MIDTERM-WORKSHOP

The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N° 958374.

Optimized Cr-steels for Creep Resistance

Lorena M. Callejo - TECNALIA

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OBJECTIVES & APPLICATIONS

- To develop novel Cr-steel grades with improved creep resistance (by 15%) with respect to standard martensitic steel grades, such as X20, P91, P92, T115 (reference)
- To optimize heat treatment conditions for an improved control of the microstructure and precipitates (carbides, Laves phase, etc) during tempering

Oil and gas, chemical and petrochemical, power generation, heat transfer, automotive, mechanical and construction industries

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CASE STUDY

Petrochemical use case description (TUPRAS)	
Industry overview	**Main targets of the demonstration**
Location: Izmit /(Turkey) **Sector**: Petrochemical **Main product:** Gasoline., diesel, LPG **Production figures:** - Processing of 1 million tons/year crude oil **Installation figures:** - Hydrocracker Unit - Distillation crude oil unit	- To improve the service life of pipelines and components within the petrochemical installation due to increased corrosion and erosion resistance of PDC coatings applied in the interior side of equipment and piping components. - To improve the energy efficiency of the boiler due to a reduction of heat required to produce steam by using Cr-steel pipes with higher creep resistance that enable to use reheater tubes with lower wall thickness. - To improve efficiency of the oil refinery process by generating steam inside the boiler with less fuel consumption due to reduce corrosion of the boiler steel pipes made of new Cr-steel. - To reduce CO_2 emissions due to less energy required to generate steam in the boiler inside the petrochemical plant and release throughout the installation due to failure of piping components.

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Tüpraş

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DILATOMETRY→LAB SCALE→PREINDUSTRIAL SCALE

Laboratory castings

Forging

Dilatometry:
Design processing routes
Characterization
Select processing route

Lab scale:
Application selected processing route
Characterization
Select steel & process

Preindustrial scale:

PRODUCE

selected steel
&
selected processing route

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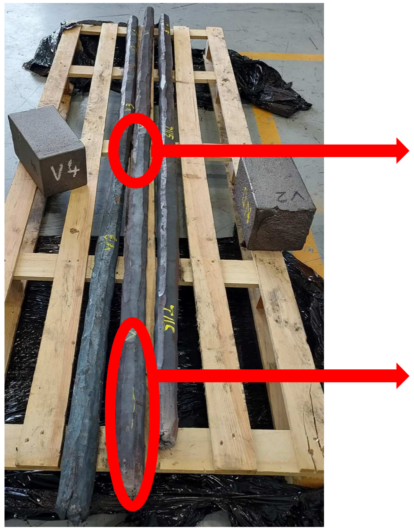
LABORATORY CASTINGS

- **Laboratory ingot castings** of 4 new Cr-steel grades (1-30 kg ingots) WP2
- **T115 steel grade** (reference) will be also cast at lab-scale for comparison
- **Analysis of chemical compositions**

Element	T115 (wt-%)	V1 V2 V3 V4 (wt-%)
Fe	Balanced	Balanced
Al	0.015 max	0.015 max
Cr	10.4-11.2	10.4-11.2
Cu	0.2 max	0.2 max
Mn	0.35-0.55	0.35-0.55
Mo	0.4-0.5	0.3-0.6
Nb	0.02-0.06	0.02-0.17
Ni	0.1-0.2	0.1-0.2
Si	0.20-0.40	0.20-0.40
Ti	0.01 max	0.01-0.050
V	0.19-0.24	0.15-0.27
W	0.1 max	0.1 max
B	0.001 max	0.001-0.0045
C	0.08-0.12	0.04-0.12
N	0.035-0.065	0.035-0.11
P	0.02 max	0.02 max
S	0.010 max	0.010 max

- **Forging** each ingot into a bar of 45 mm in diameter



- **Cutting** into 2 sets of samples

1st set of samples
Design processing routes:
Dilatometry + Characterisation
Decide the optimum processing route

2nd set of samples
Apply the optimum processing route:
Processing at lab-scale

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DILATOMETRY STUDY

DILATOMETRY:

NORMALIZING → CRITICAL TEMPERATURES → THERMAL TREATMENTS DESIGN

THERMAL TREATMENTS APPLICATION → CHARACTERIZATION → SELECTION OF THERMAL TREATMENTS

⇩

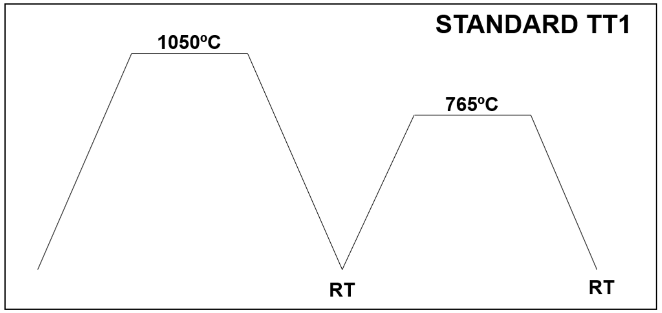
Standard thermal treatment TT1, similar for all the steel grades: based on a normalizing & tempering treatment

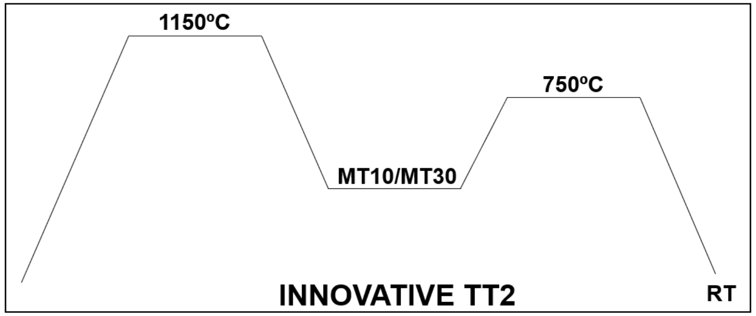
Two innovative thermal treatments TT2, also based on a normalizing & tempering treatment but interrupted by a martensitization step at:
i) MT10 to retain 10% of austenite
ii) MT30 to retain 30% of austenite

Name of the steel grade-TT1
Name of the steel grade-TT2-MT10
Name of the steel grade-TT2-MT30

Martensitization temperatures (ºC) at which a 10% of austenite (MT10) or a 30% of austenite (MT30) is retained

T115		V1		V2		V3		V4	
MT10	MT30	MT10	MT30	MT10	MT30	MT10	MT30	MT10	MT30
271	307	289	323	303	337	286	321	283	319





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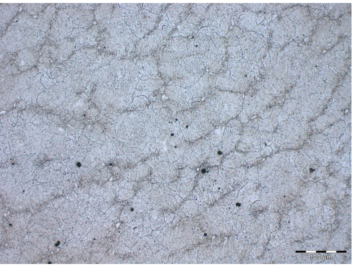
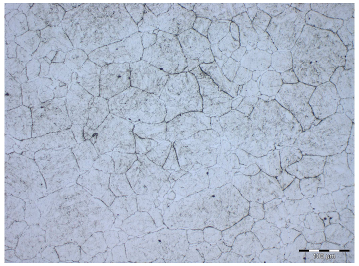
DILATOMETRY SAMPLES

Microstructure of the different steel grades after the thermal treatments applied by dilatometry:

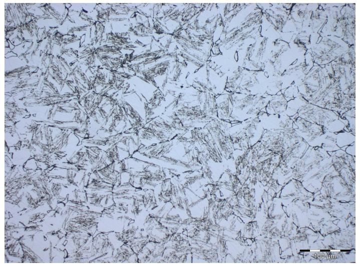
T115-TT1



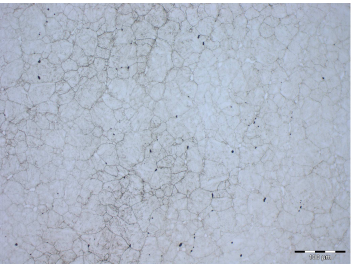
T115-TT2-271



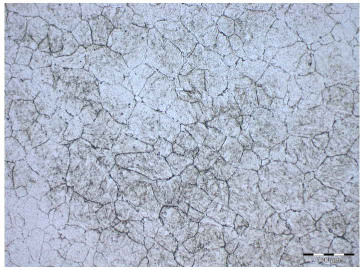
T115-TT2-307



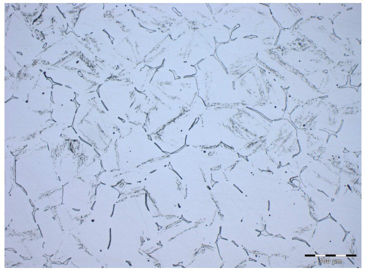
V1-TT1



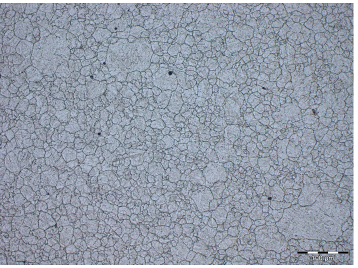
V1-TT2-289



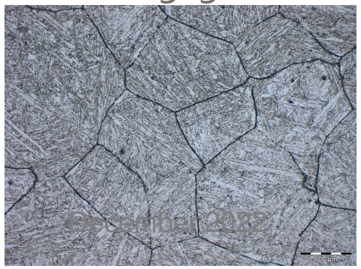
V1-TT2-323



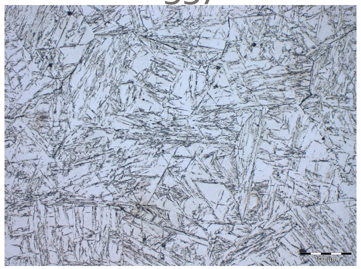
V2-TT1



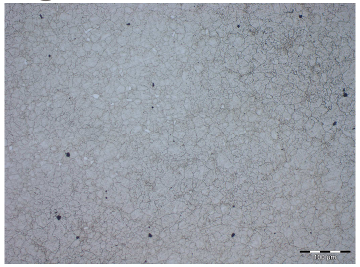
V2-TT2-303



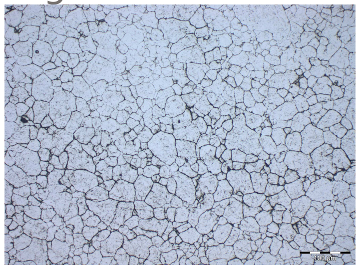
V2-TT2-337



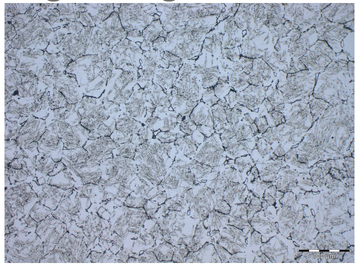
V3-TT1



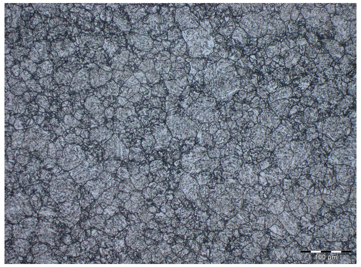
V3-TT2-286



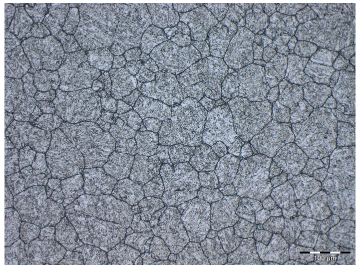
V3-TT2-321



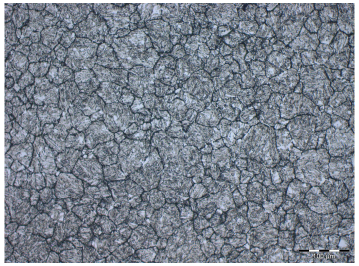
V4-TT1



V4-TT2-283



V4-TT2-319



For all the samples, TT1 & TT2-MT10: tempered martensite within PAGs of different size.

TT2-MT30: tempered martensite and fresh martensite are formed within PAGs of different size.

In particular, in V2-TT2-337, very angular tempered and fresh martensites are observed.

For high MT TT2, ferrite traces are found in the PAG boundary of T115, V1, V3 dilatometry samples.

DILATOMETRY SAMPLES

Vickers hardness (HV100) of the dilatometry samples after thermal treatments:

T115-TT1	**V1-TT1**	**V2-TT1**	**V3-TT1**	**V4-TT1**
251.94	255.54	254.58	236.56	236.86
T115-TT2-271	**V1-TT2-289**	**V2-TT2-303**	**V3-TT2-286**	**V4-TT2-283**
296.74	277.56	279.38	288.06	298.94
T115-TT2-307	**V1-TT2-323**	**V2-TT2-337**	**V3-TT2-321**	**V4-TT2-319**
318.54	371.16	377.18	308.75	313.72

For all the steel grades, the introduction of a martensitization step increases the hardness.
Especially for higher MT (MT30 vs. MT10) due to more retained austenite and thus a higher percentage of fresh martensite after tempering.

Based on hardness, the higher MT thermal treatment (TT2-MT30) was selected instead of the lower MT thermal treatment (TT2-MT10) to be applied at lab-scale.
Additionally, the standard thermal treatment TT1 for comparison.

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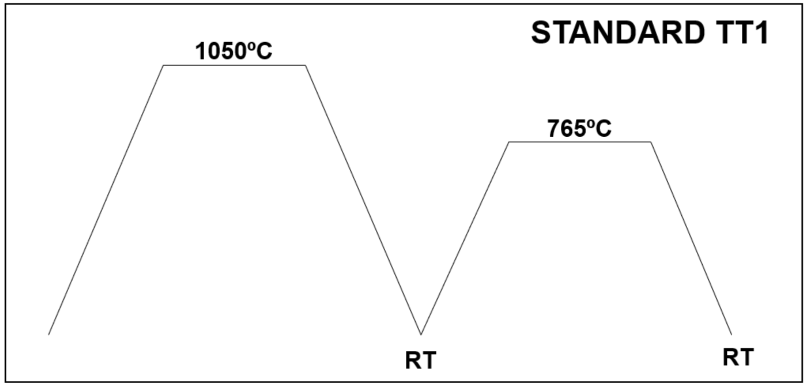
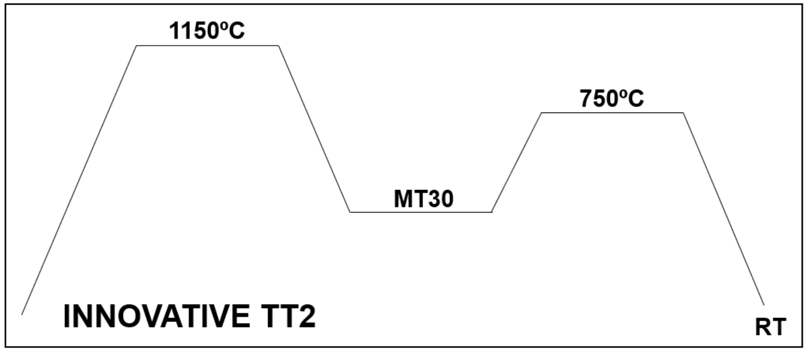
DILATOMETRY SAMPLES → LAB SCALE

APPLICATION OF SELECTED THERMAL TREATMENTS AT LAB SCALE: (optimum processing route)





- The thermal treatments selected for each steel grade (optimum processing route) are applied later on at lab-scale
- On cylinders of approximately 45 mm in diameter and 100 mm in length (second set of samples taken from the forged bars).
- By means of muffle-type furnaces and a salt-bath installation (processing at lab-scale)

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LABORATORY SCALE

Microstructural and mechanical characterization of samples after lab-scale processing

Elemental Analysis
LOM
FEG-SEM
XRD
Tensile (RT, T)
Hardness
Corrosion

Short-time creep tests at 1 temperature, and 3 different stress values. Time values for creep tests will be extended to 3300 h as a minimum. At least one of the creep tests will exceed 3300 h

Selection of steel grade (composition + processing route) offering the best overall creep behaviour
→ → for fabrication at preindustrial scale

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LABORATORY SCALE

LAB SCALE CYLINDERS:

T115, V1, V2, V3, V4 with TT1 & TT2: 10 lab samples:

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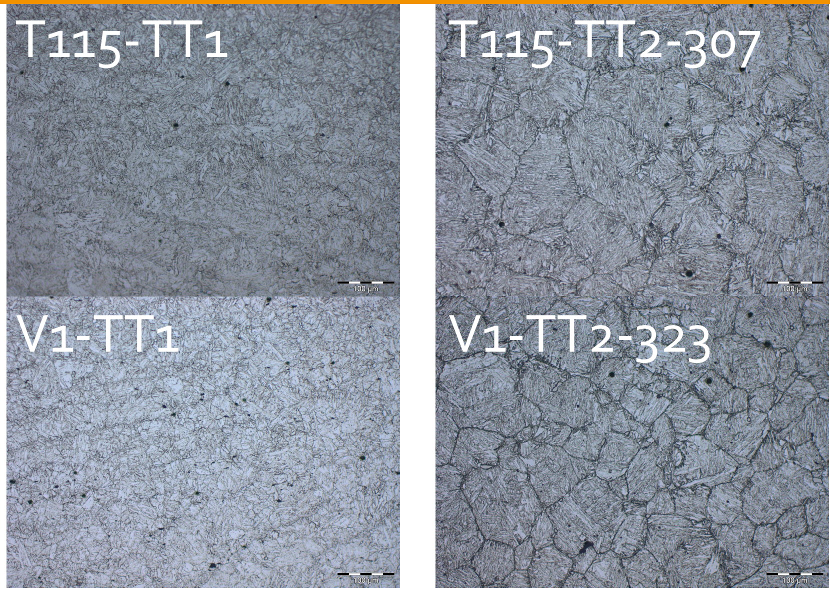
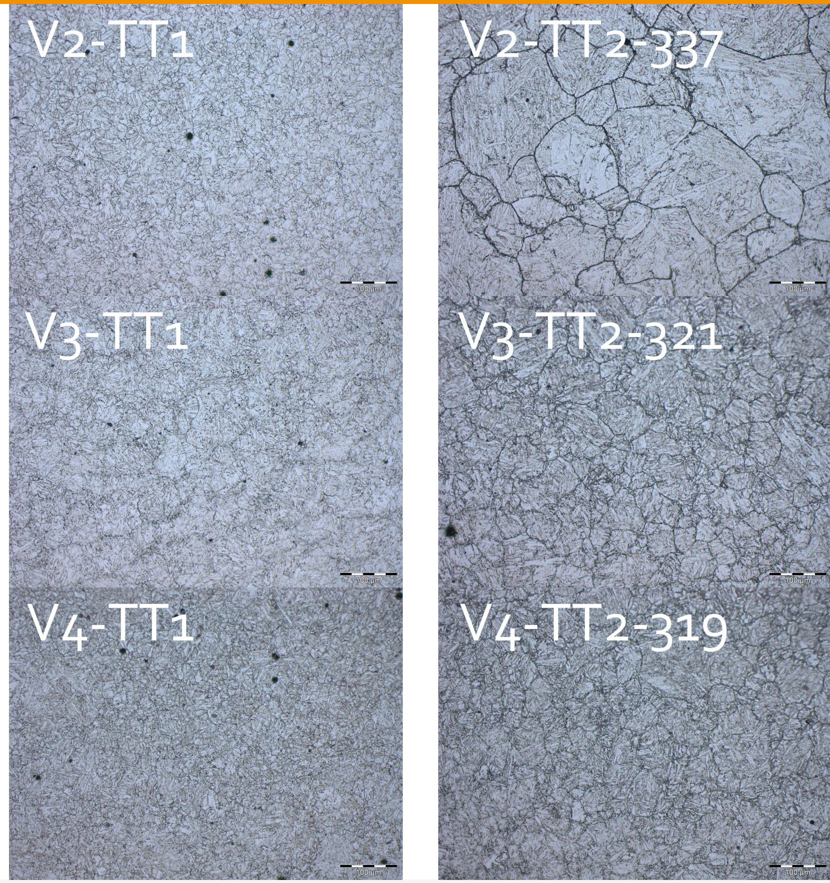
LABORATORY SCALE

LAB SCALE MICROSTRUCTURES SIMILAR TO DIL





LAB SCALE MICROSTRUCTURES: TT2 larger PAGS than TT1
Fresh & Tempered Martensite

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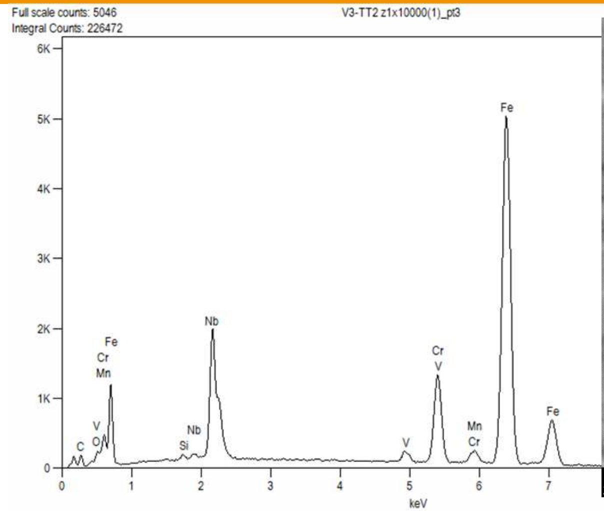
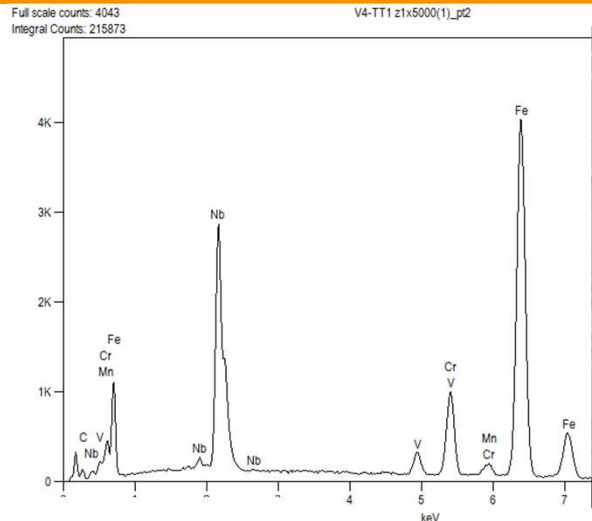
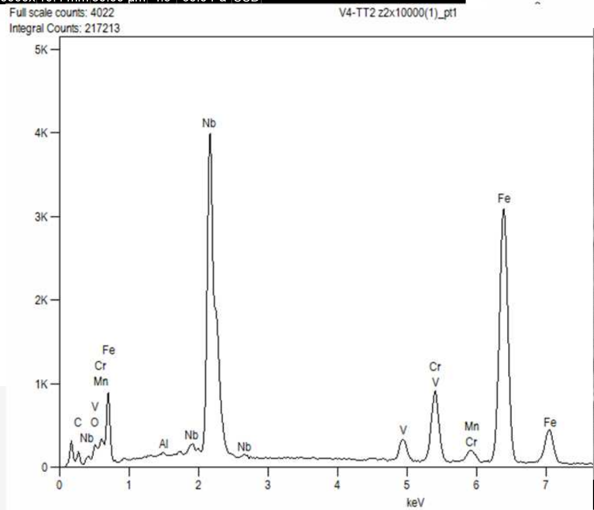
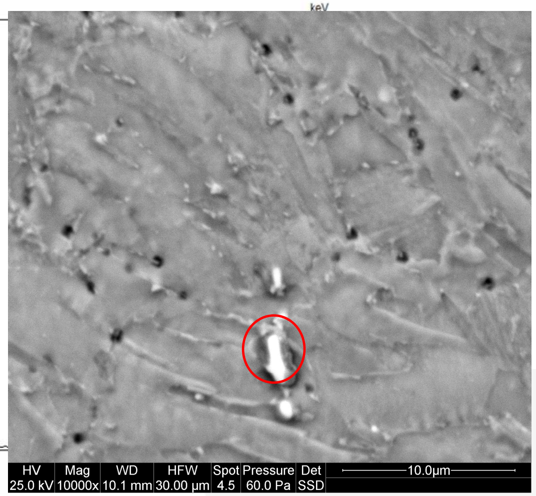
LABORATORY SCALE





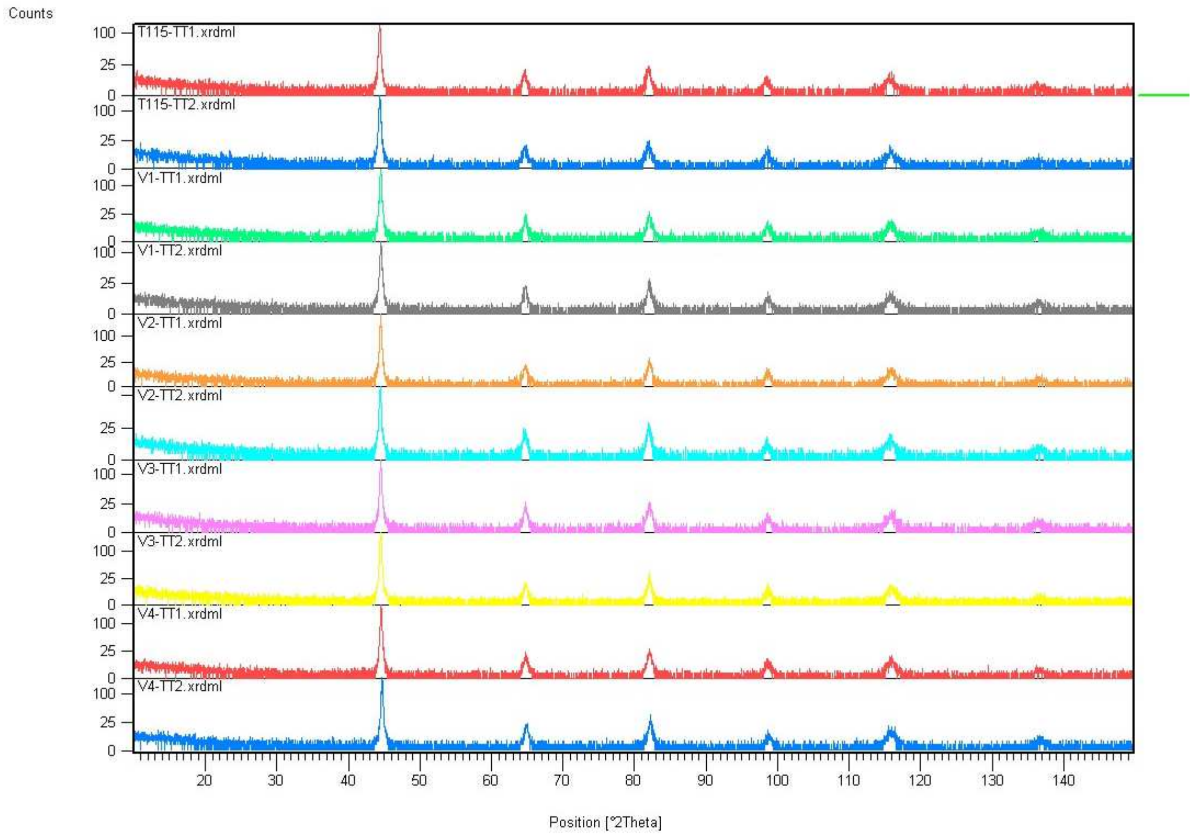
Confirms:
Martensitic microstructure
Nb-precipitates in V3 & V4





LABORATORY SCALE

Martensite



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LABORATORY SCALE

	T115-TT1	T115-TT2	V1-TT1	V1-TT2	V2-TT1	V2-TT2	V3-TT1	V3-TT2	V4-TT1	V4-TT2
HV (10)	260	266	251	**280**	243	**276**	255	258	266	262
Yield Strength (MPa)	646	627	633	**722**	621	**661**	641	629	647	600
Tensile strength (MPa)	770	801	752	**876**	736	**809**	763	795	768	777
Elongation (%)	19	21	19	**19**	19	**18**	19	20	18	19
Reduction of Area (%)	64	64	65	**65**	67	**64**	66	66	66	65

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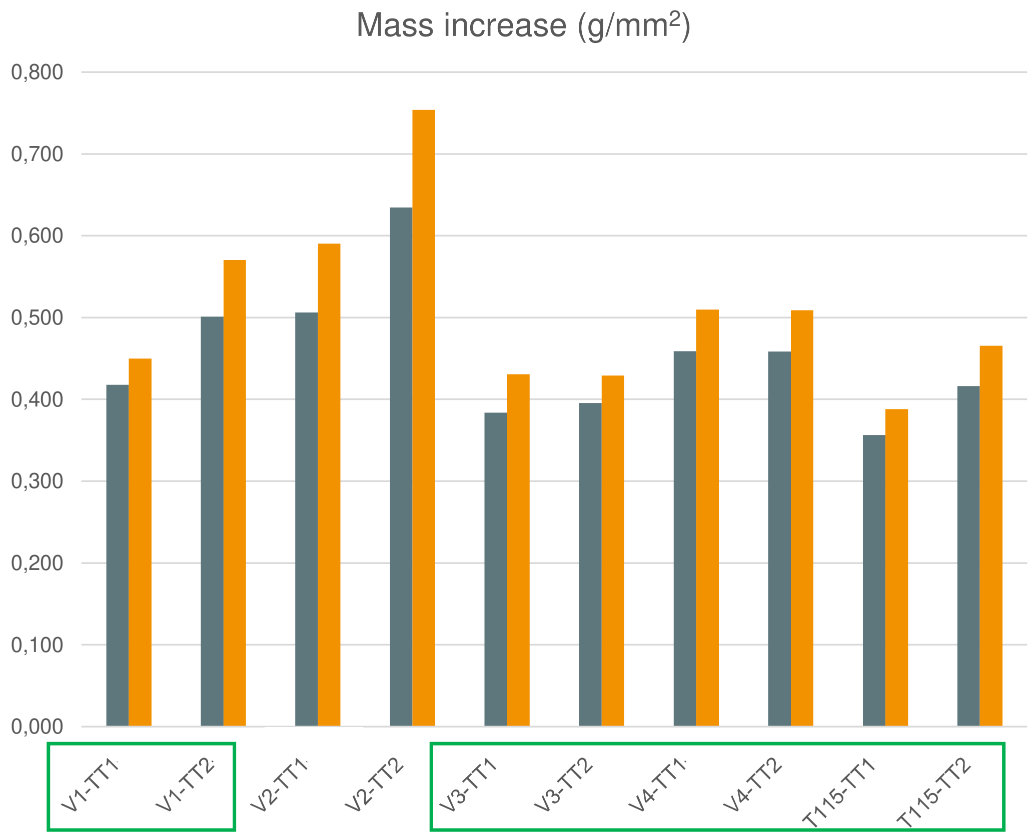
LABORATORY SCALE

Corrosion at 600ºC

Test sample	Mass increase (g/mm^2) 1000 h	Mass increase (g/mm^2) 2000 h	Mass increase between 1000 – 2000 h
V1-TT1	0.418	0.450	0.032
V1-TT2	0.501	0.570	0.069
V2-TT1	0.506	0.590	0.084
V2-TT2	0.635	0.754	0.119
V3-TT1	0.384	0.431	0.047
V3-TT2	0.396	0.429	0.034
V4-TT1	0.459	0.510	0.051
V4-TT2	0.458	0.509	0.051
T115-TT1	0.357	0.388	0.031
T115-TT2	0.416	0.465	0.049



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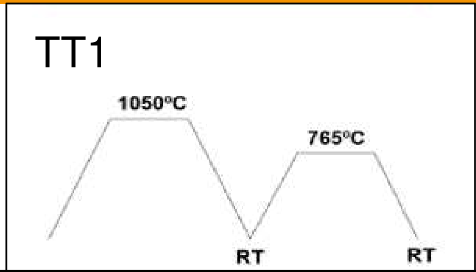
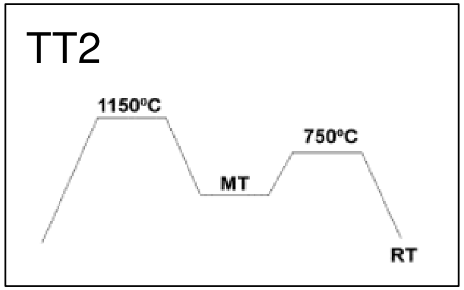
LABORATORY SCALE

Short-time creep tests at 650ºC, and 3 different stress values



Steel grade	TT1	σ(MPa)	Time (h)	Elong. (%)
T115 - ref	TT1	115	591.5	9.17
T115 - ref	TT1	88	3726	15.59
T115 - ref	TT1	72	4590.75	1.804
V1	TT1	115	1297.75	7.13
V1	TT1	88	2695.5	0.944
V1	TT1	72	2695.5	0.601
V2	TT1	115	213.75	21.44
V2	TT1	88	697.25	25.7
V2	TT1	72	1862.5	38.633
V3	TT1	115	939.75	10.45
V3	TT1	88	2372	12.58
V3	TT1	72	2695.5	1.759
V4	TT1	115	423.5	18.73
V4	TT1	88	2695.5	4.183
V4	TT1	72	2695.5	1.087



Steel grade	TT2	σ(MPa)	Time (h)	Elong. (%)
T115	TT2	115	2235.5	6.15
V1	TT2	115	4590.75	0.462
V1	TT2	88	1543	0.194
V2	TT2	115	3236.75	5.342
V2	TT2	88	1543	0.362
V3	TT2	115	2285.5	15.3249
V3	TT2	88	1543	0.411
V4	TT2	115	3745.25	12.195
V4	TT2	88	1543	0.306

Still in course with too room for elongation improvement

Finished
In course

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LAB SCALE → PREINDUSTRIAL SCALE

Selection of Best steel grade (composition + processing route) offering the best overall creep behaviour

Fabrication at Preindustrial scale

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PREINDUSTRIAL SCALE

Pre-industrial castings (700 kg) and chemical analysis

- 1 Best candidate steel grade
- 1 Reference steel grade for comparison
- Chemical analysis

Pre-industrial tubes (700 kg): ***Rolling + Thermal Treatment***

- 700 kg ingots will be used to fabricate pilot-tubes with a thickness in the range of 7 to 10 mm (the thermal treatment route defined at lab-scale will be adapted and implemented at pilot scale including the necessary hot-rolling steps)

The pilot tubes will be validated in WP7

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PREINDUSTRIAL SCALE

PREINDUSTRIAL CASTING OF T115 REFERENCE STEEL

Composition	C	Si	Mn	P	S	Cr	Ni	Mo	Al	Cu	Ti	V	W	Nb	N
PREIND T115REF	0.08-0.12	0.20-0.40	0.35-0.55	<0.02	<0.01	10.4-11.2	0.1-0.2	0.4-0.5	<0.015	<0.2	<0.01	0.19-0.24	<0.1	0.02-0.06	0.035-0.065

AFTER SHOT BLASTING, BEFORE MACHINING & ROLLING TO PILOT TUBE

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FUTURE WORK

- Complete LAB SCALE Creep characterization
- Selection of steel grade (composition + processing route) offering the best overall behaviour FOR
- Fabrication at PREINDUSTRIAL SCALE (reproduce composition & treatment as preindustrial ingots for pilot tubes to be validated in WP7)

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Thank you

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