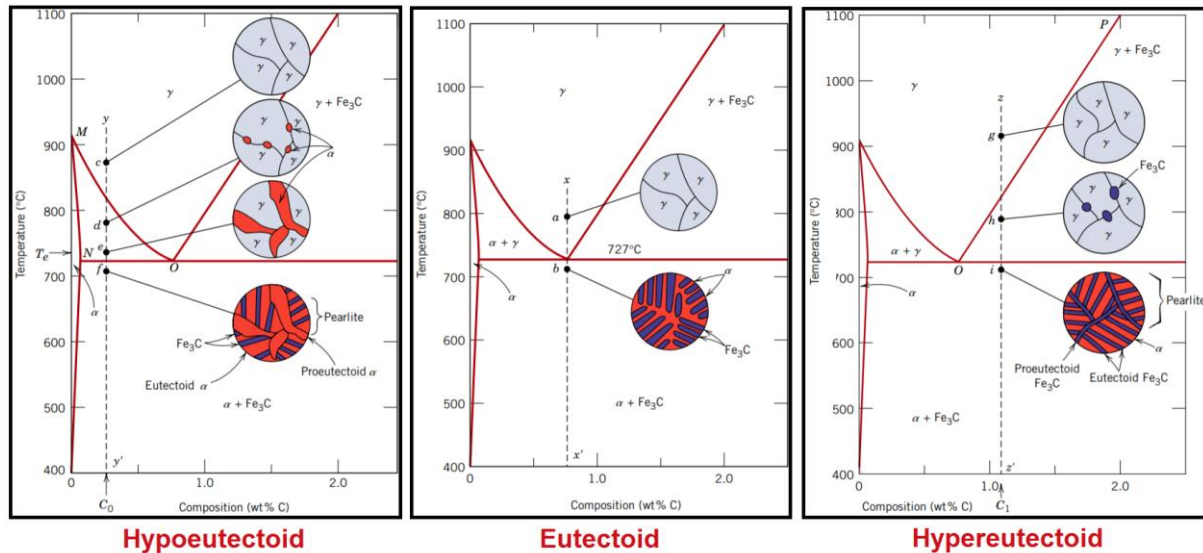


Figure 13, Schematic representation of pearlite in steel

Adapted from Fig 9.26, 9.29 & 9.32 Material Science and engineering by W. D. Callister and D. G. Rethwisch 8e.

Coarse and fine pearlite

Pearlite is further divided into two types: coarse and fine pearlite. Coarse pearlite is produced just below eutectoid temperature. The difference between those type is the thickness of ferrite and cementite layers. The lower the temperature, the slower is the diffusion rate of carbon and the layer becomes progressively thinner.

4.7.2 Martensite

Martensite is not present in the phase diagram of the iron-carbon system because it is not an equilibrium phase. Equilibrium phases are formed by slow cooling rates that allow sufficient time for diffusion, whereas martensite is formed by high cooling rates.

At high cooling rates (quenching) some carbon does get enough time to diffuse out of the austenite to form cementite. This lead to trapped interstitial carbon atoms, because of that FCC structure (austenite) are unable to transform to BCC structure (ferrite). Instead, austenite is transformed to the body-centered tetragonal (BCT) structure. A unit cell of BCT crystal structure is simply a body-centered cube that has been elongated along one of its sides, but it is distinctly different from BCC in the way that it stores much more strain energy and is much harder structure because of the trapped carbon. The mechanism of transformation is illustrated by, Figure 14. The illustration shows that the instead of long diffusion of carbon atoms they are kept in their position, forcing FCC structure (austenite) into BCT structure (ferrite), that is called "shear transformation".

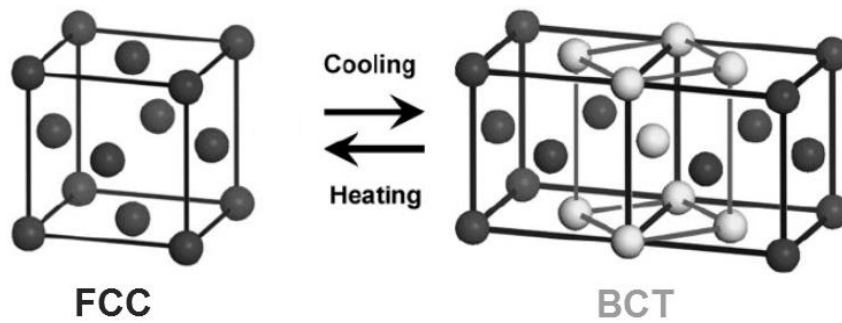
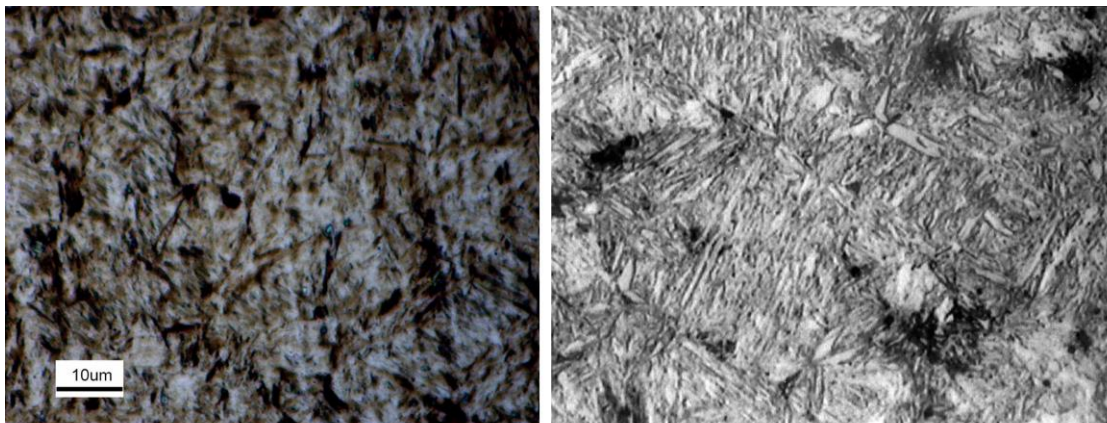


Figure 14, Austenite to martensite transformation.

Made by author, License: CC BY-SA 3.0

The mechanism of martensite transformation is not entirely clear. Though it can be said that the transformation from austenite to martensite is in a way resembles the formation of pearlite, in the way that it also starts at the cell boundaries and grow in one direction forming a layered structure, but layers are of the same, BCT structure, type. Martensite has needle-like structure, if looked at under the microscope, see Figure 15. The reason for such structure can be the fact that transformation from austenite happens at the speed of sound and starts almost simultaneously at grain boundaries during rapid cooling.



AISI 4130 Steel, oil-quenched from 850 °C

0.35%C Steel, water-quenched from 870 °

Figure 15, Martensite structure.

Adopted from Wikipedia.org. Article: Martensite. License: Public domain

Tempered martensite.

Tempering¹ is often used as a heat treatment of martensite in steels. During martensite tempering, in addition to an extensive recovery of the dislocation structure, the excess carbon in solid solution segregates and forms carbon flakes inside martensite plate. That leads to less brittle structure. Heavy (long) tempering leads to the recrystallization of the ferrite plates into equiaxed grains, see Figure 16.

¹ Tempering is a heat treatment process changes microstructure and mechanical properties as the metastable sample is held isothermally at a temperature below critical where austenite cannot form.

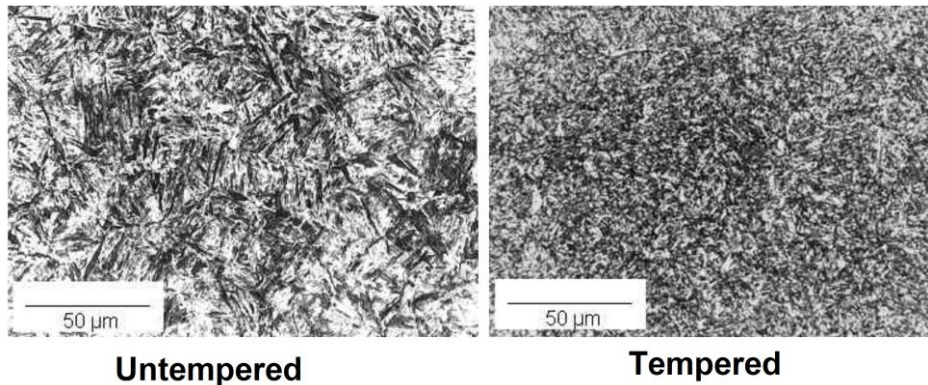


Figure 16, Martensite tempering.

Copied from <http://threepines.net/martensite.html>. Author: Peter L Berglund. Article: Martensite. License:

It can often be hard to recognize tempered martensite as it does not resemble a needle-like structure and can look more like a noise from a tv screen, see Figure 16. See also Figure 17 for high-resolution photo of tempered martensite.

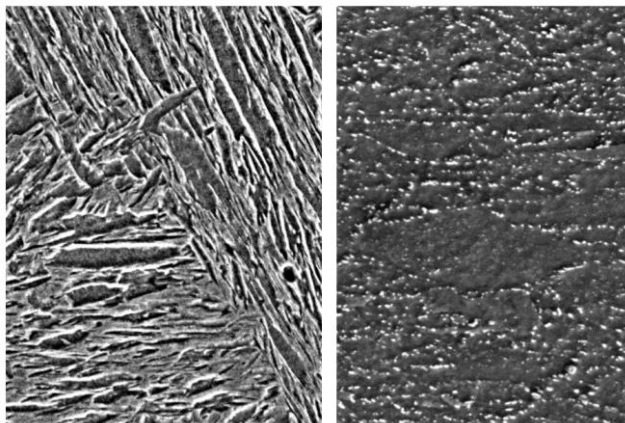


Figure 17, Tempered martensite, high resolution image.

Copied from CiteSeerx. Author: Hector Pous-Romero. Article: 'Coalesced Martensite in Pressure Vessel Steels'.

Tempering stages

1. Steel with more than 0.2% carbon produces highly unstable martensite because of supersaturation, and interstitial diffusion of carbon in BCT martensite can occur [6]. Thus, in the first stage of tempering, the decomposition of martensite into less tetragonal needles and transition carbides (ϵ -carbide, $\text{Fe}_{2.4}\text{C}$ and η -carbide, Fe_2C).
2. At the second stage of tempering retained austenite decomposes into lower bainite (see next chapter). Note that the amount of retained austenite in the as-quenched steel depends on the composition of the steel, and the temperature to which steel was quenched.
3. During the next stage of tempering martensite completely loses its tetragonality and transition carbides grow into cementite.
4. Further tempering leads to cementite coarsening and eventually to spheroidisation.

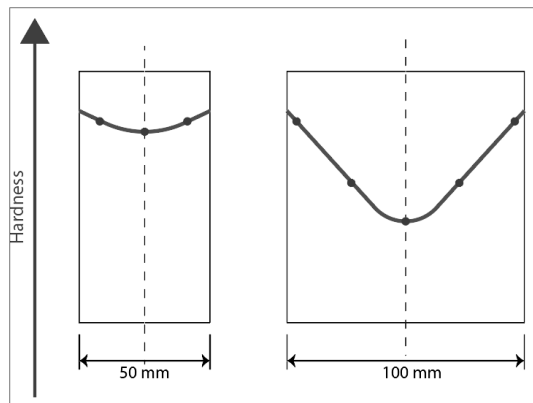


Figure 18, Hardness vs material thickness

It is also important to notice that the rate of temperature changes in thick materials varies across its cross-section, thus the cooling rate away from the quenched surface is somehow slower. Because of that higher rate of diffusion can be possible away from the surface, allowing for less martensitic structure to be formed. This can be illustrated by hardness diagram, see Figure 18

Made by author, License: CC BY-SA 3.0

Temperature range of martensite formation spreads over a range that is dependent on the material composition. For eutectoid steel, the range is somewhere between 120 and 220 °C, see Figure 19.

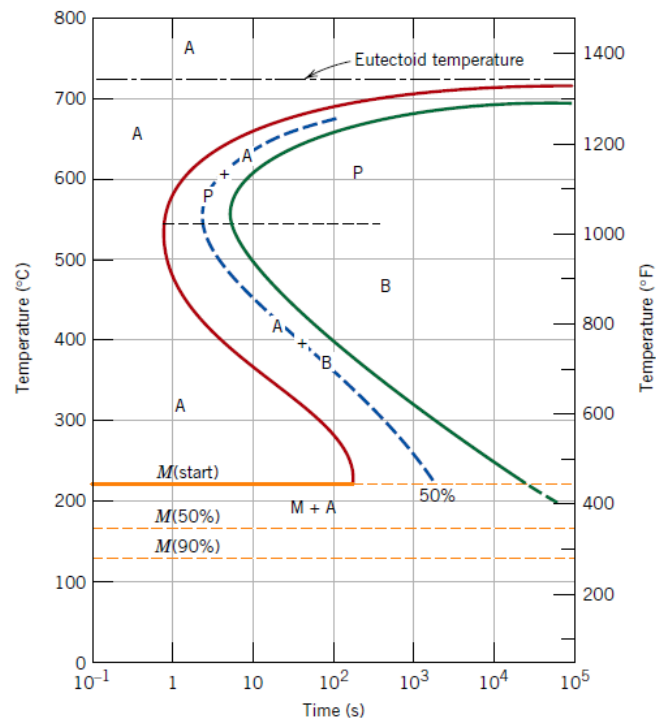


Figure 19, Isothermal transformation diagram for an eutectoid iron-carbon alloy

Copied from Fig 10.22 Material Science and engineering by W. D. Callister and D. G. Rethwisch 8e.

4.7.3 Bainite

Between the temperature range of martensite and pearlite, another microstructure can be formed called bainite, see Figure 19. The temperature range for bainite in carbon-iron steel is somewhere around 220 – 550 °C (depending on the alloy composition).

Because of a similar aspect of transformation mechanism, bainite and martensite can at first seem very similar and resemble each other as a needle-like structure. Still, mechanical properties and chemistry of microstructure are very different. While martensite is a separate phase with highly strained body-centered tetragonal structure, the bainite is a composition of ferrite and cementite.

Since the diffusion rate is not sufficient for carbon to diffuse to the grain boundaries to form separate layers of iron carbides, cementite is formed inside the ferrite layers. As the temperature falls, the diffusion rate falls with it, reducing the diffusion distance of carbon and thereby making the cementite island smaller and smaller, see Figure 20.

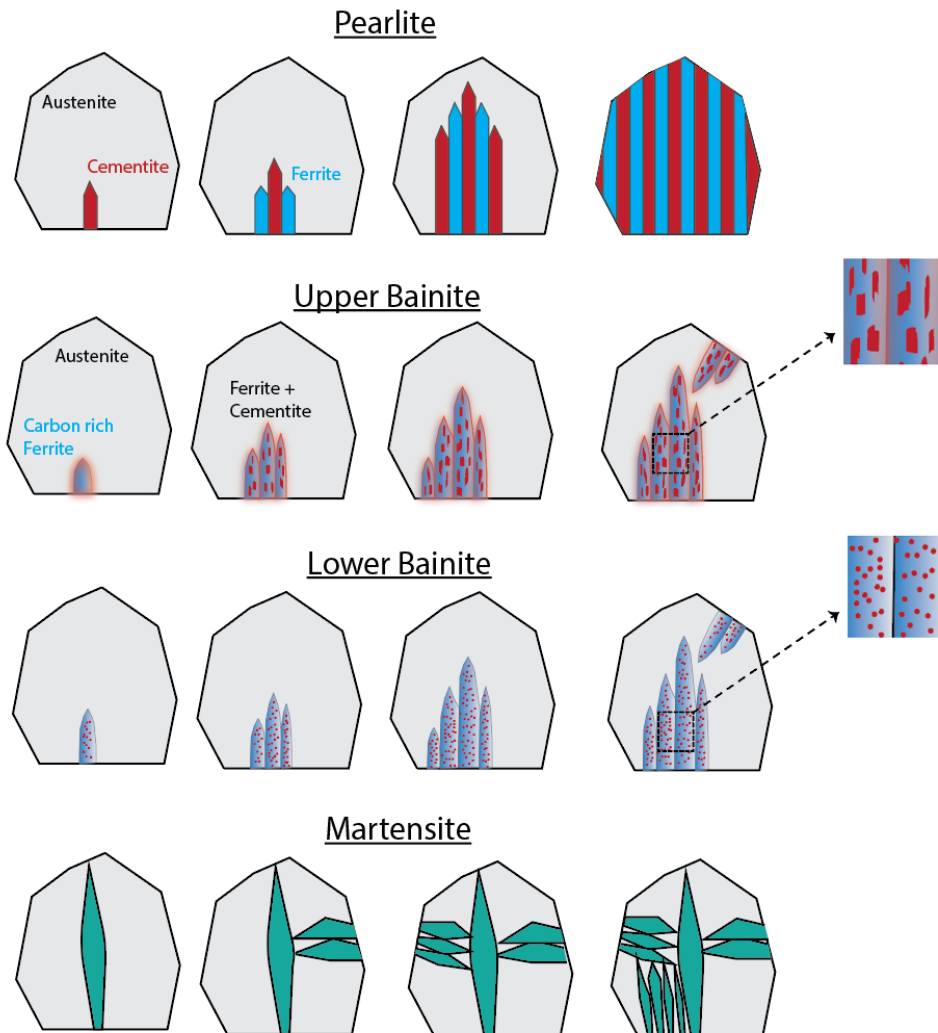


Figure 20, Formation of pearlite and bainite

Made by author, License: CC BY-SA 3.0

The Bainite can be separate into to categories of upper and lower bainite. Where upper represent structure formed at upper-temperature range with better diffusion rate, thus bigger cementite islands (elongated in the direction of ferrite plate grows), while lower bainite has lower diffusion rate leading to many small islands of cementite and thus harder structure.

The relationship between temperature and diffusion rate, thus the size of cementite island in bainite plates can be seen from microscopic pictures in Figure 21.

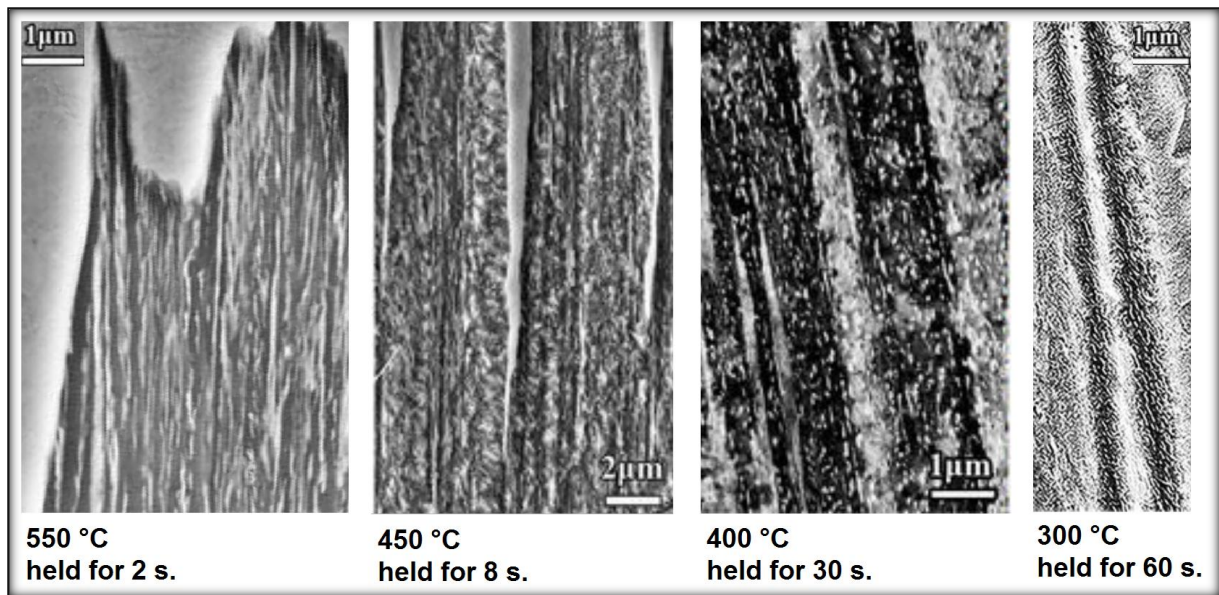


Figure 21, Bainite structures, at different formation temperatures, 0,7% C steel

Adapted from Yin, J., Hillert, M. & Borgenstam, A. *Metall and Mat Trans A* (2017) 48: 4006.

<https://doi.org/10.1007/s11661-017-4208-5>

License: CC BY-SA 4.0

Bainite starts forming at grain boundaries of austenite and usually into both neighboring grains simultaneously, and most common formation resembles chevron shape because it consisted of two legs, one in each austenite grain. The legs of a chevron consist of ferrite plates and a long series of cementite particles. It will often cover the whole length of a grain boundary, and develop into a feathery microstructure composed of parallel plates of ferrite in each grain.

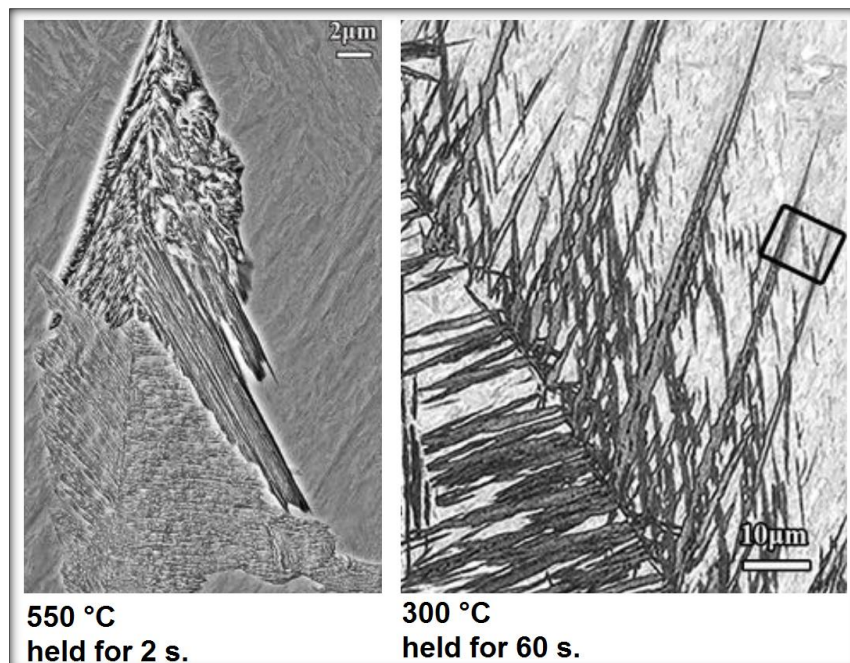


Figure 22, Feathery bainite microstructure.

Adapted from Yin, J., Hillert, M. & Borgenstam, A. *Metall and Mat Trans A* (2017) 48: 4006.

<https://doi.org/10.1007/s11661-017-4208-5>

License: CC BY-SA 4.0

It should be said that arrangement parallel ferrite plates can sometimes occur just on one side of the grain boundary and this type of bainite structure is often called semi-feathers.

4.7.4 Spheroidite

Spheroidite is the softest and most ductile steel structure. It forms when steel is heated and held for many hours at just below the lower critical temperature, A1. Since the process is diffusion driven, it can occur at lower temperatures, but the time of the transformation will drastically increase.

Structure of Spheroidite consists of cementite spheres within primary structure of ferrite or pearlite (depending on whether it is hypo or hypereutectoid type of steel).

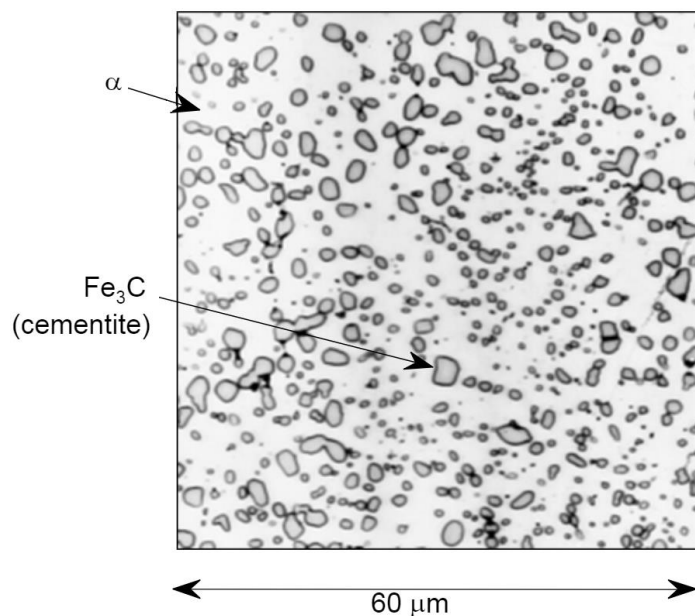


Figure 23, Spheroidite in continuous phase of α -ferrite. 1000x

Copied from Fig 10.19, Material Science and engineering by W. D. Callister and D. G. Rethwisch 8e. (Copyright 1971 by United States Steel Corporation.)

4.8 Heat treatment

4.8.1 PWHT

The heat from a welding process can cause localized expansion of HAZ in the base material, which is taken up during welding by molten metal or base material. After welding, the material will start to cool and contract. Since the heat during welding is not evenly distributed, some parts will contract more than others, leaving some residual stresses behind. This type of impurities will make the material hard and brittle, thus require treatment that can bring the material back to its design properties.

Post weld heat treatment (PWHT) is a process in which welded material is reheated to a temperature below its lower critical transformation temperature, A1 and then it is held at that temperature for a specified amount of time. The purpose of such heat treatment is to correct changes in microstructure after welding and to reduce residual stresses in the material. Process of stress relieving is called recovery, see chapter 4.5.

The process of PWHT consists of three steps: heating, holding, cooling. All steps have to be carefully planned and controlled to get the desired outcome.

The rate of heating should be based on the thickness of the component and it is specified by the governing codes. In the case with pressure piping, the most acknowledged design code is ASME B31.3. If the rate of heating is not performed properly, either by heating too quickly or not evenly, temperature changes within the component can become detrimental to the material, especially if short holding time is used. As a result, new residual stresses and stress cracks can be formed after the component is cooled to ambient temperature.

Holding time is dependent on the alloy and material thickness. It can take longer time for thick parts to achieve homogenous temperature throughout the material, thus it should be taken in consideration when deciding the holding time.

It is important to choose the PWHT temperature below the lower critical temperature, A1. To make sure that all uncertainties are covered, there is a common practice to choose the PWHT temperature 50 °C below the A1 temperature. Another important thing to consider is the original tempering temperature (tempering temperature in the material certificate), which must not be exceeded. Otherwise, the mechanical properties that is specified in the material certificate can be reduced, and in the worst case the material can become useless. In such a case where PWHT temperature exceeds the manufacturer tempering temperature, mechanical testing of the material must be performed after PWHT to make sure that the minimum design values are not exceeded.

The cooling rate is important to avoid any detrimental temperature gradients that could cause cracking or introduce new stresses.

Effect of Post weld heat treatment will generally result in a modification of the microstructure of both the weld metal and heat affected zone. The effect of short-term post weld heat treatment on the carbide is generally beneficial, whereas longer times can result in a reduction in toughness due to spheroidisation effects, see chapter 4.7.4.

4.8.2 Tempering

Tempering is a heat treatment technique applied to steel, mainly to increase toughness and decrease hardness of the alloy. The reduction in hardness is accompanied by an increase in ductility, thereby decreasing the brittleness of the metal. Tempering is usually performed after quenching, which is rapid cooling of the metal to put it in its hardest state. Tempering is accomplished by controlled heating of the quenched work-piece to a temperature below its lower critical temperature, A1, see Figure 24. Heating above this temperature will destroy martensite structure and can trigger the recrystallization process.

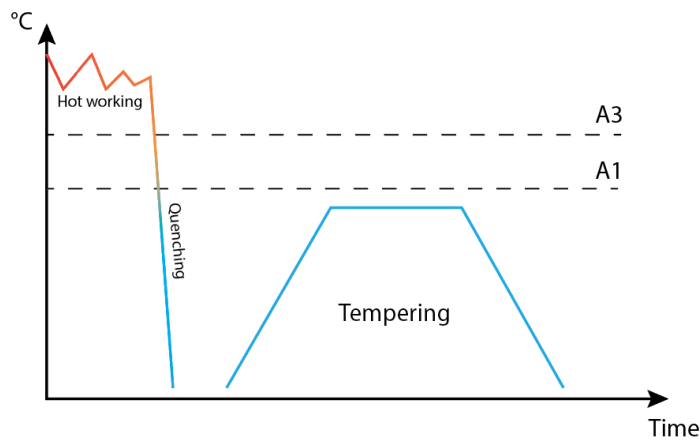


Figure 24, Tempering process

Made by author, License: CC BY-SA 3.0

Precise control of time and temperature during the tempering process is crucial to achieve the desired balance of physical properties. Low tempering temperatures may only relieve the internal stresses, decreasing brittleness while maintaining a majority of the hardness. Higher tempering temperatures tend to produce a greater reduction in the hardness, sacrificing some strength for an increase in elasticity and plasticity. However, in some low alloy steels, containing other elements like chromium and molybdenum, tempering at low temperatures range may produce an increase in hardness, while at higher temperatures the hardness will decrease.

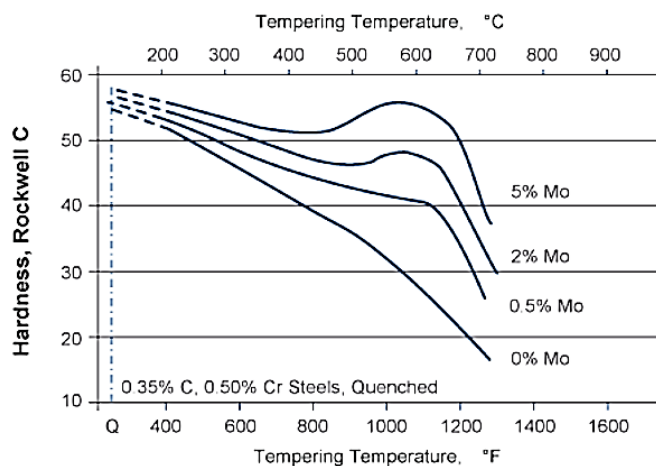


Figure 25, Influence of Mo content on temper softening (after E.C. Bain)

Copied from *imoa.info*, Copyright © 2008 International Molybdenum Association.

In carbon steels, tempering alters the size and distribution of carbides in the martensite, forming a microstructure called "tempered martensite", see chapter 4.6.2

Temperature stages of the tempering process

30 to 200 °C : The structure etches dark and is sometimes known as black martensite. The original as-quenched martensite is beginning to lose its tetragonal crystal structure by the formation of a hexagonal closed-packed transition carbide (epsilon carbide, Fe₂₄C) and low-carbon martensite.

150 to 300 °C : The retained austenite transforms into lower-bainite containing ε-carbon rather than cementite (archaically referred to as "trostite").

200 + °C : Changes the epsilon carbide to cementite (Fe₃C), the low-carbon martensite becomes BCC ferrite and any retained austenite is transformed to lower bainite.

400 to 650 °C : Continues growth of the cementite particles. This clustering of the carbide particles allows more of the ferrite matrix to be seen, giving fine mixture of ferrite and cementite. It causes the sample to etch lighter than the low-temperature product. This structure was formerly known as "sorbite".

650 to 720 °C : Highest range of steel tempering temperatures can do more harm than good and usually is avoided. During highest temperature ranges below lower critical steel will continue losing but the rate of toughness increase is low.

If high temperatures is held for long enough, globular cementite particles is produced. This structure is soft and tough, see chapter about spheroidite, chapter 4.7.4 and Figure 28. Process of spheroidisation is often listed under annealing and called spheroidized annealing.

4.8.3 Normalizing

The definition of normalizing is a process that makes something more normal or regular. That definition is reflected in the purpose of the normalizing process which is to remove coarse and uneven grain structure, creating more fine and even structure.

Normalizing is done by heating the steel 20-50 °C above the upper critical temperature, A₃ (or A_{cm} for hypereutectoid steels). During the holding time, nucleation of austenite grains will take place. The holding time of temperature must be carefully chosen to avoid extensive grows of new grains that can lead to increased ductility and unwanted strength reduction.

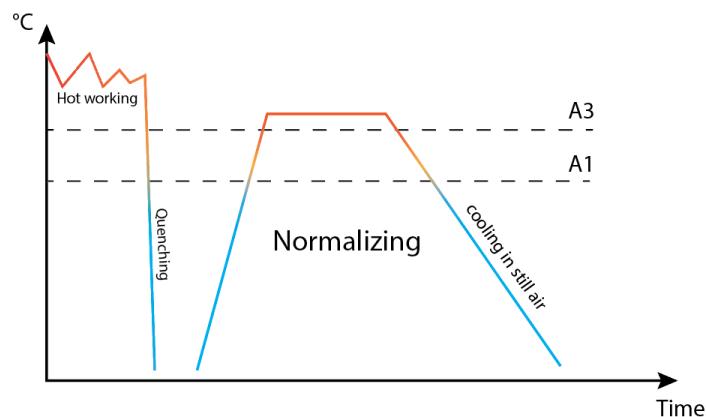


Figure 26, Normalizing process

Made by author, License: CC BY-SA 3.0

Main stages of normalization are recovery and recrystallization, see Figure 6 and Figure 7.

4.8.4 Annealing

The main purpose of annealing is to remove all residual stresses and increase the ductility. Here the steel is heated slightly above the upper critical temperature for hypoeutectoid steel and just above the lower critical temperature for hypereutectoid steel. The purpose is to transform all ferrite into austenite phase, relieving all the stresses and cooling it very slowly down in a furnace. The slow cooling allows for complete diffusion and fully annealed steel with no residual stresses.

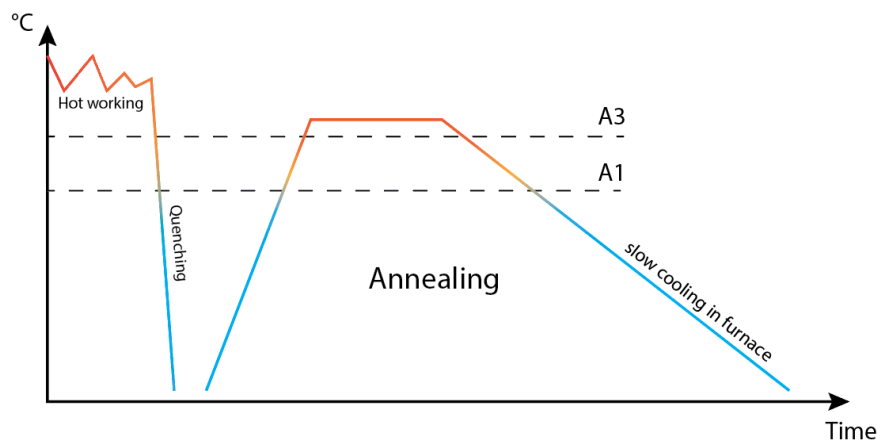


Figure 27, Annealing process of hypoeutectoid steel

Made by author, License: CC BY-SA 3.0

In addition to a full annealing process that is used to bring still to its final state, an intermediate annealing process is often used between cold working stages, it is also called process annealing. The temperature range for process annealing is between 260 °C and 760 °C (depending on the alloy in question). It is always under the lower critical temperature and is performed only to partially restore material ductility allowing for further cold working without breaking the material. Visualization of annealing and normalizing processes inside the phase diagram is illustrated in Figure 28.

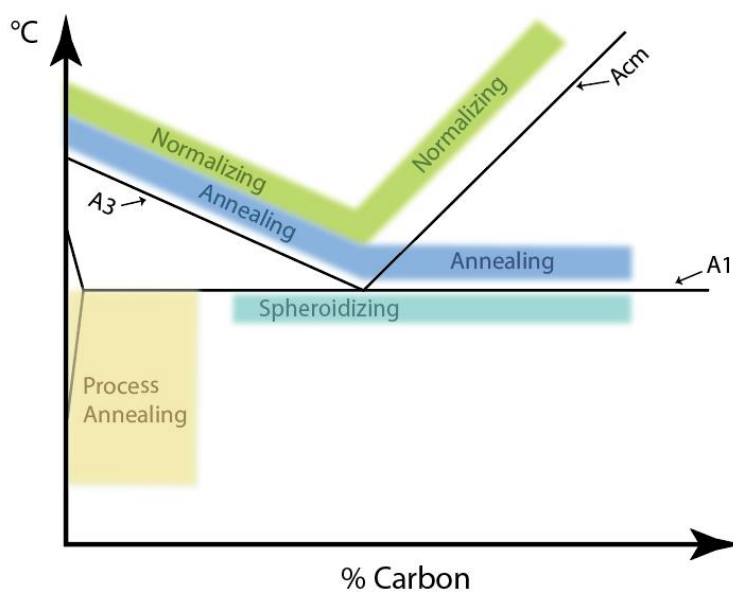


Figure 28, Annealing and Normalizing process

Made by author, License: CC BY-SA 3.0

4.9 Schaeffler diagram

The Schaeffler diagram provides some information on the welding properties of the various types of microstructure, as a function of what alloying elements they contain. Chromium equivalent is calculated using the weight percentage of ferrite stabilizing elements and Nickel equivalent is calculated using the weight percentage of austenite stabilizing elements.

Formula for Nickel equivalent:

$$\text{Ni (eq)} = \text{Ni} + (30 \times \text{C}) + (0.5 \times \text{Mn})$$

Formula for Chromium equivalent:

$$\text{Cr (eq)} = \text{Cr} + \text{Mo} + (1.5 \times \text{Si}) + (0.5 \times \text{Nb})$$

By entering the Ni-equivalent over the Cr-equivalent for stainless steel into a diagram according to Schaeffler one is able to find the content of martensite, austenite and ferrite in the resulting microstructure.

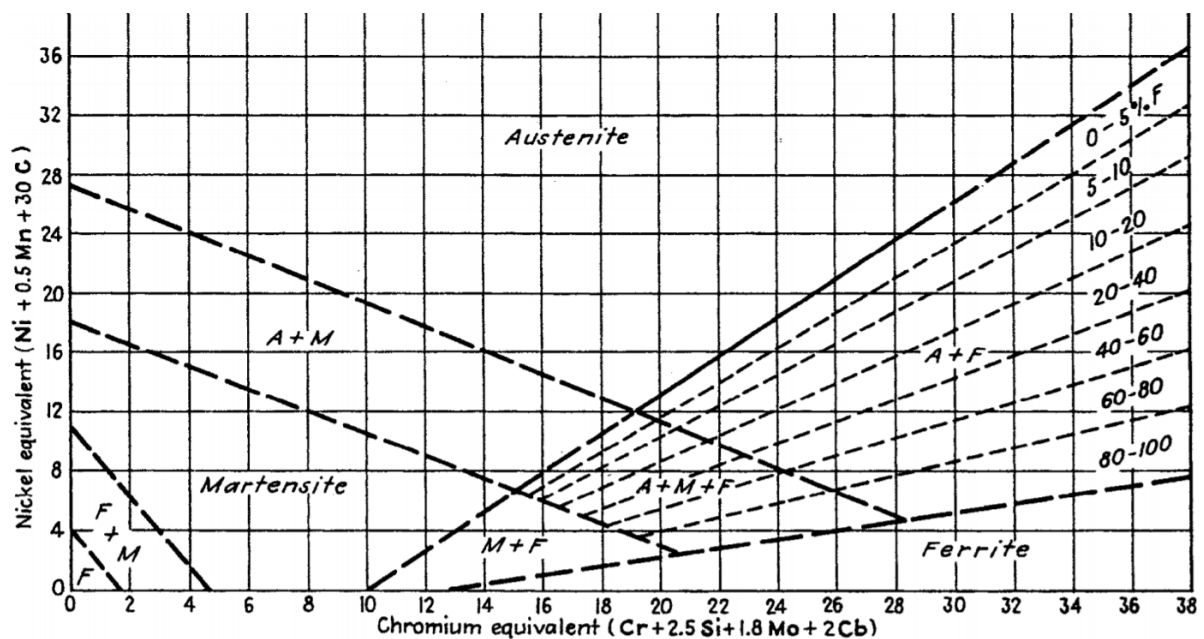


Figure 29, Schaeffler diagram

Copied from "The genesis of the Schaeffler diagram in the history of stainless steel" by Pierre Guiraldenq and Olivier Hardouin Duparc.

Although Schaeffler diagram was not design for plain steel, it may still be used to indicate expected amount of martensite based on nickel and chromium equivalents in steels.

It should be mentioned, without going in any details, that Schaeffler diagram is not applicable for steels with nitrogen additions, where Deloge diagram is rather used. Nitrogen must be taken into account as strong austenite stabilizer, leaving chromium equivalent unchanged.

4.10 Transformation diagrams, TTT and CCT

The phase transformation diagram is a so called equilibrium diagram of Fe-C composition) and only provides information about phase transformation given that a sufficient amount of time is given for diffusion of carbons and complete phase transformation. In other words, the Fe-C diagram does not show transformation under non-equilibrium conditions when faster cooling rates are applied, not allowing for equal and complete diffusion throughout the material.

Structures that are not listed in the Fe-C diagram but can be observed in steel structure is mainly bainite and martensite. TTT and CCT diagram graphically explains the effect of temperature and time on austenite transformation to pearlite, bainite or martensite. It also gives an idea about the percentage of each phase on completion of chosen temperature cycle.

There are two types of transformation diagram:

- Time Temperature Transformation diagram (TTT)
- Continuous cooling transformation diagram (CCT)

4.10.1 Isothermal Transformation diagram

Time Temperature Transformation (TTT) diagram is also called isothermal transformation diagram. This diagram is designed for a constant temperature rate transformation. Here we bring the temperature to a desired value and keep it constant until transformation of austenite to the required amount of transformation product (pearlite, bainite or martensite) have taken place.

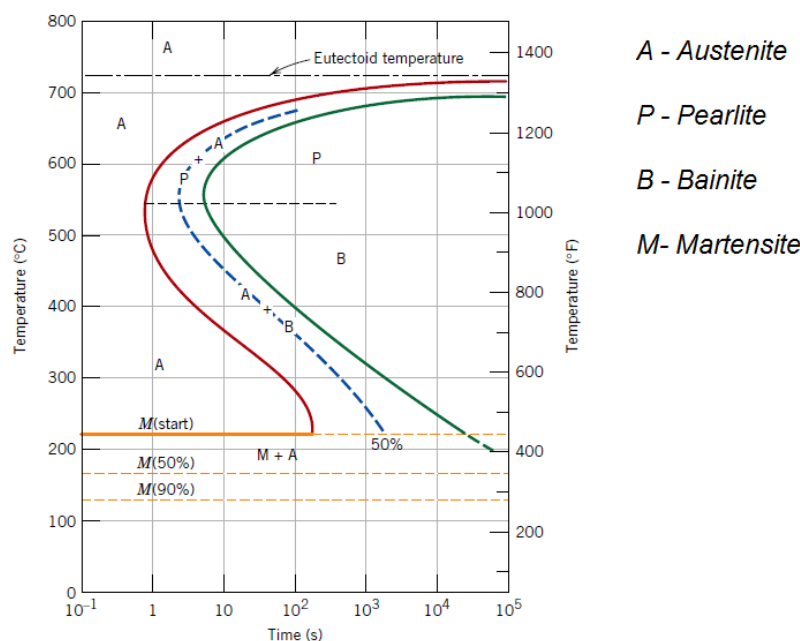


Figure 30, Isothermal transformation diagram for an eutectoid iron-carbon alloy.

Adapted from Fig 10.22 "Material Science and engineering" by W. D. Callister and D. G. Rethwisch 8e.

It is common to use multiple cooling steps, when we want to introduce multiple phases in the material.

Since the martensitic transformation does not involve any carbon diffusion, the transformation from austenite to martensite occurs almost instantaneously. The martensite grains nucleate and grow at the velocity of sound within the austenite matrix. Thus the martensitic lines are drawn horizontally in ITT and CCT diagrams. It should be also noticed that not all austenite transforms to martensite at ones, it is dependent on alloy composition and happens in stages, as indicated in Figure 30.

4.10.2 Continuous Cooling transformation, CCT

While with constant temperature transformation we can achieve more control of the transformation process, most steels are continuously cooled as it is more economically beneficial for the manufacturing process. For material transformation process at temperature that is continuously changing, we must use continuous cooling transformation (CCT) diagram.

For continuous cooling, the time required for a reaction to begin and end is delayed. This delay is usually indicated with a stippled line as it is shown on the left side of Figure 31. On the right side of the same figure, two cooling rates are introduced to demonstrate the dependence of the final microstructure on the cooling rate.

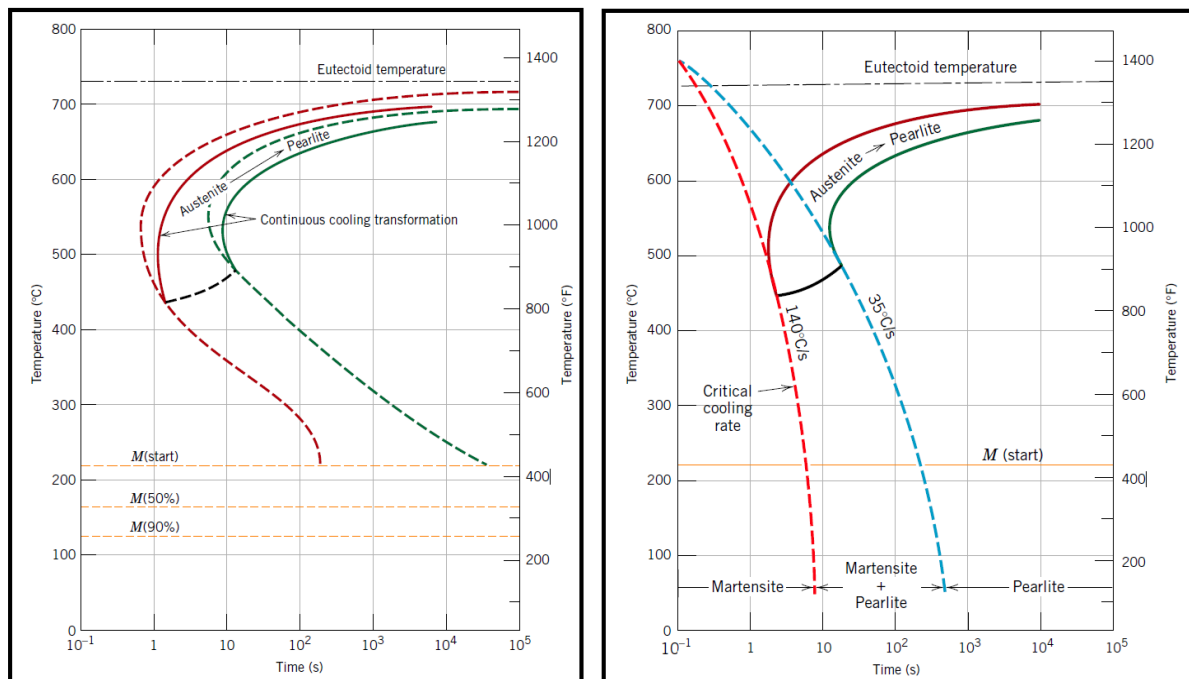


Figure 31, Continuous cooling diagram for an eutectoid iron-carbon alloy

Adapted from Fig 10.25 and 10.27, "Material Science and engineering" by W. D. Callister and D. G. Rethwisch 8e.

For the continuous cooling of a steel alloy, a term: 'critical quenching rate' is often used, which represents the minimum rate of quenching that will produce a totally martensitic structure. This critical cooling rate, when included on the continuous transformation diagram, will just miss the nose at which the pearlite transformation begins. Just as for complete pearlite structure we must just above the line at which pearlite transformation ends. Both of those rates are illustrated on the right side of Figure 31.

4.11 Metallographic etching

Since metallurgical features are microscopic in size, they cannot be seen or analyzed without optical magnification of at least 50x to 1000x times. With such enlargement it is impossible to get even optical focus of the material surface unless it is polished to a very fine mirror-like finish. Unfortunately, under a microscope, such a finely polished surface will still be useless and look like a plain white field. To allow the viewer to see parts of material microstructure, chemical solution known as etchants are used.

The idea is to expose the metal surface to a strong acid that will accelerate its rate of corrosion. Because of different compositions in material structure, rates of corrosion will also be different, creating a color contrast where more corroded phases show up as darker regions.

One of the most used acids for steel etching is the nitric acid. Typical solutions are 3% concentration of nitric acid in water. The strength of the acid determines the speed of the etching process. The etching process is also known as biting, where biting refers to idea of acid biting into the metal surface, creating minor pitting.

Etching was originally used by metal-workers in order to decorate metal items such as guns, armor, cups and plates. It is known in Europe since the Middle Ages and is popular still.



Figure 32, Example of etching art on a metal plate

Copied from Wikipedia.org. Article: Etching. Author: Wolfram Gotbe, 1989. License: Public domain

4.12 SAE/AISI Carbon Steel

Since the 1930s the American Iron and Steel Institute (AISI) and SAE were both involved in efforts to standardize a numbering system for steel grades. For several decades the systems were united into a joint system and designated as AISI/SAE steel grades, but since 1995 AISI turned over all future maintenance of the system to SAE.

SAE designation	Type
1xxx	Carbon steels
2xxx	Nickel steels
3xxx	Nickel-chromium steels
4xxx	Molybdenum steels
5xxx	Chromium steels
6xxx	Chromium-vanadium steels
7xxx	Tungsten steels
8xxx	Nickel-chromium-vanadium steels
9xxx	Silicon-manganese steels

Molybdenum steels	
40XX	Mo 0.20-0.25%
44XX	Mo 0.40-0.52%
Chromium-molybdenum steels	
41XX	Cr 0.50-0.95%, Mo 0.12-0.30%
Nickel-chromium-molybdenum steels	
43XX	Ni 1.82%, Cr 0.50-0.80%, Mo 0.25%
47XX	Ni 1.05%, Cr 0.45%, Mo 0.20-0.35%
Nickel-molybdenum steels	
46XX	Ni 0.85-1.82%, Mo 0.20-0.25%
48XX	Ni 3.50%, Mo 0.25%

The first digit indicates the type of steel, the second indicates modification of the steel and the third tells about carbon concentration in 0,01%.

41xx steel is a family of SAE steel grades that includes elements chromium and molybdenum, and as a result, these materials are often informally referred to as chromoly steel (common variant stylings include chrome-moly, CrMo, CRMO, CR-MOLY, and similar). They have an excellent strength to weight ratio and are considerably stronger and harder than 10xx plain carbon steel, but are not as easily welded and in most applications requires preheat and post weld heat treatment to relieve the residual stresses and avoid cold cracking.

5 Research question

The research question consists of two parts. The first part is directly based on the outcome of the preliminary project, which is about alternative materials [1] and the second (which is the main part of this thesis) is about optimization of welding procedure of AISI 4130 steel.

Part 1

First, smaller part consists of clarification of whether NACE MR0175/ISO 15156 requirements can be lifted for parts of the system and whether Duplex steel could be a better material alternative for mud system. Following tasks is thereafter specified:

- Conclude on whether the Sour Service requirements can be lifted for parts of the high-pressure mud system.
- Consider the use of Duplex steel instead of AISI 4130 (low alloyed steel).

Since those tasks demanded a relatively small amount of effort to conclude, they are not central in this thesis. To avoid the mixture of subjects and tasks in the main body of this document, they were awarded a separate chapter, chapter 8 . Tasks from part one is discussed and concluded on in this chapter.

Part 2

The company has decided to get some insights into the welding procedures and improve the cost and quality of WPS¹ used for welding of AISI 4130 pipes. WPS is an expansive procedure that includes the choice of the welding method, filler metal, input heat, preheat temperature, interpass temperature and PWHT². The main part of this paper is thereby to uncover the theory around the effect of welding on low alloyed steel and based on that, suggest the best PWHT practices.

Post weld heat treatment suggestion must be based on sour service requirements covered by NACE MR0175/ISO 15156 and in accordance with design standard for high-pressure piping ASME B31.3.

¹ Welding Procedure Specification (WPS) is the formal written document that describes welding procedures, which provides direction to the welder or welding operators for making sound and quality welds as per the code requirement

² Post Weld Heat Treatment heat treatment process that is performed after welding, usually to relieve residual stresses in the weld and heat effected material.

6 Case and materials

This chapter includes relevant information on material data and mandatory requirements that have to be considered when specifying a welding procedure for steel pipes that will be exposed to hydrogen sulfide medium.

We do not look into the filler metal in this work but the choice of the electrode was made in accordance with the strength and composition requirements. Data on filler material is attached, see Attachment 3 – 48.08 electrode.

6.1 Material AISI 4130

AISI or SAE 4130 grade is a low-alloy steel containing chromium and molybdenum as strengthening agents. As it is low-alloy steel, it has relatively good toughness, weldability and machinability, although it requires to be heat treated because of the relatively high chromium content. Exaptions for PWHT is mentioned in table 331.1.3 of ASME B31.3 [7].

6.1.1 Material Certificate

Engineering company Nymo kindly provided two 150 mm long pipe samples for this work.



Figure 33, Raw AISI 4130 pipe samples from Nymo

Pictures were taken by the author. License: CC BY-SA 3.0

Pipe samples are made of low alloyed carbon steel AISI 4130 with outer diameter 66,33 mm (2 inches) and wall thickness 11,07 mm (Schedule: XXS). Material certificate is attached, see Attachment 2 – Material Certificate.

Data in the material certificate is entirely in compliance with the Material Data Sheet from MHWirth, see chapter 6.1.4.

Heat treatment

Heat treatment specified in the material certificate for our samples is illustrated with the Time-Temperature diagram in Figure 34.

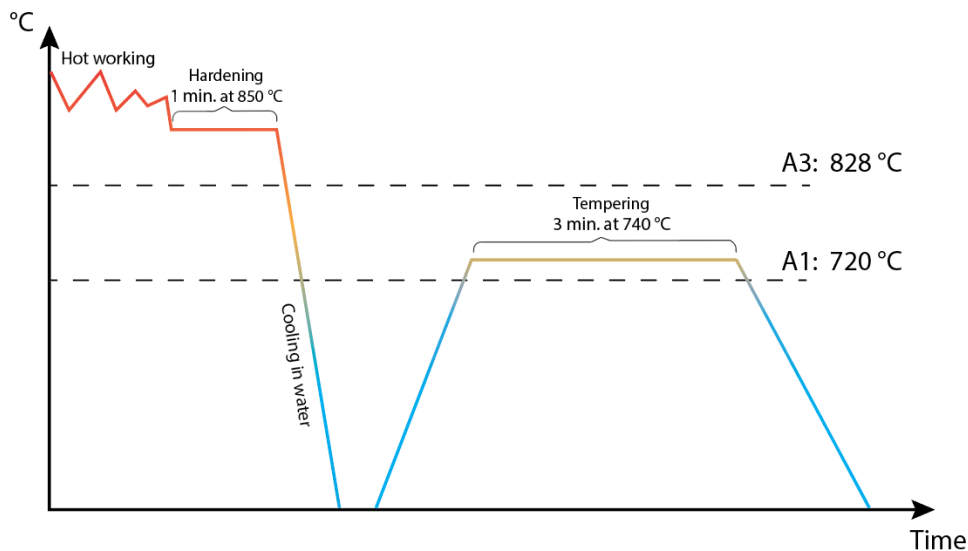


Figure 34, TT diagram for AISI 4130 pipe

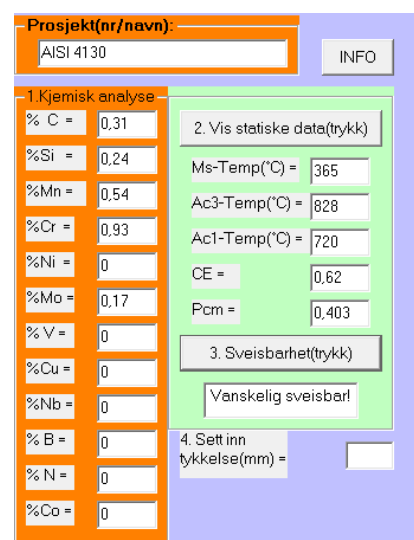
Made by author, License: CC BY-SA 3.0

6.1.2 Heat treatment

As previously described, the heat from welding process will partly remove the effect of heat treatment performed by the manufacturer during production, making the material hard and brittle. PWHT is tend to restore mechanical properties to an acceptable level by relieving some of the residual stresses after welding, but the temperature should never exceed the temperature at which material was tempered during the production phase or the lowest critical temperature, A1.

To specify the heat treatment temperatures for our AISI 4130 steel, see the attached material certificate for the sampled material (*Attachment 2 – Material Certificate*), we can use a simulation program called Welding Note. The program was developed as an aiding tool for welders to clarify welding parameters for the welding procedures, see Figure 35.

The temperature parameters in **Error! Reference source not found.** can be clarified with the help of Phase Diagram, see Figure 2. Knowing the critical temperatures of AISI 4130, we can specify the temperature conditions for different heat treatments of AISI 4130 based on theory chapter 4.8.



Prosjekt(nr/navn):		INFO
AISI 4130		
1. Kjemisk analyse		
% C =	0,31	2. Vis statistiske data(trykk)
% Si =	0,24	
% Mn =	0,54	
% Cr =	0,93	
% Ni =	0	
% Mo =	0,17	
% V =	0	
% Cu =	0	
% Nb =	0	
% B =	0	
% N =	0	3. Sveisbarhet(trykk)
% Co =	0	
		Vanskelig sveisbar!
		4. Sett inn tykkelse(mm) = <input type="text"/>

Figure 35, Welding parameters for AISI 4130

Made by author, License: CC BY-SA 3.0

Annealing

Annealing of 4130 forgings may be carried out by heating the material to a temperature above 828 °C, holding for a suitable time then furnace cooling. In this way, the material microstructure will fall in fully austenitic phase and by slow cooling rate, the material shall consist of Pearlitic-Ferritic microstructure.

Normalizing

This process is defined as heating a steel to a temperature above the ferrite to austenite transformation temperature A3, then cooling in air. Normalizing serves to refine the grain structure of the material that might have cooled not uniformly during the production process. The nominal normalizing temperature for 4130 grade is 850 - 880 °C. In practice, if normalizing is to be followed by other heat treatment processes, the upper range of normalizing temperatures is used. When normalizing is the final heat treatment, the lower temperature range preferred.

Tempering

Tempering is carried out primarily to obtain mechanical properties required for the final application and to relieve residual stresses from the hardening process. The actual tempering temperature is often chosen using trial and error method.

Tempering temperature for AISI 4130 steel shall be below 720 °C.

6.1.3 Cooling transformation diagram

Company Timken Steel is one of the leading steel manufacturers. The company has developed isothermal and continuous cooling transformation diagrams for SAE 4130 steel.

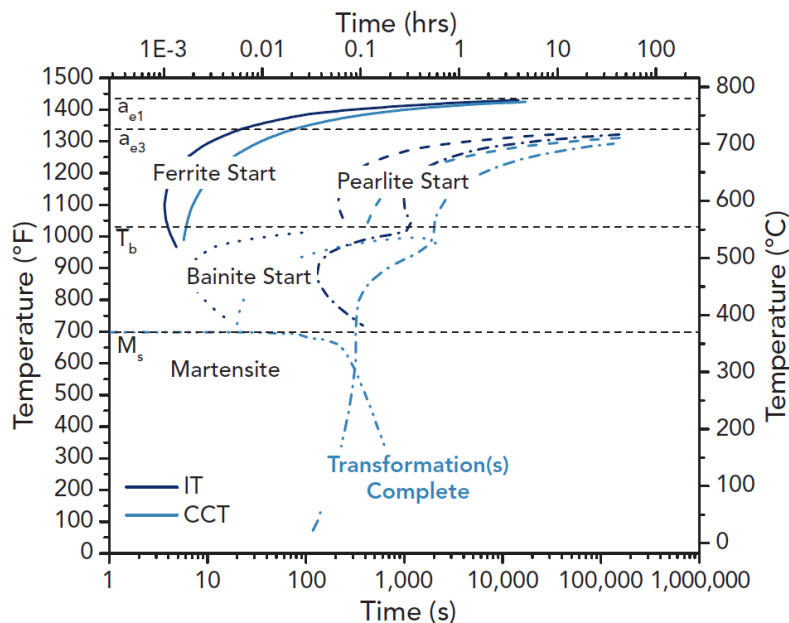


Figure 36, Isothermal and continuous cooling diagram for SAE 4130 steel

Copied from TimkenSteel.com. Copyright ©2014 TimkenSteel Corporation

Based on Figure 36 and expected cooling rates of low alloyed steel in water and still air, CCT diagram of AISI 4130 with cooling curves for water and still air was developed, see Figure 37.

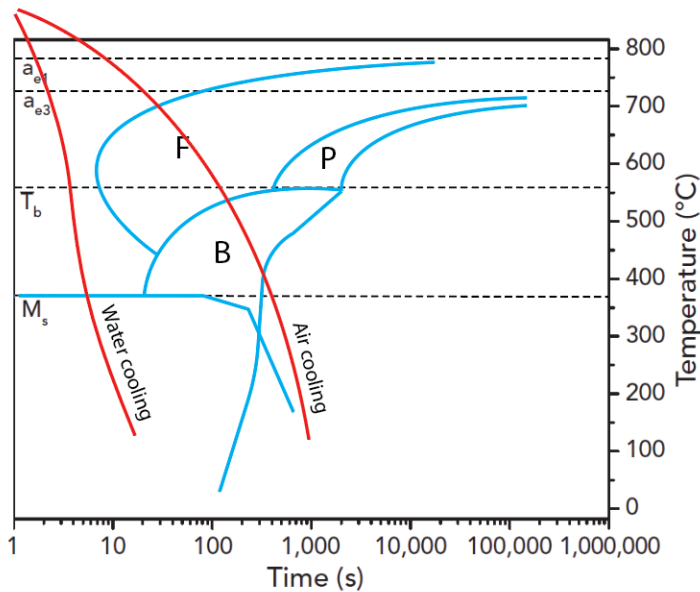


Figure 37, CCT diagram for cooling rates of AISI 4130 in water and in still air.

Made by author, License: CC BY-SA 3.0

6.1.4 Material Data Sheet

Material Data Sheet (MDS) for AISI 4130 used in high-pressure mud system by MHWirth is X21 for pipes and wrought fittings and X24 for forged fittings. Those are attached, see Attachment 1 – Material Data Sheet. There are some minor differences between those material data sheets like requirements to testing and certification, but there is no different that of concern for welding.

MDS requirements for AISI 4130:

- ✓ The maximum hardness shall be in accordance with the NACE MR0175 / ISO 15156 standard. That is no more than 22 HRC or 250 HV.
- ✓ Fittings and pipes shall be delivered in the liquid quenched and tempered conditions. The tempering temperature shall be a minimum of 650 deg C.
- ✓ Requirements to chemical composition specify restrictions to Sulphur and Phosphorous content of less than 0,025%.
- ✓ Mechanical properties are specified to a minimum of the following values:
Minimum yield strength: $R_{eH} \Rightarrow 517$ MPa (75 KSI), Minimum tensile strength: $R_m \Rightarrow 690$ MPa (100 KSI), Minimum elongation: $A_5 \Rightarrow 17\%$, Minimum reduction of area: $Z \Rightarrow 35\%$

6.2 Mandatory requirements

Following requirements is considered mandatory:

- Technical requirements of ASME B31.3 [7]
- Welding and inspection of piping, Norsok M601.
- Requirement for sour service (H2S) piping, NACE MR 0175 /ISO 15156.

The following chapters list relevant parts of applicable standards.

6.2.1 ASME B31.3

Preheat

According to ASME B31.3, chapter 330.1.1, alloy steel with Chromium content between 0,5% and 2% (materials with P-No 4), require preheating temperature not less than 120 °C.

Base Metal P-No. [Note (1)]	Base Metal Group	Greater Material Thickness		Additional Limits [Note (2)]	Required Minimum Temperature	
		mm	in.		°C	°F
1	Carbon steel	≤25	≤1	None	10	50
		>25	>1	%C ≤ 0.30 [Note (3)]	10	50
		>25	>1	%C > 0.30 [Note (3)]	95	200
3	Alloy steel, Cr ≤ 1/2%	≤13	≤1/2	SMTS ≤ 450 MPa (65 ksi)	10	50
		>13	>1/2	SMTS ≤ 450 MPa (65 ksi)	95	200
		All	All	SMTS > 450 MPa (65 ksi)	95	200
4	Alloy steel, 1/2% < Cr ≤ 2%	All	All	None	120	250
5A	Alloy steel	All	All	SMTS ≤ 414 MPa (60 ksi)	150	300
		All	All	SMTS > 414 MPa (60 ksi)	200	400
5B	Alloy steel	All	All	SMTS ≤ 414 MPa (60 ksi)	150	300
		All	All	SMTS > 414 MPa (60 ksi)	200	400
		≤13	≤1/2	%Cr > 6.0 [Note (3)]	200	400
6	Martensitic stainless steel	All	All	None	200 [Note (4)]	400 [Note (4)]
9A	Nickel alloy steel	All	All	None	120	250
9B	Nickel alloy steel	All	All	None	150	300
10I	27Cr steel	All	All	None	150 [Note (5)]	300 [Note (5)]
15E	9Cr-1Mo-V CSEF steel	All	All	None	200	400
...	All other materials	None	10	50

NOTES:
(1) P-Nos. and Group Nos. from BPV Code, Section IX, QW/QB-422.
(2) SMTS = Specified Minimum Tensile Strength.
(3) Composition may be based on ladle or product analysis or in accordance with specification limits.
(4) Maximum interpass temperature 315°C (600°F).
(5) Maintain interpass temperature between 150°C and 230°C (300°F and 450°F).

Table 1, Preheat Temperatures, ASME B31.3

Copied from ASME B31.3 – 2016, Table 330.1.1. Copyright ASME International

PWHT

Requirements for post weld heat treatment is given in table 331.1.1. According to which holding temperature range is 650 – 705 °C and minimum holding time is set to 1/25 Hour/mm, but not less than 15 min.

Table 331.1.1 Postweld Heat Treatment

P-No. and Group No. (BPV Code Section IX, QW/QB-420)	Holding Temperature Range, °C (°F) [Note (1)]	Minimum Holding Time at Temperature for Control Thickness [Note (2)]	
		Up to 50 mm (2 in.)	Over 50 mm (2 in.)
P-No. 1, Group Nos. 1–3	595 to 650 (1,100 to 1,200)	1 h/25 mm (1 hr/in.); 15 min min.	2 hr plus 15 min for each additional 25 mm (in.) over 50 mm (2 in.)
P-No. 3, Group Nos. 1 and 2	595 to 650 (1,100 to 1,200)		
P-No. 4, Group Nos. 1 and 2	650 to 705 (1,200 to 1,300)		
P-No. 5A, Group No. 1	675 to 760 (1,250 to 1,400)		
P-No. 5B, Group No. 1	675 to 760 (1,250 to 1,400)		
P-No. 6, Group Nos. 1–3	760 to 800 (1,400 to 1,475)		
P-No. 7, Group Nos. 1 and 2 [Note (3)]	730 to 775 (1,350 to 1,425)		
P-No. 8, Group Nos. 1–4	PWHT not required unless required by WPS		
P-No. 9A, Group No. 1	595 to 650 (1,100 to 1,200)		
P-No. 9B, Group No. 1	595 to 650 (1,100 to 1,200)		
P-No. 10H, Group No. 1	PWHT not required unless required by WPS. If done, see Note (4).		
P-No. 10I, Group No. 1 [Note (3)]	730 to 815 (1,350 to 1,500)		
P-No. 11A	550 to 585 (1,025 to 1,085) [Note (5)]		
P-No. 15E, Group No. 1	705 to 775 (1,300 to 1,425) [Notes (6) and (7)]	1 h/25 mm (1 hr/in.); 30 min min.	1 h/25 mm (1 hr/in.) up to 125 mm (5 in.) plus 15 min for each addi- tional 25 mm (in.) over 125 mm (5 in.)
P-No. 62	540 to 595 (1,000 to 1,100)	...	See Note (8)
All other materials	PWHT as required by WPS	In accordance with WPS	In accordance with WPS

GENERAL NOTE: The exemptions for mandatory PWHT are defined in Table 331.1.3.

NOTES:

(1) The holding temperature range is further defined in para. 331.1.6(c) and Table 331.1.2.

(2) The control thickness is defined in para. 331.1.3.

Table 2, Postweld Heat Treatment, ASME B31.3

Copied from ASME B31.3 – 2016, Table 331.1.1. Copyright ASME International

Holding time can be further reduced according to table 331.1.2 with up to 55 °C for steels with P-No 4, but minimum holding time is then sufficiently increases.

Table 331.1.2 Alternate Postweld Heat Treatment Requirements for Carbon and Low Alloy Steels, P-Nos. 1 and 3

Decrease in Specified Minimum Temperature, °C (°F)	Minimum Holding Time at Decreased Temperature, h [Note (1)]
30 (50)	2
55 (100)	4
85 (150) [Note (2)]	10
110 (200) [Note (2)]	20

NOTES:

(1) Times shown apply to thicknesses ≤25 mm (1 in.). Add 15 min/25 mm (15 min/in.) of thickness for control thicknesses >25 mm (1 in.) (see para. 331.1.3).

(2) A decrease >55°C (100°F) below the minimum specified temperature is allowable only for P-No. 1, Group Nos. 1 and 2 materials.

Table 3, Alternate Postweld Heat Treatment, ASME B31.3

Copied from ASME B31.3 – 2016, Table 331.1.2. Copyright ASME International

Heating and cooling rate

Chapter 331.1.4 states that the heating method shall be uniformly applied and may include an enclosed furnace, local flame heating, electric resistance, electric induction, or exothermic chemical reaction. Above 315°C, the rate of heating and cooling shall not exceed 335°C/h divided by one-half the maximum material thickness in inches at the weld, but in no case shall the temperature change rate exceed 335°C/h.

6.2.2 NACE MR0175

The consequences of sudden failures of metallic components in oil and gas industry is often associated with their exposure to H₂S-containing production fluids. Understanding of that led to the preparation of the first edition of ANSI/NACE MR0175, which was published in 1975 by the National Association of Corrosion Engineers, now known as NACE International. NACE MR0175 is covered by ISO 15156 standard and often refers to as NACE MR0175/ ISO 15156.

ISO version splits the standard into three:

- ISO 15156-1: General principles for selection of cracking-resistant materials.
- ISO 15156-2: Cracking-resistant carbon and low alloy steels, and the use of cast irons.
- ISO 15156-3: Cracking-resistant CRAs (corrosion-resistant alloys) and other alloys.

It has been concluded not to consider duplex steels for high-pressure piping in this work, see chapter 8.2, thus ISO 15156-3 will not be discussed further.

ISO 15156-1 is in a way an introduction part to ISO 15156 series and mainly clarifies terms, abbreviations and general principles of material selection for sour service.

ISO 15156-2 specifies the requirements for testing methods and qualification of low alloy steels.

PWHT

A minimum PWHT temperature of 620 °C (1 150 °F) shall be used for low alloy steels.

Hardness testing

Hardness testing for welding procedure qualification shall normally be carried out using the Vickers HV 10 or HV 5 method in accordance with ISO 6507-1, or the Rockwell method in accordance with ISO 6508-1 using the 15N scale.

Weldments that do not comply with other paragraphs of this subclause shall be post weld heat treated after welding. The heat treatment temperature and its duration shall be chosen to ensure that the maximum weld zone hardness shall be 250 HV or 22 HRC.

Hardness test methods	Hardness test locations for welding procedure qualification	Maximum acceptable hardness
Vickers HV 10 or HV 5 or Rockwell HR 15N	Weld root: Base metal, HAZ and weld root metal as shown in Figure 2 , Figure 3 or Figure 4	250 HV 70,6 HR 15N
	Base metal and HAZ for weld overlays as shown in Figure 6 ; see also A.2.1.5 b)	250 HV 70,6 HR 15N
	Weld cap: Base metal, HAZ and weld metal of unexposed weld cap as shown in Figure 2 or Figure 4	275 HV ^a 73,0 HR 15N
Rockwell HRC; see 7.3.3.2	As shown in Figure 5	22 HRC
	Base metal and HAZ for weld overlays as shown in Figure 6 ; see also A.2.1.5 b)	22 HRC
^a The maximum shall be 250 HV or 70,6 HR 15N unless all three of the following conditions are met: <ul style="list-style-type: none"> — equipment user agrees the alternative weld cap hardness limit; — parent material(s) are over 9 mm thick; — weld cap is not exposed directly to the sour environment. 		

Table 4, Acceptable hardness values for carbon-manganese steel, ISO 15156-2

Copied from ISO 15156-2 – 2015, Table A.1. Copyright International Organization for Standardization

Nickel content

Welding consumables:

Welding consumables and procedures that produce a deposit containing more than 1 % mass fraction nickel are acceptable after successful weld SSC qualification by testing in accordance with Annex B.

Parent material:

Carbon and low-alloy steels are acceptable at 22 HRC maximum hardness provided they contain less than 1 % mass fraction nickel.

6.2.3 NORSOK M-601

Interpass temperature

The interpass temperature shall be measured within the joint bevel. The minimum interpass temperature shall not be less than the specified preheat temperature. The maximum interpass temperature shall not exceed the maximum temperature during qualification or in no case above as stated below:

- 250 °C for carbon steels;
- 150 °C for stainless steels and nickel base alloys.

6.2.4 ISO 14745

This Technical Report provides recommendations for post-weld heat treatment (PWHT) of steels with recommendations for holding temperatures and holding times for different materials and material thicknesses.

According to Table 1 from the standard, holding temperature for AISI 4130 should be 620 – 680 °C. Holding time is set to 60 min + material thickness in minutes.

Material group (ISO/TR 15608)	Material	Holding temperature	Material thickness ^a <i>t</i>	Holding time	<i>P</i> _{crit} (see Clause 6)
		°C	mm	min	
5.1	Cr-Mo-steels free of vanadium with, by mass: 0,75 % < Cr ≤ 1,5 %; Mo ≤ 0,7 % ^{a, f}				
	— 25CrMo4, 26CrMo4-2	— ^e			
	— 13CrMoSi5-5	620 to 680 ^b	<i>t</i> ≤ 15	30	18,7
	— 13CrMo4-5	630 to 700 ^g	15 < <i>t</i> ≤ 60	2 <i>t</i>	
	— All others	620 to 680 ^g	<i>t</i> > 60	60 + <i>t</i>	18,5

^a For thickness ≤ 35 mm, post-weld treatment is normally only necessary in special cases [e. g. to reduce the danger of stress corrosion cracking or hydrogen-induced cracking (sourgas)].

^b Higher temperatures can be applied, subject to conditions given in application standards.

^e Normally welded with austenitic filler metal; in view of possible carbon diffusion, PWHT should be avoided.

^f According to EN 13480-4, [2] no PWHT is required if all the following conditions are fulfilled:

- tubes with outside diameter ≤ 114,3 mm;
- nominal wall thickness ≤ 7,1 mm;
- minimum pre-heating temperature 200 °C.

^g According to EN 13445-4, [3] no PWHT is required if all the following conditions are fulfilled:

- tubes with nominal diameter < 120 mm;
- nominal wall thickness < 13 mm.

Figure 38, PWHT parameters for steel. ISO 14745

Adopted from ISO 14745 – 2015, Table 1. Copyright International Organization for Standardization

Heating and cooling rate

In the case of PWHT in furnace, the temperature of the furnace at the time when the product or component is placed in or taken out of the furnace should not exceed:

- 400 °C for simple products or components of uncomplicated shape and $t < 60$ mm thickness;
- 300 °C for complex products or components of complicated shape or $t \geq 60$ mm thickness.

The rate for heating or cooling of the product or component should not exceed the following:

- for thickness $t \leq 25$ mm: 220 °C/h;
- for thickness $25 \text{ mm} < t \leq 100 \text{ mm}$: $(5\ 500/t)$ °C/h;
- for thickness $t > 100$ mm : 55 °C/h.

7 Method

This chapter describes the sample preparation in chronological order. Followed by a description of methods for hardness testing, heat treatment and microscopy.

7.1 Welding

In order to be able to discuss the effect of mandatory preheating specified by AISI B31.3 standard, two welded samples was prepared. Two 150 mm long samples that was received from Nymo was cut into four 75 mm long pipes and welded at ambient temperature. One set without preheating and one with preheating to 120 °C.

7.1.1 Preparation

Pipes groove was prepared with following parameters:

Groove angle: 60°C

Root gap: 3mm

Root face: 2mm



Figure 39, Butt Weld End Preparation

Pictures were taken by the author. License: CC BY-SA 3.0

7.1.2 Preheating

Two welded connection was prepared, but in order to be able to document the effect of preheating only one welded connection was preheated to a temperature of 120 °C with a flame torch.



Figure 40, Preheating with flame torch

Picture was taken by author. License: CC BY-SA 3.0

According to the standard, preheat temperature shall be checked by use of temperature indicating crayons, thermocouple pyrometers, or other suitable means to ensure that the temperature specified in the WPS is obtained prior to and maintained during welding. Thus interpass temperature must be above preheat temperature.

A digital thermometer was used to ensure that preheat temperature of 120 °C was achieved and that interpass temperature does not fall long below 250 °C.



7.1.3 SMAW Welding

Welding was performed in accordance with welding procedure for low alloy API 5LX52 steel. See Attachment 4 – WPS for API 5L52 pipe.



Figure 41, Pictures of welding process. Root and cap welding stages

Pictures were taken by the author. License: CC BY-SA 3.0

Welding log with heat input data for the weld with 120 °C preheat is attached, see Attachment 5 – Heat input data. The other welding sample (the one without preheat) is welded in the same conditions and have the same average heat input. Average heat input is 3,02 kJ/mm. Heat input is higher than suggested by the WPS, but it is because carbon equivalent for AISI 4130 steel is higher than the API 5L G52 material that the used WPS was originally developed for. For AISI 4130, higher Ampere and voltage is used.

7.1.4 Polishing

After welding (and between every string) slag is removed and welded area is polished to remove impurities and prepare the surface for next weld string.

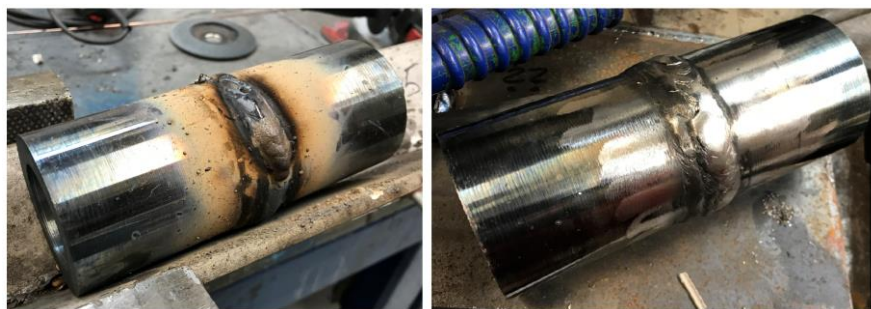


Figure 42, Polishing of welded sample.

Pictures were taken by the author. License: CC BY-SA 3.0

7.2 Cutting

To expose the cross-section of the weld and to prepare sample pieces for hardness testing and microscopy, welded pipe samples were cut out using a band saw:



Figure 43, Cutting of samples

Pictures was taken by author. License: CC BY-SA 3.0

7.3 Polishing

Polishing was done in two stages: milling and polishing with waterproof silicon carbide paper.

Milling was necessary to achieve flatness and parallelism of sample surfaces. ISO 6507-3¹ states that maximum deviation in the flatness of the test and support surfaces shall not exceed 0,005 mm. The maximum error in parallelism shall not exceed 0,010 mm in 50 mm long samples.



Figure 44, Use of milling machine for surface correction and smoothing

Pictures were taken by the author. License: CC BY-SA 3.0

¹ ISO 6507-3 standard specifies requirements for preparation of test piece for Vickers hardness testing.

ISO 6507-3 further states that the test surface shall be free from scratches that interfere with the measurement of the indentations. The test surface roughness, R_a , shall not exceed $0,05 \mu\text{m}$.

That can be archived with the help of extra fine grinding paper. According to Table 5, grinding paper with grind grade P2400 should be sufficient and gives surface roughness of $0.025 \mu\text{m}$.

Standard ANSI grit	European (P-Grade)	Median Diameter (microns)	Surface Roughness on Steel, Rc 30 (Ra -nm)
60	P60	250	-
80	P80	180	1140
120	P120	106	1050
180	P180	75	880
240	P220	63	300
320	P360	40.5	230
400	P800	25.8	120
600	P1200	15.3	110
800	P2400	6.5	25
1200	P4000	2.5	20

Table 5, Grinding paper grid overview

Copied from SiC paper brochure on mtallographic.com.

We perform polishing with help of polishing machine Knuth Rotor in following steps: P320, P800, P1200, P2400:



Figure 45, Grinding machine and polished samples

Pictures were taken by the author. License: CC BY-SA 3.0

7.4 Etching

Two types of etching were tried: 3 % Nitric acid (HNO_3) and 50% Hydrogen chloride (HCl). Nitric acid (also called Nital) gave clear results within 10 seconds. Hydrogen chloride gave less clear results and it took approximately 2 minutes. A clear difference between those two methods allowed us to make a choice in favor of 3% Nital.

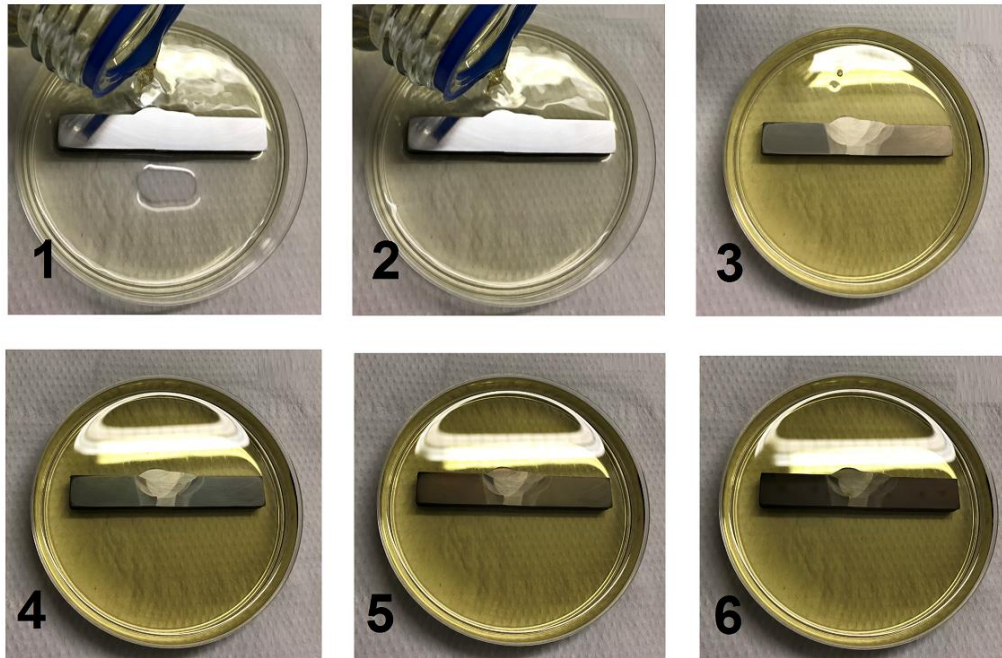


Figure 46, Etching with 3% Nital, time from 1 to 6 is 6 seconds.

Pictures were taken by the author. License: CC BY-SA 3.0

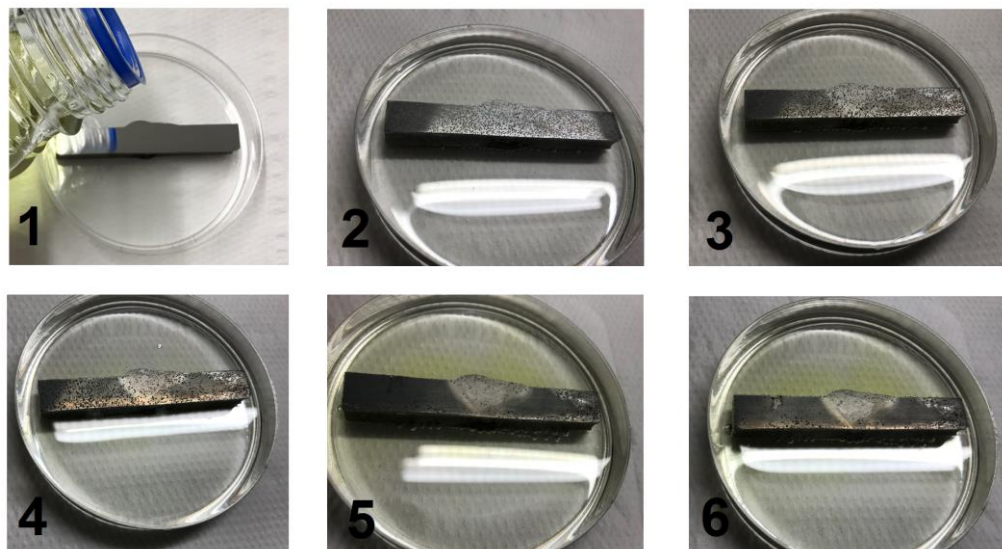


Figure 47, Etching with 50% HCl, time from 1 to 6 is 2 minutes.

Pictures were taken by the author. License: CC BY-SA 3.0

The etching was done with 3% Nital for all samples, of which we have eight. Four from welded pipes without preheating and four from welded pipe that was preheated with flame torch to 120 °C.

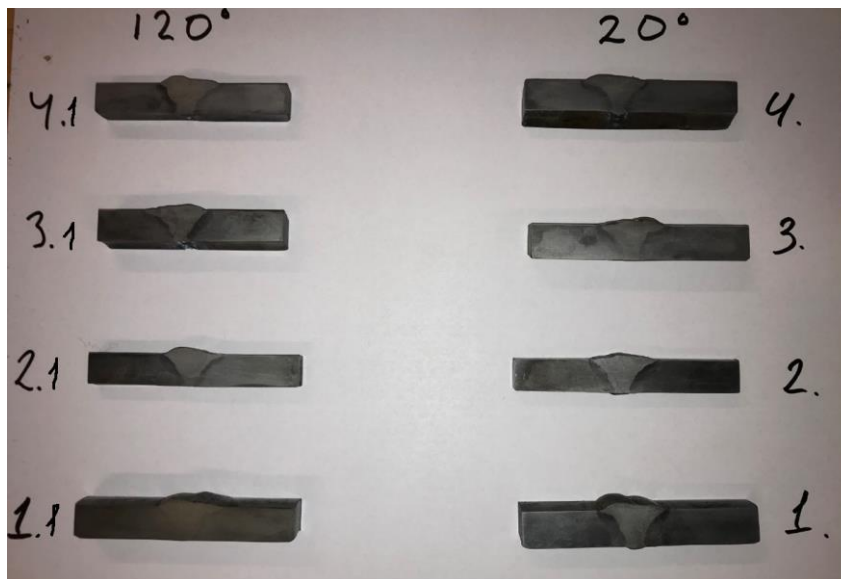


Figure 48, Etched samples

Pictures were taken by the author. License: CC BY-SA 3.0

Note that preheated sample 1.1 stand out from the rest. This sample was etched with Hydrogen chloride on this picture, but was at a later stage (before microscopy) polished and etched with Nitric acid.

Etching defects

After etching with nitric acid, the samples was covered with a dark, easily removable layer that is formed on the base material when etched with nitric acid. Since the layer is dark-blue and it was only observed on the surface of base metal when etched with Nital, it may be Chromium nitrate since filler metal had no chromium in it. It is therefore very important that etched samples are gently wiped after etching to remove the dark layer that covers the metal surface so that it won't be confused with the microstructure of the sample. The layer is visible on element 2 of Figure 49.

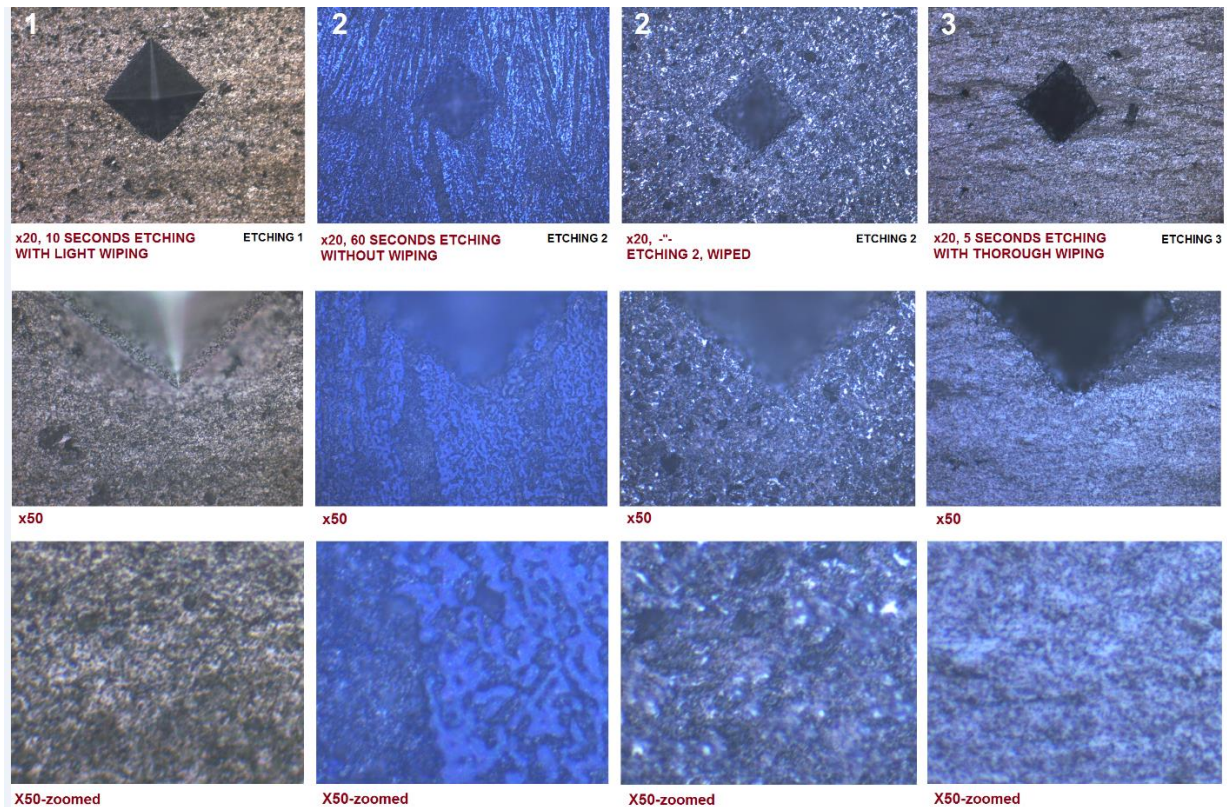


Figure 49, Different etching results

Pictures were taken by the author. License: CC BY-SA 3.0

We can also see some dark spots on the metal surface after etching, the concentration of which is not consistent and seems to be effected by etching time. Those are likely to be caused by the pullout of inclusions in the steel during grinding and polishing or localized corrosion attacks from etching. Those can often be mistaken with the microstructure of the material.

7.5 Samples

We have prepared 8 samples in total, see Figure 48. Four samples was preheated to 120 °C, those samples have suffix *.1. From now on, when referring to a specific sample we will use sample no. as they are marked on the Figure 48 and listed in Table 6.

Sample no.	Preheat °C	PWHT °C	Holding time min.
1	na	na	na
2	na	na	na
3	na	na	na
4	na	na	na
1,1	120	na	na
2,1	120	650	20
3,1	120	650	120
4,1	120	705	20

Table 6, Table of samples

Sample purpose

Samples 1 to 4 was not preheated. Those will be compared to samples 1,1 to 4,1 to see how preheating temperature of 120 °C effect the material hardness.

Preheated sample 1,1 was not heat treated after welding. Microstructure and hardness of this sample will be compared with different PWHT temperatures and holding times.

7.6 Hardness Testing

Hardness testing was done with hardness testing machine ZHU250CL, from Indentec. Calibration of this machine was done 2,5 years ago, which it is less then recommended schedule of 12 month by ISO 6507-2. However it should be sufficient given that the machine is seldom used and verification of overall performance of the testing machine by means of calibrated reference blocks was performed in accordance with the mandatory schedule.



Figure 50, Hardness testing machine

Pictures were taken by the author. License: CC BY-SA 3.0

Hardness survey is performed in accordance with ISO 15156-2. It is carried out using the Vickers HV10 and impression was made as suggested by figure 2 in the standard, see Figure 51.

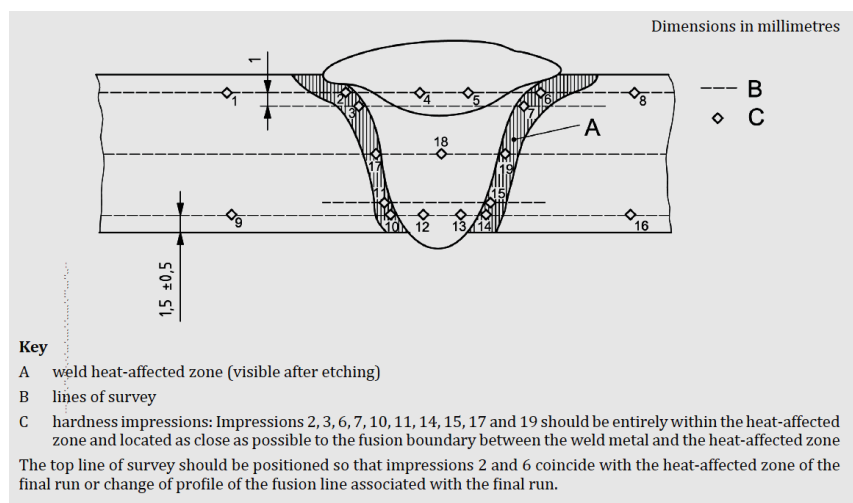


Figure 51, Butt-weld survey method for Vickers hardness measurement, ISO 15156-2.

Copied from ISO 15156-2 – 2015, Copyright International Organization for Standardization

7.7 Heat treatment

To achieve even heat distribution and heat/cooling rate of 335 °C/h, as in accordance with ASME B31.3. standard, a programmable furnace was used.



Figure 52, Controlled heat treatment in furnace

Pictures were taken by the author. License: CC BY-SA 3.0

The heating rate was around 250 °C/h, while the cooling rate could not be controlled in this furnace, thus uncontrolled cooling in turned off furnace was used. The cooling rate was timed and noted for every 50 °C (see Table 7), giving the following treatment diagrams:

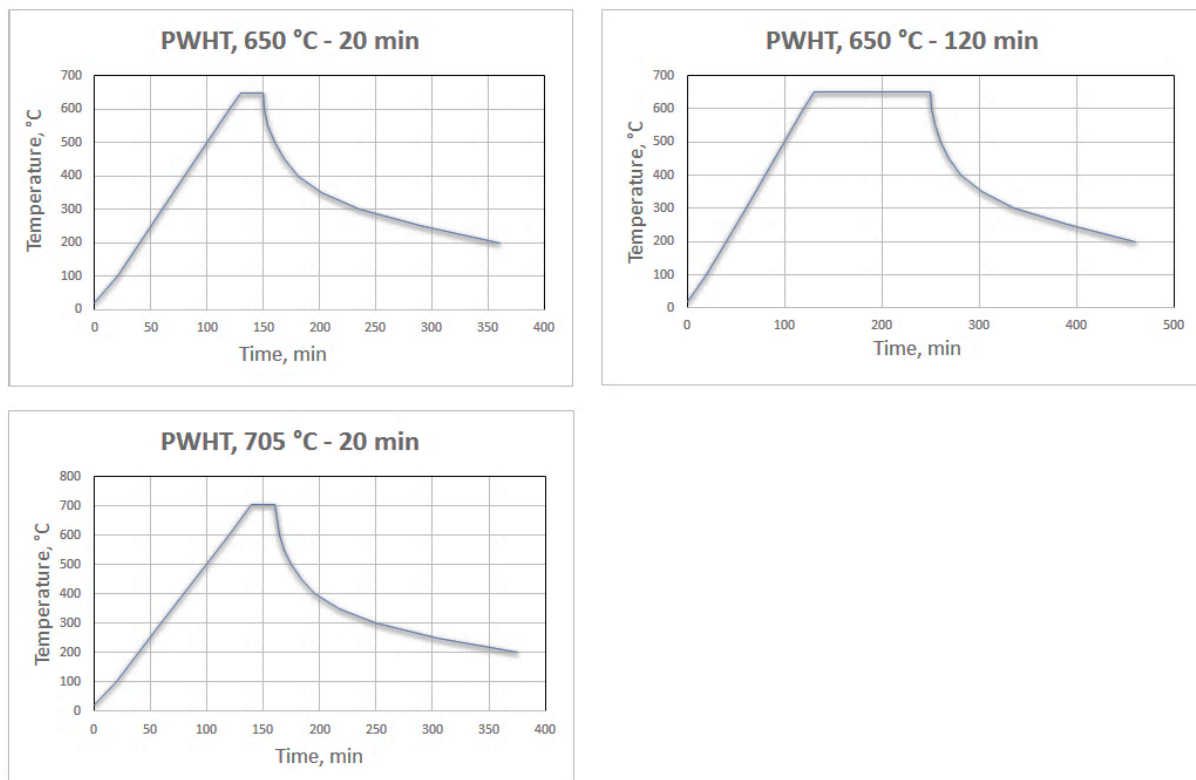


Figure 53, PWHT diagrams

Made by author, License: CC BY-SA 3.0

650 °C - 20 min		650 °C - 120 min		705 °C - 20 min	
temp. °C	time, min.	temp. °C	time, min.	temp. °C	time, min.
20	0	20	0	20	0
100	20	100	20	100	20
300	60	300	60	300	60
600	120	600	120	600	120
650	130	650	130	705	140
650	150	650	250	705	160
600	151	600	251	600	165
550	154	550	254	550	169
500	160	500	260	500	175
450	169	450	269	450	184
400	181	400	281	400	196
350	202	350	302	350	217
300	235	300	335	300	250
250	290	250	390	250	305
200	360	200	460	200	375

Table 7, Cooling rate in furnace

Made by author, License: CC BY-SA 3.0

7.8 Microscopy

The microstructural analysis was prepared with the help of scanning electron microscope (SEM) and energy dispersive (X-ray) spectroscopy (EDS) equipment, Jeol JSM-7200F.



Figure 54, Picture of Jeol JSM-7200F microscope at University of Agder.

The JSM-7800F incorporates multiple types of electrode detectors. For this analysis pictures was taken, using the backscattered electron detector (BED), and a lower electron detector (LED).

LED

Lower electron detector is used for topography images that does not tell so much about different compositions in material structure.

BED

Backscattered electron detector detects different elements in material structures telling us more about the composition of the sample. It is particularly useful for samples composed of different materials like alloy steel.

Backscattered electron gave two types of images: BED-C or BED-S. Bed-C reveals the composition of the material under the microscope, while BED-S is a shading mode that combines both topographical and compositional information in the same image due to the greater sample interaction depth than with LED detector.

EDS Analyses

Is an analytical technique used for analysis or chemical composition of a sample that can be performed with Jeol JSM-7200F. Working principle relies on fundamental principle that each element has a unique atomic structure. The energy from electron beam pushes some of objects electrons to a higher orbit and when those electrons fall back to its original position, they release X-ray radiation that is unique for all elements. That allows for recognition of different elements and generation of different set of peaks that allows for calculation of composition relationships in the sample.

8 Secondary tasks

This chapter is entirely devoted to part one of the research question.

8.1 Split of Services

To clarify whether it is possible or not to avoid NACE MR0175 / ISO 15156 requirements after drilling fluid from the well have been treated, we will assume the use of a standard set of equipment in an offshore mud treatment system.

Although the treatment system can be somehow different from installation to installation, the following equipment is present on most offshore installations [1]:

- Shale-shaker
- Degasser
- Sandtrap
- Desilter
- Centrifuge
- Active Pit
- Mud-Gas separator (emergency equipment for separation of gas in drilling fluid)

Gasses are continuously removed during every step of the treatment process, but only the degasser is specifically designed to remove gas bubbles from the drilling fluid. Since we have no data on the degassing effect of other equipment in the treatment system, we will only consider efficiency of the degasser during normal operation and mud-gas separator + degasser during kick¹ situation.

Sour service

In order for us to suggest whether there are any advantages in splitting the piping specifications in two, non-hydrogen and hydrogen-containing service, we must also argue the difference between them. As it was earlier discussed in the pre-project, the main reason for existing restrictions of material use in hydrogen containing environment in oil and gas production is the danger of material cracking

¹ Kick is a situation of sudden rise of well pressure because of penetration of high pressure zones of formation or gas pockets. See preliminary work for this thesis for more information

that is provoked by hydrogen contamination and its propagation in the material lattice. Thereby the main difference would be the pollution of drilling fluid with hydrogen sulfide.

Not all hydrogen-containing services can trigger the requirements, it is dependent on the concentration of hydrogen and operational pressure. In the case with drilling fluid, it is NACE MR0175/ISO 15156 document that regulates those requirements. In accordance with the standard, no precautions are required for the selection of steels when the partial pressure of H₂S is smaller than 0,3 kPa (0,05 psi). So the introduction of alternative non-sour service specification is only relevant in the parts of the system where the partial pressure of hydrogen sulfide is lower than 0,05 psi.

8.1.1 Partial pressure

Determination of H₂S partial pressure is done using the following formula:

$$P_{H_2S} = p * \frac{X_{H_2S}}{100}$$

p is the system total absolute pressure, expressed in megapascals

X_{H₂S} is the mole fraction of H₂S in the gas, expressed as a percentage.

From this formula, we can see that NACE requirements can be triggered by increased operational pressure and/or increased concentration of H₂S.

Using this formula, a range of H₂S concentration values in units of ppm¹ was generated for pressure class ranges from 150 psi to 15000 psi, see Figure 55.

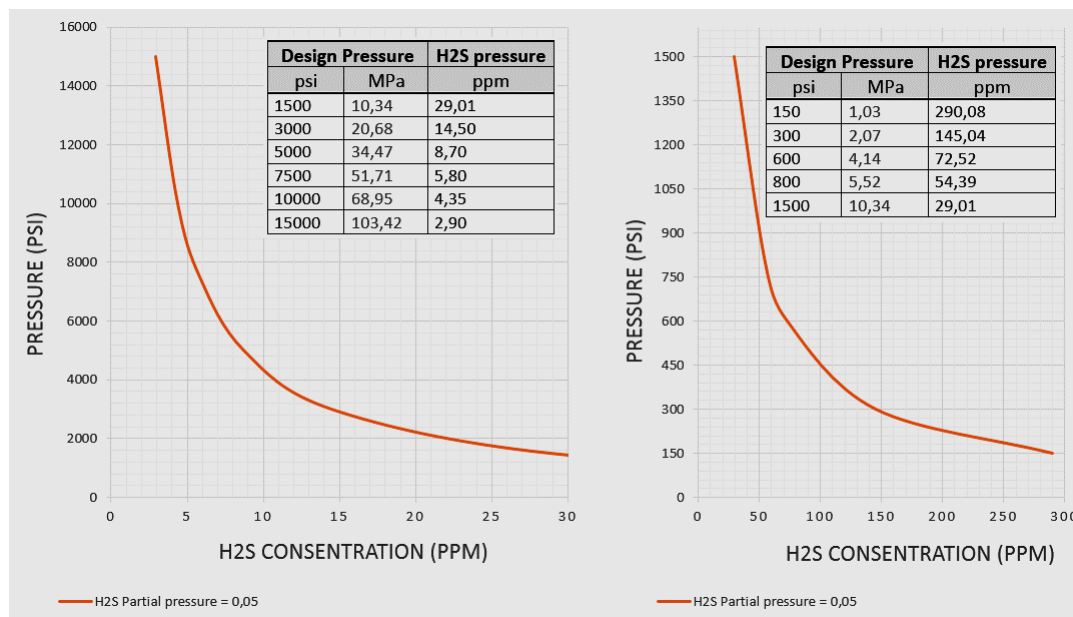


Figure 55, H₂S partial pressure = 0,3 KPa (0,05 psi)

Made by author, License: CC BY-SA 3.0

Based on data from Figure 55, it can be concluded that low-pressure mud (150 psi) system must have H₂S values below **290 ppm**, while for high-pressure mud (10.000 psi) the hydrogen sulfide concentration must be below **4,3 ppm**.

¹ ppm stand for "part per million by volume". This value corresponding to percent volume fraction times 10⁴.

To give us some perspectives the Hydrogen sulfide has an immediately danger to life and health at concentration of 100 ppm, while at concentration over 1,000 ppm (0,1 % per volume gas) it causes nearly instant death. ACGIH¹ recommends a threshold limit value of 1 ppm as an 8-hour time weighted average and a short-term exposure limit of 4,3 ppm. It is also mandatory with monitoring equipment of H₂S concentration in air at drilling sites to make sure that workers are not exposed to health threatening amounts of this substance.

8.1.2 Process values of H₂S

Drilling fluid may be contaminated with various amounts of hydrogen sulfide gas that may vary from a few parts per million (ppm) to over 10,000 ppm. Very high ppm may be an indication of gas pockets in the well and will at certain point lead to a kick² situation that will send the fluid to Mud Gas separator equipment to remove the gas and lower the pressure. Therefore degassers do not experience high contaminations of H₂S, but contaminations of 100 ppm are very common, and 500 ppm can occur.

In order for us to make the considerations of dropping the NACE requirements, we must argue the level of degassing in the mud treatment system. As it is said in the previous chapter, the concentration must be reduced to 4,3 ppm for 10.000 psi system and bellow 290 ppm for 150 psi.

The efficiency rate of single vacuum degasser is usually between 95% and 99,9%. Since only the minimum efficiency rate can be guaranteed by the producer, it can be concluded that degasser alone would not be sufficient to guaranty amount of H₂S bellow 4,3 ppm.

We could consider the possibility of connecting two degassers in series to increase the total efficiency, but according to a degasser supplier AIPU Solids Control (referring to a mail conversation), the efficiency of equipment is affected not so much by the quantity of gas but by it appearance. A lot of gas bubbles with a diameter smaller than 3mm are covered by drilling fluid and is hard to break out. Therefore connecting vacuum degassers in series would give little effect and does not seems like a good solution.

8.1.3 Conclusion on service split

Sour Service requirements are in most cases unnecesery precoution since:

- Hydrogen sulfide content would usually be below 100 ppm in the mud and efficiency of degasser is generally higher than 95%, leading to H₂S contamination below 4,3 ppm.
- Gasses are removed from the drilling fluid at many steps during treatment, see Figure 56: Shale-shaker → Vacuum Degasser → Centrifuge → Active Pit. Making it pretty safe to assume that H₂S concentration will be reduced to below 4,3 ppm by the time they reach mud pump.

Still, there are no specific requirements to the treatment system or mandatory practice like passements after treatment that can guaranty H₂S concentration bellow 4.3 ppm.

¹ ACGIH - Association Advancing Occupational and Environmental Health

² Kick is a situation of sudden rise of well pressure because of penetration of high pressure zones of formation or gas pockets. Se preliminary work for this thesis for more information

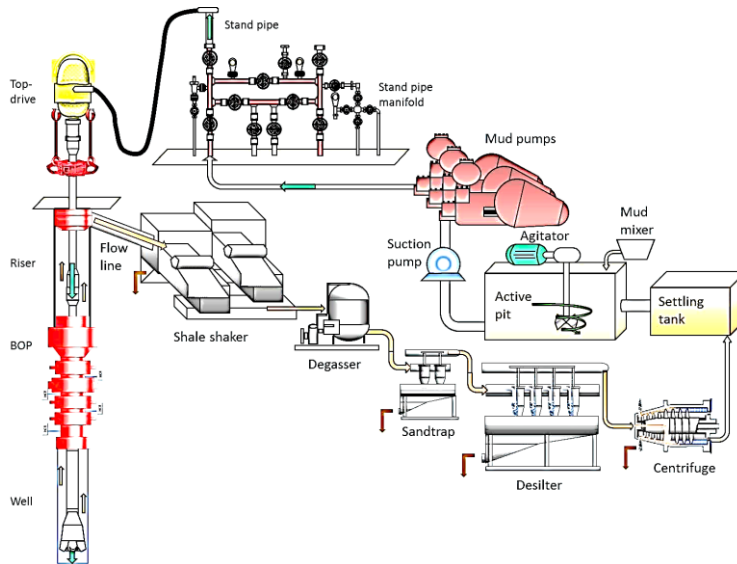


Figure 56, Treatment of drilling fluid

CC-BY-SA-4.0 Author: Sissel Paaske: Adopted from <https://ndla.no>

On that account, we would have to conclude that today's practice does not allow for safe removal of NACE MR0175/ISO 15156 requirements from any parts of high-pressure drilling fluid system. In low-pressure mud, on the other hand, the concentration of H₂S will with most certainty not exceed 300 ppm. Thus the NACE MR0175/ISO 15156 requirements can be ignored for the low-pressure mud systems.

The Figure 57 illustrates different parts of drilling fluid system and as it was concluded above, only low pressured treated mud (150 psi) is out of danger for hydrogen-caused cracking. Unfortunately, the rest of the system could not be guaranteed H₂S contamination that is low enough (see ppm values in Figure 55 and Figure 57) and it is thereby suggested to keep NACE MR0175/ISO 15156 requirements for those systems.

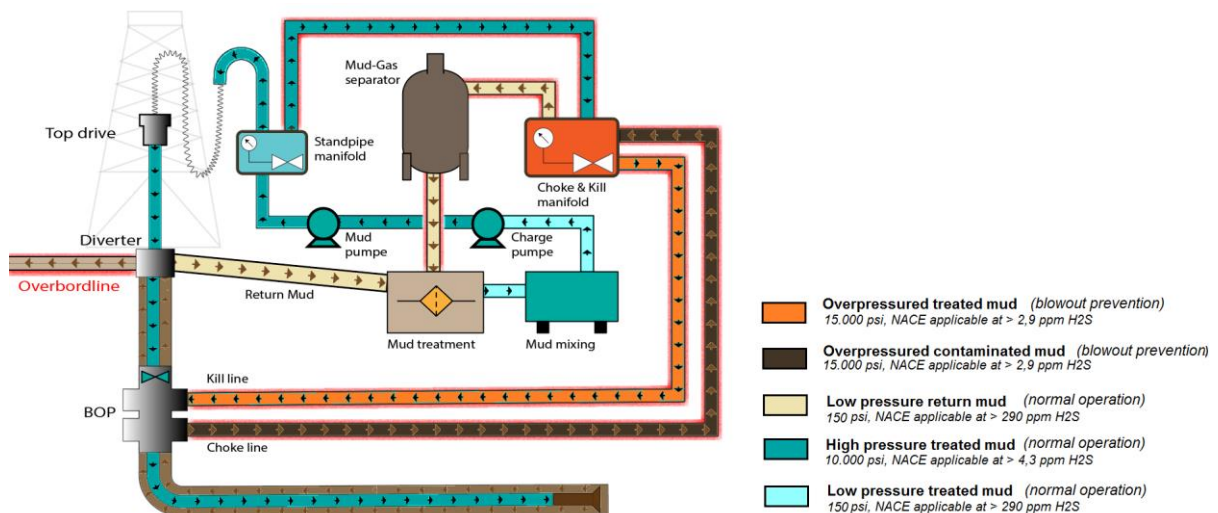


Figure 57, Simplified Mud system map.

Made by author, License: CC BY-SA 3.0

Thereby NACE requirements of 250 Vickers must apply to all high-pressure lines (both 10.000 and 15.000 psi).

8.2 Duplex steel

As it was discussed in pre-project, the duplex steel would be a good alternative to AISI 4130 as it does not have to include corrosion tolerances, which partly compensates for lower mechanical properties. It would also be economically beneficial as it does not require costly surface treatment which is mandatory for carbon steels.

There are many types of duplex steel available, usually divided into two groups: duplex and super duplex steels. Without going into details we can choose one common type from each group to consider. Standard duplex alloy would be super duplex UNS S32760 and duplex UNS 31803 (*main difference in chromium content, 27% in super duplex and 22% in duplex steel*).

8.2.1 Weight reduction

To see if we can get some weight reduction by choosing duplex steel over AISI 4130 we will have to perform wall thickness calculations in accordance with ASME B313, as it was described in chapter 9.2.1 and Attachment 5 of pre-project. Since the calculation method is well described in the pre-project it is not included in this work. Results from the calculation are further listed in the table and plotted on the graph, see Figure 58.

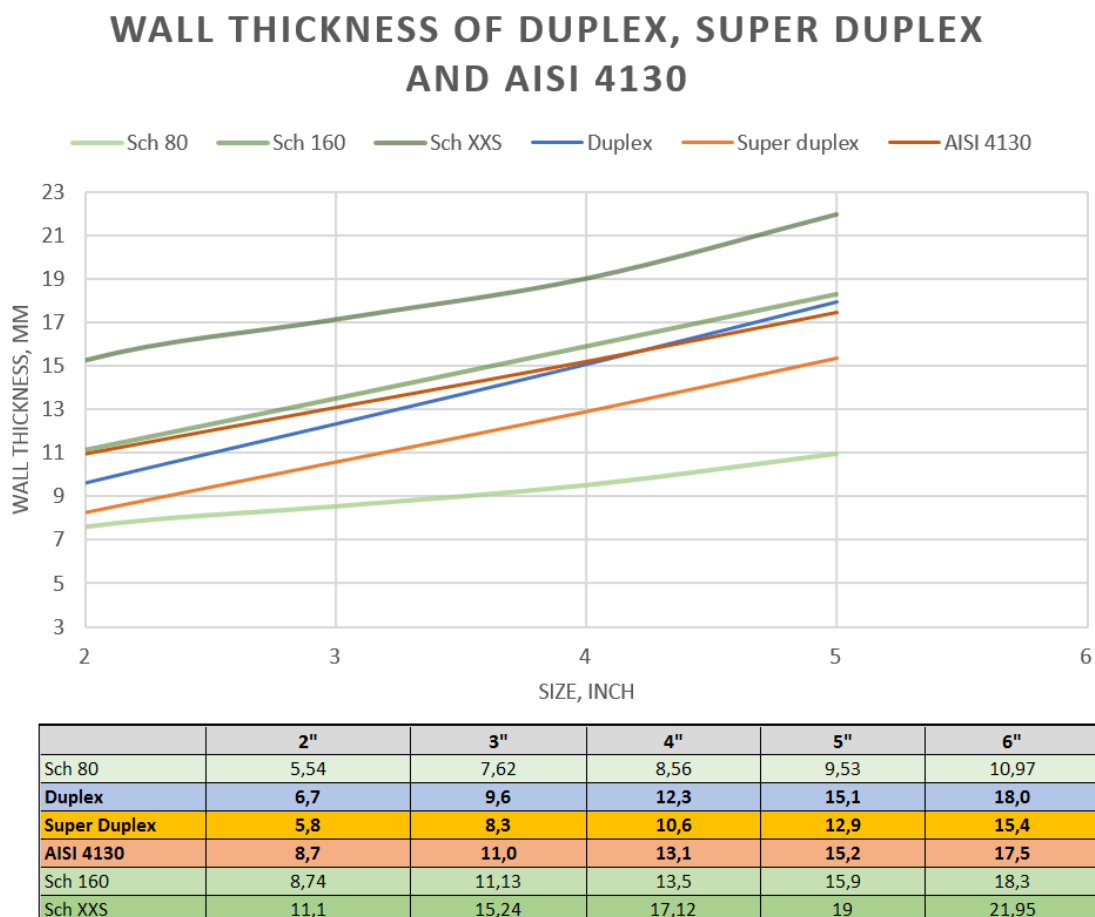


Figure 58, Wall Thickness comparison (UNS 31803, UNS S32760 and AISI 4130)

Made by author, License: CC BY-SA 3.0

From the graph, it can be seen that the wall thicknesses of super duplex steel is reduced with approximately 20% on average. That would be great, but we still have to round it up to the nearest standard wall thickness (schedule) and as it is illustrated all three candidates fall in between Sch 80 and Sch 160, thus either way schedule 160 must be used in all three cases.

8.2.2 Conclusion on duplex steel

By using Duplex steel we will avoid expansive surface treatment, but the spending on the painting of pipe cannot be considered to be higher than its double price (although it can be argued that it may be dependent on the region and the price of working an hour). Therefore, with no weight reduction, double pricing and more demanding welding procedures (those are not discussed in this work) duplex steel do not appear to be an economically correct choice for that purpose.

9 Results

This chapter includes following:

- Description on expected microstructure and comparison to electrode microscope photos.
- Comparison of harness measurement data for preheated and not preheated samples.
- Comparison of harness measurement data for different PWHT conditions.
- EDS analysis

9.1 Microstructure

Water is the most effective quenching medium (of those that is commercially used) and cools steel with a cooling rate above critical, thereby leading to fully martensitic structure. Carbon cannot diffuse out of martensite at room temperature, thus making the steel hard and brittle. Tempering process allows for carbon to diffuse out of martensite by participating out as carbide, see step 2 in Figure 59. Since tempering temperature is above the lowest critical temperature, the formation of austenite at the grain boundaries will take place, see step 3. Cooling of tempering steel in still air leads to a relatively quick cooling rate and will miss pearlite formation, see Figure 37. Austenite will be transformed into ferrite and bainite, see step 4. For an enlarged illustration of microstructure, see Attachment 7 – Illustrations.

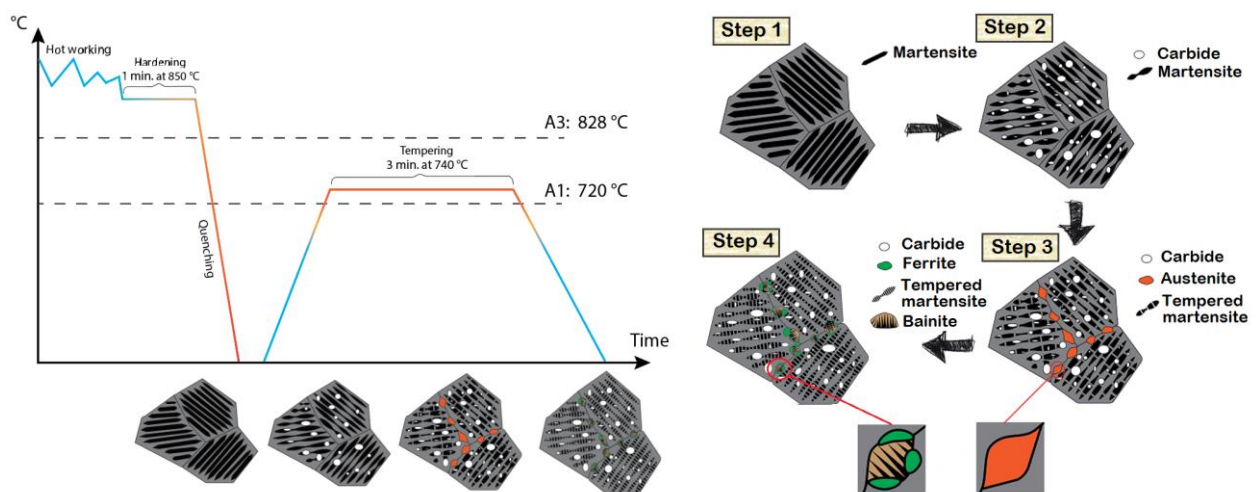
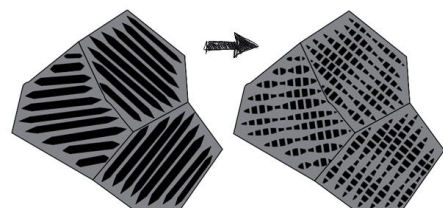


Figure 59, Microstructure of AISI 4130 at different heat treatment steps

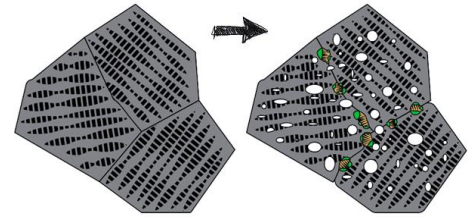
Made by author, License: CC BY-SA 3.0. See attachment 7 for a high-resolution image.

It should be mentioned that the illustration does not include retained austenite after quenching and its decomposition during tempering. That is because the amount of austenite is highly dependent on the temperature to which the steel was quenched. Whatever amounts of austenite were present after quenching it have decomposed into lower bainite.

It is hard to make out the grain boundary lines of quenched steel because of the martensite needles that blend in and defuses the boundaries. When martensite is tempered, the needles defuses out into flakes of carbides, making the structure even more confusing.



In addition to tempered martensite, we have carbides. Mostly it is cementite (Fe_3C), but iron is not the only carbide former in AISI 4130 alloy. Chromium, molybdenum and manganese are carbides formers and can also be participating out as carbide. Because of tempering temperature above A_1 , bainite and ferrite will also be present at grain boundaries, making those boundaries almost impossible to spot on the microscope photos, see Figure 61 and Figure 62.



We were also able to spot some transition carbides such as Fe_2C or $Fe_{2.4}C$, called respectively η (eta) or ϵ (epsilon) carbides. Those are very small particles, ranging in size 1-5 nm and requires very high-resolution images to be spotted. x100.000 enlargement of the base material of sample 3, revealed some small circular flakes of transition carbides that will continue to grow if the temperature is kept high enough and eventually they will be replaced by cementite.

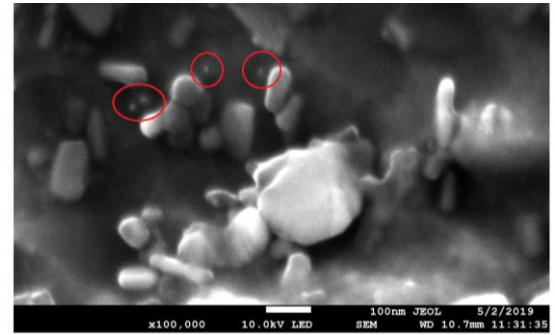


Figure 60, Transition carbides. Sample 1.1, base material, x100.000.

Pictures was taken with SEM microscope. License: CC BY-SA 3.0

Microscopy of AISI 4130 at x2500 enlargement, see Figure 61, reveals parts of microstructure that were previously described and drawn under step 4 of microstructure illustration in Figure 59. It shows both topographical and compositional information, while Figure 62 is a pure compositional image generated by the backscatter electron detector. The pictures were taken from the base material of sample 1.1, that was not heat treated after welding.

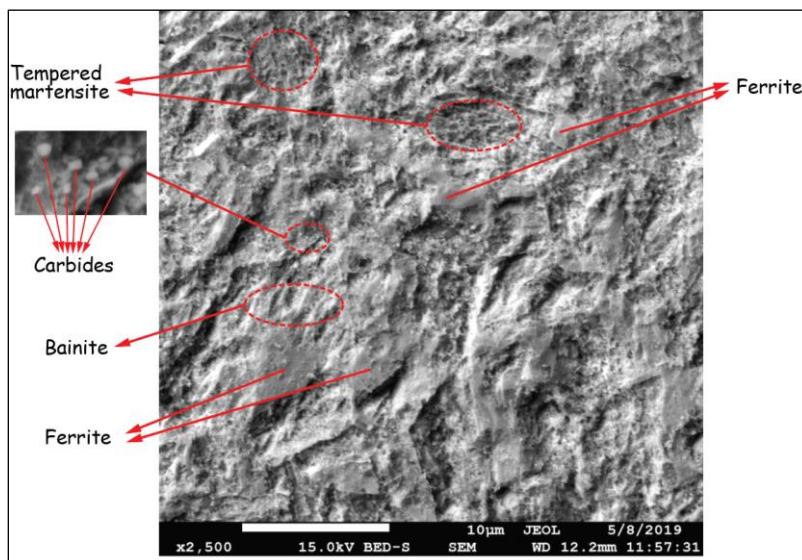


Figure 61, Microscopy of AISI 4130 steel. Sample 1.1, base material, x2500.

Pictures were taken with SEM microscope. License: CC BY-SA 3.0

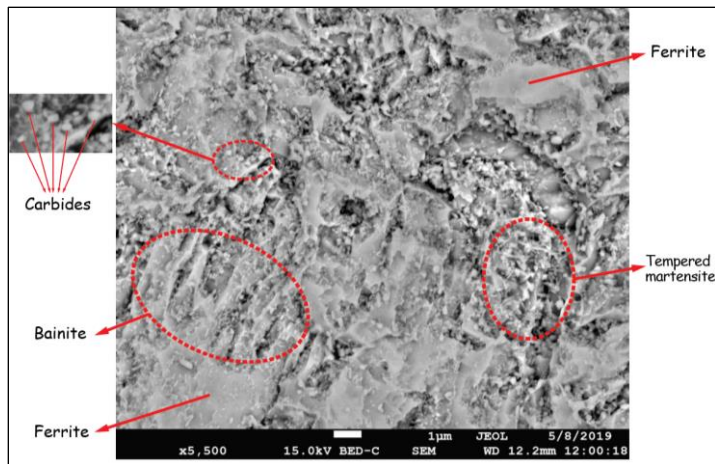


Figure 62, Microscopy of AISI 4130 steel. Sample 1.1, base material, x5500.

Pictures were taken with SEM microscope. License: CC BY-SA 3.0

Figure 63 reveals bainite in heat affected zone of 4.1 sample.

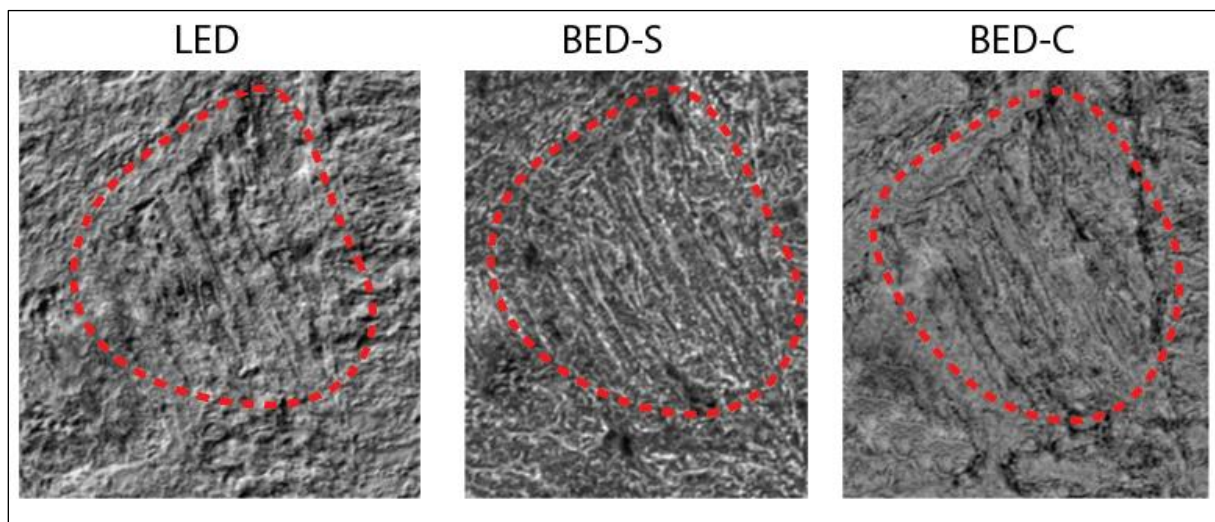


Figure 63, Tempered bainite, at x1200 enlargement. Sample 4.1, HAZ, x5500

Pictures were taken with SEM microscope. License: CC BY-SA 3.0

9.2 Preheat

The minimum required preheat temperature of 120 °C was used to preheat the material prior to welding, see Figure 40.

One of the arguably most important reasons for the preheat is to lower the cooling rate, producing more ductile and less hard structure in heat affected zone. Thereby hardness measurements can be a good indication on whether there is any positive effect from the preheating.

Table of hardness measurements that was prepared in accordance with ISO 15156-2 is attached, see Attachment 6 – Hardness measurements. Average data from all measurements were used to generate graphical representation in Figure 64.

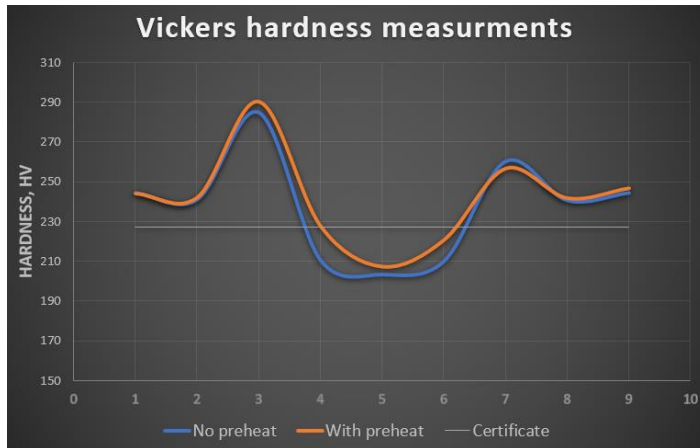


Figure 64, Hardness measurements with and without preheating to 120°C.

Made by author, License: CC BY-SA 3.0

Based on these results it is quite clear that preheat of 120 °C does not have any visible effect on hardness and toughness of the material. However, for confirmation on that assertion, Charpy testing should be carried out.

9.3 PWHT

Three different PWHT conditions were used, see Table 6. The result from hardness measurement of those samples is attached, see part 2 of Attachment 6 – Hardness measurements.

Hardness measurements revile extensive loss of hardness in heat affected zone, to a value below the one that is specified in the material certificate. There is also some indication of a slight hardness reduction in the base material.

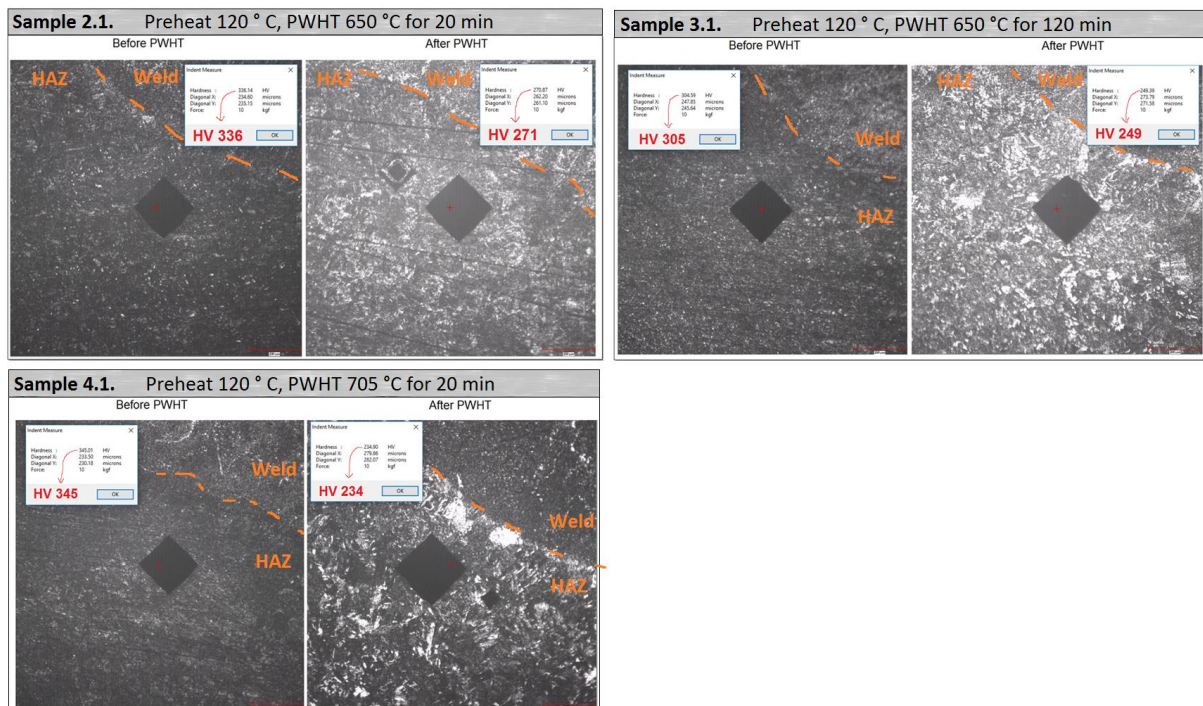


Figure 65, Hardness measurements of HAZ.

Pictures was taken with $\times 10$ microscope on hardness testing machine. License: CC BY-SA 3.0

Microscope picture of hardness indentation point reveals clear changes in HAZ with an increase of post weld heat exposer, see Figure 65. As the microstructure of base material seems to be unaffected by the increase of heat treatment holding time or temperature, the HAZ seems to be losing a lot of hardness and become visually courses as temperature exposure increases.

Extensive loss of hardness at high tempering temperature, even for a shortest possible holding time is graphically illustrated in Figure 66. The chart is based on the results from attached hardness measurements.

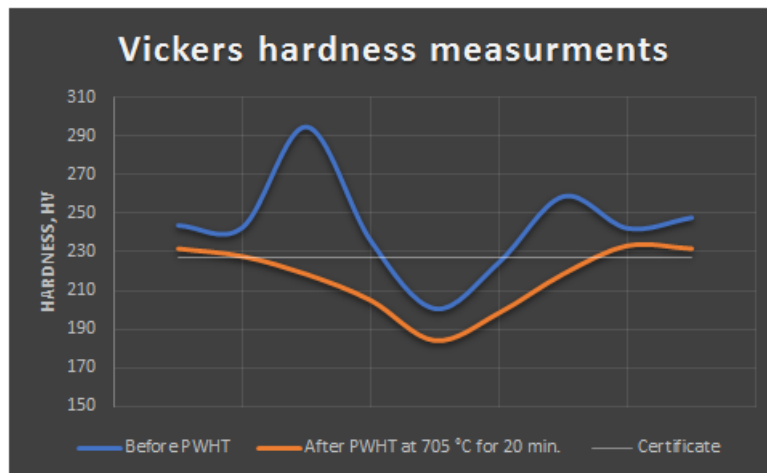


Figure 66, Results from hardness testing after PWHT at 705 °C.

Made by author, License: CC BY-SA 3.0

The HAZ of the weld is indicated by the peaks in the blue line chart, which indicates residual stresses in the heat affected zone. The orange line chart represents hardness distribution across the welded material from which we can clearly see the effect of PWHT on residual stresses after welding. It is clear from hardness results that it was reduced far below the certificate values and thereby, the material can be regarded as overtreated.

9.3.1 Tempered martensite

In order to understand the mechanism of visual changes of HAZ that is visible in Figure 65 and to understand the extensive loss of hardness, several pictures of samples with different PWHT temperatures was taken with SEM microscope, see Figure 67.

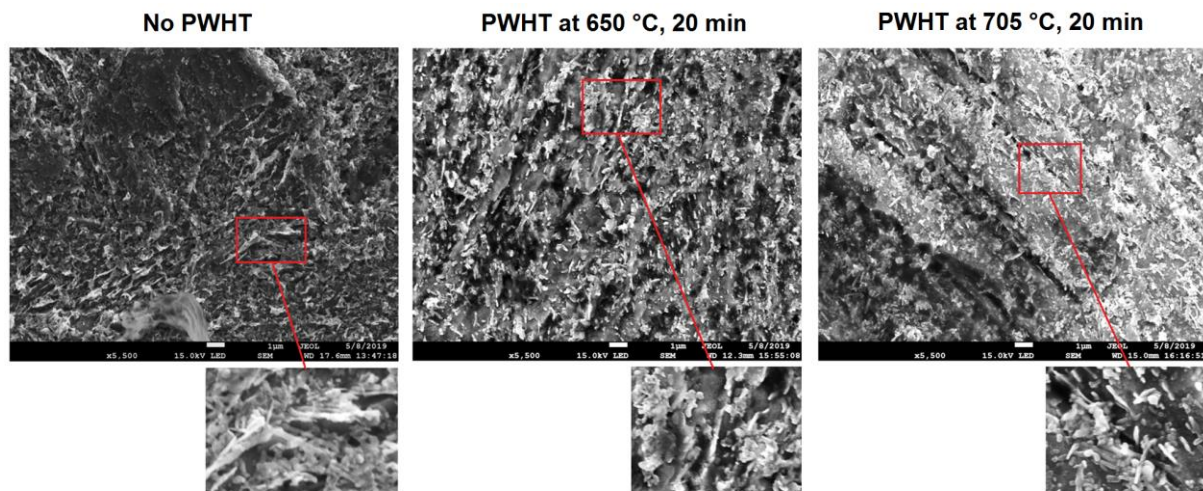


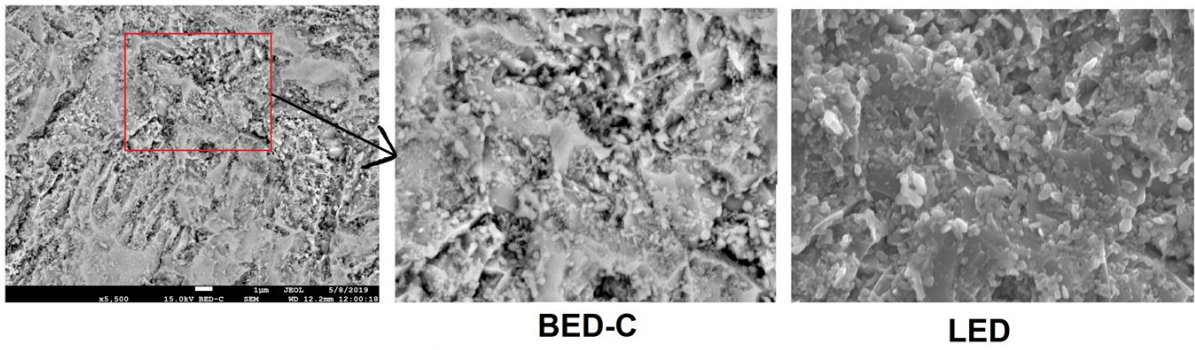
Figure 67, Effect of PWHT on martensite in HAZ

Pictures were taken with SEM microscope. License: CC BY-SA 3.0

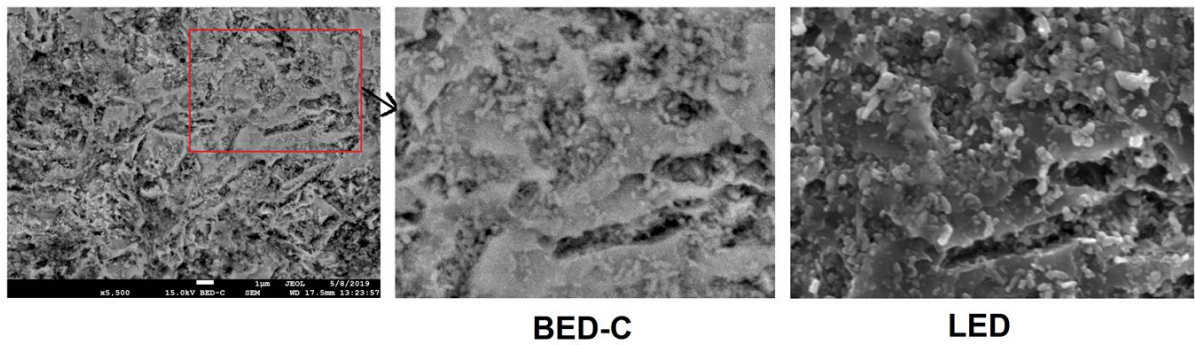
It is quite clear that martensite has lost its tetragonality in HAZ already before PWHT, which can be explained by the tempering effect that welding has on itself. Post weld heat treatment at 650 °C for 20 minutes have allowed for further carbon diffusion and participation of carbides, thus we can see more white particles and less needle-like structure. The sample that was heat treated at temperature of 705 °C (*maximum allowed PWHT temperature for this steel according to ASME B31.3*) have clearly experienced high carbide coarsening because of high diffusion of carbon and gathering of carbides along grain boundaries. That explains the growing amount of white flakes under x10 microscope in Figure 65.

SEM pictures in Figure 68 reveals the growth of carbide particles in the base material, as the PWHT temperature increases (*see also Attachment 9 – Microscopy, where PWHT at 650 °C for 120 minutes is also included*).

Sample 1.1. No PWHT



Sample 2.1. PWHT at 650 °C for 20 min



Sample 4.1. PWHT at 705 °C for 20 min

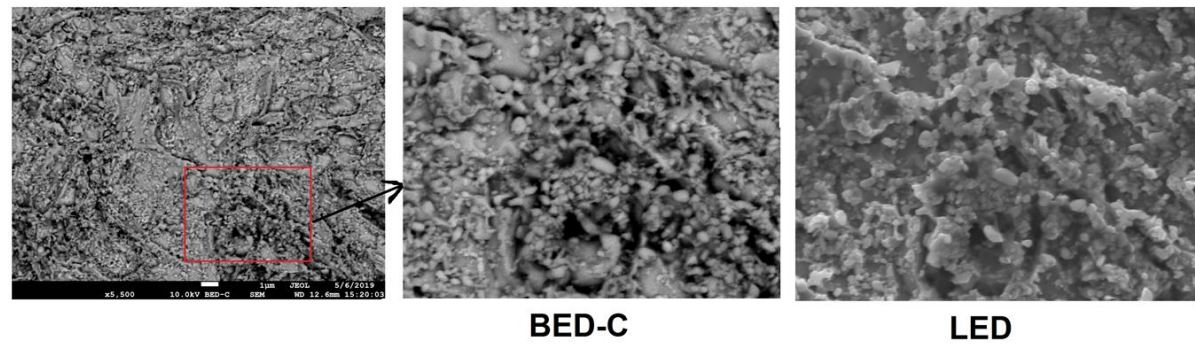


Figure 68, Participation of carbides in base material of AISI 4130.

Pictures were taken with SEM microscope. License: CC BY-SA 3.0

9.4 EDS analysis

EDS analysis was used to determine the composition of the chemical elements and estimate their proportion in the steel. The accuracy of this analysis of sample composition is affected by various factors

- Elements can have overlapping X-ray emission peaks.
Solved by forcing out elements that are not expected in this steel.
- Chemistry of the etchant.
Solved by preparing sample with not etching.
- Contaminated sample.
Sample was handled with gloves and EDS analysis was done shortly after polishing of the sample.

Multiple other sources of accuracy issue can be affecting our results, which should be at some extent covered by the error factor in Table 8.

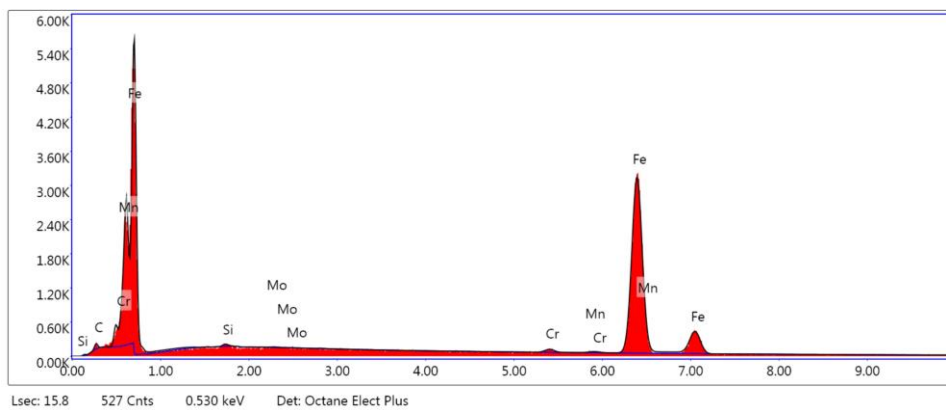


Figure 69, EDS spectrum of AISI 4130

Prepared with Energy Dispersive X-Ray Spectroscopy. License: CC BY-SA 3.0

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
C K	1.4	6.2	29.3	24.2	0.0043	1.3497	0.2264	1.0000
SiK	0.2	0.4	21.0	46.5	0.0013	1.1757	0.5514	1.0028
MoL	0.1	0.1	4.9	68.4	0.0007	0.8807	0.8865	1.0014
CrK	0.9	1.0	45.6	23.8	0.0108	0.9991	0.9929	1.1471
MnK	0.3	0.3	11.9	66.0	0.0033	0.9782	0.9972	1.1330
FeK	97.0	92.1	2889.4	2.9	0.9637	0.9939	0.9991	1.0002

Table 8, EDS results

The results from EDS spectroscopy reveals relatively accurate results for all composition elements of AISI 4130 steel sample except for carbon. Carbon content is show to be very high even if we account for the expected error of 24 %, the content is still three times over the certificate value.

Graphite

Another sign of high carbon content in this steel is graphite flakes that seem to be present in a high amount at steel surface.

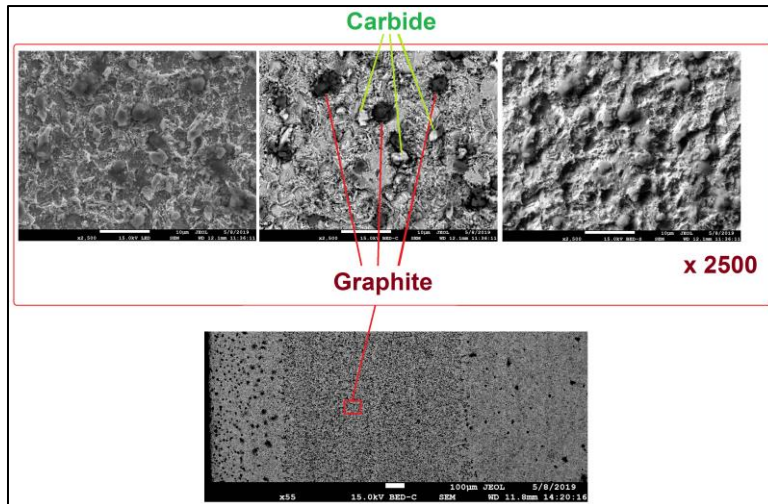


Figure 70, Graphite flakes in AISI 4130

Pictures were taken with SEM microscope. License: CC BY-SA 3.0

The explanation of high carbon content in EDS result could be contamination of the internal walls of the vacuum chamber with carbon, especially if it was used to inspect organic material earlier. Inaccurate EDS data of carbon content is a common problem, thus the carbon can often be disregarded.

Another explanation could be the material of windows used in the EDS detector, that has an absorption edge very close to the C X-ray energy, resulting in artificial carbon (C) peak [8].

Graphite, however, is not something we expect to see in low alloyed steel. To explain these phenomena, further investigation is required, that will not be a part of this work.

10 Discussion

In this chapter, we will discuss the following:

- The microstructure, based on hardness measurements and microscopy.
- Tempering effect on the hardness and microstructure
- Hardness difference of bainite and martensite
- Required holding time for PWHT temperature

10.1 Hardness testing

Base material

When heat treating welded steel below its original tempering temperature, no changes in the base material should be expected. Thus the heat treatment should only effect and release stresses from the weld and heat affected zone. Since during the production stage the tempering temperature was held for a very short amount of time, it seems to allow for further decomposition of martensite at lower temperatures with longer holding time that leads to further martensite tempering in the base material and small loss of hardness, see part 2 of Attachment 6 – Hardness measurements.

HAZ

HAZ consists of different regions that are dependent on heat input from the welding process, see chapter 4.6. For simplicity, we can divide those regions into two: tempering region (below critical temperature) and the region of austenitization (above critical temperature).

Tempered regions will experience tempering of the original structure and as long as the temperature is below the lowest critical temperature and the original tempering temperature, no significant changes in the material structure should be possible since the holding time of temperature splices caused by welding is relatively low.

Austenized region will experience recrystallization and possibly some grain grows. The extend of grain grows is dependent on the heat input. The closer to the fusion line the faster will be the grain grows.

Deviation of hardness

According to ISO 15156, Individual HRC (Rockwell hardness) readings exceeding the value permitted by this part of ISO 15156 may be considered acceptable if the average of several readings taken within close proximity does not exceed the value permitted by this part of ISO 15156 and no individual reading is greater than 2 HRC above the specified value. Equivalent requirements shall apply to other methods of hardness measurement.

2 HRC corresponds to approximately 14 HV in Vickers scale. Those the acceptable deviations from the 250 HV allowing for single splices of hardness to 264 HV, as long as the average values does not exceeds permitted limits of ISO 15156. That makes small deviations in hardness of sample 3.1 (PWHT at 650 for 120 min.) fully acceptable since the average value is far below the accepted value of 250 HV.

10.2 Tempering temperature

Tempering is a process in which the microstructure approaches equilibrium under the influence of thermal activation, thereby tempering of microstructure that is in equilibrium will have limited effect in contrast to not equilibrium phases like martensite. Since the tendency to temper depends on how far the starting microstructure deviates from equilibrium, the carbon concentration is a decisive factor for the tempering temperature. The higher the carbon concentration the harder and less stable will be the martensite formations in the steel, allowing for carbide participation at lower temperatures.

Tempering during manufacturing

Since the base material was heat treated just above critical temperature for a short amount of time, new grains of austenite was formed, but they did not have time for grain growth. That leads to a material with longer grain boundaries that can counteract dislocations movements better. See Attachment 7 – Illustrations.

This tempering effect should be present on parts of the heat affected zone that was heated to above critical temperature, A1.

Quenching effect

Parts of the HAZ experience more martensite formation than others, see Figure 71.

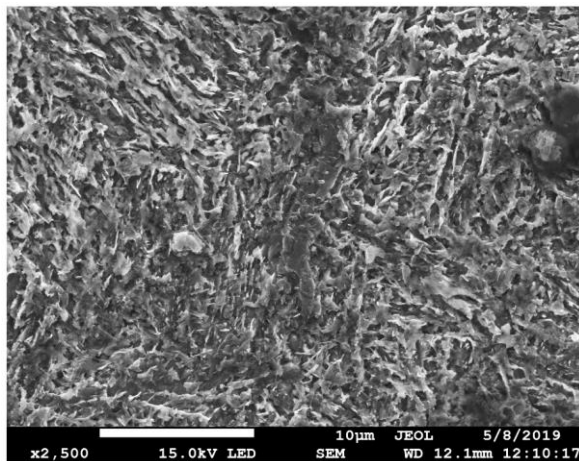


Figure 71, Martensite formations in HAZ of sample 3

Pictures were taken with SEM microscope. License: CC BY-SA 3.0

Those martensite islands could be explained by spikes of higher heat input from the welding followed by the high cooling rate. Those regions would give the highest hardness in HAZ.

10.3 Bainite vs martensite

The hardness of bainite and martensite is directly dependent on carbon content. Although martensite has a higher maximum hardness for a given carbon content, martensite can be tempered to the same hardness as bainite. Lower bainite and tempered martensite can thereby end up with same hardness values [9]:

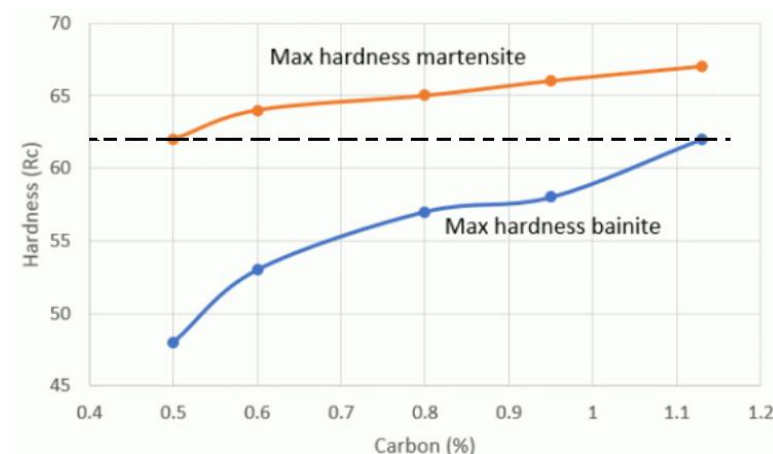


Figure 72, Carbon content vs hardness of martensite and bainite

Adopted from <https://knifesteelnerds.com>. Article: Bainite vs Martensite – The Secret to Ultimate Toughness.

Thus it is fair to assume that in the case with low alloy steel that was tempered above its lower critical temperature, the martensite will experience extensive tempering during heating and tempering stage. While bainite will start forming from new grains during cooling after tempering, thus martensite will be heavily tempered by the time when lower bainite will form. Thereby martensite could end up with hardness that is relatively close to the hardness of newly formed lower bainite.

Since bainite is less brittle and has higher impact toughness than martensite, lower bainite should be preferred structure. Especially if the same strength can be achieved.

10.4 Holding time

Holding time of 120 minutes gave good results for 11mm thick pipe, but it is more practical to qualify one temperature for a range of pipe thicknesses. The range of pipe thicknesses in piping specifications for high-pressure mud system is 11-22mm, see Attachment 10 – Piping specification. To convert holding time of 120 minutes for 11 mm to time that is required for 22 mm thickness, we use ASME B31.3 requirements of 1 hour holding time per 25mm. That gives us 26,4 minutes that we should add for those extra 11mm $((60min/25mm)*(22mm-11mm)=26,4mm)$. Thereby optimal holding time for pipes with thickness range 11-22mm should be 150 minutes.

It should be noted that in the preliminary part for this thesis, it was decided to reduce the wall thickness from Schedule XXS to Schedule 160 for the next revision of 10.000 psi piping specification [1]. That will reduce wall thickness range from 9 -18 mm. However, we choose to qualify this work for SCH XXS, that will not add many minutes to holding time of PWHT, but will qualify for both XXS and 160 schedule pipe.

11 Conclusion

Choice of PWHT temperature is highly effected by heat treatments that is applied during the production process. When the tempering temperature was above lower critical temperature A1, it could have led to some amount of recrystallization (austenization). Those new grains will take the form of lower bainite with hardness values close to values of tempered martensite, giving us finer grain structure with the same hardness, thus making the material harder and stronger.

Based on that mechanism we want to keep the hardness of bainite as high as possible after post weld heat treatment in order to keep the intended properties of the material. Thereby we must choose the lowest possible temperature and holding time. Heat treatment at 650 °C with holding time of 120 minutes gave us a good result. Although a couple of indentation points was very close to and at one point slightly above the critical value of 250 HV specified by NACE / ISO 15156, still, the average value of HAZ was 233 HV.

By increasing the temperature to 705 °C we are risking the extensive loss of hardening by two mechanisms:

HAZ < A1

Heat affected zone that was not brought to temperatures above critical did not change its material structure during welding, thus it still partly consists of bainite that may experience extensive tempering and weaken the material.

HAZ > A1

Recrystallized part of HAZ will have coarser grains than the rest of the material. Those will mainly consist of martensite if the preheat temperature was not brought high enough to allow for slower cooling rate. If this type of structure, with coarser grains and martensitic structure heated up to much it may lose its hardness to values below acceptable, making the HAZ to a weak spot.

Thereby everything suggests that we keep PWHT temperature as low as possible, which in our case is 650 °C. The holding time of 120 min is shown to be sufficient for our 11 mm thick pipe and should not be increased to unnecessary high values that will do more harm than good, but to qualify the whole range of pipe thicknesses (11-22mm) holding time of 150 minutes is chosen.

That concludes the main task with that following PWHT parameters should be used for removal of residual stresses and bringing the average hardness values below 250 HV:

PWHT temperature:	650 °C
PWHT holding time:	150 min.

This conclusion applies for material that was preheated to 120 °C, which is the minimum required temperature by ASME B31.3. Although the effect of this preheating temperature could not be detected either by hardness measurements or microscopy. For higher preheat temperatures (see next chapter) it is possible that we could reduce the holding time of PWHT to even lower time value.

12 Suggestions and afterthoughts

Heat input

Heat input from the welding is an essential factor in the quality of the weld, it governs the cooling rate in welds and thereby affects the microstructure of the heat-affected zone.

It is also important to use as low heat input as possible to avoid unnecessary recrystallization and tempering of the base material. By reducing the heat input we will reduce the formation of martensite island in HAZ and will avoid extensive tempering of bainite. Tempering of bainite will have a negative effect on material properties by reducing its strength, while the formation of martensite will reduce its toughness.

Heat input can be reduced by choosing a welding procedure that allows for high arc travel speed or by lowering welding current and arc voltage.

Preheat and interpass temperature

Preheating temperature is also of high importance. Low preheating can result in high cooling rates and formation of martensite, while too high preheat temperature in combination with heat input from the weld can cause extensive grain growth and thereby deterioration of mechanical properties for carbon steel. It should therefore not be held to height either.

The preheat temperature should ideally be held throughout the whole welding process, thus interpass temperature should be the same as preheat temperature. Maximum allowed interpass temperature by Norsok M-601 is 250 °C for carbon steel, thus this is the temperature that should be used for preheating. Heat input should then be reduced accordingly.

The low cooling rate of recrystallized part of HAZ will also lead to the formation of bainite instead of martensite, which is preferable for increased toughness of steel without reducing its strength, see chapter 10.3.

Etching

Certain steps should be followed during the etching process to avoid impurities in the material that can be confused with material structure. Based on the experience of this work, it is suggested to use 2-3% Nitric acid. Etch for 5-10 seconds and wipe the surface clean of dark coating from etchant with a piece of non-organic cloth that leaves no lint or dust (microfiber wipes should be a good choice).

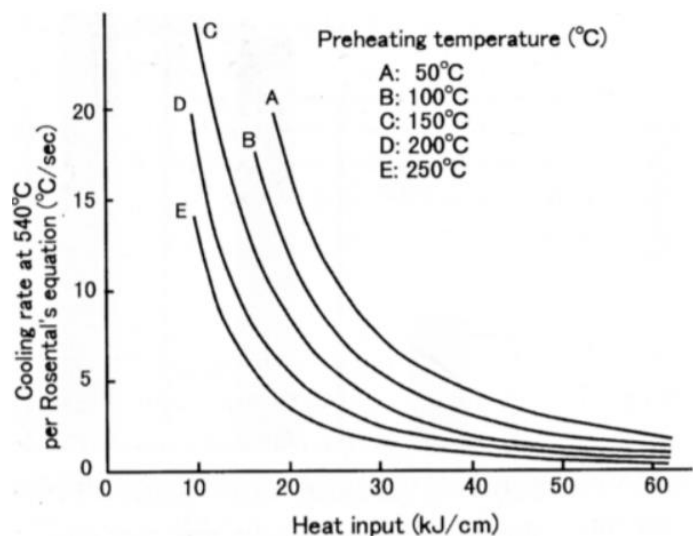


Figure 73, The effect of heat input on cooling rates in welds as a function of preheating temperatures

Copied from
<https://www.kobelco.co.jp/english/welding/files/kwt2000-04.pdf>
 Article: KOBELCO Welding today.

13 Bibliography

- [1] D. Rybakov, Rør materialer i offshore borevæske system, Grimstad: UIA, 2018.
- [2] MHWirth, *Standard piping specification*, Doc No.: MHSTD-L-ZZ50-SA17-0001, 2018.
- [3] *Materials for use in H2S-containing environments in oil and gas production*, NACE MR0175 / ISO 15156, 2015.
- [4] Y. N, *Carbon equivalents for hardenability and cold cracking susceptibility of steels*, 1990.
- [5] W. Callister and D. Rethwisch, *Materials Science and Engineering*, 2011.
- [6] J. S, *Tempering of Steel: 4 Stages*, <http://www.engineeringenotes.com>.
- [7] *ASME Code for Pressure Piping, B31.3*, 2016.
- [8] "EDS Measurement of Carbon," [Online]. Available: <http://www.globalsino.com/EM/page1853.html>.
- [9] J. D. Verhoeven, "Steel metallurgy for the non-metallurgist," ASM International, 2007.

14 Attachments

14.1 Attachment 1 – Material Data Sheet

MDS X21

MATERIAL DATA SHEET			MDS X21	Rev. 01
TYPE OF MATERIAL: Low Alloyed Steel, Type 4130				Page 1 of 1
PRODUCT	STANDARD	GRADE	ACCEPT. CLASS	SUPPL. REQ.
Seamless pipes Wrought fittings (seamless)	ASTM A519 ASTM A234	AISI 4130 AISI 4130	- -	- S2
1. SCOPE	This MDS specifies the selected options in the referred standard and additional requirements which shall be added or supersede the corresponding requirements in the referred standard.			
2. MANUFACTURING PROCESS	Pipes shall be manufactured by means of the hot finished (HF) sizing method. Only seamless fittings are acceptable.			
3. HEAT TREATMENT	Fittings and pipes shall be delivered in the liquid quenched and tempered conditions. The tempering temperature shall be minimum 650 deg C.			
4. CHEMICAL COMPOSITION	Max. sulphur content: S<=0.025% Max. phosphorous content: P<=0.025%			
5. TENSILE TESTING	Minimum yield strength: Reh=>517 MPa (75 KSI) Minimum tensile strength: Rm =>690 MPa (100 KSI) Minimum elongation: A5 => 17% Minimum reduction of area: Z => 35%			
6. IMPACT TESTING	Charpy V-notch impact testing shall be carried out according to ASTM A370 for thicknesses t=>6 mm. Full sized Charpy V-notch specimens shall be used wherever possible. The notch shall be perpendicular to the surface. The test temperature shall be -30 deg C. The minimum absorbed energy for full size specimens shall be 42 J average and 30 J single. Reduction factors for sub sized specimens shall be: 7.5 mm – 5/6 and 5 mm – 2/3.			
7. HARDNESS	The hardness shall be maximum 22 HRC. NACE MR – 175			
8. EXTENT OF TESTING	One set of tensile and impact tests shall be carried out for each lot. A lot is defined as all products of the same type, normal size and wall thickness, produced from the same heat and heat treatment load. For pipes heat treated in continuous furnace the maximum lot size shall be 60 m.			
9. TEST SAMPLING	Samples for production testing shall realistically reflect the properties in the actual component. Fittings: According to supplementary requirement S2.			
10. NON DESTRUCTIVE TESTING	Pipes: All pipes shall be 100% tested in accordance with API 5L supplementary requirement 4 (SR4). Alternatively, ultrasonic testing according to SEL 1915 may be carried out. Fittings: Fittings shall be 100% magnetic particle tested in accordance with ASME VIII, div. 1, Appendix 6. All products: Hardness tested to maximum 22 HRC and shall satisfy NACE MR 0175.			
11. REPAIR OF DEFECTS	Weld repair is not acceptable.			
12. MARKING	The product shall be marked to ensure full traceability to melt and heat treatment lot.			
13. CERTIFICATION	EN 10204 type 3.1. Heat treatment temperature, soaking time and cooling medium should be stated in the certificate.			

MDS X24

MATERIAL DATA SHEET			MDS X24	Rev. 01
<i>TYPE OF MATERIAL:</i> High Strength Low Alloyed Steel Type 4130				Page 1 of 2
<i>PRODUCT</i>	<i>STANDARD</i>	<i>GRADE</i>	<i>ACCEPT. CLASS</i>	<i>SUPPL. REQ.</i>
Forgings	API 6A	75K (AISI4130)	Product Specification Level (PSL) 3	-
<i>1. SCOPE</i>	This MDS specifies the selected options in the referred standard and additional requirements which shall be added or supersede the corresponding requirements in the referred standard.			
<i>2. MANUFACTURING PROCESS</i>	The forgings shall be finished hot-worked. The flanges shall be forged to shape. Flanges machined out of bar and or plate are not accepted.			
<i>3. HEAT TREATMENT</i>	The forgings shall be austenitised, liquid quenched and tempered.			
<i>4. CHEMICAL COMPOSITION</i>	The steel chemistry shall comply with the requirements of AISI 4130, however modified in accordance with the requirements PSL 3 given in API 6A. The chemical composition shall be agreed.			
<i>5. TENSILE TESTING</i>	Minimum yield strength: Reh=>517 MPa (75 KSI) Minimum tensile strength: Rm =>690 MPa (100 KSI) Minimum elongation: A5 => 17% Minimum reduction of area: Z => 35%			
<i>6. IMPACT TESTING</i>	Charpy V-notch impact testing shall be carried out at -30 deg C. The minimum absorbed energy for full size specimens shall be 42 J average and 30 J single.			
<i>7. HARDNESS</i>	The hardness shall be maximum 22 HRC. NACE MR – 175			
<i>8. EXTENT OF TESTING</i>	One set of tensile and impact tests shall be carried out for each melt, section thickness according to API 6A, PSL 3, and heat treatment load. A test lot shall not exceed 2000 kg.			
<i>9. TEST SAMPLING</i>	Samples for production testing shall realistically reflect the properties in the actual component. Test samples shall be from prolongations on actual components. Sacrificial forgings shall be used for die forged components. However, special agreements may be made for die forged components with as forged weight exceeding 50 kg. Test specimens shall be cut at the ¼ T location from the surface where T is the thickness of the test samples as heat treated. Sketches shall be established showing type, size and location of test samples and extraction of test.			
<i>10. DIMENSIONAL TOLERANCES</i>	Flanges to MSS SP-44 shall have a maximum wall thickness under tolerance of 0.3 mm for the hub at the welding end.			
<i>11. NON DESTRUCTIVE TESTING</i>	NDT shall be carried out after final heat treatment: - 100% MT according to ASME VIII, Div.1, App.6, shall be carried out. - 100% UT according to ASTM A388, shall be carried out. The acceptance criteria shall be according to ASTM A388 para 8.			
<i>12. REPAIR OF DEFECTS</i>	Weld repair is not acceptable.			
<i>13. MARKING</i>	The component shall be marked to ensure full traceability to melt and heat treatment lot.			
<i>14. CERTIFICATION</i>	EN 10204 type 3.1. Heat treatment temperature, soaking time and cooling medium should be stated in the certificate.			

14.2 Attachment 2 – Material Certificate

REVIEWED BENTELER
 By stein roar egge at 6:44 am, Dec 17, 2012

ABNAHMEPRÜFZEUGNIS EN 10204-3.1
 INSPECTION CERTIFICATE EN 10204-3.1
 CERTIFICAT DE RECEPTION EN 10204-3.1
 EN 10204:2005-01

Dokument-Nr.: 65-706733/001/P
 Document No.:
 No. du document:

Prüf-Nr.:
 Inspection No.:
 No. du certificat:

Blatt: 1 / 3
 Page:

Kunden-Bestell-Nr.: 16383/0000
 Purchase Order No.:
 No. de commande du client:

Hersteller:
 Manufacturer:
 Producteur:

Warmrohrwerk Dinslaken
 (DIN EN ISO 9001, ISO/TS-16949 CERTIFIED BY TÜV NORD CERT)
 (PED 97/23/EC CERTIFIED BY TÜV NORD SYSTEMS)

Benteler Auftrags-Nr.: 1568398
 Benteler Order No.:
 No. de commande Benteler:

Herstellerzeichen:
 Manufacturer's brand:
 Marque du producteur:

Stempel des Abnahmebeauftragten: WA
 Stamp of the inspection representative:
 Poignon du contrôleur:

Versandanzeigen-Nr.: 6565471
 Dispatch Note No.:
 No. d'avis d'expédition:

Produkt: NAHTLOSE STAHLROHRE
 Product: SEAMLESS STEEL TUBES
 Produit: TUBES D' ACIER SANS SOUDURE

Stahlschmelzungsverfahren: ELEKTROSTAHL
 Steelmaking process: ELECTRIC FURNACE
 Procédé d'élaboration de l'acier: FOUR ELECTRIQUE

Lieferbedingungen: Techdrill Specification No. TDA519-4130 rev. C with agreed deviations: Without pt. 3, tensile strength, protection, NDT, ASTM-A 519-2006, NACE Standard MR0175-97
 Terms of delivery:

Maße - Toleranzen: Techdrill specification no. TDA519-4130 rev. B
 Dimensions-tolerances:

Stahlsorte: GRADE 4130
 Steel grade:

Nuance d'acier: QT
 Delivery condition:

Produktkennzeichnung: FS: BENTELER SIGN 4130 DIMENSION HEAT-NO.
 Product marking:

Marquage du produit:

ABZ = Ätzintensivbeschriftung, Etching ink marking, Givres à l'encre . FK = Farbkennzeichnung, colour marking, marquage par couleur . FS = Farbschublenierung, paint stencilling, marquage par peinture . FSD = Farbstahlstrahldrucker, Colour jet printer, Imprimante à jet d'encre de couleur . LK = Laserkennzeichnung, Laser marking, Marquage laser . PKE = Etikettenkennzeichnung, tag marking, marquage sur étiquette . PS = Prägestempel, die stamp, marquage par poinçonnage . TS = Tintenstrahlkennzeichnung, ink jet spray marking, Imprimante à jet d'encre .

Pos.	Stück	Maße	Länge	Gewicht	Schmelzen-Nr.	Prüfdruck	Rohr-Nr.-Gruppe	Vielfachlängen
Item	Number	Dimensions	Length	Weight	Heat No.	Test pressure	Tube number group	Multiple lengths
Poste	Nombre	Dimensions	Longueur	Poids	No. de coulée	Pression d'épreuve	Série de no. des tubes	Longueurs multiples
0010	22	60,330 X 11,070 mm	5000 - 7000	134,29	1811	571644		

ITEM 49

ENTRY/CERT. X7008

ABNAHMEPRÜFZEUGNIS EN 10204-3.1
 INSPECTION CERTIFICATE EN 10204-3.1
 CERTIFICAT DE RECEPTION EN 10204-3.1

Dokument-Nr.: 65-706733/001/P
 Document No.:
 No. du document:

Prüf-Nr.:
 Inspection No.:
 No. du certificat:

Blatt: 2 / 3
 Page:

Schmelzenanalyse [%] / Heat analysis [%] / Analyse sur coulée [%]

Pos.	Schmelzen-Nr.	C	SI	MN	P	S	CR	MO
Item	Heat No.							
Poste	No. de coulée							
0010	571644	0,310	0,240	0,54	0,006	0,001	0,93	0,17

Prüfergebnisse / Test results / Résultats des essais

Die Rohre wurden zerstörungsfrei geprüft: UT-long.imperfections: acc. to API 5L 42nd Edition, SR 4.3; Outside notch depth: 12,5
 The tubes are non destructive tested: %; Inside notch depth: 12,5 %
 Les tubes ont passé un essai non destructif:

Augensichtkontrolle: PASSED Maßkontrolle: PASSED
 Visual inspection: Dimensions examination:
 Examen visuel: Vérification des dimensions:

Ergebnisse der mechanischen Prüfung / Results of mechanical testing / Résultats des essais mécaniques

Die Probenahme erfolgte an Vielfachlängen.
 The sampling was carried out on multiple lengths.
 L'échantillonnage était réalisé aux longueurs multiples.

Zugversuch längs Streifenprobe / Tensile test longitudinal Strip test specimen / Essai de traction longitudinale Bande decoupee sur tube

Pos.	Proben-Nr.	Schmelzen-Nr.	Probenabmessung	Streckgrenze	Zugfestigkeit	Dehnung	Einschnürung
Item	Specimen No.	Heat No.	Specimen dimensions	Yield strength	Tensile strength	Elongation	Area reduction
Poste	No. de l'éprouvette	No. de coulée	Dimensions de l'éprouv.	Limite élastique	Résistance à la traction	Allongement	Coefficient de striction
0010	000002	571644	19,00 X 10,70	645	745	32	72,00

Anforderungen
 Requirements
 Exigences

ReH	Rm	A2"	1. Formula
N/mm²	N/mm²	%	1. Formula
MIN 586	MIN 690	MIN 18	1. Formule
			MIN 35

Härteprüfung / Hardness test / Essai de dureté

Pos.	Proben-Nr.	Schmelzen-Nr.	Härte
Item	Specimen No.	Heat No.	Hardness
Poste	No. de l'éprouv.	No. de coulée	Dureté
0010	000002	571644	020

Anforderungen
 Requirements
 Exigences

HRC	HB	HV	HRB	HBW
MAX 22,0				

ITEM 49

Benteler Steel/Tube GmbH
 Postfach 13 40
 33043 Paderborn
 Deutschland
 Tel.: + 49.5254.81-0 Fax: + 49.5254.13666

BENTELER
 Steel/Tube

ABNAHMEPRÜFZEUGNIS EN 10204-3.1
 INSPECTION CERTIFICATE EN 10204-3.1
 CERTIFICAT DE RECEPTION EN 10204-3.1

Dokument-Nr.: 65-706733/001/P
 Document No.:
 No. du document:

Prüf-Nr.:
 Inspection No.:
 No. du certificat:

Blatt: 3 / 3
 Page:
 Page:

Kerbschlagbiegeversuch Notched bar impact test / Essai de flexion par choc (résilience) [1 CHARPY_V]

Pos. Item	Proben-Nr. Specimen No.	Schmelzen-Nr. Heat No.	Probenabmessung Specimen dimensions			Probenlage Specimen position	Prüftemperatur Test temperature	Kerbschlagarbeit Absorbed energy		Kerbschlagzähigkeit Impact strength		Laterale Breitung Lateral expansion
Poste No. de l'éprouv.	No. de coulée		Dimensions de l'éprouvette			Position de l'éprouvette	Température d'essai	Energie absorbée		Résistance au choc		Expansion latéral
Anforderungen Requirements Exigences			Länge Length Longueur	Breite Width Largeur	Höhe Height Hauteur	längs (L) longitudinal (L) longitudinal (L)	GRAD °C	einzel single individuelle	mittel average moyenne	einzel single individuelle	mittel average moyenne	mm
			mm	mm	mm	quer (Q) transversal (Q) transversal (Q)		J	J	J/cm²	J/cm²	min.
0010	000004	571644	55	7,50	10,00	L	-50	MIN 024	MIN 032			0,38
				7,50	10,00			123				
				7,50	10,00			126				1,80
				7,50	10,00			130	126			

Wärmebehandlung / Heat treatment / Traitement thermique

Hardening temperature: 850°C, Holding time: 1 min, Cooling: water / Tempering temperature: 740°C, Holding time: 3 min, Cooling: air

Verkäufer(in) / Salesman/ woman in charge / Personne chargée : Mr Pospischil, Tel.: 05254/81-4348, Fax: 4394

NOV 2012

ITEM 49

Dinslaken, 25.07.2012, TEL.: 02064.623-537 FAX: 02064.623-539

Abnahmebeauftragter
 Inspection representative
 Contrôleur
 DR. BASEL KEITA / Thei

Es wird bestätigt, daß die gelieferten Erzeugnisse den techn. Lieferbedingungen des Auftrages entsprechen. Dieses Dokument wurde mittels EDV erstellt und ist ohne Unterschrift rechtsgültig.
 We certify that the supplied products comply with the order specification. This document was prepared by means of electronic data processing and is valid without signature.
 Nous attestons que les produits livrés sont conformes aux stipulations de la commande. Ce document a été établi par traitement électronique de l'information et est valide sans signature.

14.3 Attachment 3 – 48.08 electrode



C03

OK 48.08

SMAW

OK 48.08 is an LMA electrode with very good mechanical properties suitable for demanding applications, such as offshore. The weld metal contains approximately 1% Ni for high impact values down to -40°C. The coating is of the latest LMA type for optimum resistance to porosity and hydrogen cracking. OK 48.08 is CTOD tested.

Recovery

125%

Welding Current

AC, DC + (-) OCV 65 V



PACKING/ORDERING INFORMATION				
Part Number	Dia (mm)	Inner Carton (kg)	Carton Weight (kg)	Pallet Weight (kg)
VacPac				
4808253NV0	2.5	2.9	11.6	580.0
480832HNV0	3.2	3.5	14.0	700.0
480840HNV0	4.0	3.7	14.8	740.0
480850HNV0	5.0	4.0	16.0	800.0

CLASSIFICATIONS	APPROVALS	TYPICAL ALL WELD METAL COMPOSITION (%)		TYPICAL MECH. PROPERTIES ALL WELD METAL	
<u>SFA/AWS A5.5</u>	ABS 3H5 3Y H5	C	0.06	<u>Yield Stress, Mpa</u>	
E7018-G	DNV 4 Y40H10	Si	0.4	540	
<u>EN ISO 2560-A</u>	GL 4YH5	Mn	1.2	<u>Tensile Strength, Mpa</u>	
E 46 5 Ni B 32 H5	LR 4Y40M H5	Ni	0.8	600	
				<u>Elongation, %</u>	
				26	
				<u>Charpy V</u>	
				Test Temps, °C	Impact Values, J
				-20	160
				-40	130
				-50	100
				-60	60

WELDING PARAMETERS							
Diameter (mm)	Length (mm)	Welding Current, A	Arc Voltage, V	N. Kg Weld Metal/(kg) Electrodes	B. No. Of Electrodes/(kg) Weld Metal	H. Kg Weld Metal/(hour) Arc Time	T. Burn-off time, (secs)/ Electrode
2.0	300	55-80	22	0.57	135.1	0.6	42
2.5	350	75-110	27	0.57	88.2	1.0	41
3.2	350	110-150	22	0.62	42.3	1.3	66
3.2	450	110-150	22	0.66	30.0	1.4	85
4.0	350	150-200	22	0.66	26.5	2.0	68
4.0	450	150-200	22	0.69	20.3	2.0	90
5.0	450	190-275	23	0.69	14.0	3.0	85
6.0	450	220-360	26	0.66	10.0	3.8	95



TEL : +009-507 4002 FAX : +009-500 4450
EMAIL : sales@netlite.com.my

14.4 Attachment 4 – WPS for API 5L52 pipe



DET NORSKE VERITAS WELDING PROCEDURE QUALIFICATION RECORD

According to

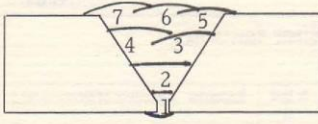


DNV's ref. : KRS 280

NORSOK M-601

Manufacturer's welding procedure No.

(Standard / code)

WPQ 003-2006

Manufacturer : Herman Hansen Mek. Verksted		Place and date: Kristiansand 2006-01-10															
Purchaser's spec. No. : -		Project : -															
Requirements beyond standard / code : EN-ISO 15614-1																	
Joint type, preparation, dimensions and welding sequence (Sketch). State rolling direction, if applicable Groove angle 60° Root gap 2-4 mm Root face 0-1 mm		Material spec/grade(s): API 5L X 52 Delivery condition(s): Normalized Material group(s) acc. to standard: 1.2															
		<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th>Grade</th> <th>C %</th> <th>C eq%</th> <th>to</th> <th>Grade</th> <th>C %</th> <th>C eq%</th> </tr> <tr> <td>API 5LX52</td> <td>0,13</td> <td>0,41</td> <td></td> <td>API5L X52</td> <td>0,13</td> <td>0,41</td> </tr> </table>		Grade	C %	C eq%	to	Grade	C %	C eq%	API 5LX52	0,13	0,41		API5L X52	0,13	0,41
Grade	C %	C eq%	to	Grade	C %	C eq%											
API 5LX52	0,13	0,41		API5L X52	0,13	0,41											
If applicable, tick off type of C eq (ladle analysis) : <input checked="" type="checkbox"/> $C_{eq} = C + \frac{Mn}{6} + \frac{Cr}{5} + \frac{Mo}{5} + \frac{V}{15} + \frac{Cu}{15} + \frac{Ni}{15}$ (%) <input type="checkbox"/> $C_{eq} (P cm) = C + \frac{Si}{30} + \frac{Mn}{20} + \frac{Cu}{60} + \frac{Cr}{15} + \frac{Ni}{15} + \frac{Mo}{10} + \frac{V}{10} + 5B$ (%)																	
Diameter and/or thickness, mm : Ø168,3 x 12		Welders name/id. of test piece: Kurt Fredriksen/DNV.KRS.WPQ003-200															
Welding process(es) : 111		Welding position : H-LO45															
Single/double sided welding : ss																	
WELDING CONSUMABLES																	
Index	Consumable(s) trade name and manufacturer		Classification														
A	ESAB OK 48.08		AWS A5.1 E7018-G														
B																	
C																	
WELDING PARAMETERS																	
Pass No.	Index	Diam. mm	Gas shielding composition	Gas l/min.	Current, Polarity	Amps A	Voltage V	Travel speed mm/min.	Wire feed m/min.	Run-out length mm	Heat input kJ/mm						
1	A	2,5	-	-	DC-	90	20	142			0,76						
2	A	3,25	-	-	DC+	120	23	115			1,44						
3-4	A	3,25	-	-	DC+	115	22	150			1,01						
5-7	A	3,25	-	-	DC+	115	22	135			1,12						
OTHER INFO (Groove prep., backing, weaving, classification of gas EN 439, W-electrode size and type EN 26848, pulse details etc.)																	
Groove preparation: Machine bevelling/grinding. Max weaving 5mm																	
SPECIAL REQUIREMENTS																	
Preheat min.°C	20	Interpass max°C	250														
Drying of consumables °C and h(rs)		Soaking h(rs)															
PWHT/Ageing °C		Heating/cooling rate °C/h															
Procedure																	
Others:																	
EXTENT OF QUALIFICATION		Material group(s)	1.2 / 1.1														
		Diam. mm	>84														
		Thickn. mm	3 to 24														
Fillet weld throat thickness, mm		All multirun	Positions All														
Other limitations		Heat input +/- 25%															
We certify that the statements in this record are correct and that the test piece was prepared, welded and heat treated in accordance with above specified Code/Standard and/or Purchaser's requirements.																	
Manufacturers signature and stamp		DNV survey station Kristiansand															
 Herman Hansen Mek. Verksted A/S Etablert 1837		 Surveyors signature Ole Jørgen Haugen															

14.5 Attachment 5 – Heat input data

Heat input data for weld with 120 ° preheat.

Run Number	Process	Process thermal efficiency	Current (A)	Voltage (V)	Weld length	Weld length units	Weld length (mm)	Welding time	Welding time units	Welding time (s)	Travel speed (mm/s)	EN heat input (kj/mm)
1	Shielded metal arc welding (SMAW)	0,8	80	22	8	mm	8	19	seconds	19	0,42	3,34
1	Shielded metal arc welding (SMAW)	0,8	80	22	8	mm	8	19	seconds	19	0,42	3,34
1	Shielded metal arc welding (SMAW)	0,8	80	22	8	mm	8	18	seconds	18	0,44	3,17
1	Shielded metal arc welding (SMAW)	0,8	80	22	6	mm	7	16	seconds	16	0,44	3,22
1	Shielded metal arc welding (SMAW)	0,8	80	22	6	mm	7	14	seconds	14	0,50	2,82
2	Shielded metal arc welding (SMAW)	0,8	80	22	8	mm	8	18	seconds	18	0,44	3,17
2	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	7	14	seconds	14	0,50	2,82
2	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	17	seconds	17	0,47	2,99
2	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	7	14	seconds	14	0,50	2,82
2	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	22	seconds	22	0,41	3,44
3	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	20	seconds	20	0,45	3,13
3	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	17	seconds	17	0,53	2,66
3	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	18	seconds	18	0,50	2,82
3	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	16	seconds	16	0,56	2,50
3	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	6	12	seconds	12	0,50	2,82
4	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	22	seconds	22	0,41	3,44
4	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	27	seconds	27	0,33	4,22
4	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	23	seconds	23	0,39	3,60
4	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	18	seconds	18	0,44	3,17
4	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	20	seconds	20	0,45	3,13
5	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	17	seconds	17	0,47	2,99
5	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	18	seconds	18	0,50	2,82
5	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	16	seconds	16	0,50	2,82
5	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	19	seconds	19	0,47	2,97
5	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	18	seconds	18	0,44	3,17
6	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	7	15	seconds	15	0,47	3,02
6	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	7	14	seconds	14	0,50	2,82
6	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	17	seconds	17	0,47	2,99
6	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	9	16	seconds	16	0,56	2,50
6	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	6	12	seconds	12	0,50	2,82
7	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	16	seconds	16	0,50	2,82
7	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	17	seconds	17	0,47	2,99
7	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	17	seconds	17	0,47	2,99
7	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	16	seconds	16	0,50	2,82
7	Shielded metal arc welding (SMAW)	0,8	80	22	9	mm	8	15	seconds	15	0,53	2,64

14.6 Attachment 6 – Hardness measurements

Part 1

Comparison of material hardness with and without preheating

Sample 2 No preheat					
Without preheat					
Base	HAZ	Weld		HAZ	Base
242	303	210	222	316	242
	302			347	
236	266	208		286	242
	242			235	
234	230	206	203	220	233

Sample 2.1 Preheat 120 ° C					
Preheat 120 ° C					
Base	HAZ	Weld		HAZ	Base
244	336	229	234	274	246
	349			273	
245	294	218		253	245
	230			235	
240	232	220	210	226	231

Sample 3 No preheat					
Without preheat					
Base	HAZ	Weld		HAZ	Base
247	338	217	229	201	251
	346			298	
250	255	197		238	248
	240			220	
245	236	192	189	218	239

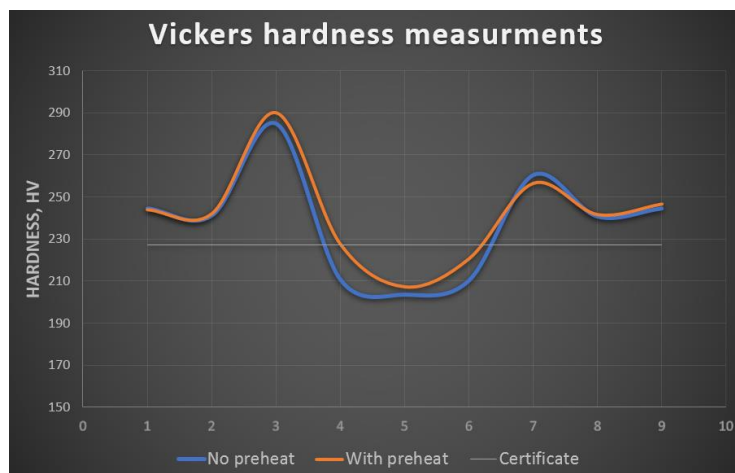
Sample 3.1 Preheat 120 ° C					
Preheat 120 ° C					
Base	HAZ	Weld		HAZ	Base
247	324	222	211	290	244
	333			274	
243	281	203		267	247
	270			234	
235	227	223	219	227	244

Sample 4 No preheat					
Without preheat					
Base	HAZ	Weld		HAZ	Base
244	375	246	222	308	242
	390			287	
247	299	205		285	243
	230			223	
234	217	192	195	220	236

Sample 4.1 Preheat 120 ° C					
Preheat 120 ° C					
Base	HAZ	Weld		HAZ	Base
250	345	234	213	270	245
	345			287	
244	307	201		271	248
	245			230	
236	233	238	237	237	240

Graphical representation of preheating to 120 °C.

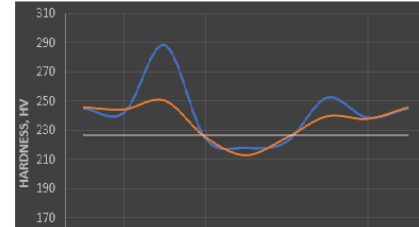
Average values from all three samples was used to generate the graph.



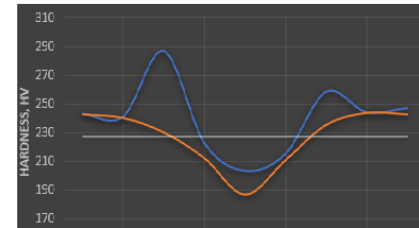
Part 2

Comparison of material hardness after different PWHT temperatures.

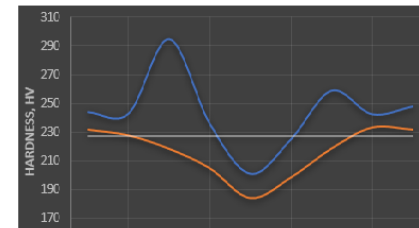
Sample 2.1 Preheat 120 ° C PWHT 650 ° C for 20 min										
Before PWHT						After PWHT				
Base	HAZ	Weld		HAZ	Base	Base	HAZ	Weld	HAZ	Base
244	336	229	234	274	246	245	271	239	238	273
	349			273			277			242
245	294	218		253	245	246	245	213		234
	230			235			229			221
240	232	220	210	226	231	244	231	212	212	229
										227



Sample 3.1 Preheat 120 ° C PWHT 650 ° C for 120 min										
Before PWHT						After PWHT				
Base	HAZ	Weld		HAZ	Base	Base	HAZ	Weld	HAZ	Base
247	324	222	211	290	244	243	249	215	206	253
	333			274			245			250
243	281	203		267	247	243	220	187		226
	270			234			226			222
235	227	223	219	227	244	238	212	210	216	227
										243



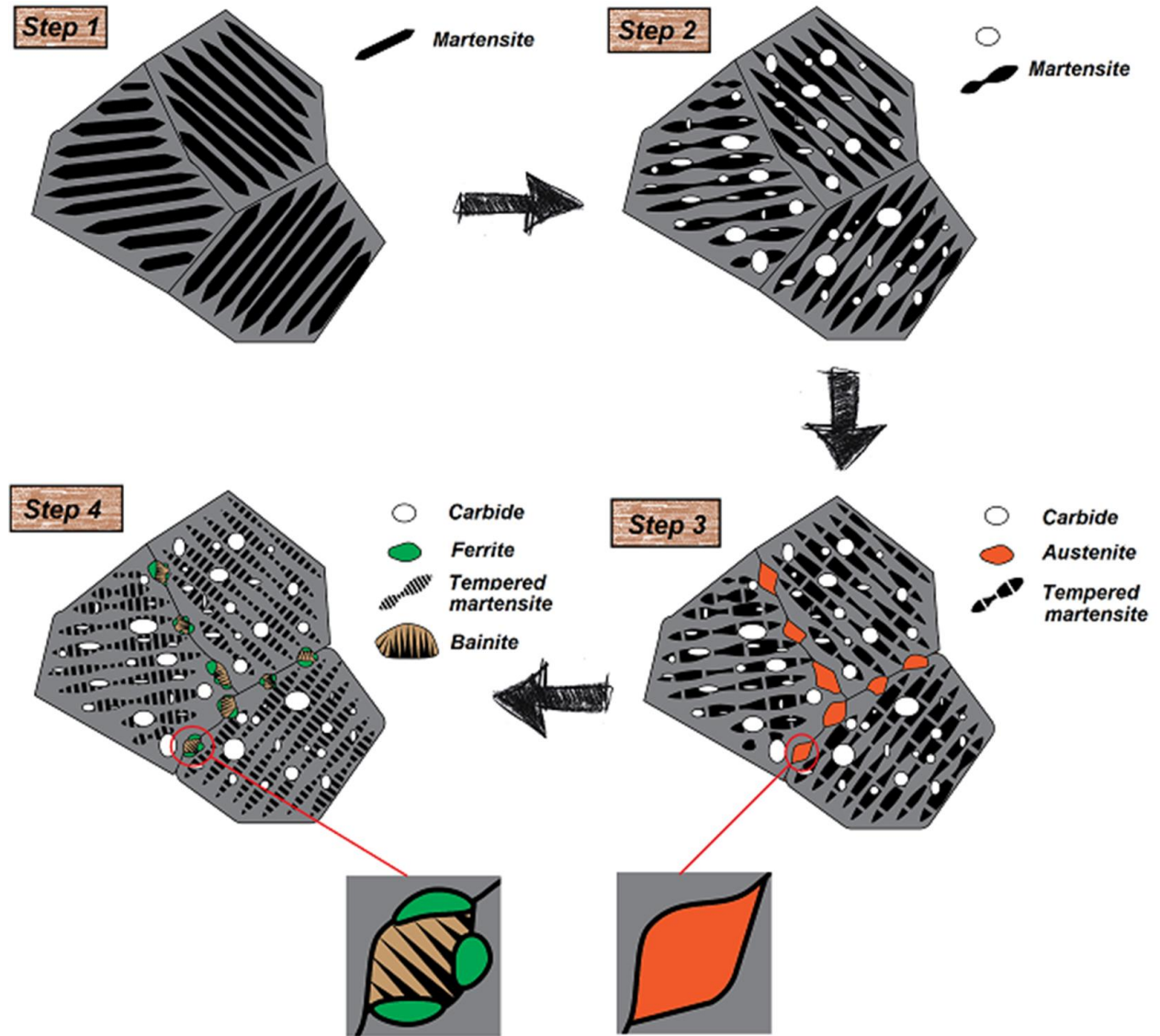
Sample 4.1 Preheat 120 ° C PWHT 705 ° C for 20 min										
Before PWHT						After PWHT				
Base	HAZ	Weld		HAZ	Base	Base	HAZ	Weld	HAZ	Base
250	345	234	213	270	245	230	235	216	202	248
	345			287			230			213
244	307	201		271	248	232	217	184		216
	245			230			209			208
236	233	238	237	237	240	226	202	194	195	209
										231



14.7 Attachment 7 – Illustrations

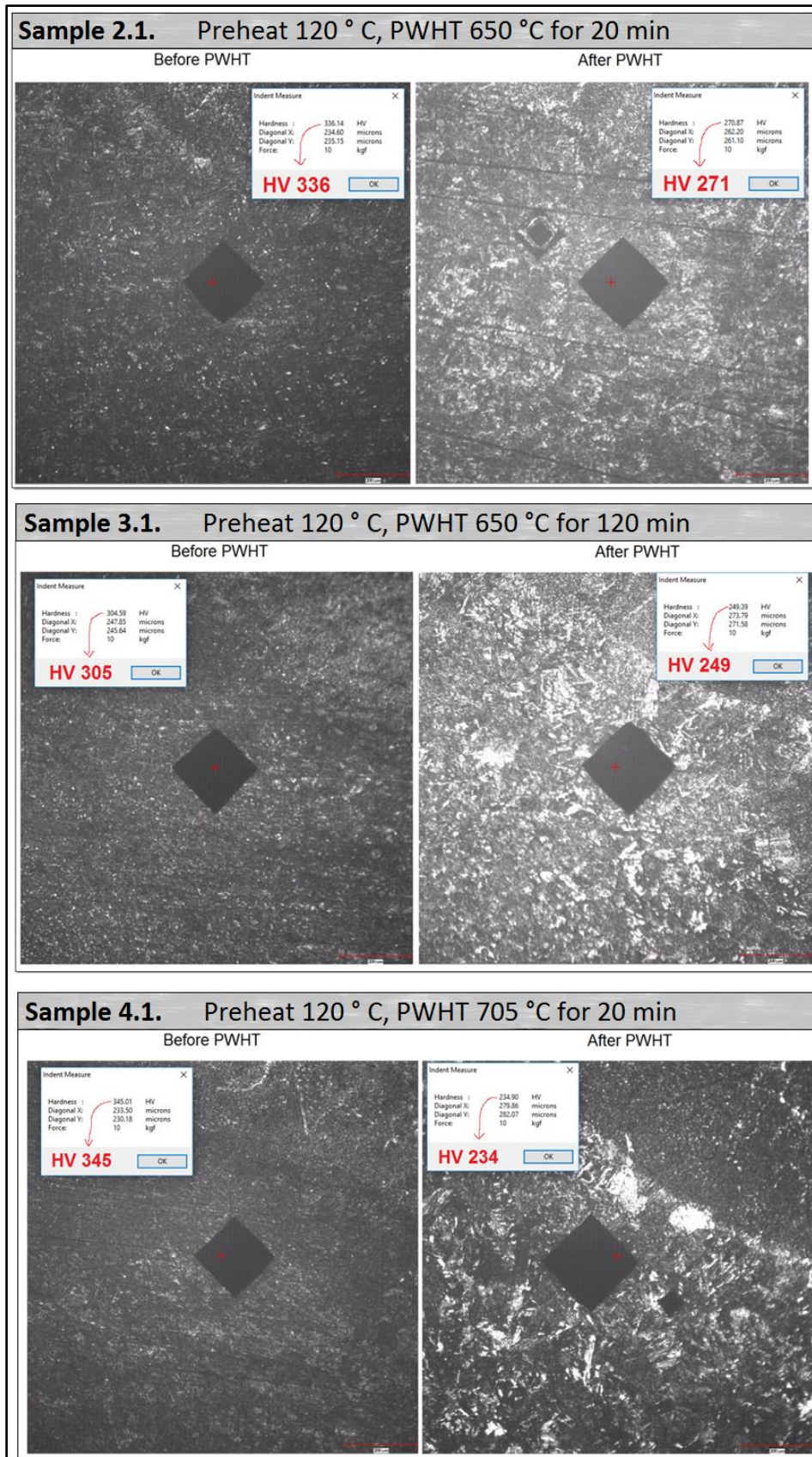
Illustration 1

Expected changes in microstructure of AISI 4130 when heat treated in accordance with attached certificate.



14.8 Attachment 7 – Hardness measurements

Hardness measurements at impression point number 2, see ISO 15156-2

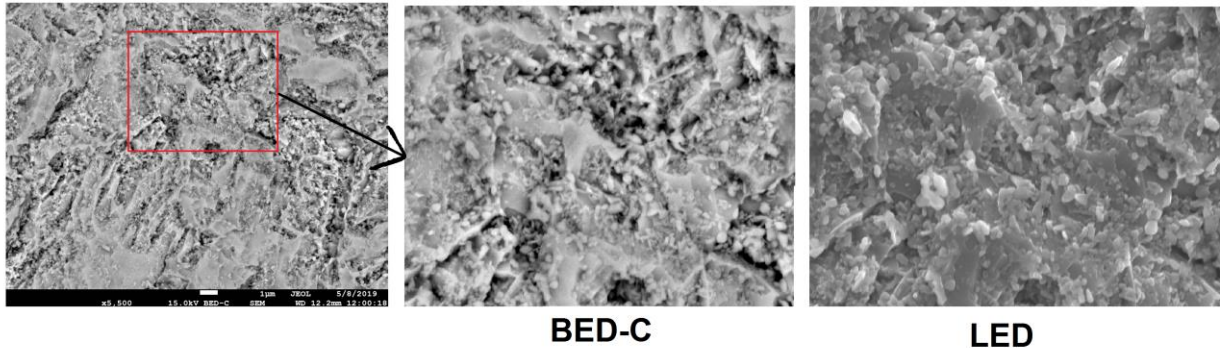


14.9 Attachment 9 – Microscopy

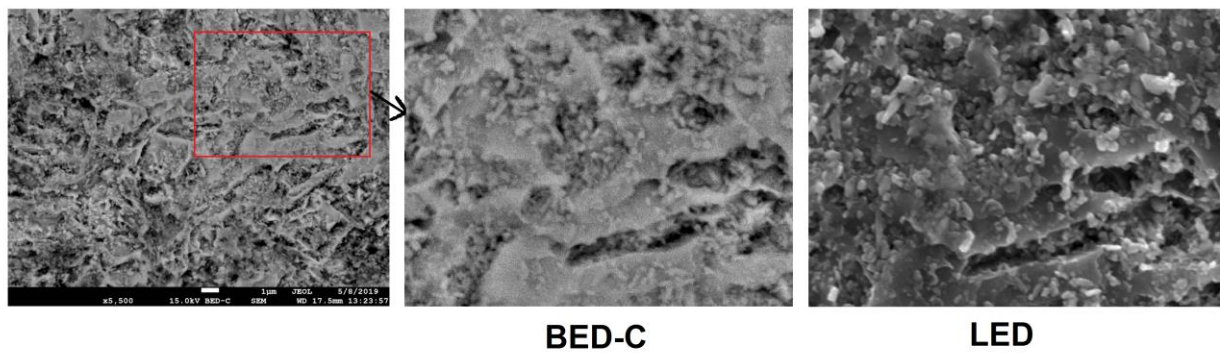
Picture 1.

Formation of carbides in base material of welded AISI 4130 joint

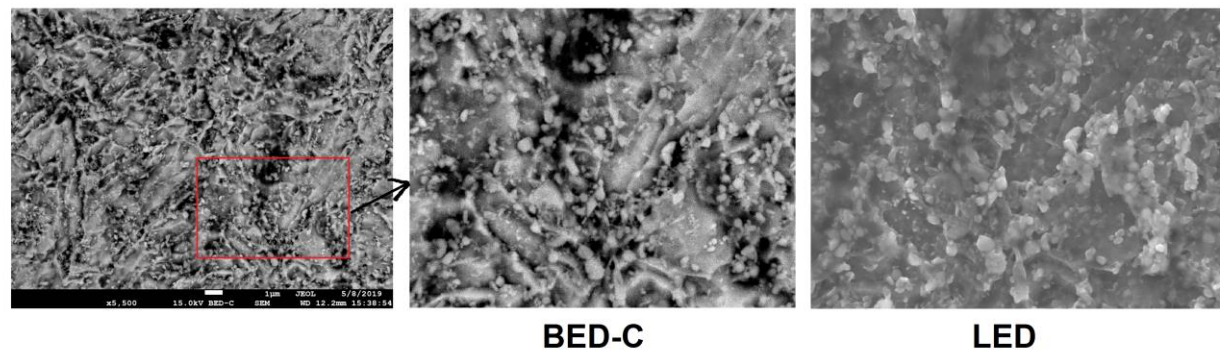
Sample 1.1. No PWHT



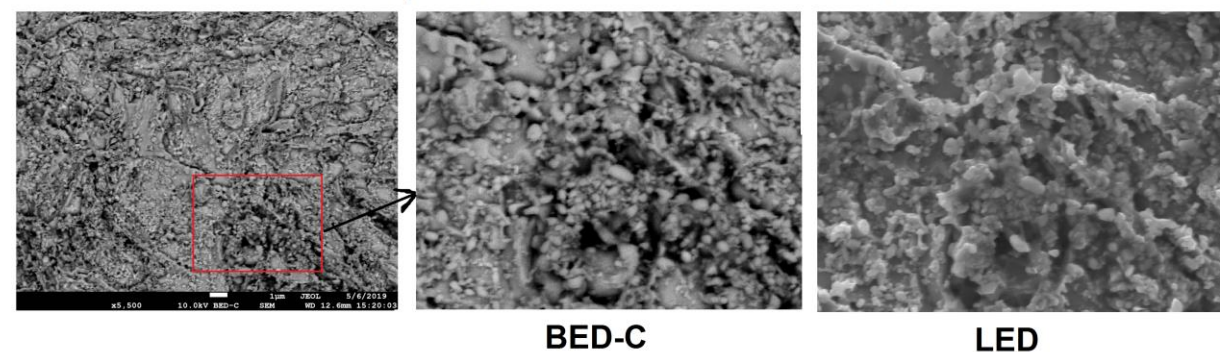
Sample 2.1. PWHT at 650 °C for 20 min



Sample 3.1. PWHT at 650 °C for 120 min



Sample 4.1. PWHT at 705 °C for 20 min



14.10 Attachment 10 – Piping specification



Title:	Piping Class Sheet	Doc. No.	MHSTD-L-ZZ50-SA17-0001	Appendix:	1	Class	KX10MH
Project:	MHWirth Standard Piping Specification	Company:	MHWirth	Rev. Date:	25.09.18	Rev.:	05

Max Design Pressure at Temperature (See note 02)						API Pressure rating	7 500 psi
Pressure (Barg)	517.0	517.0	517.0	517.0	517.0	-	-
Temperature (Deg. C)	-29	38	50	80	90	Service Code	Chap. 4.4

Design Code	ASME B31.3 Chapter IX High Pressure Piping (See note 52)
Corr. Allow. (mm)	3.0
Wall Thickness Under Tolerance	12.5 %, (See note 01)
Longitudinal Weld Efficiency	1
PWHT	Yes
NDT Class	3
Painting	MHSTD-Z-ZZ50-SA16-0001, SYSTEM No. 1 (See note 49)

ND (inch)	2	3	4	5	6
OD (mm)	60.30	88.90	114.30	141.30	168.30
ID (mm)	38.16	58.42	80.06	103.20	124.40
THK (mm)	11.07	15.24	17.12	19.05	21.95
SCH	-	-	-	-	-
WEIGHT	XXS	XXS	XXS	XXS	XXS

ITEM TYPE	RANGE FROM TO (inch)		GEOMETRIC STANDARD	EDS/ VDS	END CONN #1 #2		MATERIAL DESCR.	MDS	RATING	SCHD.	NOTES
PIPE SMLS	2	6	ASME B36.10M		BE	BE	A519 AISI 4130	X21			
MANIFOLD FITTINGS	2	6	MANUFACTURERS STANDARD	HMF1	BE	BE	API 6A 75K AISI 4130	X24			03
CAP	2	6	ASME B16.9		BE		A234 AISI 4130	X21			
CONCENTRIC REDUCER	2	6	ASME B16.9		BE	BE	A234 AISI 4130	X21			
ECCENTRIC REDUCER	2	6	ASME B16.9		BE	BE	A234 AISI 4130	X21			
ELBOW LONG RADIUS 45 DEG	2	6	ASME B16.9		BE	BE	A234 AISI 4130	X21			03
ELBOW LONG RADIUS 90 DEG	2	6	ASME B16.9		BE	BE	A234 AISI 4130	X21			03
STRAIGHT TEE	2	6	ASME B16.9		BE	BE	A234 AISI 4130	X21			03
REDUCING TEE	2	6	ASME B16.9		BE	BE	A234 AISI 4130	X21			03
FLANGE BLIND	2	6	API 6A TYPE 6BX		RJ		API 6A 75K AISI 4130	X24	10000psi		17
FLANGE WN	2	6	API 6A TYPE 6BX		RJ	BE	API 6A 75K AISI 4130	X24	10000psi		15, 17
SPADE	2	6	API 6A TYPE 6BX		RJ	RJ	API 6A 75K AISI 4130	X24	10000psi		17
SPACER	2	6	API 6A TYPE 6BX		RJ	RJ	API 6A 75K AISI 4130	X24	10000psi		15, 17
GASKET	2	6	API 6A TYPE BX RING				316SS HB83 PRESS. ENERGIZED				
MECHANICAL JOINT BLIND HUB	2	6	GRAYLOC TYPE	NMJ1			API 6A 75K AISI 4130	X24			
MECHANICAL JOINT HUB	2	6	GRAYLOC TYPE	NMJ1	BE		API 6A 75K AISI 4130	X24			
MECHANICAL JOINT CLAMP	2	6	GRAYLOC TYPE	NMJ1			A788 AISI 4130	X02			



ITEM TYPE	RANGE FROM TO (inch)		GEOMETRIC STANDARD	EDS/ VDS	END CONN #1 #2		MATERIAL DESCR.	MDS	RATING	SCHD.	NOTES
MECHANICAL JOINT SEAL RING	2	6	GRAYLOC TYPE	NMJ1			AISI 4140 PTFE COAT				
HAMMERLUG UNION	2	4	WECO FIG.1502 SOUR SERVICE		BE	BE	API 6A 75K AISI 4130	X24			
HAMMERLUG UNION	3	4	WECO FIG 1002 SOUR SERVICE		BE	BE	API 6A 75K AISI 4130	X24			
HAMMERLUG UNION	5	6	WECO FIG 1004 SOUR SERVICE		BE	BE	API 6A 75K AISI 4130	X24			
STUD BOLT C/W NUTS	2	6	API 6A				A193 B7/A194 2H				19, 21
MUD GATE VALVE	2	6	API 6A	GTKX1XJ	RJ	RJ	API 6A class E-E				17, 44, 103
MUD GATE VALVE	2	6		GTKX1XC	CL	CL	API 6A class E-E				44
CHECK VALVE SWING	2	6	API 6A	CHKX1XJ	RJ	RJ	API 6A class D-D				17, 44, 103
PLUG VALVE	2	4	WECO FIG.1502	PGKX1YX	FS	MS	API 6A class D-D				

PIPE BRANCH SPECIFICATION

		NOMINAL HEADER SIZE (IN)				
		6	5	4	3	2
NOM BRANCH SIZE	2		WRT	WRT	WRT	WT
	3	WRT	-	WRT	WT	
	4	WRT	WRT	WT		
	5	WRT	WT			
	6	WT				

WT = EQUAL LONG SWEEP FULL FLOW TEE BW
WRT = RED LONG SWEEP TEE BW

NOTES:

01	THE WALL THICKNESS TOLERANCE IS USED FOR DESIGN AND IS NOT PART OF THE PURCHASE SPECIFICATION WHEN DEVIATING FROM THE INDIVIDUAL PIPING ITEMS.
02	ALL WETTED PARTS SHALL BE RATED FOR SOUR SERVICE ACCORDING TO NACE MR0175.
03	IN EROSION SERVICE USE MANIFOLD FITTINGS WITH INCREASED WALL THICKNESS.
15	MACHINED TO MATCH INSIDE DIAMETER IN ACCORDANCE WITH API 6A (ISO 10423) 2"NB use 1 13/16" Flange with transition piece 3"NB use 2 9/16" Flange with transition piece 4"NB use 3 1/16" Flange with transition piece 5"NB use 4 1/16" Flange with transition piece 6"NB use 5 1/8" Flange with transition piece TRANSITION PIECE IS REQUIRED WHERE PIPE DIMENSIONS DOES NOT CORRESPOND TO API 6A REQUIREMENTS. ALTERNATIVELY FLANGE END CAN BE TAPERED TO MATCH PIPE SIZES, IN THAT CASE MANUFACTURER HAS TO DO DESIGN CALCULATIONS TO ENSURE THAT IT IS SUITABLE FOR THE RATED PRESSURE AND PROJECT HAVE TO ACCEPT THAT IT WILL NOT BE API 6A MONOGRAMMED
17	RTJ GROOVES SHALL HAVE ALLOY 625 OVERLAY.
19	WHERE NO MDS IS REFERRED TO, CERTIFICATION SHALL BE IN ACCORDANCE WITH "EN 10204 TYPE 2.1"
21	HOT DIPPED GALVANIZED ACCORDING TO ISO 10684 OR ASTM A153.
44	INSIDE DIAMETER MACHINED TO MATCH PIPE.
49	TO BE PAINTED TO AKER MH PAINT SPEC. MHSTD-Z-ZZ50-SA16-0001 SYSTEM NO.1 OR NORSOK M501 SYSTEM NO. 1.
52	DESIGN BASED ON CHAPTER IX AND APPENDIX K REQUIRES APPROVAL OF THE OWNER.
103	FLANGES ACCORDING TO ISO 10423 100psi (API SPECIFICATION 6A Type 6BX).