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EP302 Stainless Steel Procurement

S. Matera, L. Alleva, O. Tassa

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EP302 STAINLESS STEEL PROCUREMENT

S. Matera, L. Alleva, O. Tassa – CSM s.p.a.

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EP302 Stainless Steel Procurement

Ente emittente CSM Spa

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Sommario

ENEA asked CSM to perform a preliminary characterization on EP302 austenitic steel. CSM has been asked to manufacture one ingot EP302 austenitic stainless steel, to manufacture 15 mm thick hot rolled plates, and to assess microstructure and properties of these plates after a final heat treatment in order to optimize the quality of the product.
In this part of the activity two hot rolled plates with a final thickness of 15.8 mm and with dimension 145 mm x 1500 mm, in compliance with the requirements for the chemical composition of EP302 stainless steel, have been manufactured

Note

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EP302 Stainless Steel Procurement

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1. Executive Summary

In the frame of the research activities planned to support the development of a DEMO-LFR, ENEA asked CSM to perform a preliminary characterization on EP302 austenitic steel.

CSM has been asked to manufacture one ingot EP302 austenitic stainless steel, to manufacture 15 mm thick hot rolled plates, and to assess microstructure and properties of these plates after a final heat treatment in order to optimize the quality of the product.

In this part of the activity two hot rolled plates with a final thickness of 15.8 mm and with dimension 145 mm x 1500 mm, in compliance with the requirements for the chemical composition of EP302 stainless steel, have been manufactured using the following process route:

- production of one 80 kg ingot (VM2758) with the following geometries: length 300 mm, width 250 mm, thickness 125 mm whose alloy composition in wt% (0.127 C, 2.50 Si, 0.51 Mn, 15.1 Cr, 8.8 Ni, 0.87 Nb) satisfies the requirements for EP302 stainless steel;
- hot rolling of the two semi-ingot after homogenization annealing (1180 °C for 60 minutes) using an hot rolled schedule consisting of 8 passes to achieve a 35 mm plate, reheating at 1180°C with an holding time of 20 minutes and then 5 passes to achieve 15 mm final thickness. Reduction, force and temperature were respectively in the range 10÷16 %, 46÷85 t, 995÷1150 °C;

Two pieces with a length of 350 mm, cut from one plate, have been heat treated at 1060°C for 40 minutes to improve the properties of the product.

2. Background

The Lead cooled Fast Reactor (LFR) technology has been developed in Europe in the framework of the FP-6 European Lead cooled System (ELSY) and the subsequent FP-7 Lead-cooled European Advanced Demonstration Reactor (LEADER) projects. Among the open issues related to the LFR demonstration, the cladding material assessment is one of the most crucial question still under investigation.

Austenitic - high Si - stainless steel EP302 has been selected for the application as cladding material in the LFR for its promising performance in high temperature lead and under irradiated conditions (radiation resistance, mechanical properties, fuel–clad compatibility).

In the frame of the research activities planned to support the development of a DEMO-LFR, ENEA asked CSM to perform a preliminary characterization on EP302 austenitic steel.

CSM has been asked to manufacture one ingot EP302 austenitic stainless steel, to manufacture 15 mm thick hot rolled plates, and to assess microstructure and properties of these plates after a final heat treatment in order to optimize the quality of the product.

3. Scope of work

Within the aims of the ENEA project, devoted to the implementation of the manufacturing process for the production of EP302 austenitic stainless steel plates, CSM role is concerning to the production at pilot plant scale of material and its mechanical, microstructural assessment properties.

4. Planned activities

The whole work program is summarized in the following Table 1.

Task	Parameters	Remarks
1 Production of 1 ingot (80 kg) by Vacuum Induction Melting (VIM)	Si addition	Chem Comp. supplied by ENEA
2 Metallographic characterization of as cast (lollipop) and ingot	Hardness, precipitate second phase type, morphology and location	LM and SEM-EDS
3 Manufacturing of hot rolled 15 mm thick plates- chemical composition	Homogenization annealing	hot rolled schedule implemented by CSM
4 Quality Heat Treatment (Last hardening)	Annealing/quenching	Temperature and soaking conditions implemented by CSM
5 metallographic	Grain Size, hardness, precipitate	LM , SEM-EDS

characterization of as rolled and heat treated plates	second phase type, morphology and location	
6 Mechanical Testing	10 (longitudinal) tensile tests at RT and 10 (longitudinal) tensile tests at 550°C	ASTM E21 rules (constant elongation rate)
7 Creep-tests	5 creep-to-rupture tests at 550°C	constant elongation rate and imposed stress in the range 75%-85% of the yield strength
Final report	Including all the results, tests parameters and procedures	

Table 1 – Detailed activities

Tasks from 1 to 4 have been completed.

5. Experimental procedure

The main data of the procedures adopted in the laboratory activities are reported in the following.

EP302 Ingot Production by VIM

One ingot, 80 kg, of EP302 austenitic stainless steel has been cast by the Vacuum Induction Melting furnace (VIM) shown in Figure 1.
The VIM is a pilot plant for inductive melting, treating, casting and solidifying of special steels, nickel based alloys, cobalt based alloys, copper, aluminium alloys under controlled conditions.
The melting and casting process can be executed under vacuum, inert gas or ambient (air) pressure.
Charging, measuring of temperature and sampling are possible during the process via a vacuum lock valve.

The CSM VIM plant has the following main process characteristics:

Melting:	1300 – 1600 °C	(depending on alloy grade)
Atmosphere:	Vacuum, Inert gas	
Process pressure:	5×10^{-5} – 300	mbar
Overheating temperature:	1750 °C	
Temperature measurement:	750 – 1800°C (thermocouple, optical pyrometer)	
Sampling of liquid metals:	Quartz mould	
Casting mode:	Top pouring into Moulds or Ceramic shells (investment casting)	
Melting/Casting capacities:	1- 11 dm^3	

Figure 1 – VIM plant

Plate production by hot rolling

Hot rolling of the ingots to manufacture 15 mm thick plates have been performed at CSM hot rolling mill, whose picture is shown in Figure 2.
The hot rolling mill has the following characteristics:

Engine:
- Power: 445 kW
- Voltage 500 V
- Current 968 A
- RPM 1760 rpm / min

Cage Mill:
- Force up to 250 t
- Maximum speed 1.0 m / sec
- Cylinders Ø 500 mm
- Board 350 x 2400 mm

Hot rolled plates
- Maximum thickness 120 mm
- Overall width 300 mm

Figure 2 – Hot rolling mill

<u>Microstructure Investigation by LM and SEM-EDS</u>

Metallographic specimens were used for Light Microscopy (LM) characterisation. Sections of samples were embedded in epoxy (ethoxylen) resin and underwent metallographic preparation by emery paper and diamond polishing using diamond paste of 3µm, 1µm and 0.25µm grades. The microstructure characterisation was performed on the metallographic specimens after etching in an electrochemical solution of HNO_3 in water. Hardness (HV_{10}) measurements have been also performed. SEM- EDS analysis was used for characterization of second phases (morphology and chemical composition).

6. Results

6.1. Production of ingot

One ingot (Figure 3) has been produced with the following geometries: length 300 mm, width 250 mm, thickness 125 mm. The relevant data of the VIM process are collected in the Table 2. The in line chemical analysis is reported in the Table 3.

Figure 3 – VIM ingot EP302 stainless steel VM2758

Tme	Action	Temp [°C]	Power [kW]	Pressure [mbar]
12.00	Power on		20	0.11
13.30	Heting up Fe,C,Cr,Ni,Nb		50-80	0.11
13.35	Melting start + Argon addition		80	0.11/100
13.53	All charge melted	1520	35	100
13.56	Degassing			100
14.05	Degassing end + Argon addition			0.24/100
14.06	Si,Mn addition		40	100
14.09	Degassing		42	0.15/150
14.18	Degassing end	1530	42	150
14.20	1° sample		28	150
14.41	Addition Ni,Cr,Nb		33	150
14.45	2° sample			150
14.48	casting	1490	26	150

Table 2 – VIM process parameters of VM2758 ingot

EP302	**C**	**Si**	**Mn**	**Cr**	**Ni**	**Nb**	**P**	**S**
VM2758	0.127	2.58	0.51	15.10	8.8	0.87	<0.01	0.0056

Table 3 – Chemical composition (wt%) of VM2758 ingot

The alloy composition of VM2758 satisfies the requirements in wt% for EP302 stainless steel.

6.1.1. Metallographic characterization of as cast (lollipop sample)

LM analysis of as cast sample has shown the solidification microstructure characterized by dendritic delta ferrite phase within austenite. Mean hardness value was 175 HV_{10}.

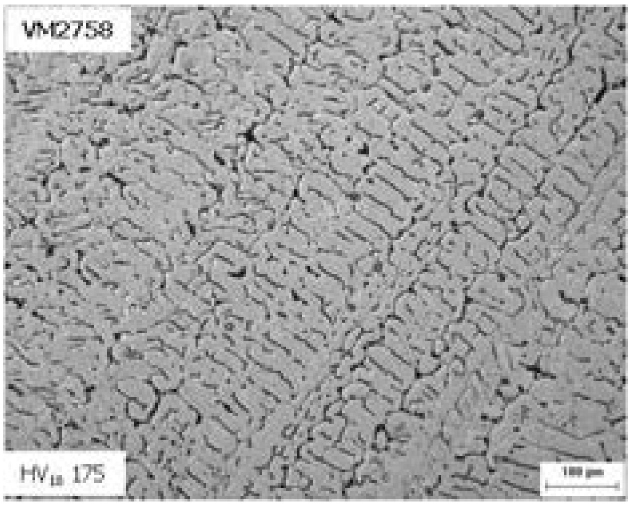


Figure 4 – LM solidification microstructure of EP302 stainless steel VM2758

SEM-EDS analysis has shown also the presence of NbC second phase with eutectoid morphology in addition to delta ferrite (Cr-rich phase) phase (Figures 5-6).

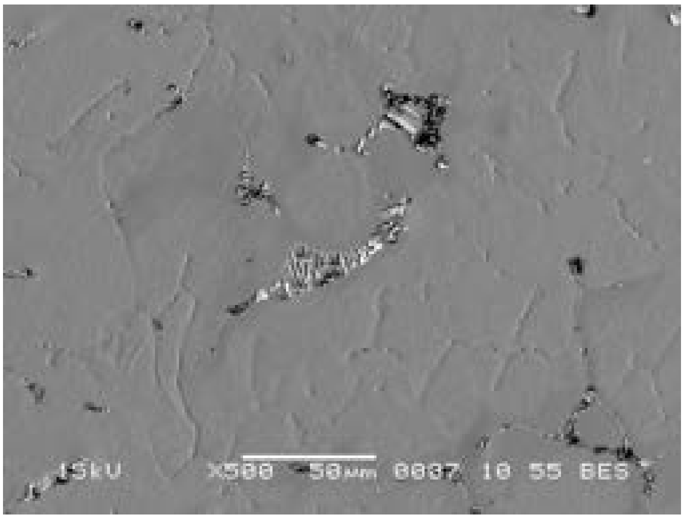
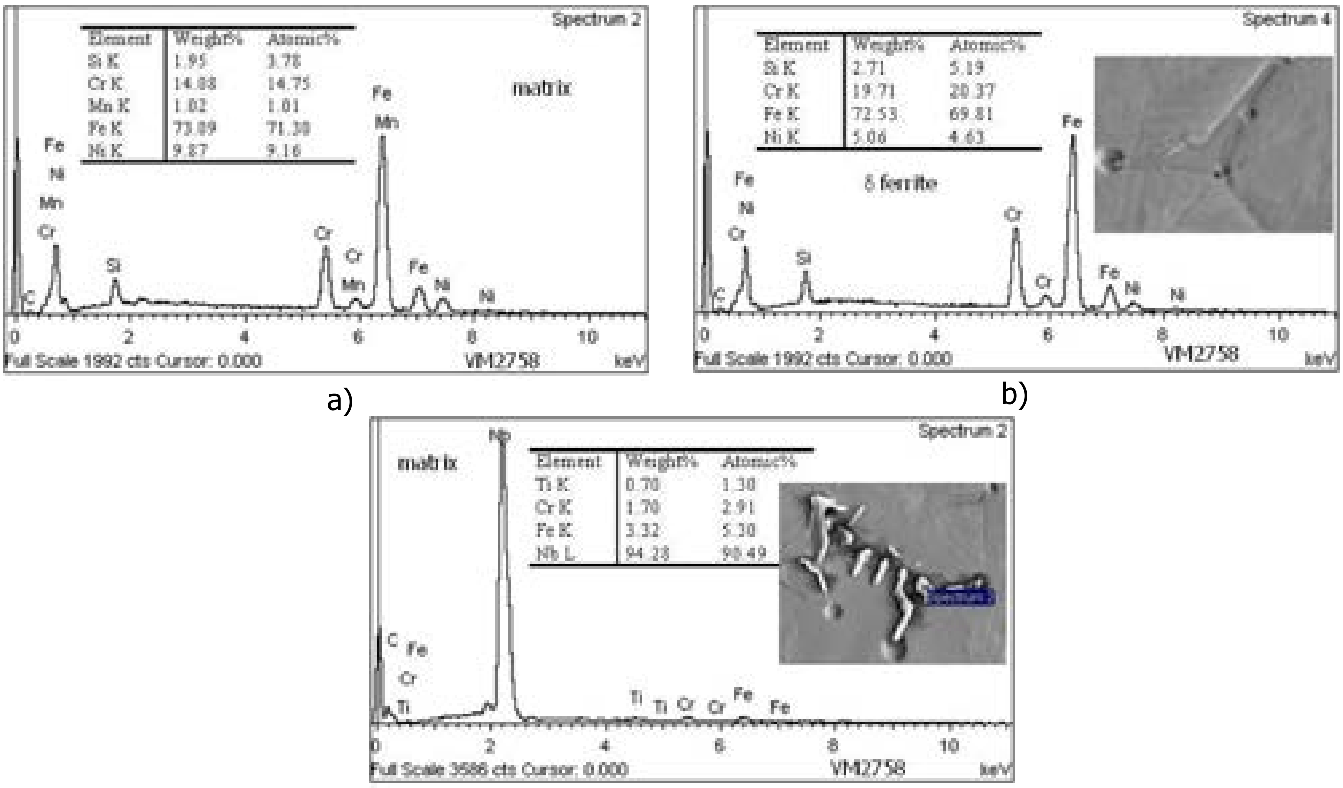


Figure 5 – SEM (BES) solidification microstructure of EP302 stainless steel VM2758



c)

Figure 6 – SEM-EDS spectrum of phases in EP302 stainless steel VM2758: a) matrix; b) δ-ferrite: c) NbC carbide.

6.2. Manufacturing of hot rolled plates

The produced ingot VM2758 has been machined to achieve parallel surfaces and has been cut in two parts (semi -ingot 2758 A and 2758 B). Semi-ingot geometry is shown in Fig. 7.

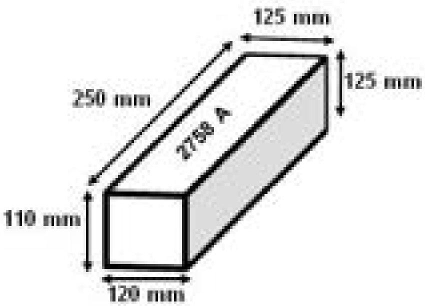


Figure 7 – Scheme of semi-ingot 2758 A

The following thermal treatment has been performed before hot rolling on the two semi-ingots:

- ingot inserting in the furnace at 1180°C and holding at 1180°C for 60 minutes (homogenization annealing) (Figure 8)

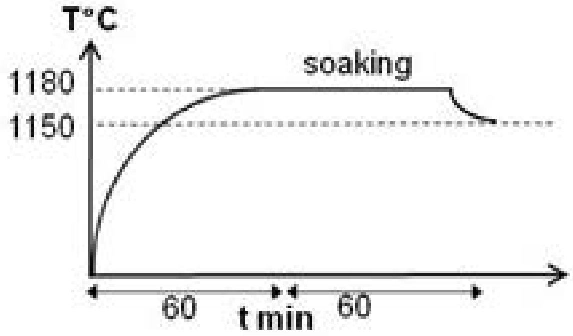


Figure 8 – Homogenization annealing cycle before hot rolling

The hot rolling schedule has been the following:

a) 8 passes to achieve a 35 mm plate,
b) reheating at 1180°C with an holding time of 20 minutes,
c) 5 passes to achieve 15 mm final thickness plates.

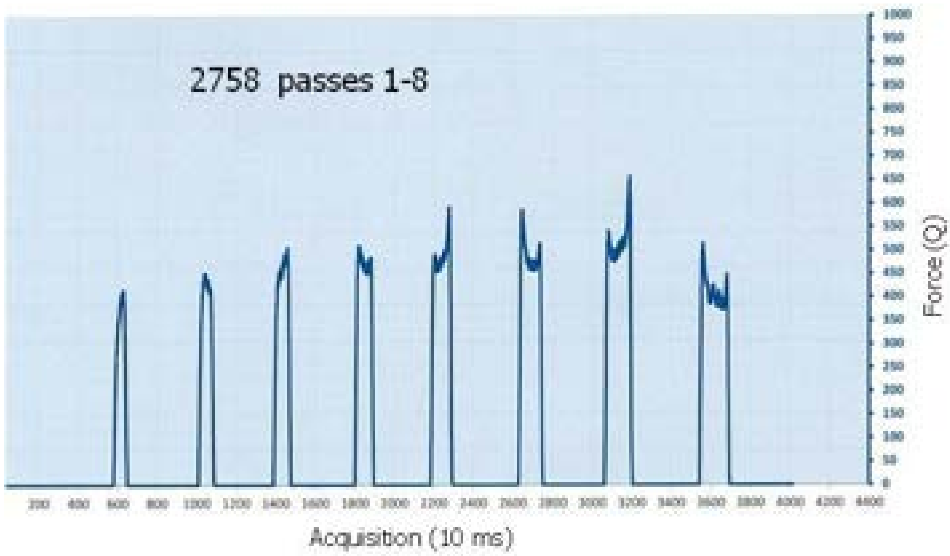
Hot rolling schedules are shown in Tables 4-5. Hot rolling temperature has been in the range 1150°C÷990°C. Force values during hot rolling of semi-ingot 2758 are shown in Figure 9.

Hot rolled plate 2758 A							
semi ingot (mm)	**N° pass**	**temperature (°C)**		**red %**	**force (t)**	**thickness mm**	
		heating	pyrometer			start	finish
110 X 120 x 250	1	Homogenization annealing 1180°C soaking 60 minutes	n.d.	15.0	41.5	120.0	102.0
	2		1091	15.0	45.1	102.0	86.7
	3		1046	15.0	50.6	86.7	73.7
	4		1036	15.1	51.1	73.7	62.6
	5		1026	15.0	59.3	62.6	53.2
	6		1022	14.8	58.7	53.2	45.3
	7		1015	15.0	65.7	45.3	38.5
	8		1005	9.9	51.4	38.5	34.7
	9	Reheating 1180°C soaking 20 minutes	nd	17.0	58.6	34.7	28.8
	10		1024	16.0	60.8	28.8	24.2
	11		1012	16.1	72.3	24.2	20.3
	12		1001	15.8	75.7	20.3	17.1
	13		996	16.4	85.6	17.1	14.3
effective final thickness							15.8

Table 4 – Hot rolling schedule of semi-ingot 2758 A

Hot rolled plate 2758 B							
semi ingot (mm)	N° pass	temperature (°C)		red %	force (t)	thickness mm	
		heating	pyrometer			start	finish
110 X 120 x 250	1	Homogenization annealing 1180°C soaking 60 minutes	n.d.	17.0	46.5	120.0	99.6
	2		1112	17.0	52.0	99.6	82.7
	3		1089	16.1	64.8	82.7	69.4
	4		1048	16.0	56.4	69.4	58.3
	5		1026	16.0	64.7	58.3	49.0
	6		1021	15,9	65.2	49.0	41.2
	7		1016	16.00	74.3	41.2	34.7
	8	Reheating 1180°C soaking 20 minutes	nd	17.0	50.4	34.7	28.8
	9		1048	16.0	55.2	28.8	24.2
	10		1021	16.1	68.4	24.2	20.3
	11		1006	15.8	70.9	20.3	17.1
	12		994	16.4	84.7	17.1	14.3
effective final thickness							15.8

Table 5 – Hot rolling schedule of semi-ingot 2758 B



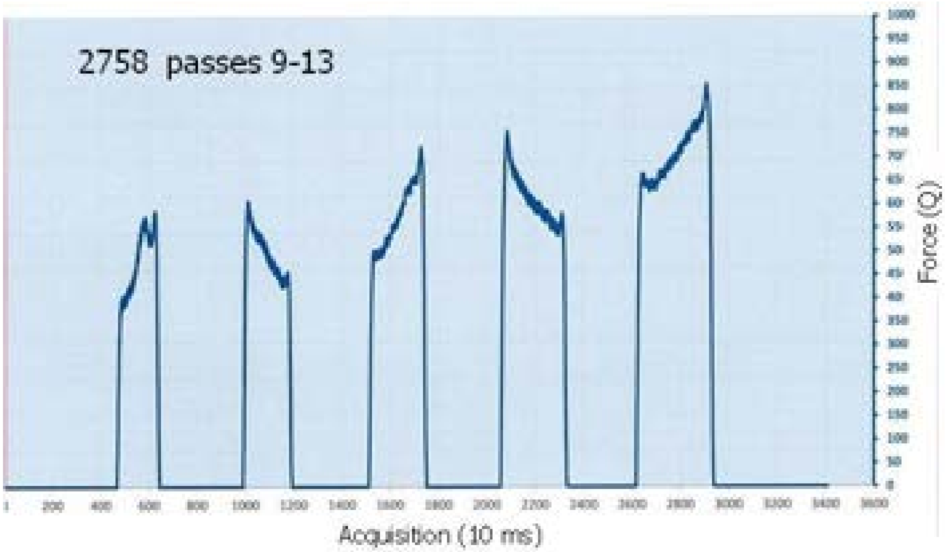


Figure 9 – Force during hot rolling of semi-ingot 278 A

Two plates with a final thickness of 15.8 mm and with dimension 145 mm x 1500 mm have been manufactured (see picture in Figure 10).

Figure 10 – Manufactured hot rolled plates 2758 A and 2758 B

6.2.1. Chemical composition of hot rolled plates

The chemical analysis of a sample of hot rolled plate 2758 A is reported in the Table 6

EP302	C	Si	Mn	Cr	Ni	Nb	P	S
VM2758	0.125	2.60	0.52	15.10	8.83	0.88	<0.01	0.0060

Table 6 – Chemical composition (wt%) of hot rolled plate 2758 A.

6.3. Heat Treatment of hot rolled plates

The plate 2758 A has been cut in 3 pieces with a length of 350 mm. A piercing of three pieces was done for thermo coupling in order to record thermal profiles during thermal treatment.

Thermal treatment condition has been defined on the base of standard specification of TP 321, TP 347, TP 348 austenitic stainless steels for which heat treatment above 1065 °C may impair the resistance to intergranular corrosion after subsequent exposure to sensitizing condition [1]. In addition heat treatment above 1120 °C (2050 F) promotes austenitic grain growth (Figure 11) [2].

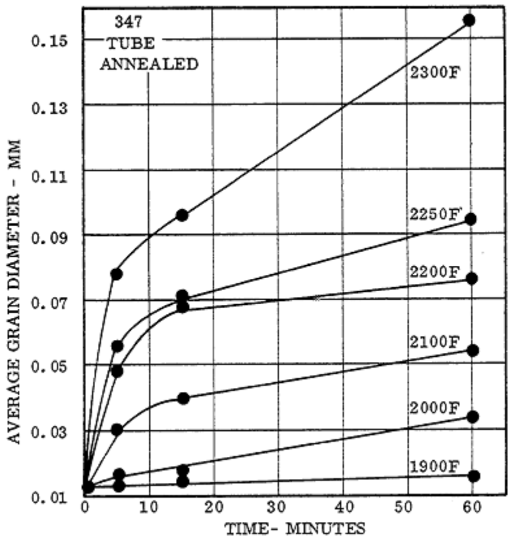


Figure 11 – Effect of time and temperature on grain growth in TP347 austenitic stainless steel [2].

The following thermal treatment has been performed on 2 pieces:

- piece inserting in the furnace at 1060°C and holding at 1060 °C for 40 minutes and the water quenching.

An example of thermal profile achieved during the treatment is reported in Figure 12.

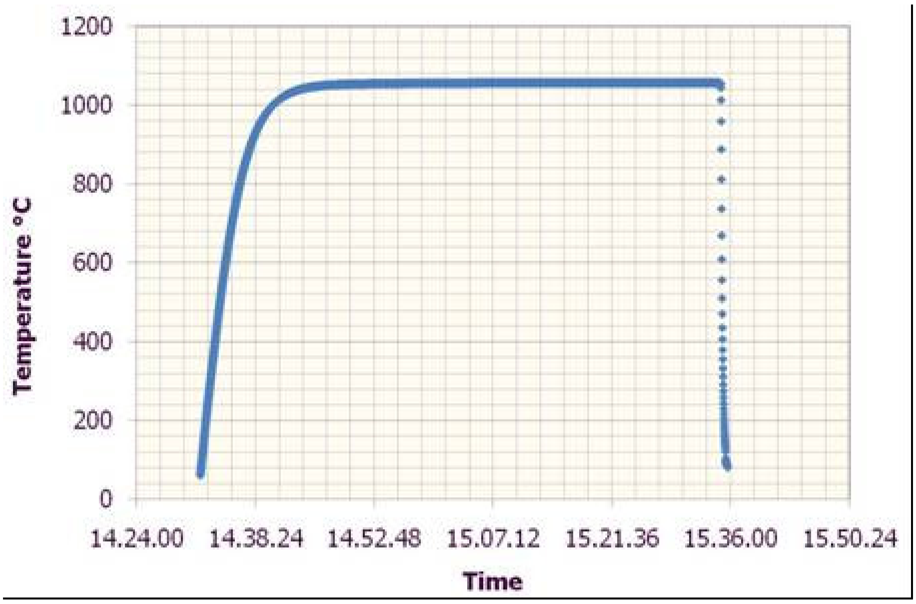


Figure 12 – Thermal profile acquired during the thermal treatment of hot rolled plate 2758 A.

7. Conclusions

Two hot rolled plates with a final thickness of 15.8 mm and with dimension 145 mm x 1500 mm, in compliance with the requirements for the chemical composition of EP302 stainless steel, have been manufactured using the following process route:

- production of one 80 kg ingot (VM2758) with the following geometries: length 300 mm, width 250 mm, thickness 125 mm whose alloy composition in wt% (0.127 C, 2.50 Si, 0.51 Mn, 15.1 Cr, 8.8 Ni, 0.87 Nb) satisfies the requirements for EP302 stainless steel;
- hot rolling of the two semi-ingot after homogenization annealing (1180 °C for 60 minutes) using an hot rolling schedule consisting of 8 passes to achieve a 35 mm plate, reheating at 1180°C with an holding time of 20 minutes and then 5 passes to achieve 15 mm final thickness. Reduction, force and temperature were respectively in the range 10÷16 %, 46÷85 t, 995÷1150 °C;

Two pieces with a length of 350 mm, cut from one plate, have been heat treated at 1060°C for 40 minutes and water quenched to enhance the properties of the product.

8. Future work

The following activities will be carried out in accordance to the agreed program described in the Technical Specification:

- Delivery of four treated plates (350 x 145 x 15.8 mm) to ENEA;
- Microstructure characterization of the as cast material;
- Microstructure characterization of as rolled and heat treated plates;
- Ten longitudinal tensile tests at room temperature and ten longitudinal tensile tests at 550°C of heat treated plates;
- 5 creep-to-rupture tests at 550°C, with a constant elongation rate and an imposed stress in the range of 75%-85% of the yield strength.

9. References

[1] ASTM A 269-04 Standard specification for seamless and welded austenitic stainless steel tubing for general service (December 2002).

[2] R. Kattus, Aerospace Structural Metals Handbook Ferrous Alloys Type 347 & 348 (December 1977).