Production of large forged parts (steels, stainless steels and nickel-based superalloys)

Prof. Marcello Gelfi - Università degli Studi di Brescia

Agenda

- Introduction of Brescia University and Metallurgy Group.
- Large steel forgings production route.
- The effect of ingot internal cleanness and chemical segregations on the quality of forgings → case studies.
- The effect of heat treatments and forging parameters on the final microstructure and mechanical properties → case studies.
- Non-conventional application of PH heat treatment on 625 Ni-based superalloy forged bars for oil & gas field applications.
- Final discussion/questions.

Brescia - Lombardy (Italy)

- 200,000 inhabitants in Town, 1,100,000 in Province
- 40% of the manufacturing capabilities of Milan with 10% population
- Unique economy: 90.000 companies
- Lombardy: Top 3 GDP region in Europe with London and Paris

Brescia University

Metallurgy Group (DIMI)

- **Full professor**
  - Prof. Annalisa Pola
  - Prof. Marina La Vecchia
- **Associate professors**
  - Prof. Marcello Gelfi
  - Prof. Michela Faccoli
- **Researchers**
  - Dr. Giovanna Cornacchia

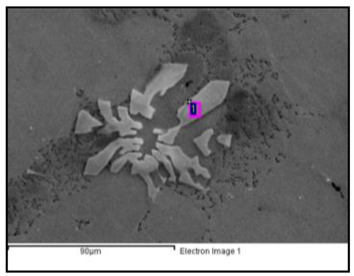
- **Research fellows**
  - Dr. Marialaura Tocci
  - M.Sc. Bojken Delibashi
  - M.Sc. Pietro Tonolini
- **Technicians**
  - Dr. Lorenzo Montesano
  - Mr. Alessandro Coffetti
- **External collaborators**
  - Prof. Roberto Roberti
  - Dr. Silvia Cecchel

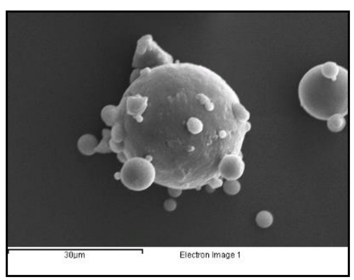
Group main activities

- **Microstructural** and **mechanical** characterization of metallic materials
- **Heat treatment** parameters optimization
- **Alloy composition** optimization
- Study of **innovative or non-conventional materials and technologies**
- **Additive Manufacturing**: characterization of powders and components
- **Failure analysis**

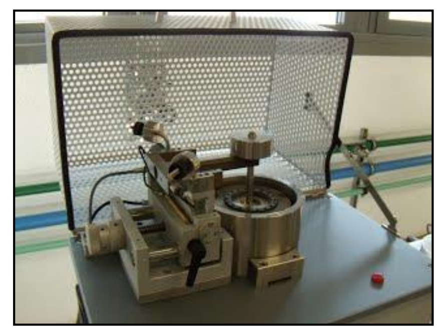
Metallurgy laboratories

Metallography





Mechanical and wear testing

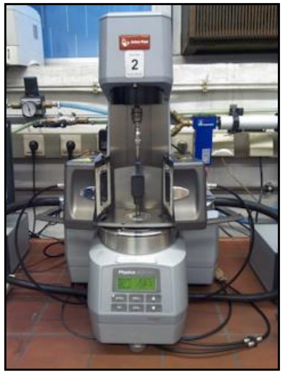


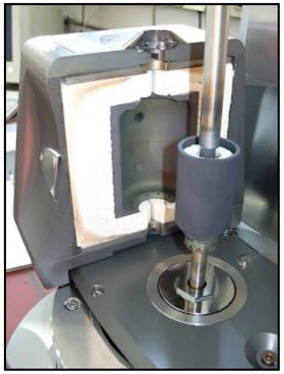
Metallurgy laboratories

Foundry process simulation

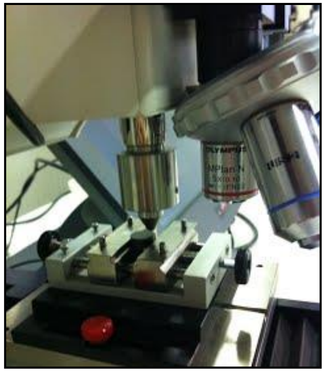
Heat treatments and foundry

Rheology of semisolid metals





Coatings characterization



Industry collaborations

Various forms of collaboration with companies:

- Third-Party analysis and testing
- Research projects
- Funding research fellowships
- Funding PhD projects (3 years)

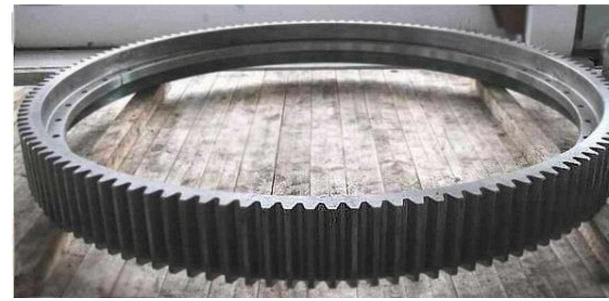
The Metallurgy group is involved in several of these activities (approx. 40-50 contracts/year).

Large steel forgings

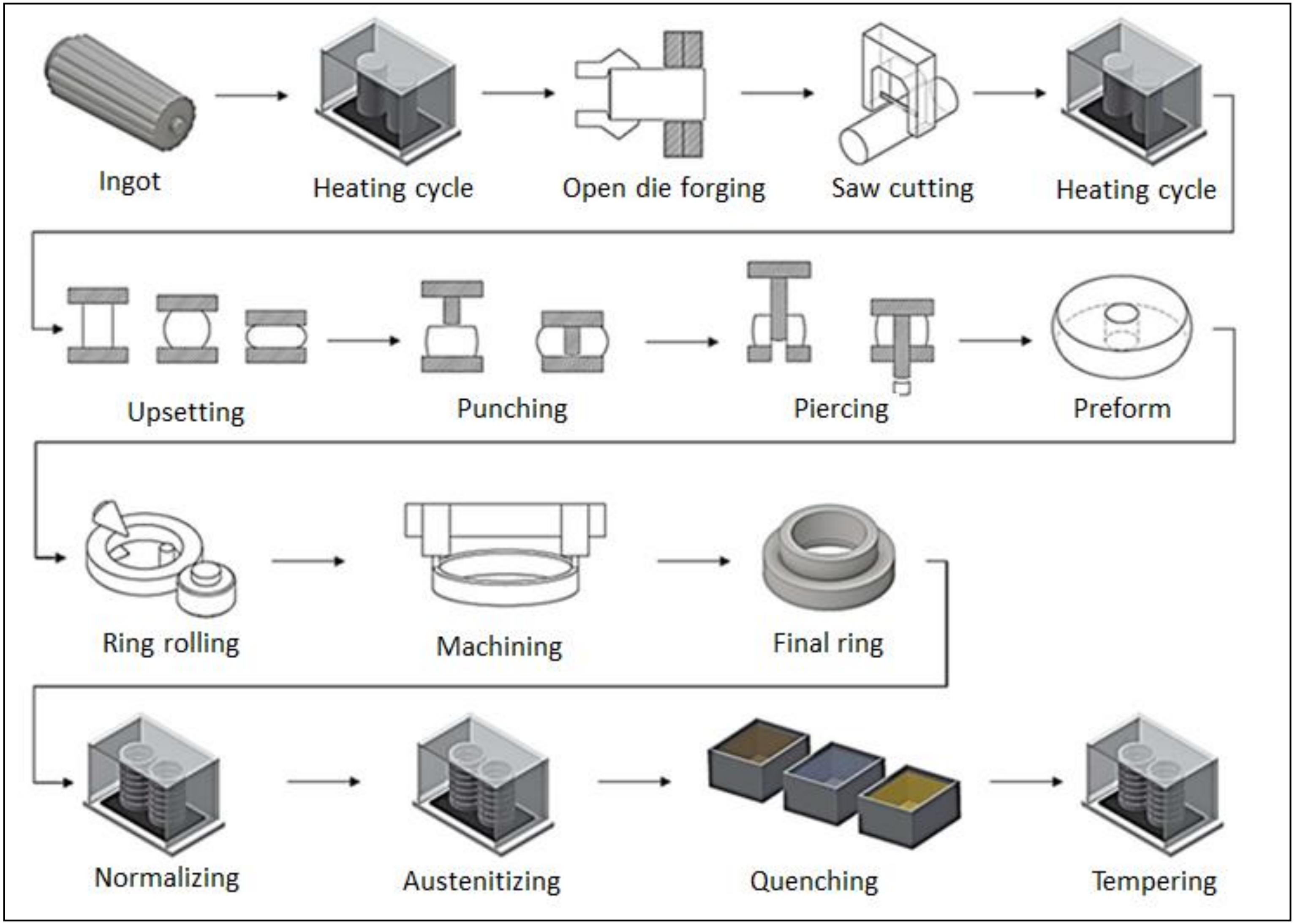
OIL & GAS INDUSTRY

AIRCRAFT & SHIP INDUSTRY

ENERGY PLANT



Large steel forgings

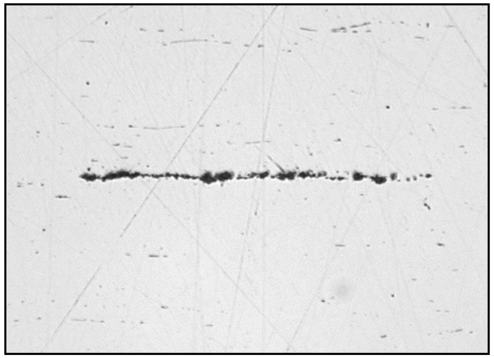


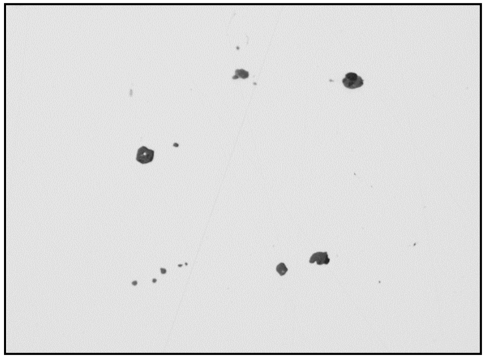
Ingot quality

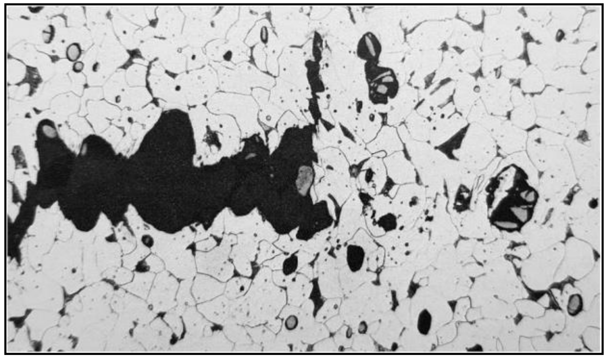
Two factors related to steel ingots can affect the forgings quality:

1. **Non-metallic macro-inclusions.**
2. **Micro- and macro-segregations.**

Non-metallic inclusions with size of few tens of microns are always present in steel (deoxidation products).

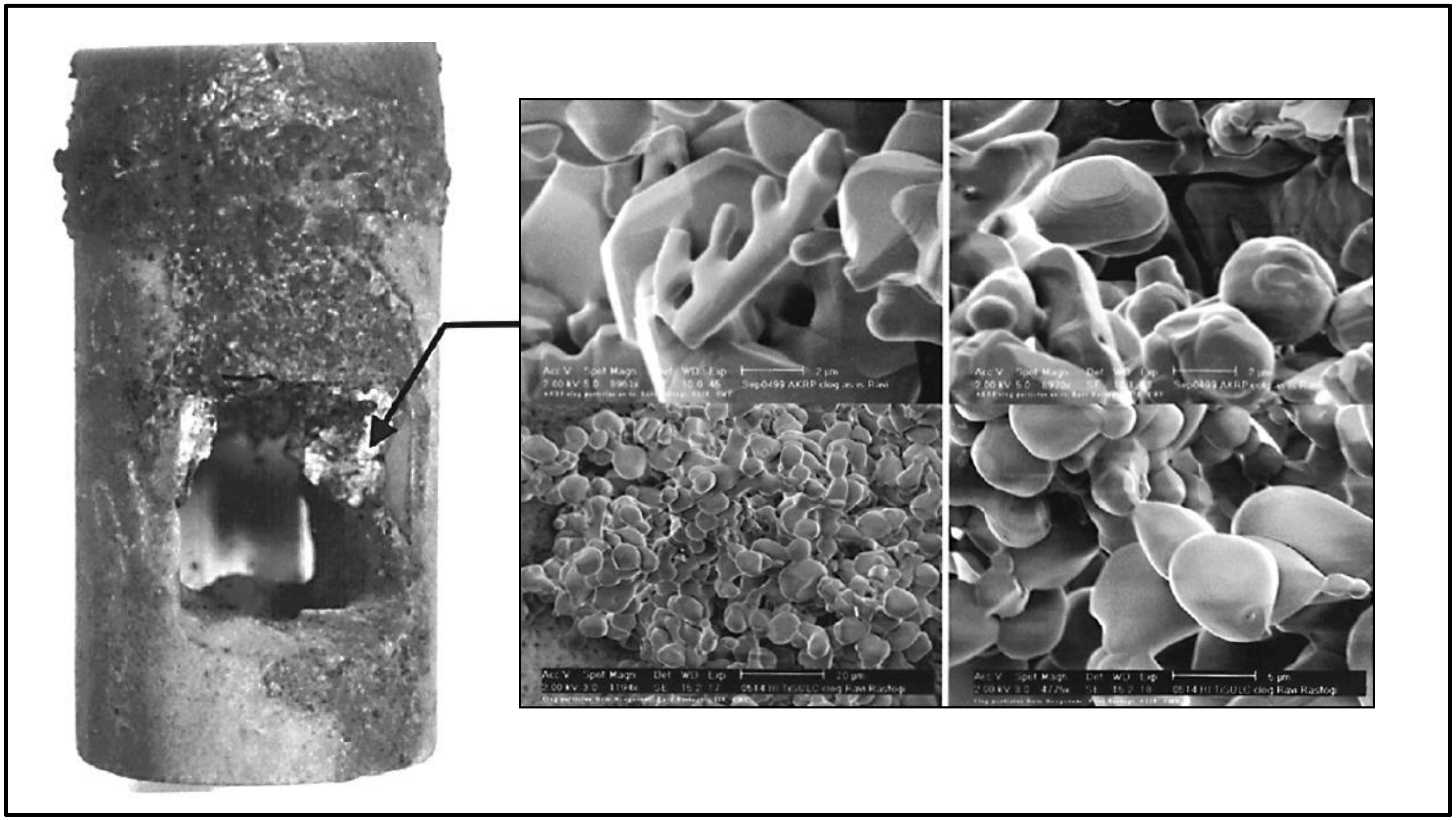






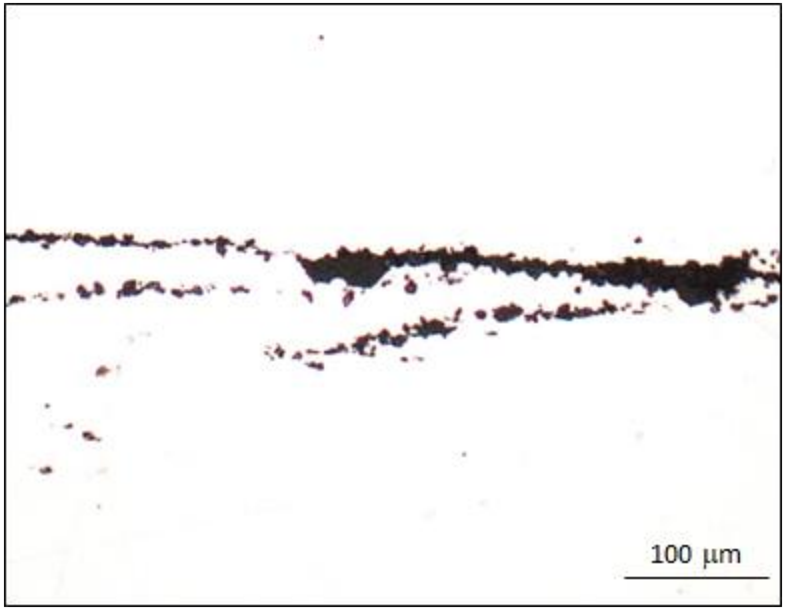
Ingot quality

Alumina-type inclusions remain in the liquid steel and tend to agglomerate during casting (e.g. at the nozzle exit) creating a problem, known as nozzle clogging.

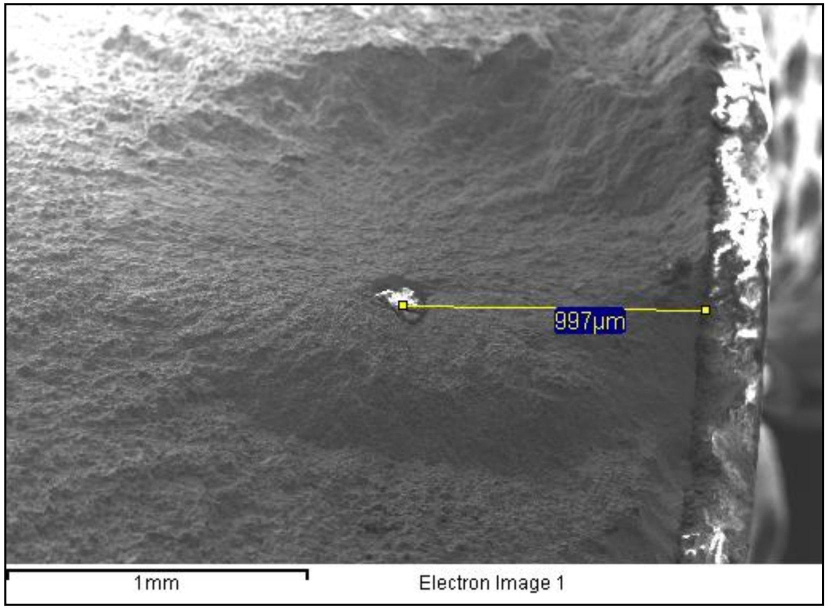


Ingot quality

An even more serious problem happens if these alumina-type agglomerates pass from the ladle to the mold, remaining entrapped in the solidified ingot → indigenous macro-inclusions.



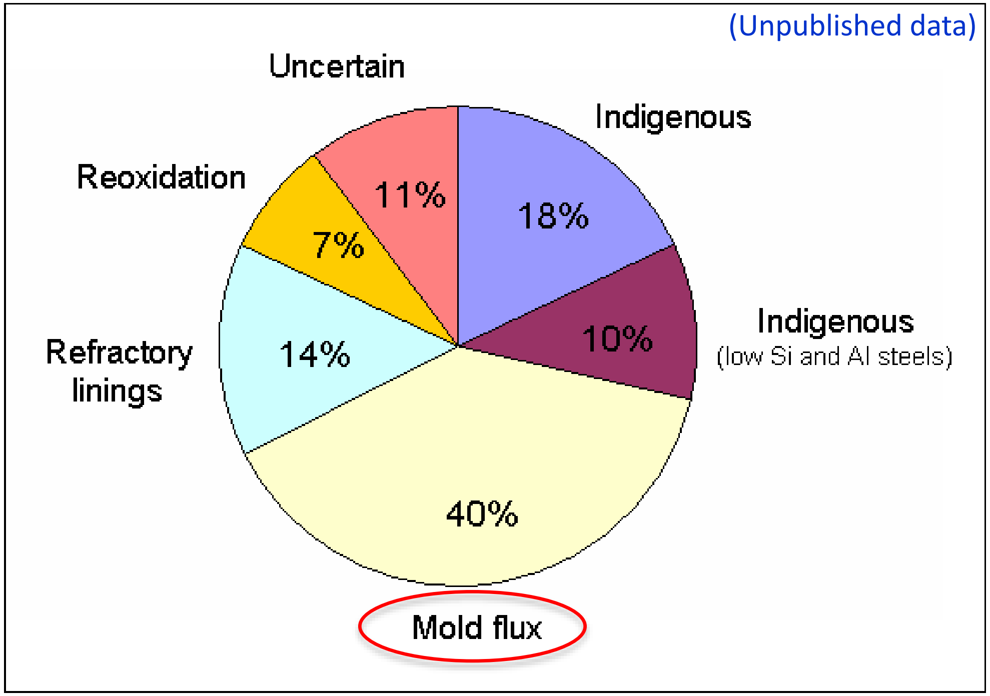
OM image of alumina-type agglomerates



SEM image of crack nucleated from alumina-type macro-inclusion

Ingot quality

Other types of macro-inclusions can come from external sources (e.g. refractories, mold flux,..) → exogenous macro-inclusions.

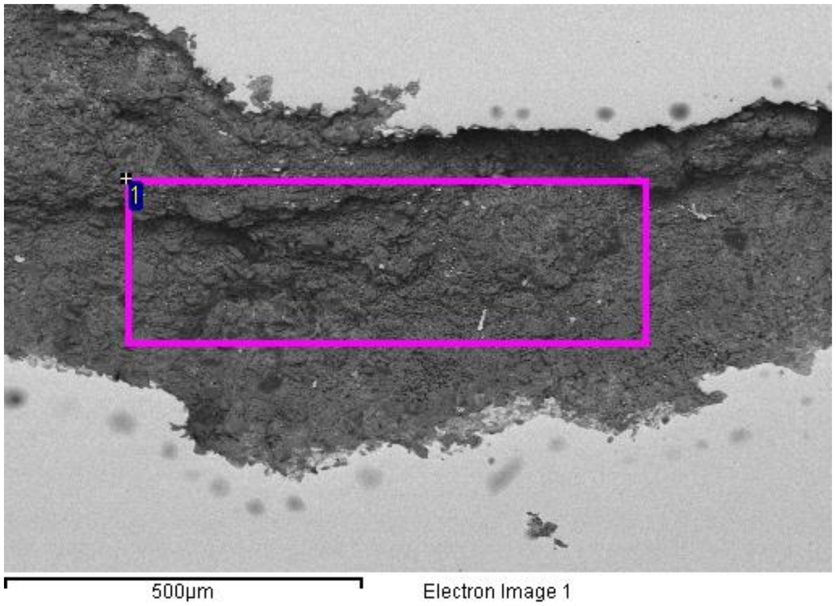


Data from 35 ton-ingots scrapped for macro-inclusions (75 ingots).

40% of ingot scraps is due to **entrapment of mold flux (powder).**

Ingot quality

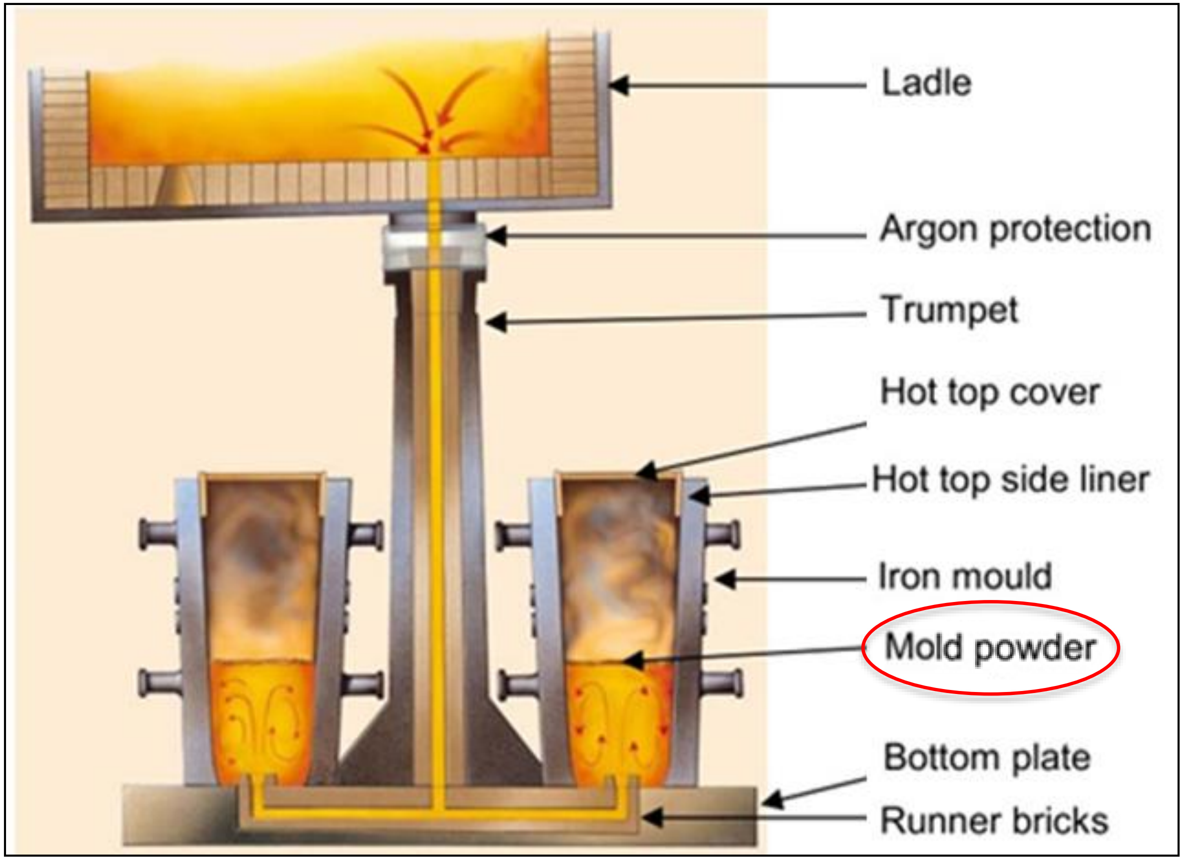
NB: mold flux macro-inclusions can be easily detected as they have specific composition and typical shape and distribution.



Spectrum	O	Na	Mg	Al	K	Ca	Fe
1	39.03	1.46	2.82	34.97	2.48	3.55	15.68

Ingot quality

To limit this problem, bags of mold powder are prepared into the mold suspended at a certain height to avoid premature release.



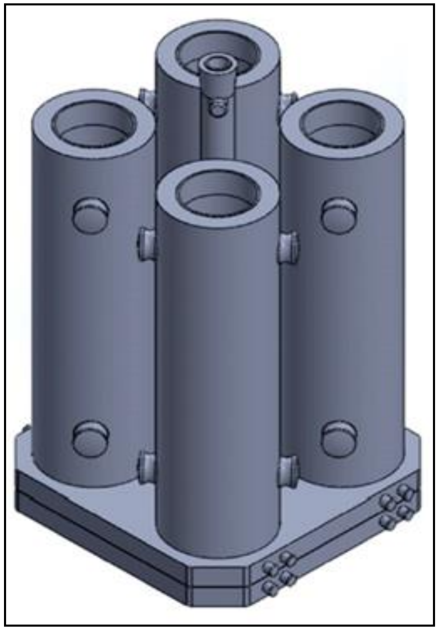
Ingot quality

CASE STUDY: numerical modelling can be conveniently applied to simulate the liquid metal flow during the mold filling.

This can help evaluating the risk of powder entrapment and defects formation.

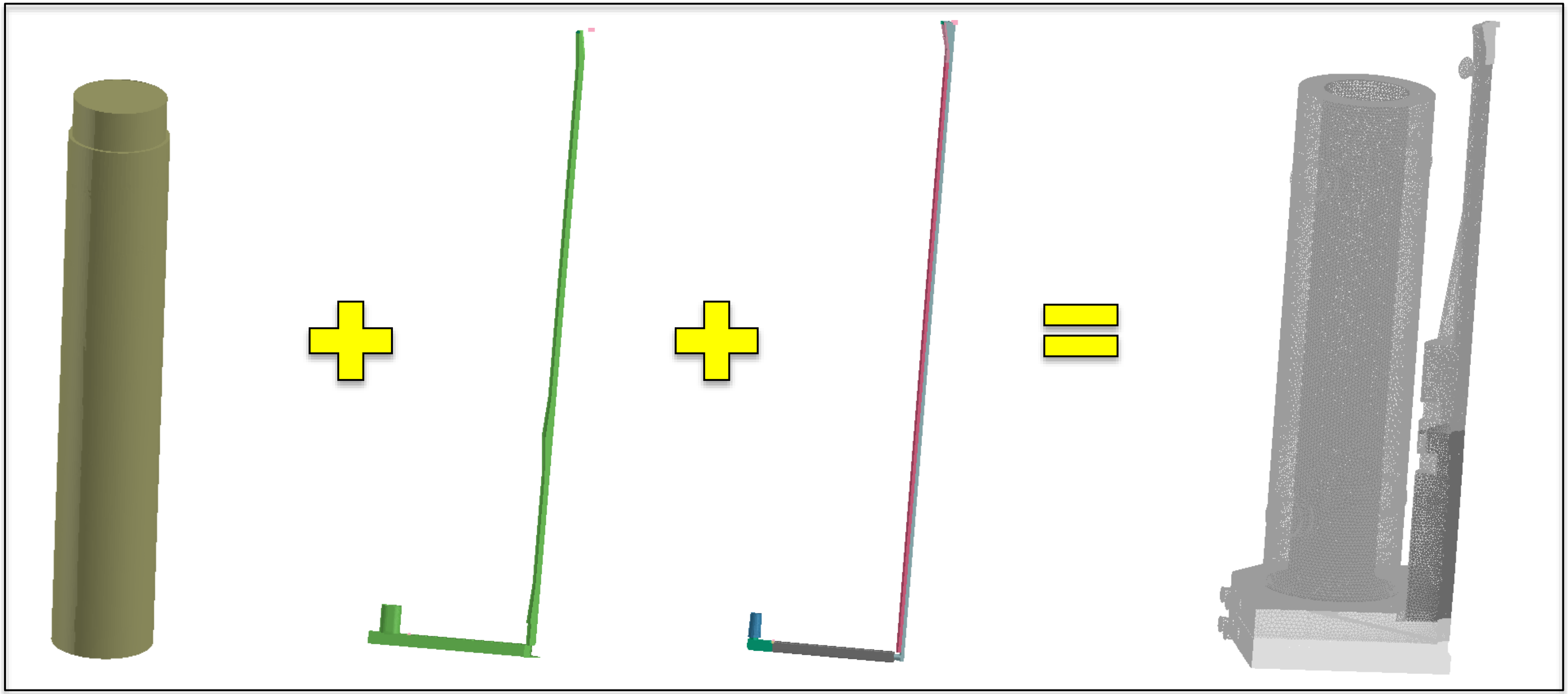
A 4-mold system for 19-ton round ingots of AISI 4140 steel was considered.

For this purpose, it is crucial using a full mold geometry, respect to conventional simplified models.



Ingot quality

Pouring basin (trumpet) and running system were included in the model, considering real geometry and refractory materials.

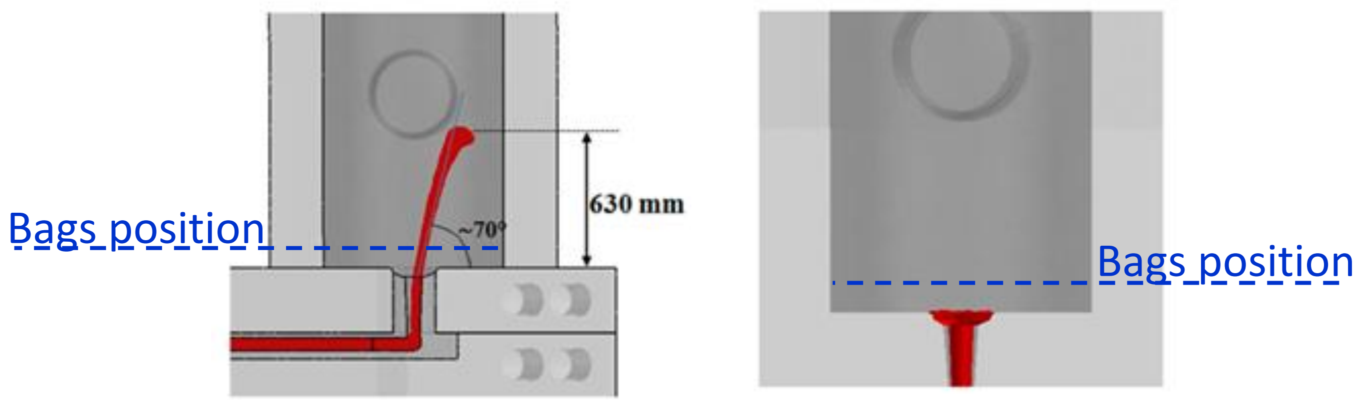


Full model

Ingot quality

Full model results show that the steel, entering the mold at high speed, reaches a height of 630 mm → risk to break the suspended powder bags → premature release of mold powder.

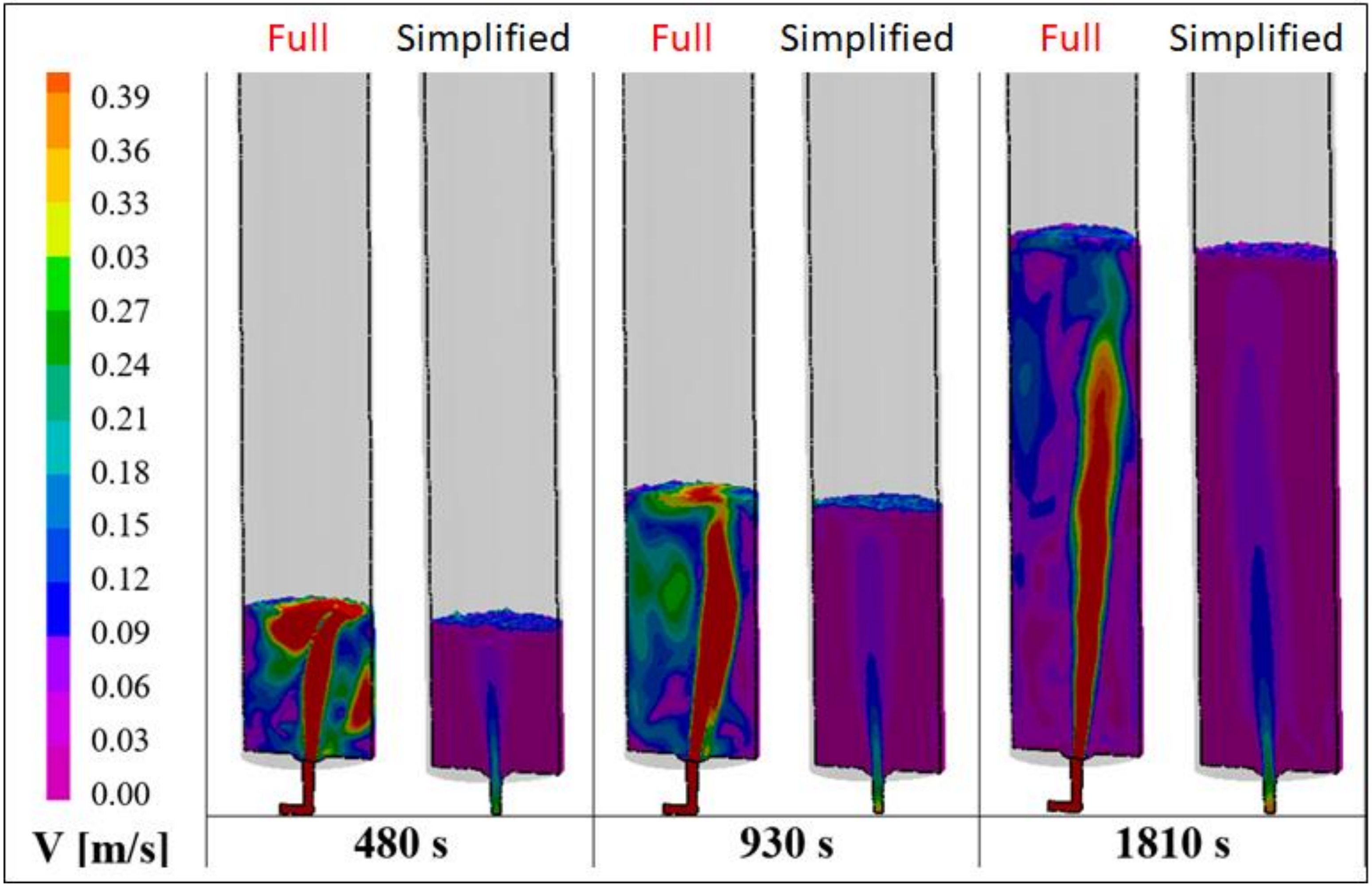
Simplified model completely neglects this problem.



Full model Simplified model

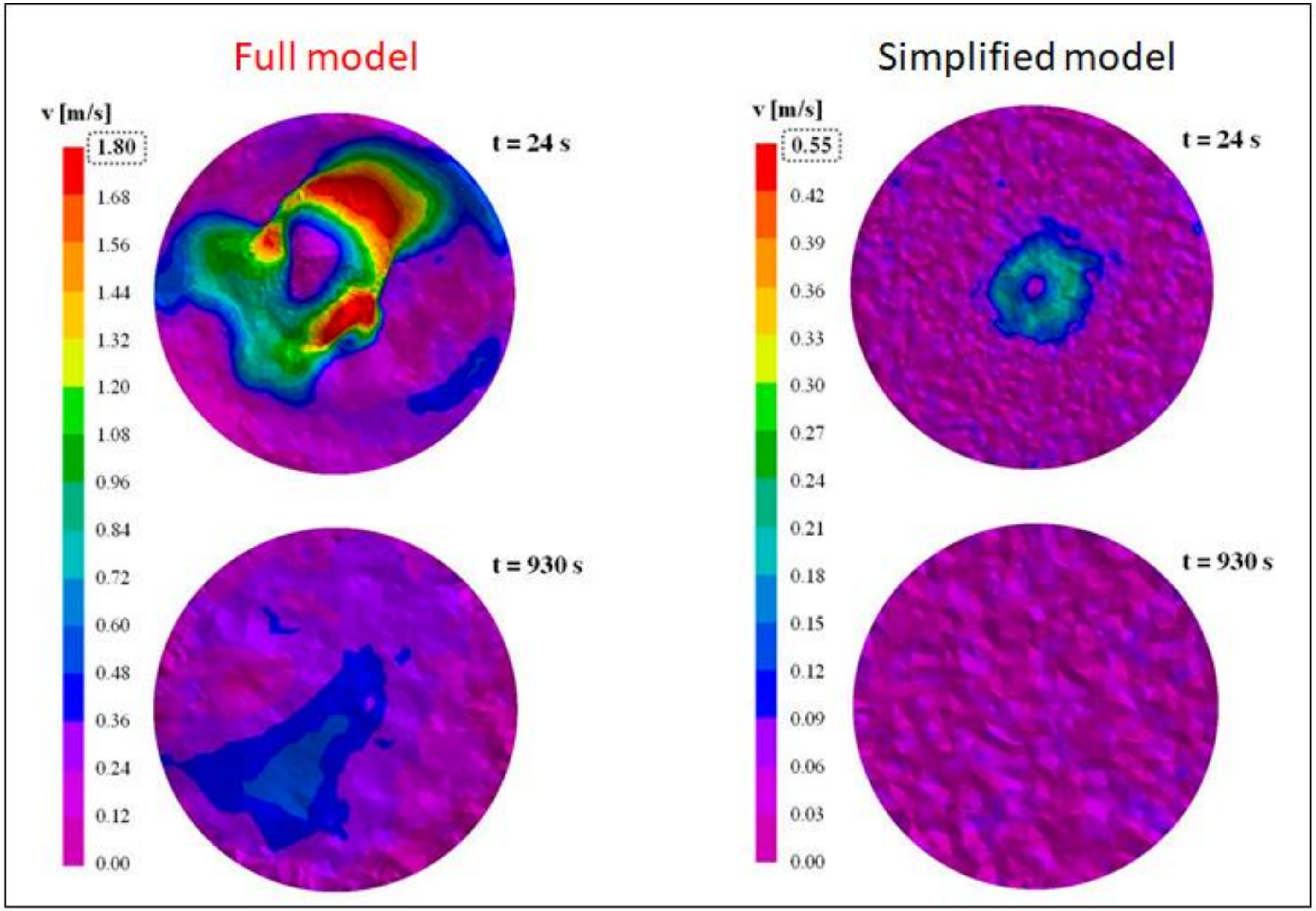
Liquid steel entering the mold: full model vs. simplified model

Ingot quality



Mold filling simulation: full model vs. simplified model

Ingot quality



Liquid metal tangential velocity after 24 s and 930 s

Ingot quality

The Weber number, W_e can be calculated to estimate the risk for powder entrapment → if $W_e > 12.3$ this risk increases.

$$W_e = \frac{u_{steel}^2 \cdot \rho_{steel}}{\sqrt{\gamma \cdot g \cdot \left(\rho_{steel} - \rho_{slag}\right)}}$$

where:

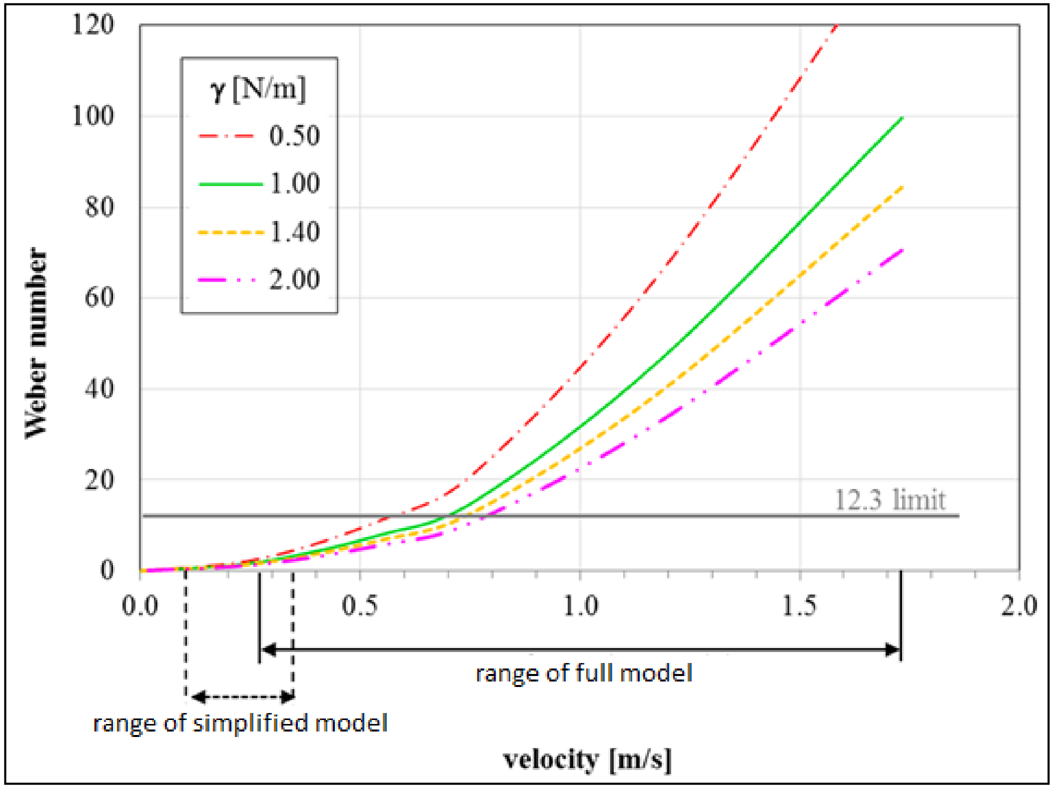
u_{steel} = tangential steel velocity

ρ_{steel} = steel density

ρ_{slag} = slag density

γ = slag-steel interfacial tension

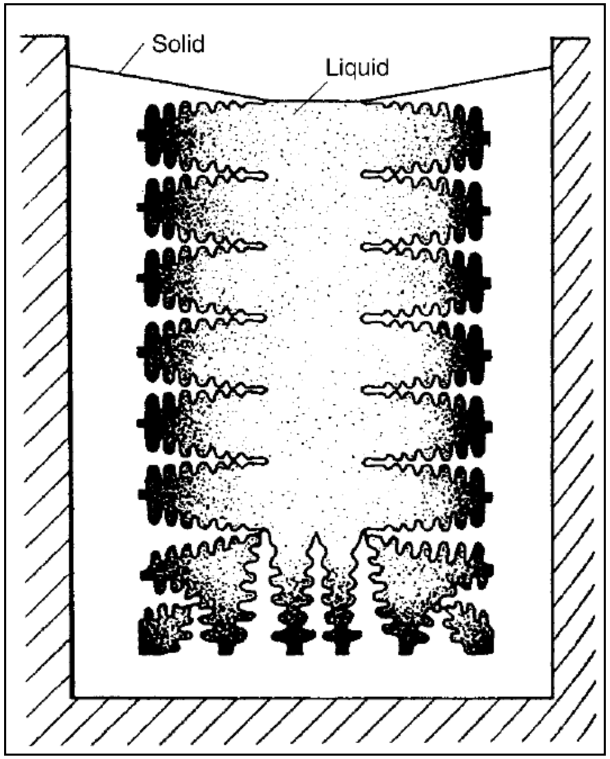
g = gravity constant

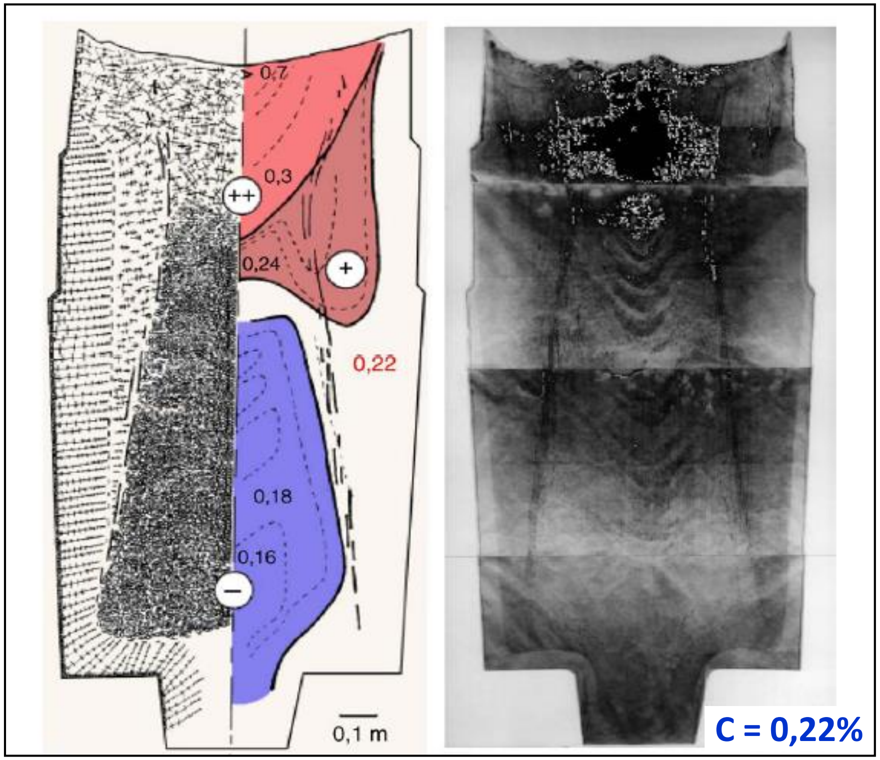


Ingot quality

Two main factors related to steel ingots affect forgings quality:

1. Non-metallic macro-inclusions.
2. **Micro- and macro-segregations.**

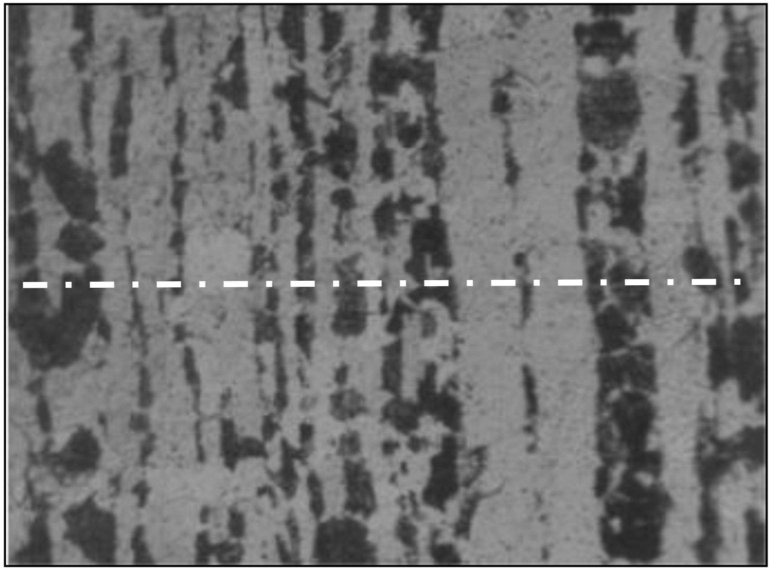


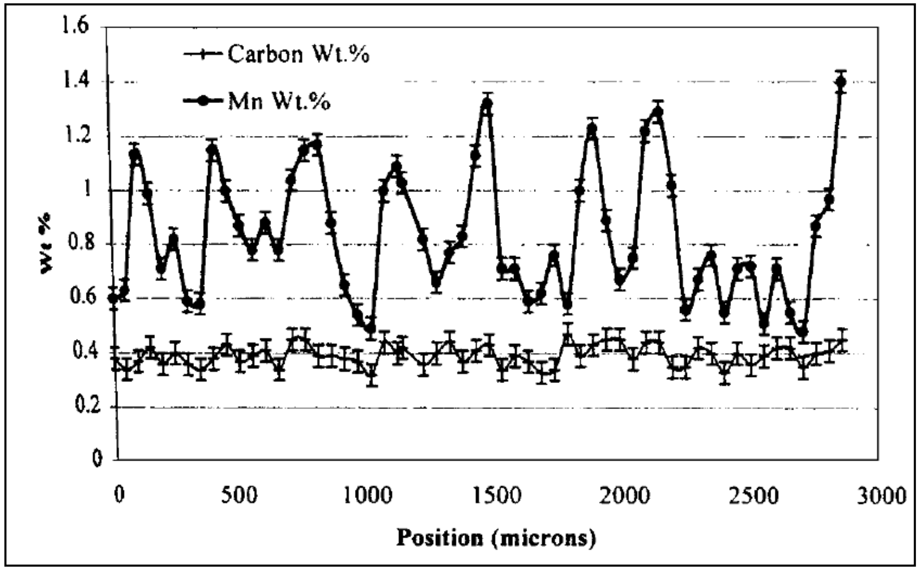


Ingot quality

Hot deformation processes align these interdendritic chemistry variations into **micro-segregation bands,** parallel to deformation.

→ alternating regions of high and low concentration of solute.





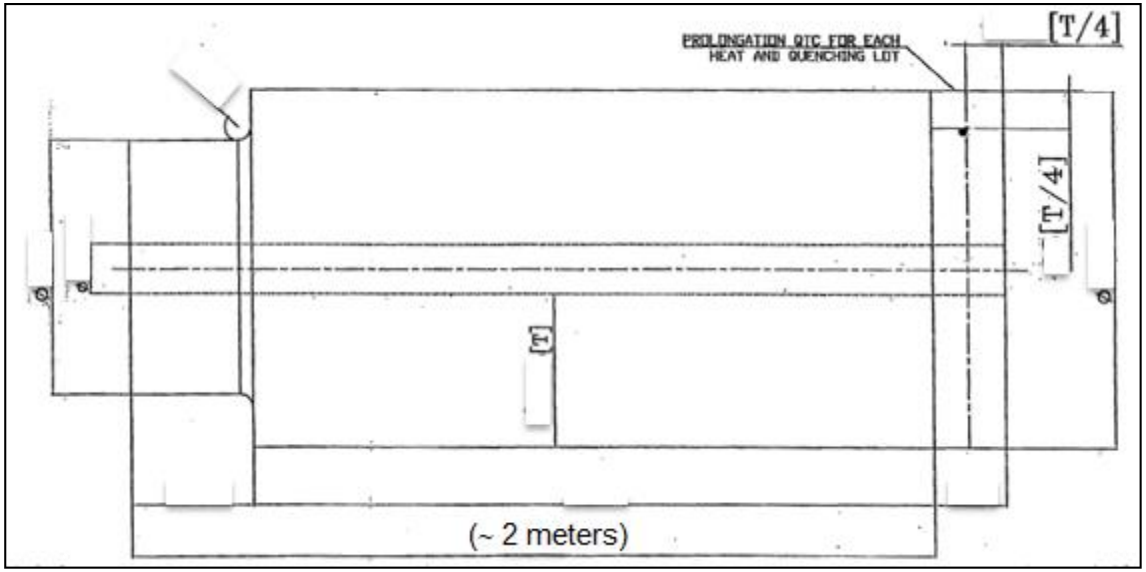
Mn and C content fluctuations across segregation bands of AISI 4140 rolled steel

Ingot quality

CASE STUDY: the effect of segregation bands on mechanical properties have been studied on heavy forgings in AISI 8630 steel.

Two forgings with different level of segregations were considered.

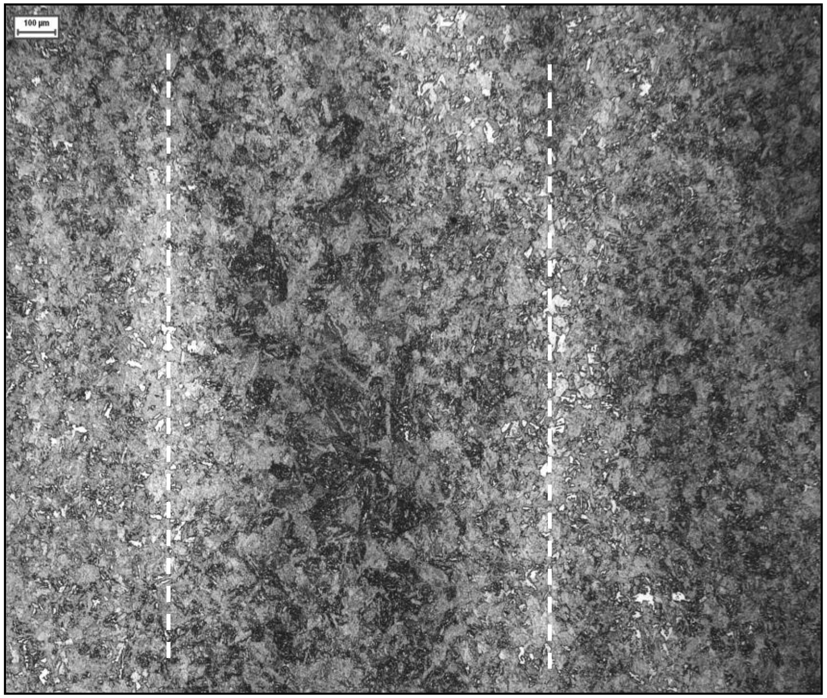
Geometry and chemical compositions



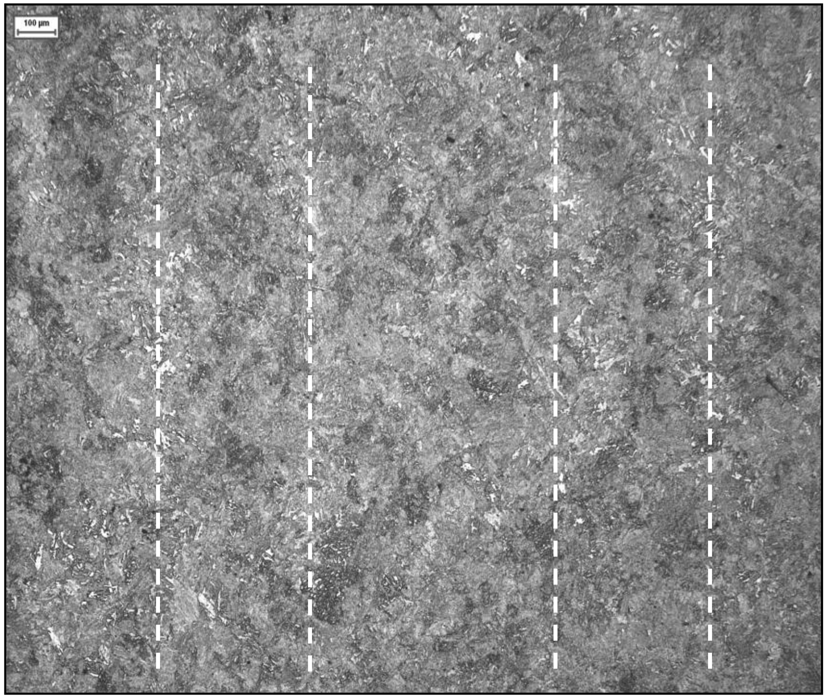
	C	Mn	Si	Cr	Ni	Mo	V	Cu	CE
HS-forging 1	0.31	1.07	0.31	0.98	0.83	0.42	0.042	0.05	**0.84**
LS-forging 2	0.322	1.08	0.3	0.96	0.82	0.4	0.027	0.13	**0.84**

Ingot quality

The alternate light/dark bands revealed by Nital2 etching on HS-forging 1 samples are clearly more intense respect to LS-forging 2.



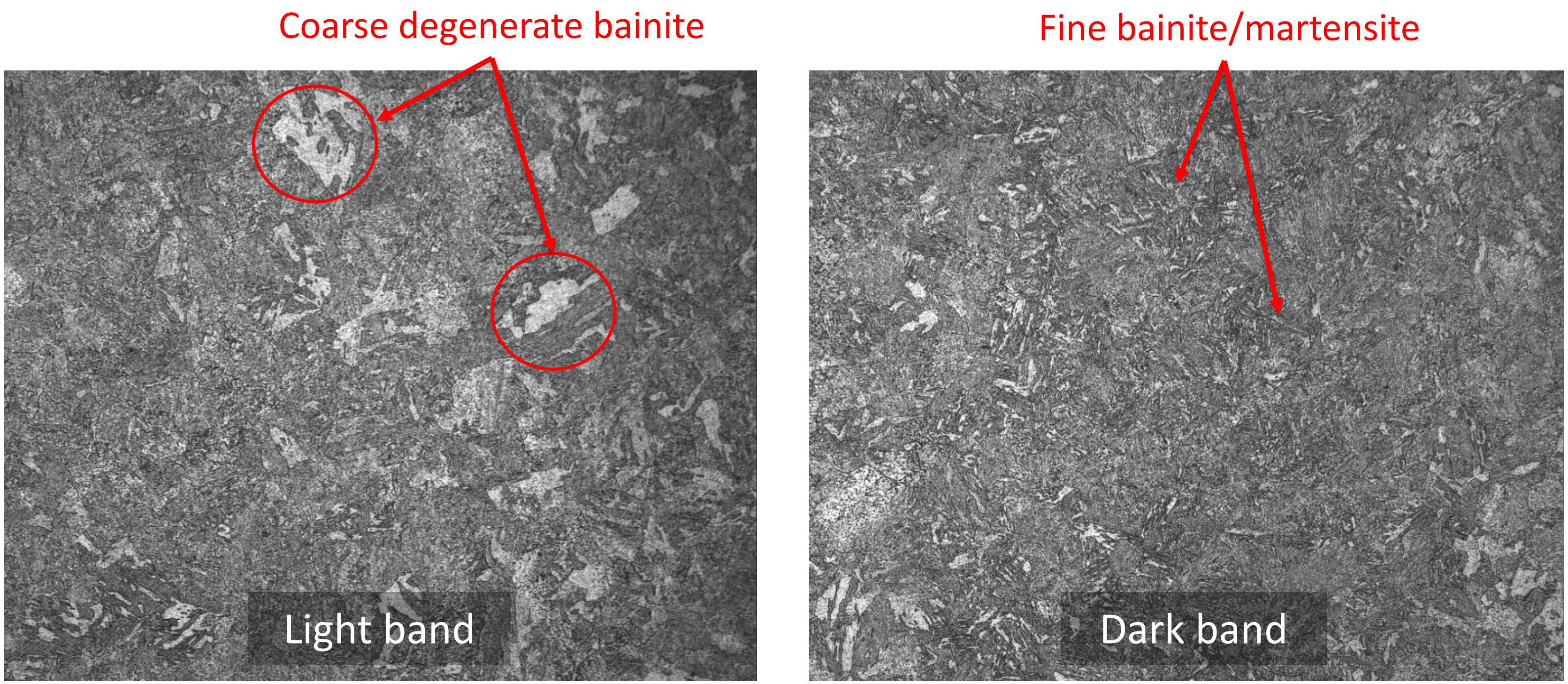
HS-forging 1 (High)



LS-forging 2 (Low)

Ingot quality

Light bands with low alloying elements have lower hardenability → anomalous microstructure after quenching and tempering.

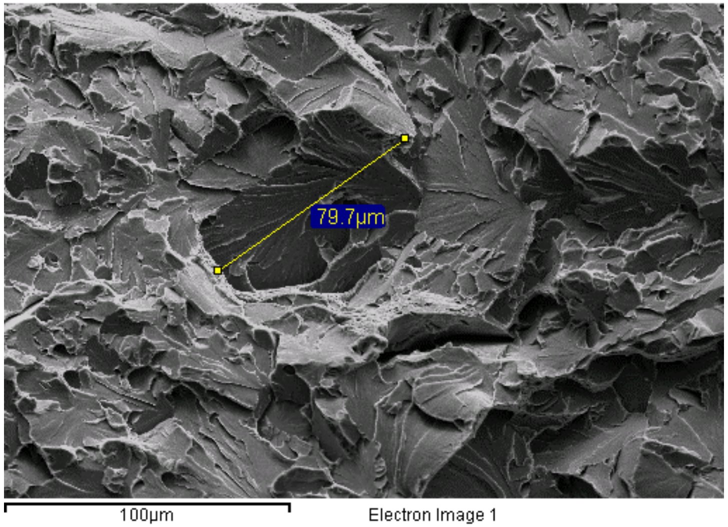


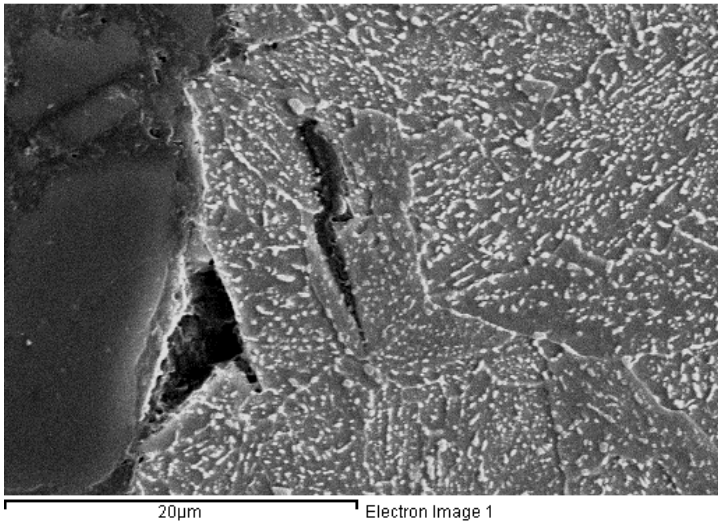
HS-forging 1 (High)

Ingot quality

In HS-forging 1, strong segregation banding significantly reduce both the tensile strength and the impact energy absorption.

	HV_{300g} light band	HV_{300g} dark band	UTS (MPa)	YS (MPa)	$KV_{-46°C}$ (J)
HS-forging 1	219 ± 9	249 ± 3	749 ± 13	589 ± 9	31
LS-forging 2	245 ± 5	265 ± 6	827 ± 14	657 ± 11	123

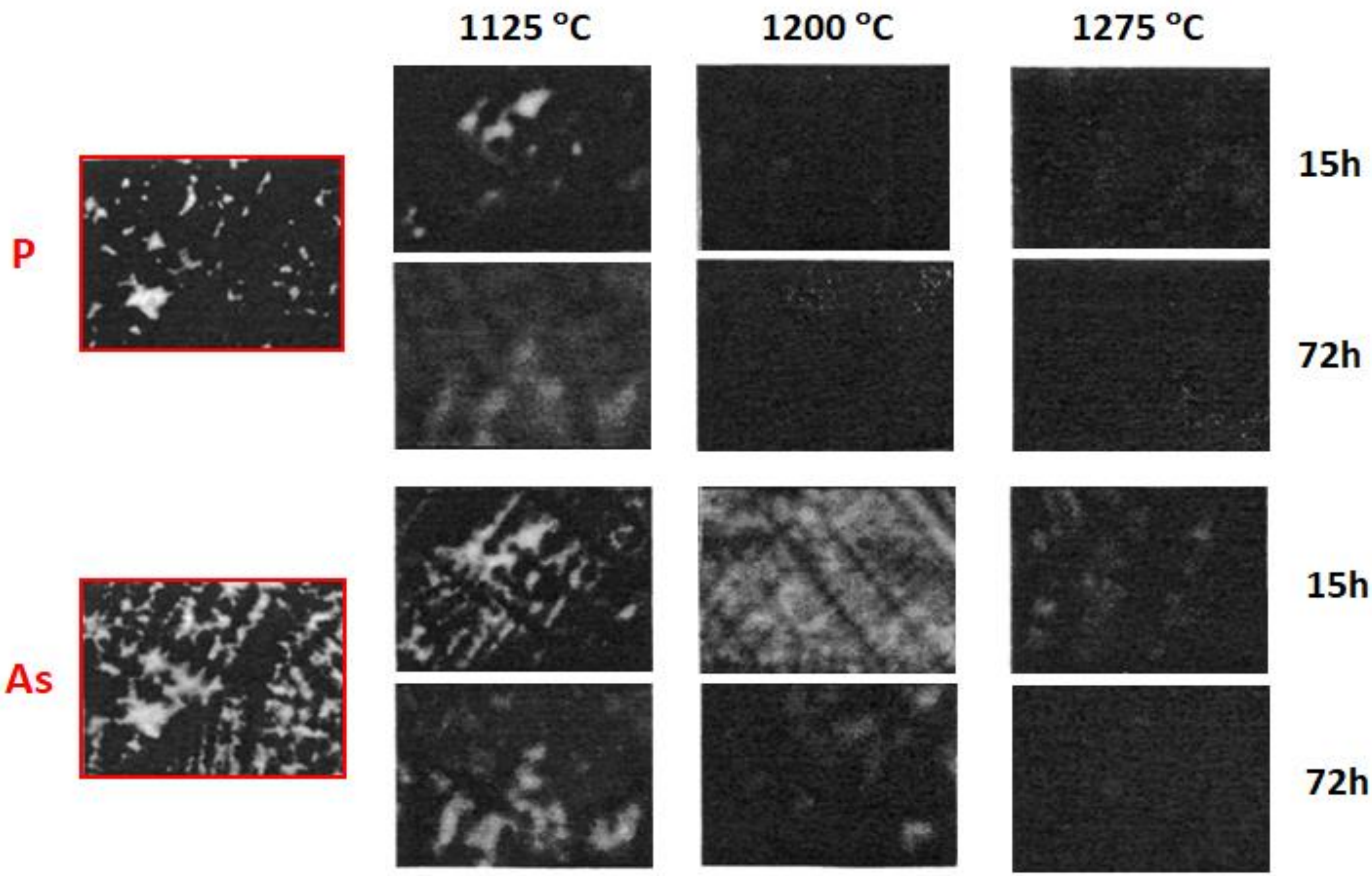




HS-forging 1 - Charpy test fracture surface and etched cross section

Forging parameters and HTs

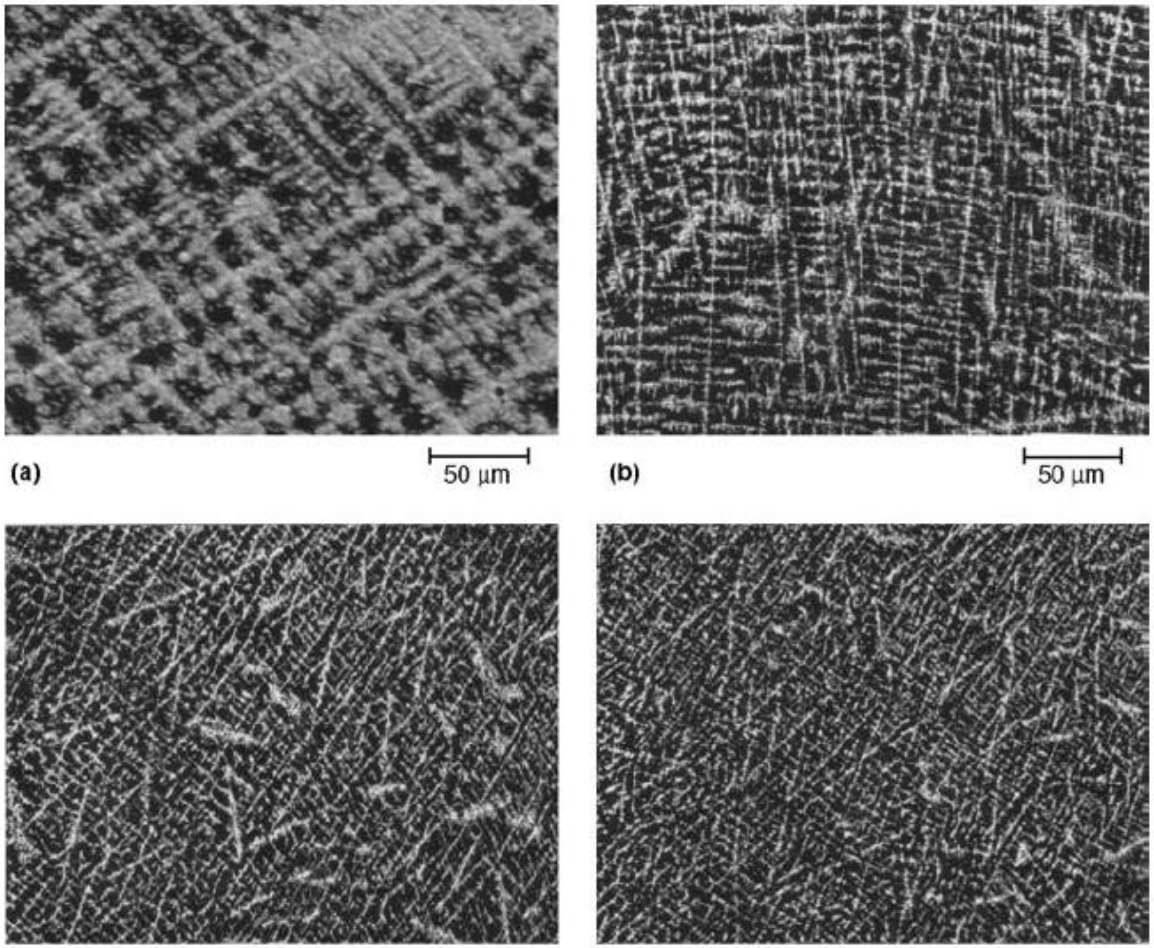
High pre-forging temperatures combined with large reductions (deformation ratios > 5:1) can help to homogenize the material.



Forging parameters and HTs

7:1

10:1



27:1

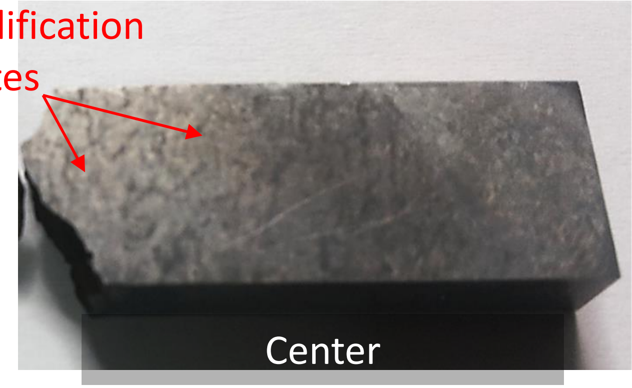
49:1

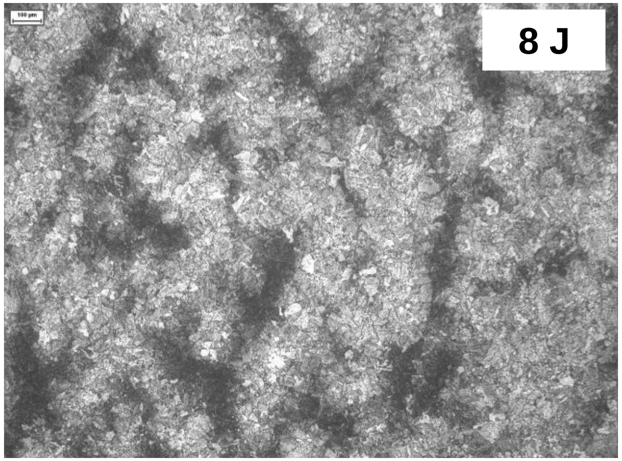
Effect of different forging ratios on dendrites size

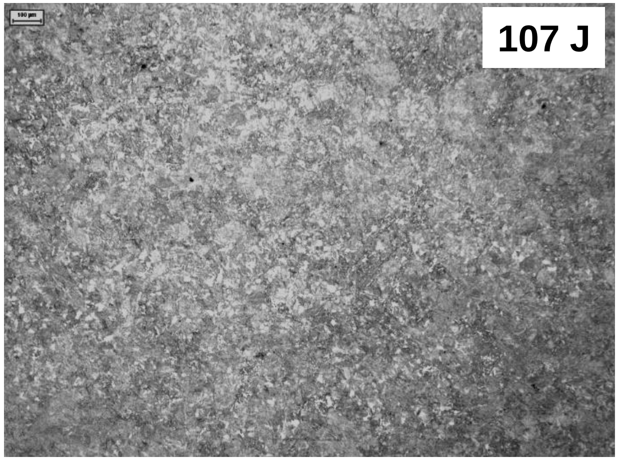
Forging parameters and HTs

Single upsetting	Double upsetting

Residual solidification dendrites





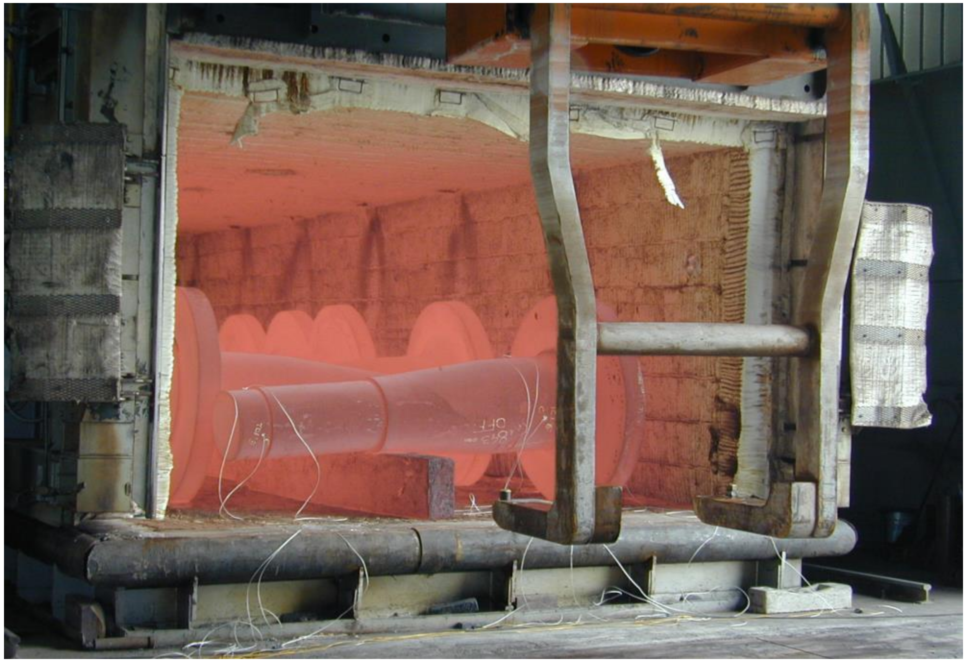


Microstructure and impact energy of AISI 8630 steel forgings (longitudinal samples)

Forging parameters and HTs

Heat treatments are also very important to determine the final microstructure and mechanical properties of heavy forgings.

Typically, special grades steel forgings are provided in quenched and tempered conditions.



Forging parameters and HTs

CASE STUDY: the proper choice of quenching medium is crucial to guarantee the expected microstructure and mechanical properties, also minimizing the risk of thermal cracks.

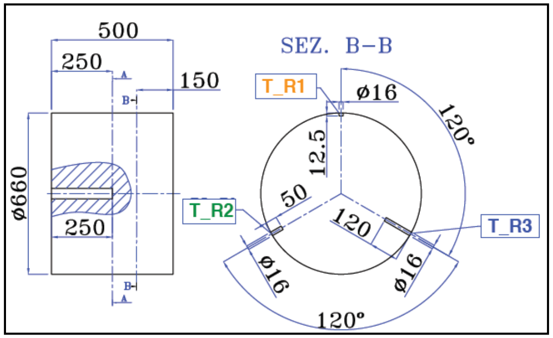
Numerical modelling is helpful in simulating forgings cooling to forecast the microstructure on the whole thickness.

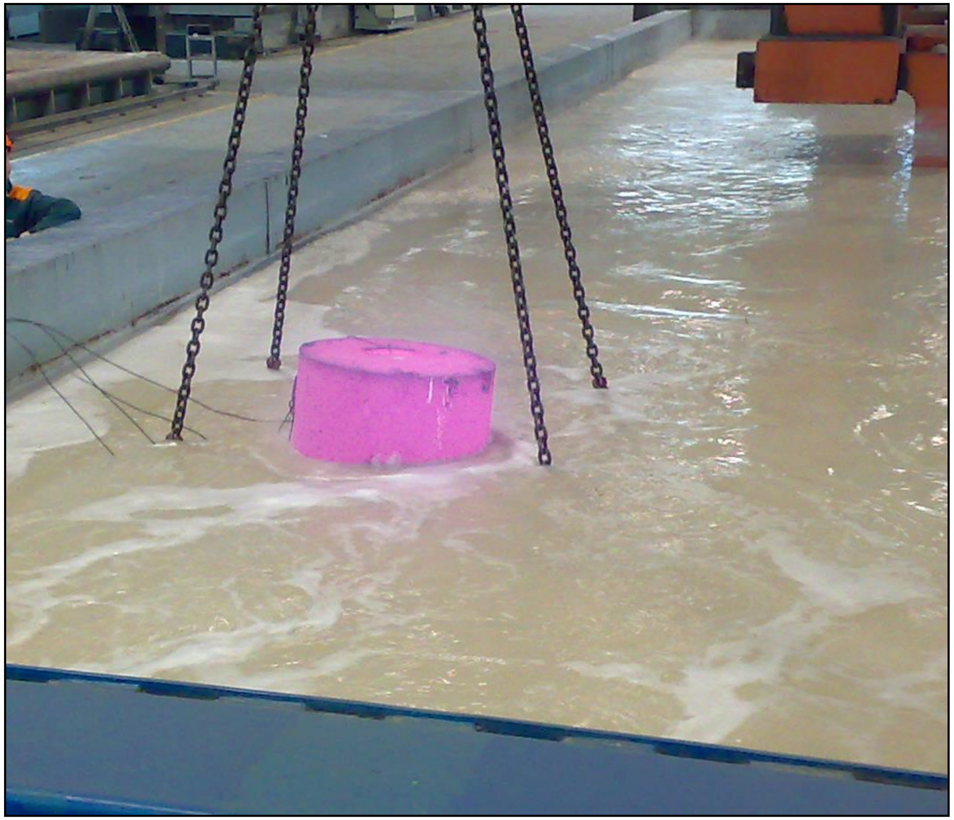
To obtain reliable results, the heat transfer coefficient, h (W/m^2K) of quenching medium has to be properly defined.

	Air	Oil	Water	Brine
No circulation of fluid or agitation of piece	0.02	0.25–0.30	0.9–1.0	2
Mild circulation (or agitation)	. . .	0.30–0.35	1.0–1.1	2–2.2
Moderate circulation	. . .	0.35–0.40	1.2–1.3	. . .
Good circulation	. . .	0.4–0.5	1.4–1.5	. . .
Strong circulation	0.05	0.5–0.8	1.6–2.0	. . .
Violent circulation	. . .	0.8–1.1	4	5

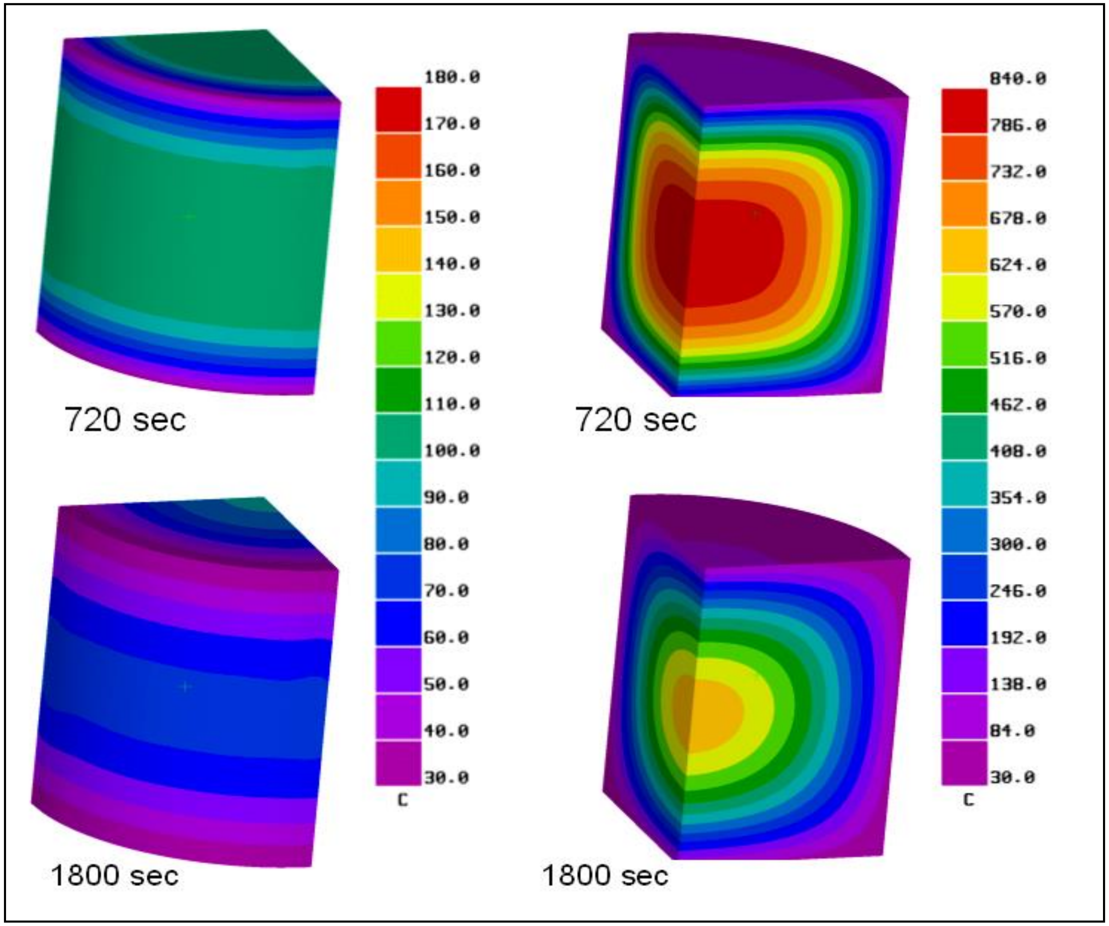
Forging parameters and HTs

In this example, the h coefficient was determined by experimental tests carried out on a AISI 4140 steel block equipped with n°4 thermocouples embedded into the material at different depths.





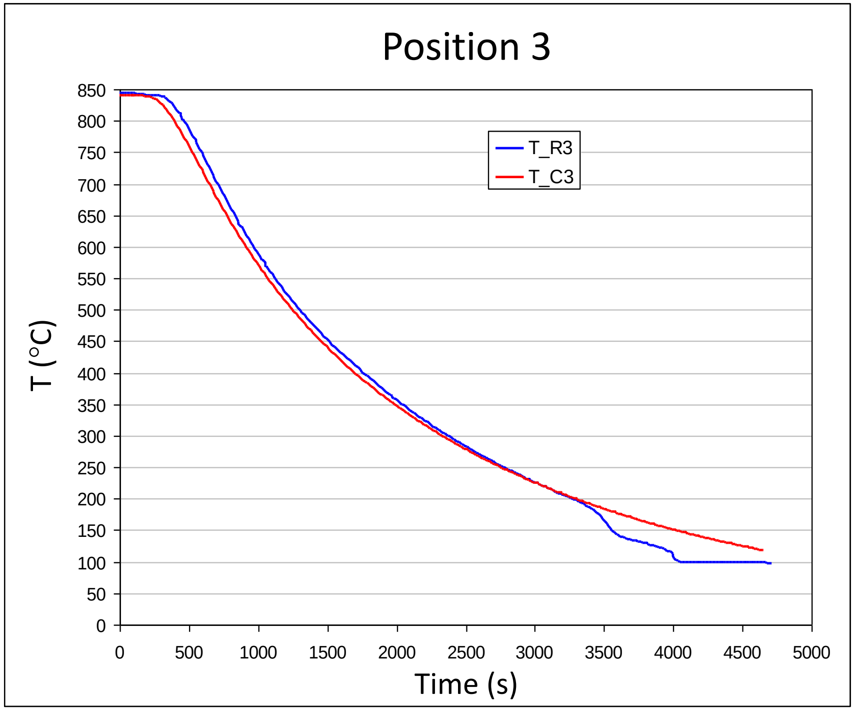
Forging parameters and HTs

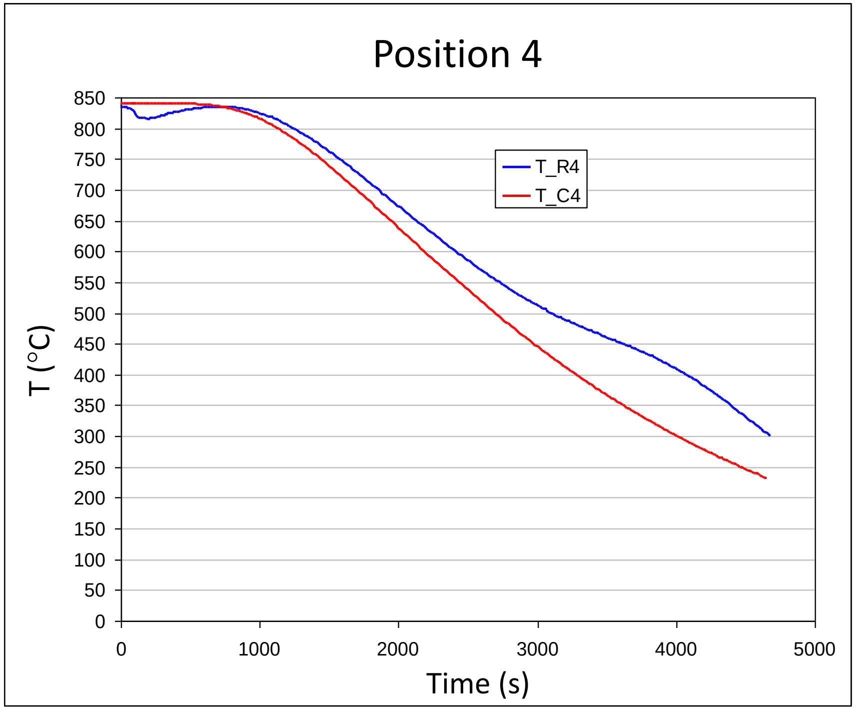


Simulated temperatures in the cross section of steel block after 720 s and 1800 s

Forging parameters and HTs

The model calibration was obtained by fitting the experimental temperatures vs. time curves with the simulated one, at different depths in the steel block.

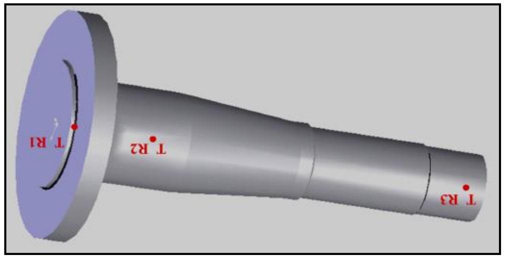


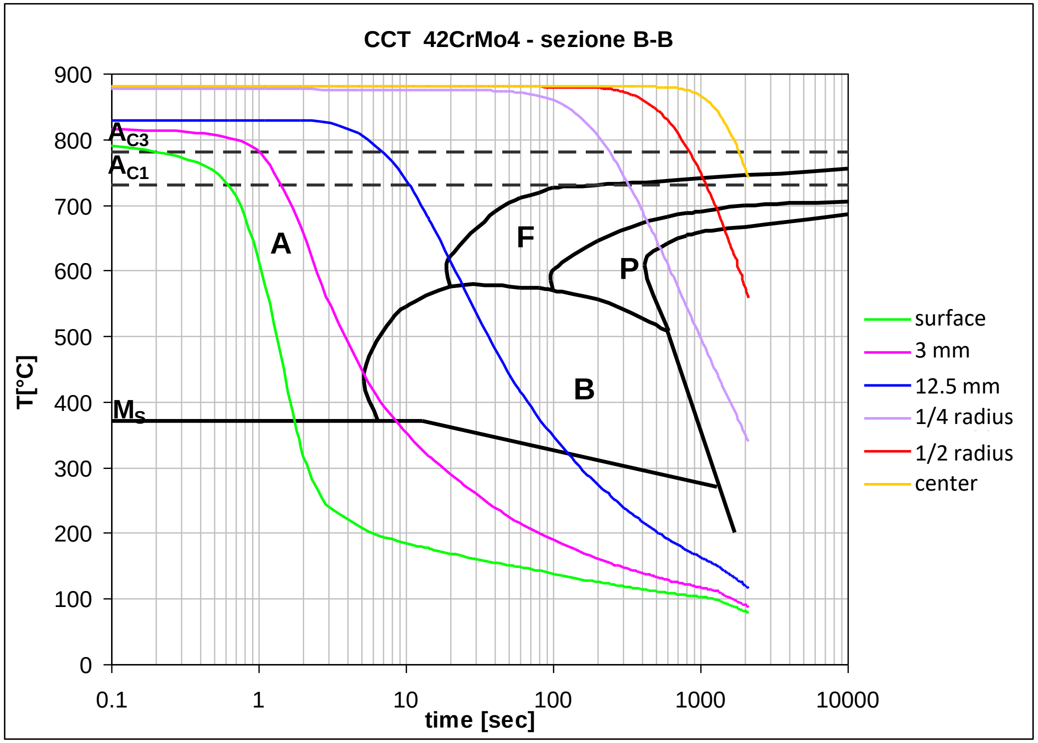


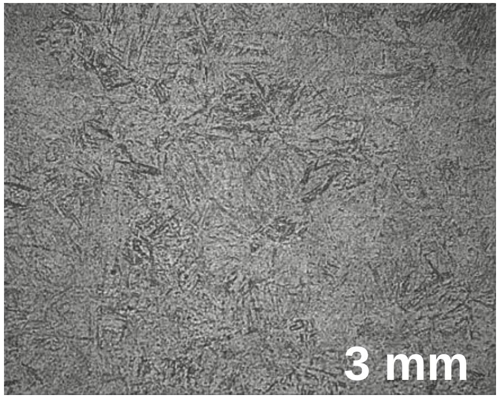
Forging parameters and HTs

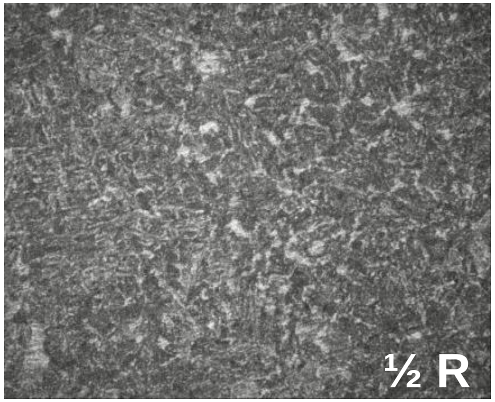
The model was successfully applied on a real forging for wind turbine in AISI 4140 steel.

The expected microstructure and hardness were confirmed by metallographic analysis.





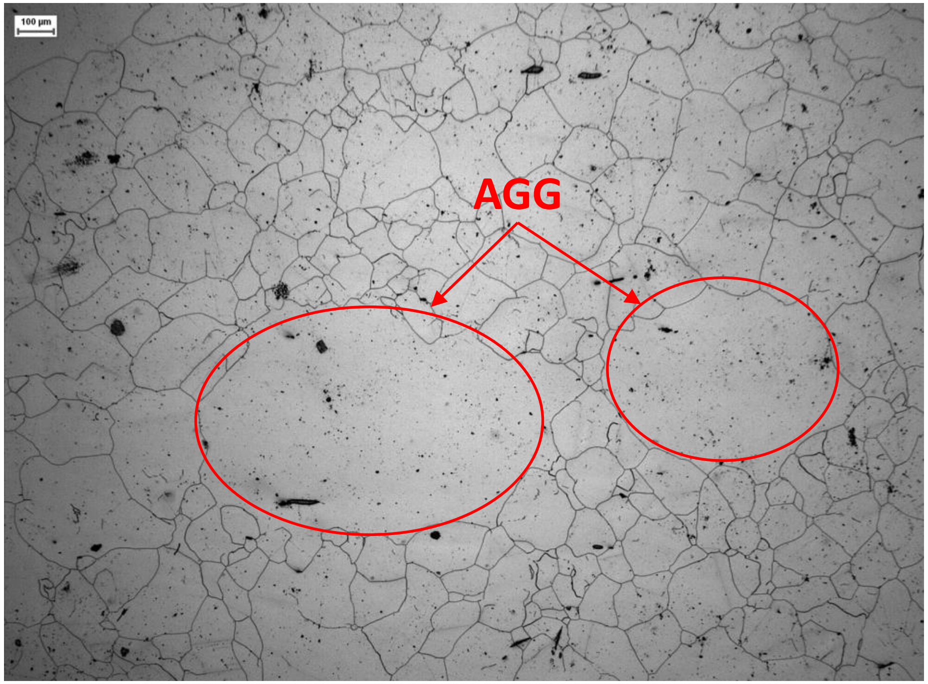




Forging parameters and HTs

<u>CASE STUDY:</u> the effect of the final solution annealing on the microstructure of AISI 316L forged bars was studied.

316L microstructure can suffer of a problem named Abnormal Grain Growth (AGG) → orange peel defects, false positives in UT,..



Forging parameters and HTs

At the end of bar forging, two different options were considered:

1) Solution annealing (1050°C-3h) followed by water quenching.

2) Direct water quenching.

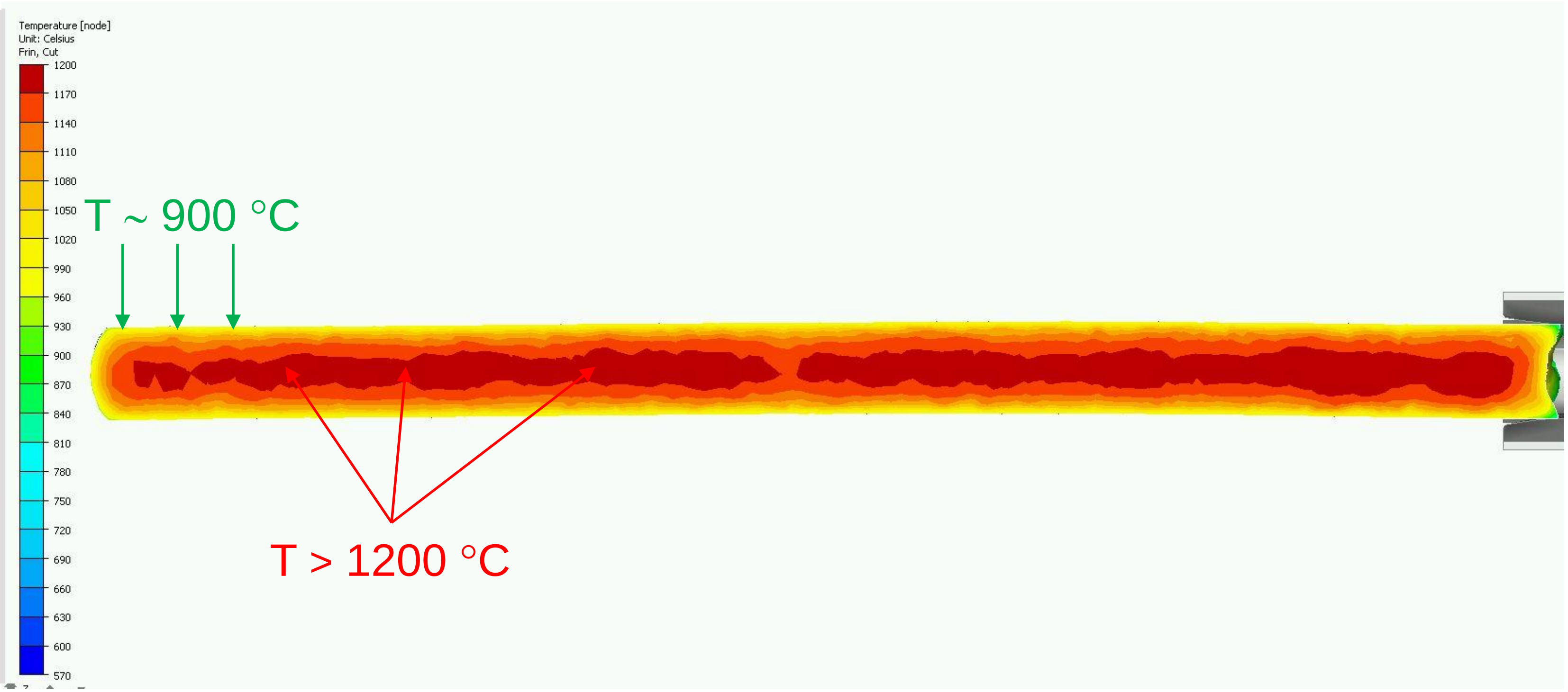
Samples were cut at the bars end in 3 positions (surface, ½ radius, center), polished and electrolytic etched with 60% HNO_3.

Microstructure grain size was measured according to ASTM E-112.

Bars	ϕ initial (mm)	ϕ final (mm)	Reduction (%)	Pyrometer final temperature (°c)	Heat treatment
1	450	360	36%	900°C	1050°C 3h + water quenching
2	450	360	36%	900°C	Direct water quenching

Forging parameters and HTs

FEM simulation of forging process showed that at the surface the temperature dropped down to ~ 900°C (close to the tips), while at the center it progressively increased above 1200 °C.



Simulated temperatures distribution in the bar at different reduction steps

Forging parameters and HTs

SYNCHROTRON THAILAND CENTRAL LAB

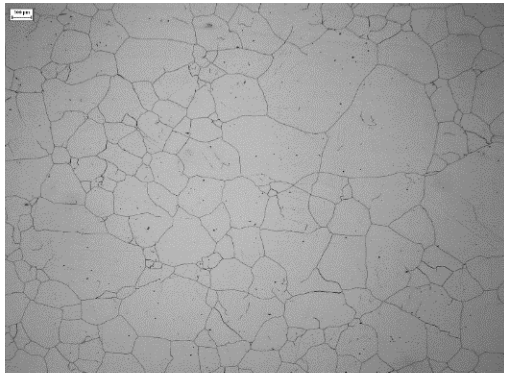
Bar 1

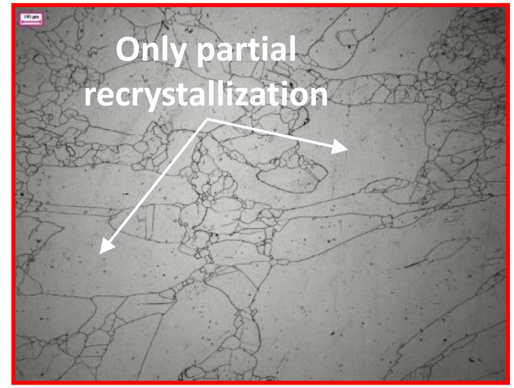
(solution annealing + quenching)

Bar 2

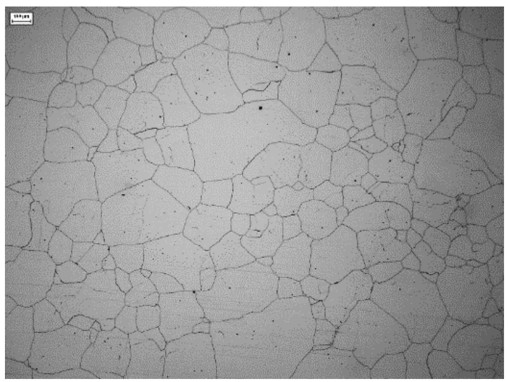
(direct quenching)

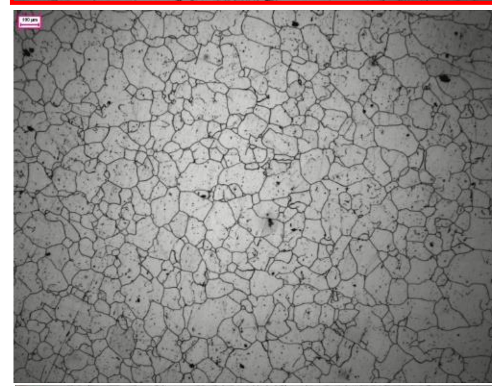
Surface



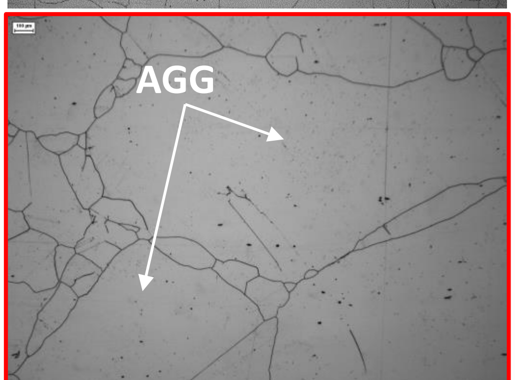


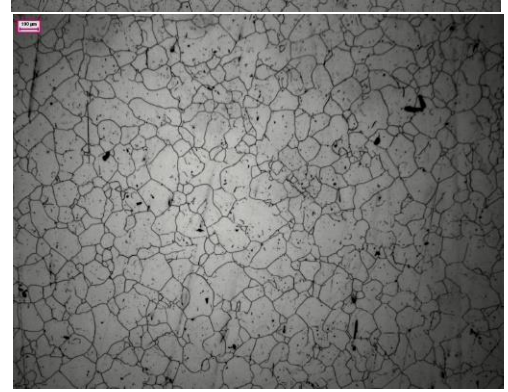
½ radius





Center

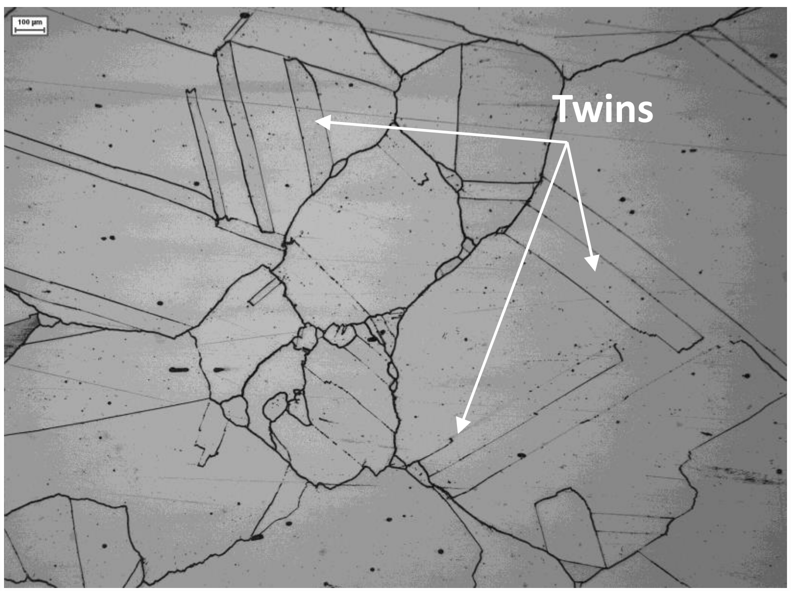




NB: partial recrystallization can be confused with AGG.

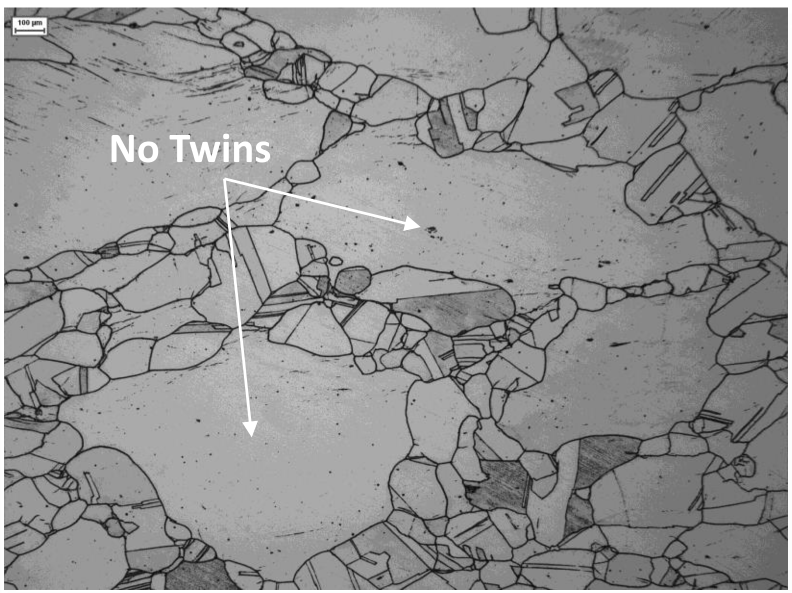
→ double electrolytic etching: 60% nitric acid + 10% oxalic acid, recrystallization twins in AGG grains are revealed.

Bar 1



Center

Bar 2

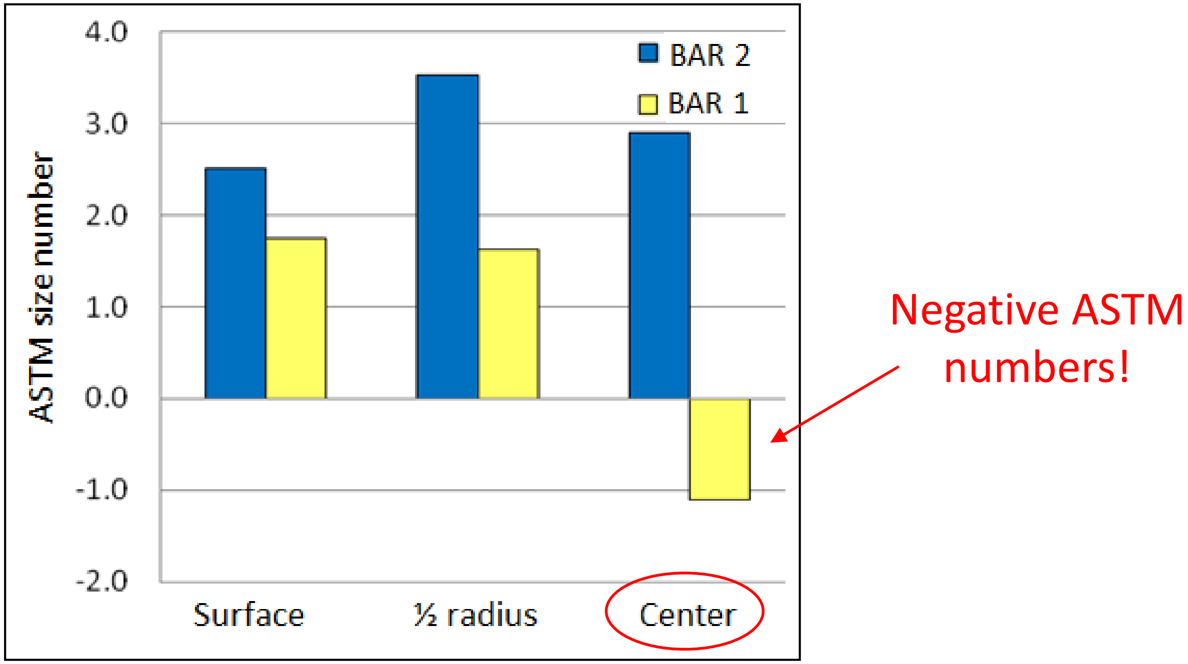


Surface

Forging parameters and HTs

Solution annealing vs. direct quenching has the advantage to ensures full recrystallization everywhere in the bar cross-section.

But, it leads to grain coarsening and AGG, especially at the bar center, where maximum forging temperatures are developed.



ASTM grain size distribution in the two forged bars

Forging parameters and HTs

CASE STUDY: the 625 alloy is an excellent corrosion resistance material for Oil and Gas field normally supplied in two conditions:

Grade 1 – Annealed

Grade 2 – Solution Annealed

The aim of this study was to evaluate the effect of precipitation hardening (PH) heat treatment on mechanical and corrosion resistance (e.g. SCC and SSC) of 625 alloy forged bars.

For this purpose 625 alloy forged bars with 3 different diameters: 152 mm, 203 mm, 254 mm (6 - 8 - 10 in.) have been produced.

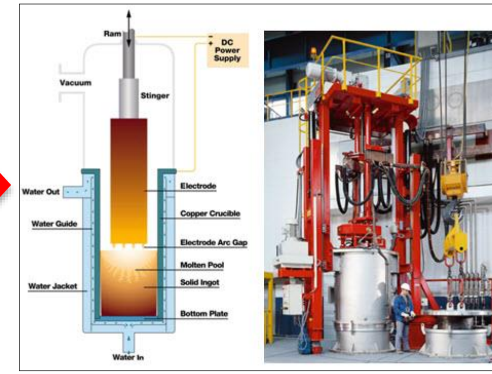
Ni	**Cr**	**Mo**	**Nb + Ta**	**Fe**	Ti	C	Mn	Si	Al	Co
58.0 min.	**20 - 23**	**8 -10**	**3.15 -4.15**	**5.0 max.**	0.40 max.	0.10 max.	0.50 max.	0.50 max.	0.40 max.	1.0 max.

Forging parameters and HTs

Melting (EAF)

Refining (AOD)

Remelting (VAR)



Forging

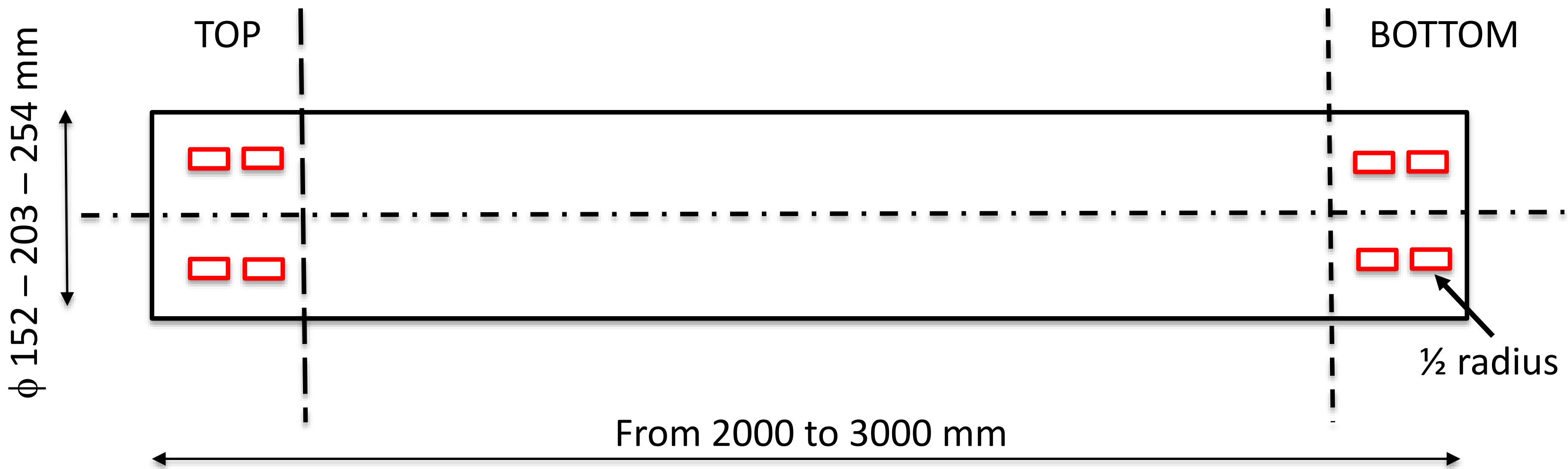
Heat Treatment

Final product

Forging parameters and HTs

Preliminary tests were performed on samples collected from top/bottom of bars at ½ radius position to find out the optimal heat treatment parameters.

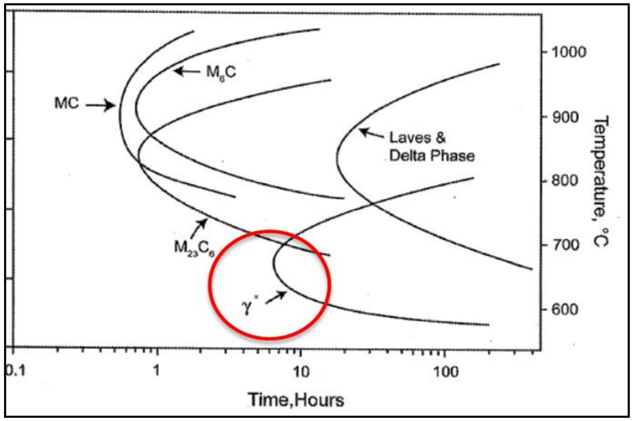
Each samples set was composed of: tensile, Charpy, hardness and ASTM G28 specimens.

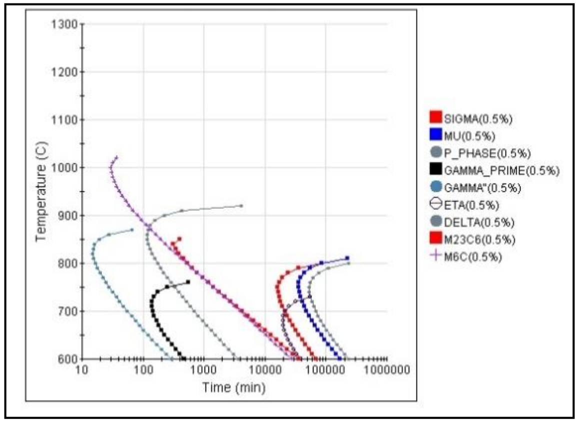


Forging parameters and HTs

HTs parameters were chosen according to literature and JMatPRO, aiming at maximizing gamma double prime γ’’ (Ni_3Nb) precipitation.

T Solubilization		T Ageing		Ageing time		
980 °C	⊗	650 °C	⊗	10 hours	→	12 test conditions
1010 °C		660 °C		16 hours		
		670 °C				



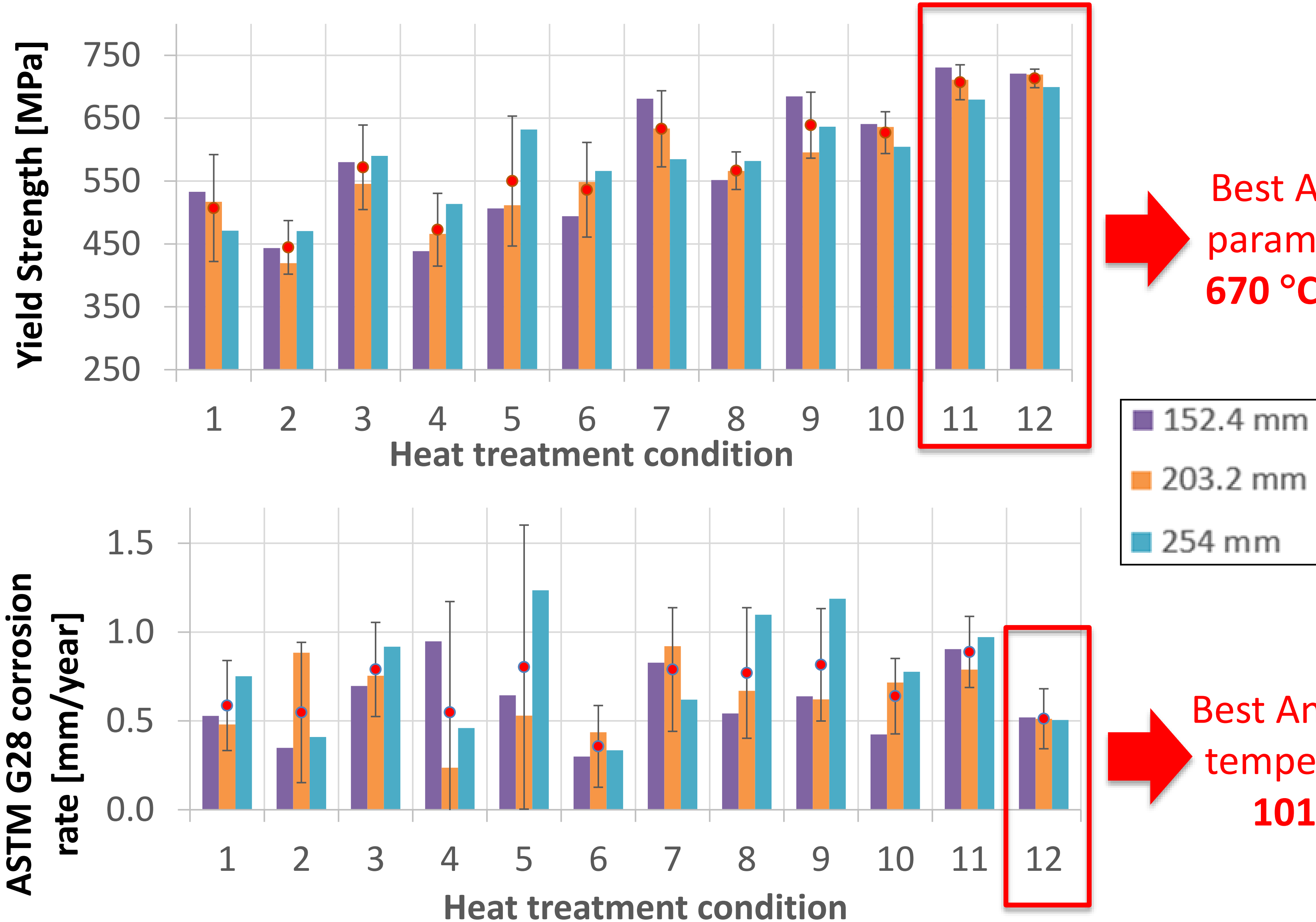


Forging parameters and HTs

HT conditions	Annealing temperature (°C)	Ageing temperature (°C)	Ageing time (h)
1	980	650	10
2	1010	650	10
3	980	660	10
4	1010	660	10
5	980	670	10
6	1010	670	10
7	980	650	16
8	1010	650	16
9	980	660	16
10	1010	660	16
11	980	670	16
12	1010	670	16

PH heat treatment conditions

Forging parameters and HTs



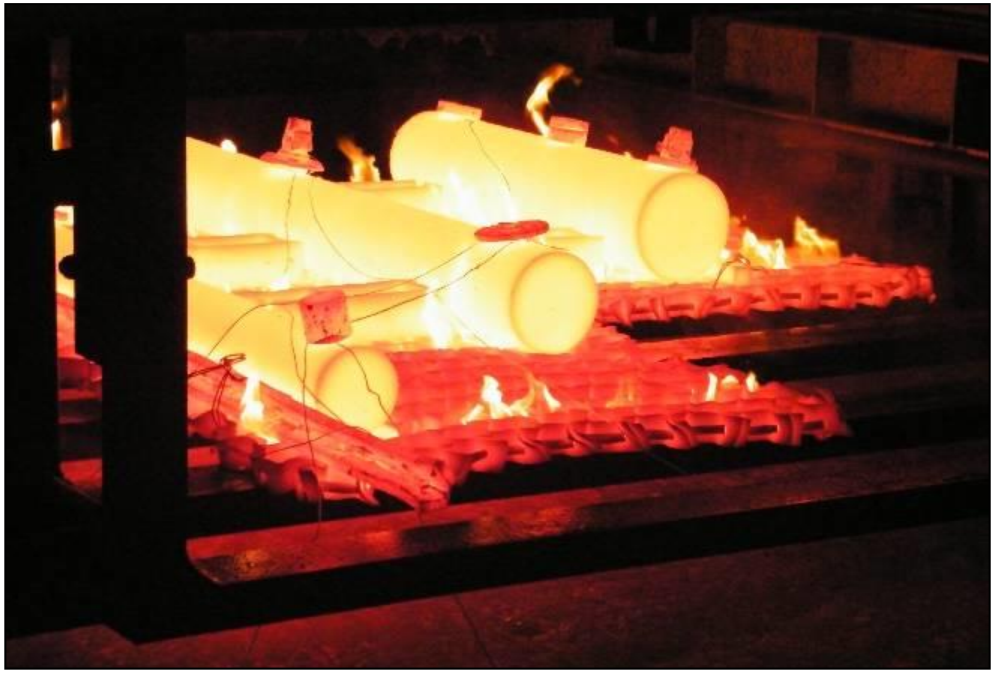
Best Ageing parameters: **670 °C - 16h**

Best Annealing temperature: **1010 °C**

Forging parameters and HTs

The heat treatment was performed on n°3 forged bars with different dimeters in an industrial furnace, as designed:

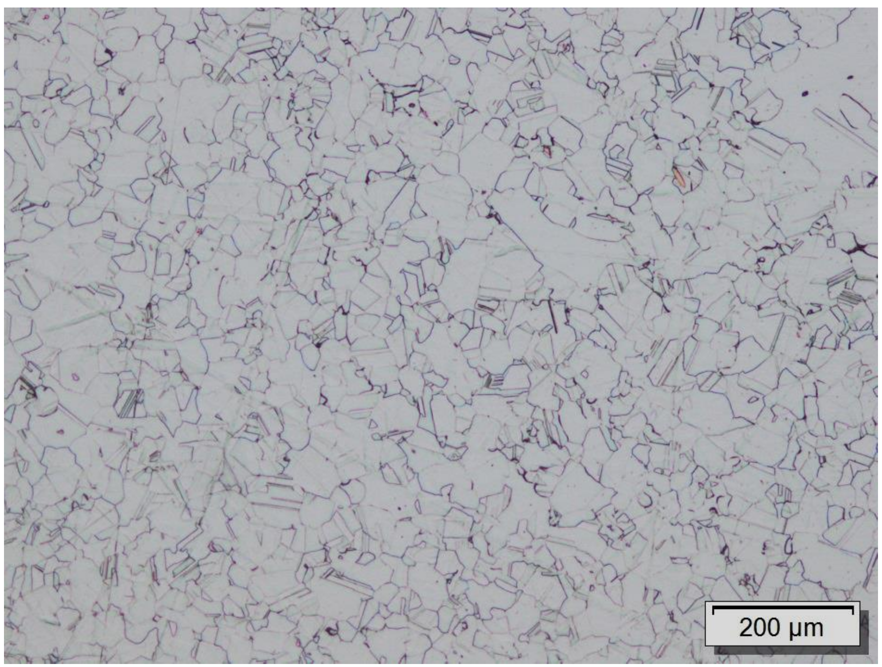
- annealing at 1010 °C for 1 hour,
- ageing at 670 °C for 16 hours followed by water quenching.

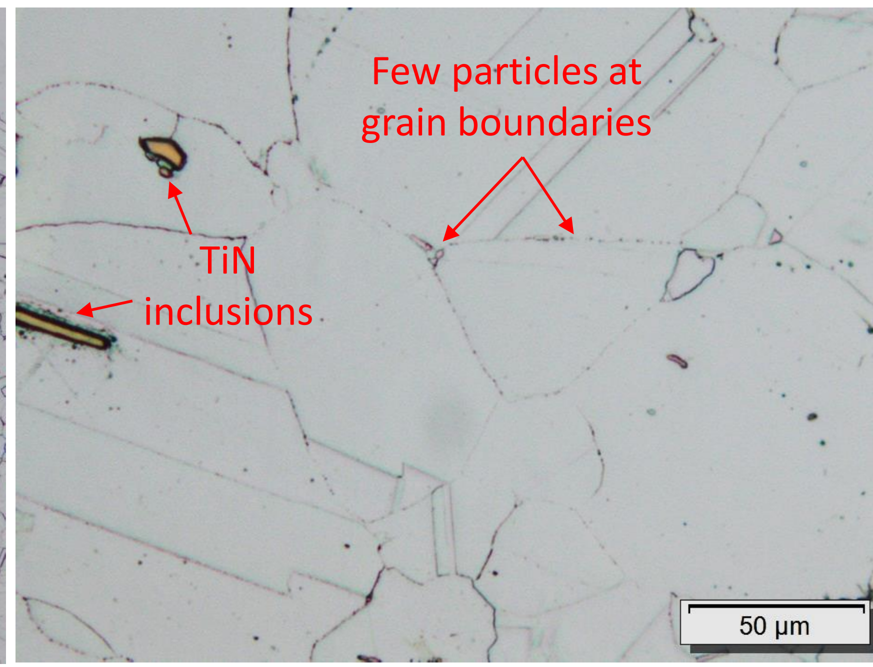


Forging parameters and HTs

OM analysis performed on top/bottom of the 3 bars, at the surface, ½ radius and center, gave the following results:

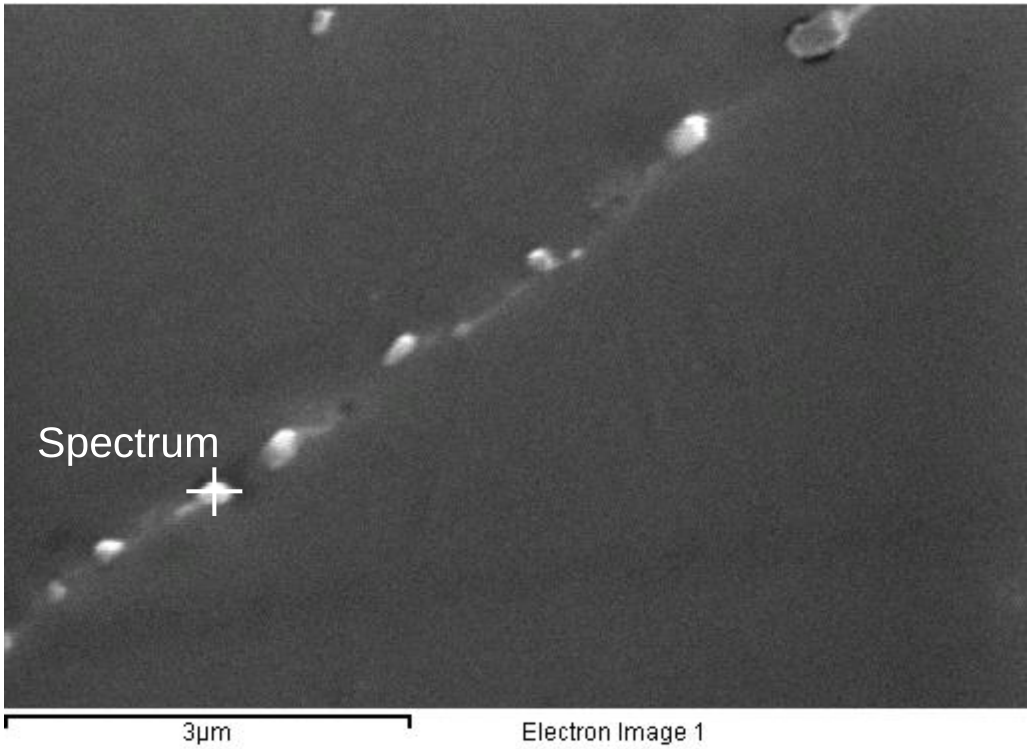
- even recrystallized microstructure with ASTM G6 grains;
- no continuous network of secondary phases (Laves, carbides..).





Forging parameters and HTs

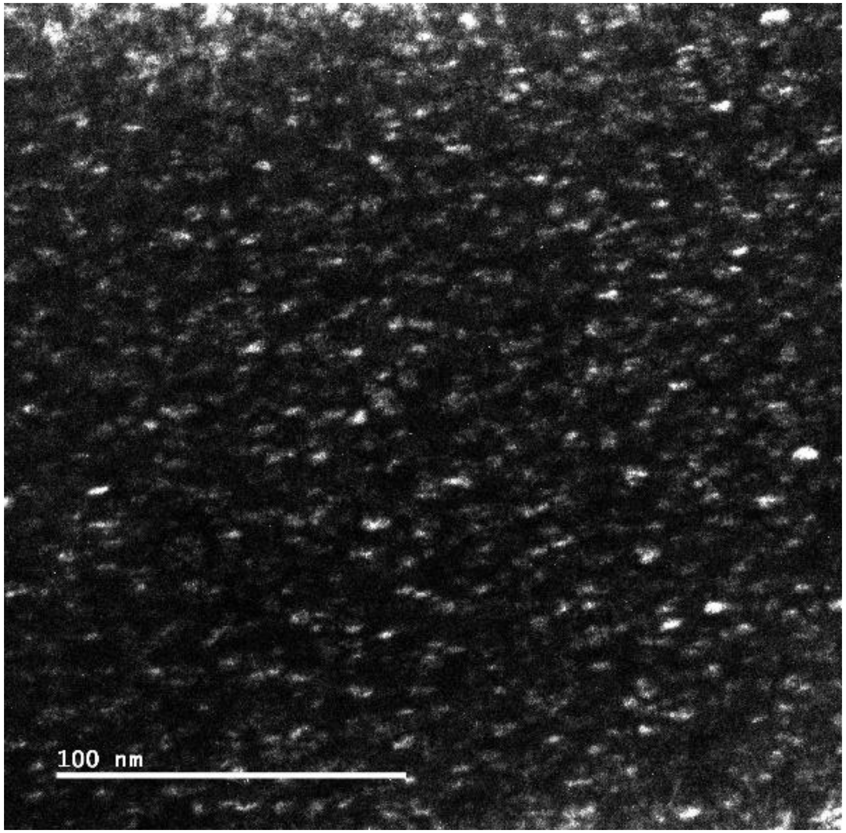
SEM-EDS analysis identified the isolated second-phase particles at grain boundaries as Nb-rich carbides.

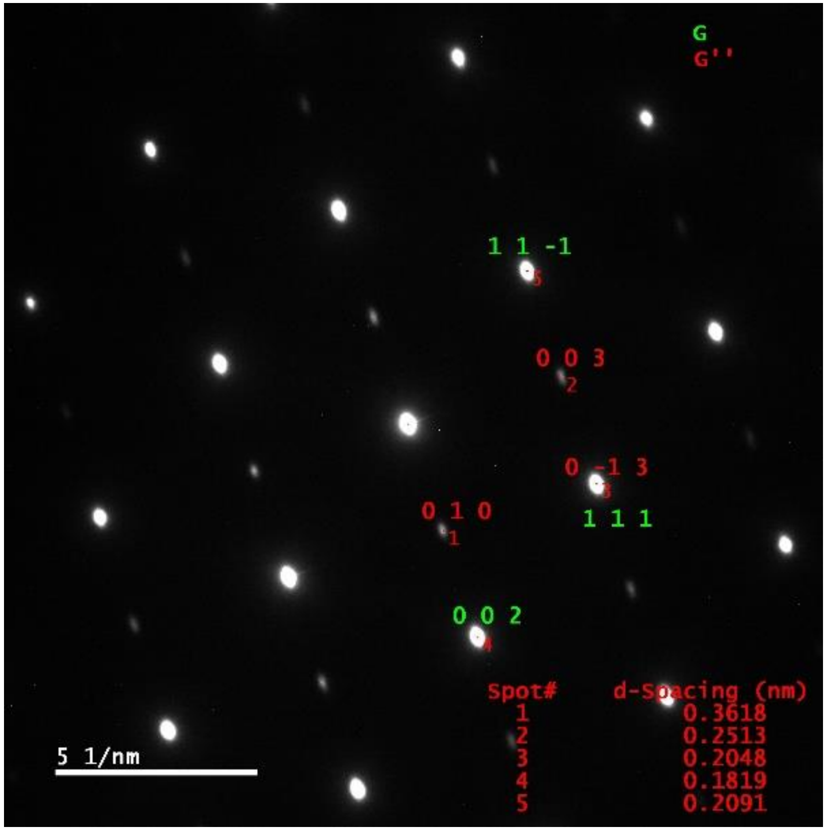


Result Type	Weight %
Elements	Spectrum
C	25.80
Cr	16.24
Fe	2.64
Ni	43.23
Nb	4.61
Mo	7.70

Forging parameters and HTs

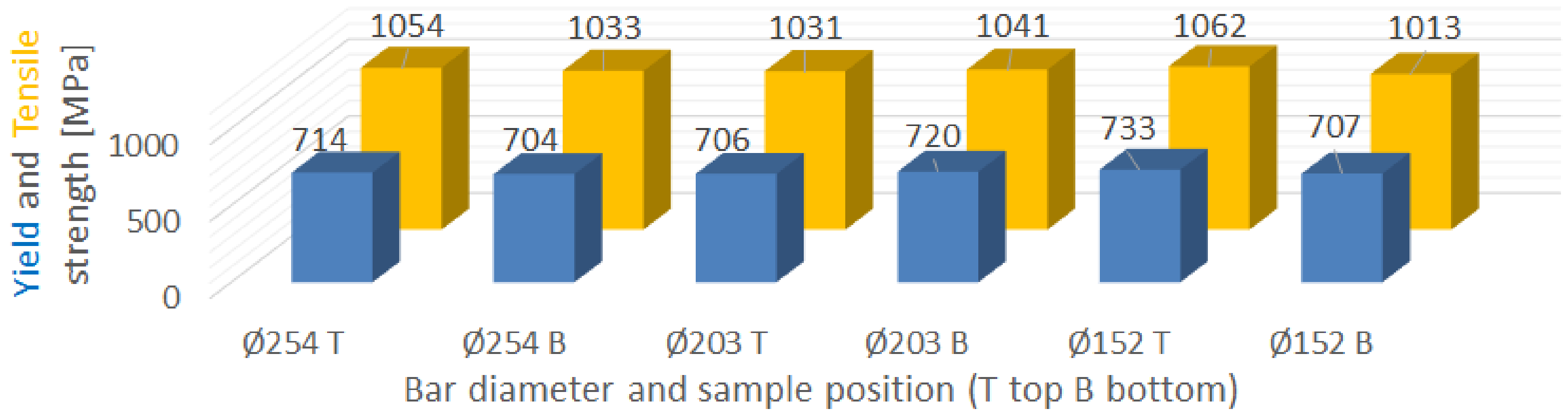
TEM diffraction patterns confirmed the presence of one phase within the grains compatible with γ'' phase (average size = 3.6 nm).

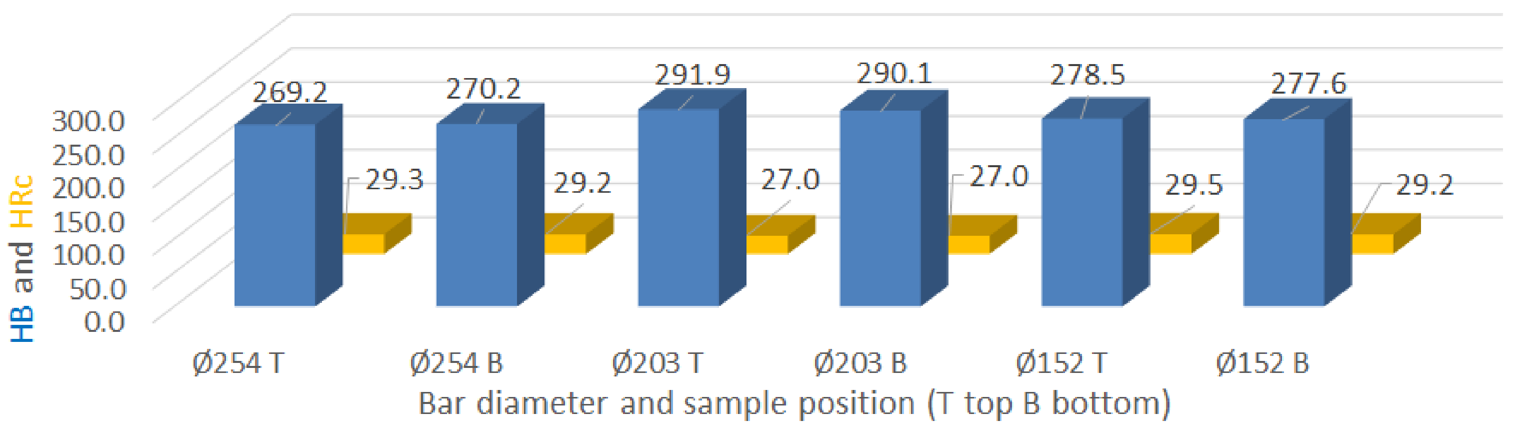




Forging parameters and HTs

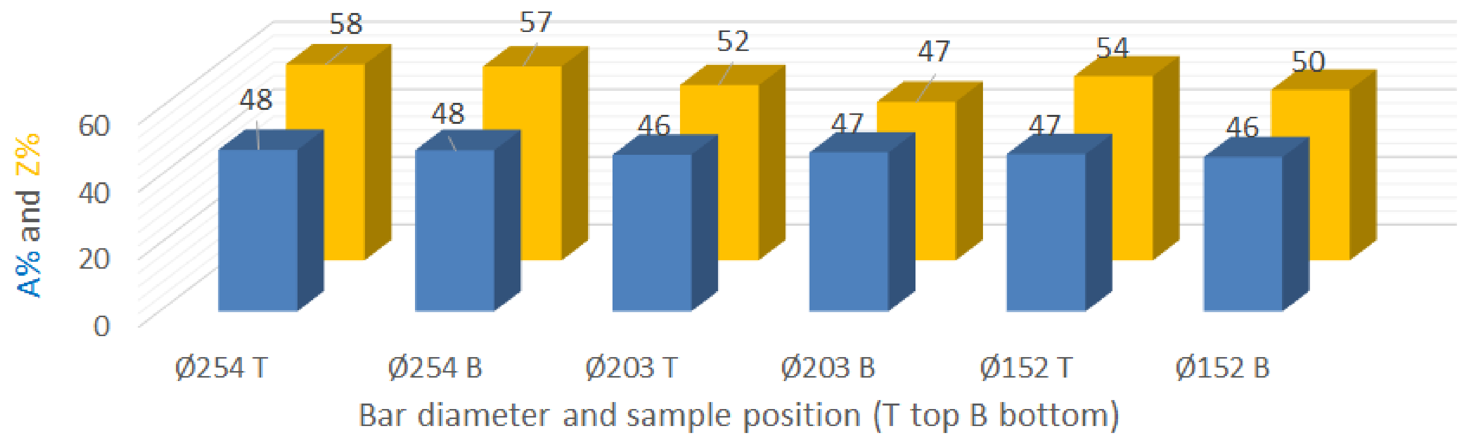
For all bar diameters and positions, the PH heat treatment significantly increases tensile strength and hardness.

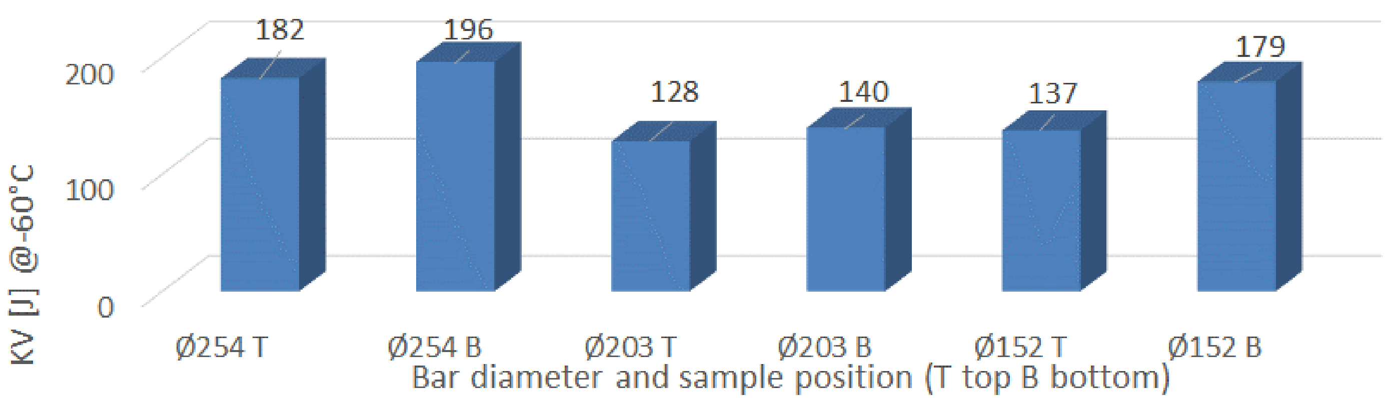




Forging parameters and HTs

On the other side, the expected decrease of ductility, expressed in terms of A%, Z% and absorbed impact energy, was quite limited.



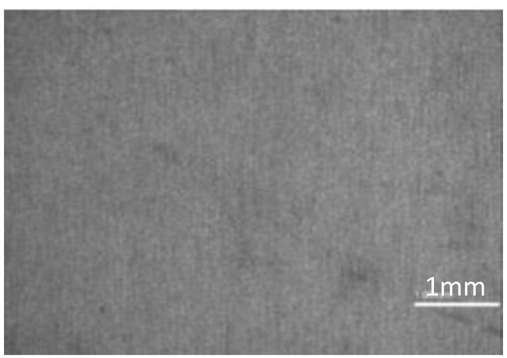


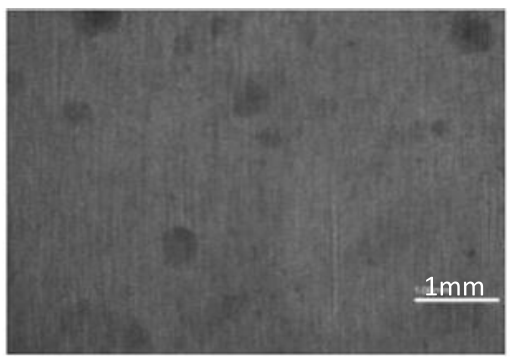
Forging parameters and HTs

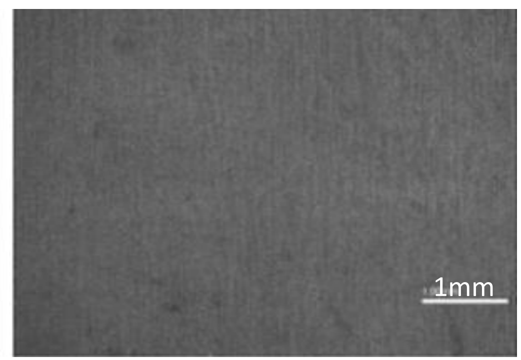
From the corrosion point of view, the age-strenghtened 625 alloy gave excellent results, comparable to the annealed condition.

Every bar passed the C-ring test, performed in the SCC- NACE VII environment → no cracks were detected after more than 2000 h.

Test	Number of bar materials	Total Number of alloy 625 specimens	Temperature (°C)	Test duration (hours)	Environment	Applied stress (%AYS)
SSC NACE VII	3	9 C-ring + 3 calibration C-ring	205	2160	180000 ppm Cl^- $ppCO_2$: 35 bar ppH_2S: 35 bar	100





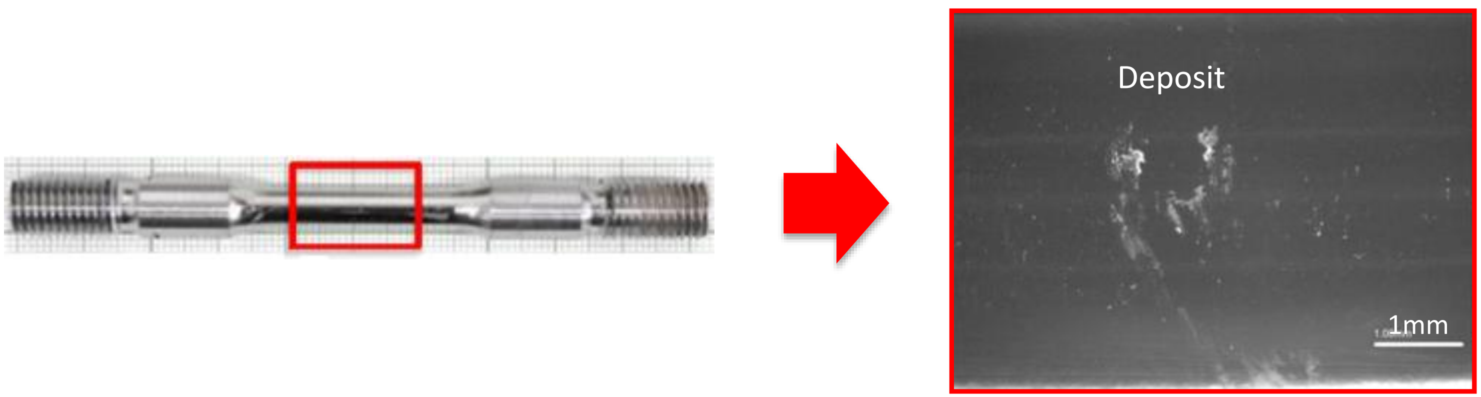


Forging parameters and HTs

The aged 625 alloy also passed the Dead weight test carried out to evaluate the resistance to SSC (sulphide stress corrosion cracking).

After more than 700 h, stereo-microscope analysis of samples surface showed the absence of cracks.

Test	Alloy 625	Number of alloy 625 specimens	Temperature (°C)	Test duration (hours)	Test gas	Solution	Applied stress (%AYS)
SSC		3	24	720	1 bar H_2S	NACE A	90
		3					
		3					



Forging parameters and HTs

In conclusion:

- The optimized precipitation hardening (PH) parameters were defined for 625 alloy forged bars.
- PH treatment produced a strong increase of yield strength respect to the annealed condition, without loss of ductility.
- NACE SCC and SSC corrosion tests were successfully passed.
- Mechanical and corrosion properties have not been influenced by bar diameter (from 6 to 10 inches).
- 625 alloy forged bars in PH condition are suitable for Oil & Gas applications.

THANK YOU FOR YOUR KIND ATTENTION!

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