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SUMMARY

DOCTORAL THESIS

EXPERIMENTAL RESEARCH FOR ESTABLISHMENT OF THE OPTIMUM TECHNOLOGY FOR THE EXECUTION OF FORGED SEMI-PRODUCTS WITH SPECIAL DESTINATION

Author: Drd. Eng. Valentina-Elena CALOIAN

PhD supervisor: Prof. Dr. Eng. Nicolae CONSTANTIN

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Keywords: forging, A694F65, semi-finished, mechanical, heating, grain size.

PART I

DOCUMENTARY ANALYSIS REGARDING THE CURRENT STAGE OF PLASTIC DEFORMING BY FORGING STEEL SEMI-FINISHES

CHAPTER 1

ELEMENTS REGARDING THE THERMAL REGIME OF THE DEFORMATION PROCESS OF METALS AND METAL ALLOYS

1.1 General notions regarding the thermal regime of the deformation process

In order for materials to be easily processed by deformation, they must have a low resistance to deformation and a high deformability. In most materials these conditions can be achieved by heating. At the temperatures at which the hot deformation takes place, the deformation resistance is less than [10… 12] times compared to the resistance of the same material in the cold state. Cold deformation ensures better part surface quality and dimensional accuracy than hot deformation. Cold deformation can only be applied to materials with lower deformation resistance and high deformability at ambient temperature and to obtain small parts.

The decrease in deformability at high temperatures is given by the beginning of the excessive growth of the grains and the beginning of the melting process at the intercrystalline limits. These phenomena occur all the more intensely as the heating temperature approaches the melting temperature of the respective material. The critical temperature at which these phenomena begin to occur intensely is found with [15-300] 0C below the solidus line (Fe-C phase diagram, figure 1.1.) [2].

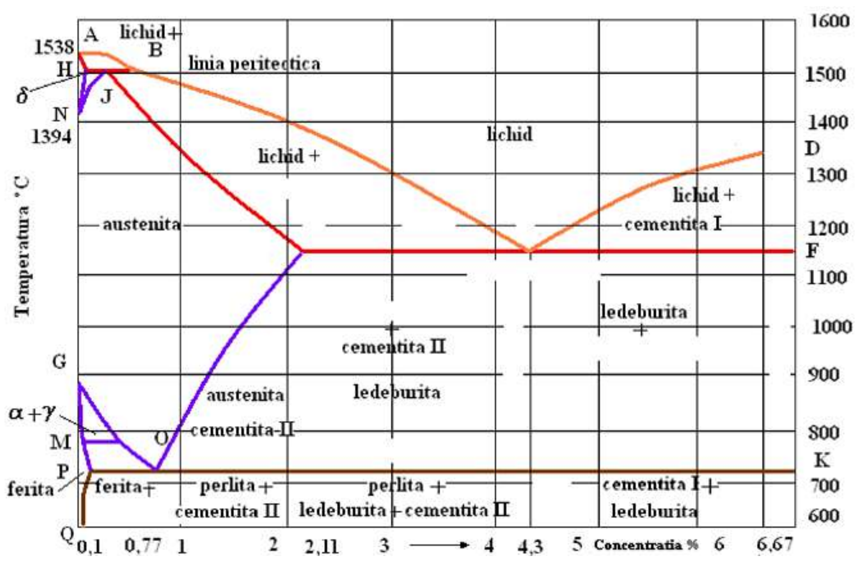


Figure 1.1 Chart Fe-Fe_3C [1].

- Duration of maintenance. -Maintenance duration. The amount of oxide increases with increasing the duration of keeping the semi-finished product in the oven at the given temperature. The connection between the amount of oxide formed and the duration of maintenance at the given temperature (in the range [600… 1200] 0C) can be expressed by the relation:

$$q = c\sqrt{\tau} e^{-\frac{9000}{T}} \quad (1.1)$$

1.2 Setting the heating rate

Heating speed means the increase of the temperature of the semi-finished product in the unit of time. There are three heating speeds: • technically possible heating speed, which depends on the heating installation; • permissible heating speed, which can be reached at a given semi-finished product; • actual heating speed, which is achieved during heating.

The thermal stresses increase in proportion to the heating rate, the expansion coefficient, the dimensions of the part and the modulus of elasticity of the heated material. A possible link between these parameters is presented below:

$$\sigma_{\max} = \frac{\alpha_0 E \Delta t}{1(1-\nu)} \quad (1.2)$$

1.3. Determining the heating time

The calculation of the heating time, relation 1.3., Is made starting from the equation of heat exchange between the semi-finished product and the heating medium:

$$\tau = \frac{M_s c}{A_s} \ln \frac{t_c - t_{si}}{t_c - t_{sf}} \quad (1.3)$$

PART II

EXPERIMENTAL RESEARCH ON TECHNOLOGY OF EXECUTION OF FORGED SEMI-FINISHED PRODUCTS

CHAPTER 2

THE PROCEDURE FOR DRAWING UP SPECIFICATIONS FOR FLANGED FORGED SEMI-FINISHED PRODUCTS OF A694F65 WITH SPECIAL PURPOSE FOR UNDERWATER OR NAVAL SYSTEMS

The procedure for drawing up specifications for flanged forged semi-finished products of A694F65 with special purpose for underwater or naval systems according to DNVGL-RP-0034 SFC 2.

The paper presents the procedure for drawing up specifications for a forged semi-finished product made of the material A694F65 (MOD).

Initially, an analysis of the chemical composition and forging procedure (starting ingot, drafting equipment) is performed followed by a simulation of the reduction degree (corrosion).

In the second part, an analysis of the heat treatment process (heat treatment furnace, contact thermocouples, charging sketch) and the mechanical characteristics obtained according to the standard will be performed.

Finally, we will evaluate the framing of the sample and the characteristics obtained with the reference standards.

In conclusion, the work shows step by step the elaboration and certification of the forged semi-finished products from A694F65 MOD in order to be used at special destinations as well as to meet the special conditions imposed by international standards.

Keywords: Open Die Forging; ASTM A694F65(MOD); MPS; DNVGL-RP-0034 SFC 2; Mechanical Tests;

2.1. Introduction

Forging means the procedure of processing a metallic semi-finished product by hot plastic deformation, without cracking, by means of static or dynamic forces exerted by presses or hammers [1, 3, 5]. Forging has the following advantages: rapid processing, low cost and simple workmanship. Disadvantages include: low dimensional accuracy, poor surface quality and the need for large deformation forces [2, 6, 9]. The main factor characterizing forging is the deformation degree (degree of strain).

Forging is classified according to the following criteria:

- the degree of freedom of the material during deformation: free forging, profiling forging on machines for limited use, forging in die;

- the working temperature: cold / hot;
- the deformation speed: low speed / high speed;
- the application of the deformation force: manual or mechanical.

The standard regulating the forging process of special purpose metallic materials for underwater systems is DNV-GL-ST-F101.

Applications of forged semi-finished products provided by DNV-GL-ST-F101 are multiple and differ depending on the industry in which the finished product operates (oil, natural gas, etc.), the used forged material, the characteristics, the shape and dimensions of the forged semi-manufactured product, the necessary mechanic characteristics of the exploitation. In this respect, a special importance is given to the ratio between stress and the degree of deformation presented in Figure 1 (with importance given to the variation between the mechanical resistance R_m and the uniform medium elongation A_{gt} marked in red on the diagram).

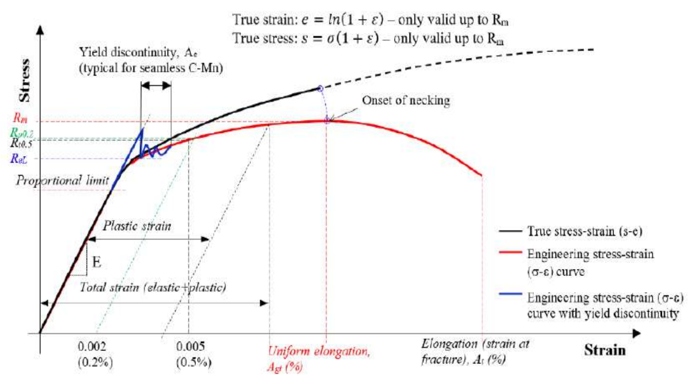


Figure 1 The dependence of the mechanical characteristics according to DNV-GL-ST-F101

The A694F65 (MOD) material is widely used in the underwater industry and is characterized by the ASTM A694/A694M standard as a steel with a content of 0.21% in carbon, in modified condition reaching 0.55-0.70 % Ni. Steel can be developed in a Vacuum Degassing (VD) electric oven, or in other development devices provided it is treated in order to obtain a fine structure.

(a fine grain practice using standard deoxidizing/grain refining elements to complete deoxidization and to refine the grain size).

Like other additional requirements, steel should not contain harmful elements. (the presence of which would indicate insufficient discard from the starting ingot).

As starting material in the forging process:: Material shall be free from burst, flakes, cracks, seams, laps, from any open discontinuities or other injurious defects detrimental to the end use of the part.

As non-destructive control requirements: All bars, blooms must be free of internal defects and to be guarantee ultrasonically according SEP 1921 class 3C/C minimum.

Breitenfeld Edelstahl AG is the European market leader of hight quality speciality steel ingots for the open die forging and ring rolling industry. These ingots of A694F65(MOD) are used for high demanding end-user segments like the power generation business, the oil and gas industry, naval and general engineering applications. The offered products are octagonal, polygonal, round, square or flat ingots (more than 70 different ingot types) from 1 to 120 ton.

In **Figure 2** the production route of Breitenfeld Edelstahl is shown. In 65 ton electric arc furnace (EAF) - the charged steel scarp is melted. After tapping in a bottom stirred ladle the process slag is removed. During the secondary metallurgy process the ignots, over the 60 tons, the double ladle technique, shown in **Figure 2b** is applied. After solidification the steel ingots have to be heat treated.

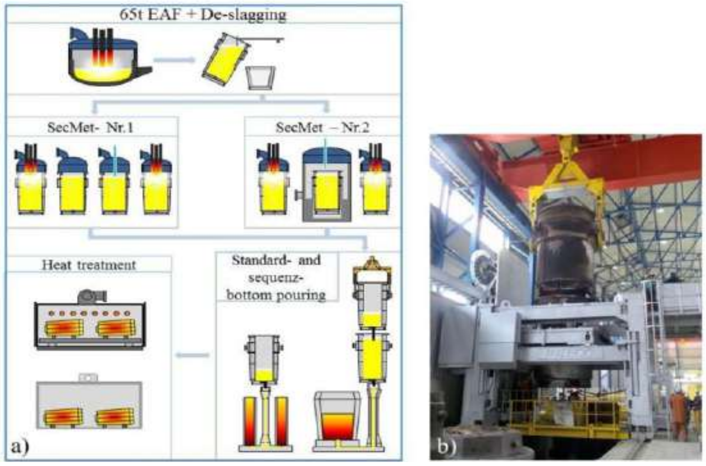


Figure 2. A - Production route Breitenfeld Edelstahl AG; B - Double ladle technique.

2.2 Experimental methodology

All raw materials purchased (ingots, rolled bars, forged bars, continuous castings, etc.) are ordered according to our internal purchasing specifications for all kind of grade of materials.

Ingot Type: A900 (9500 kg)-Polygonal Ingot; (D bottom =780 mm) – BREITENFELD Edeltahl, Mitendorf, Austria.

Material shall be melted using Electic Furnace steel making practices, fully killed & Argon Oxygien Decarburization (AOD) or Vacuum Arc Re-melting (VAR).

The chemical composition shall be tested on a ladle (heat) basis, as per ASTM A788, or an alternative recognised industry standard equivalent. The chemical composition of the raw material shall be tested on each heat in according to SPECIFICATION MS-FRO-17-A694 F65 MOD.

Elements (other than those used for grain refinement/deoxidation) not specified in table 2 shall not be intentionally added. Any element, including residuals, over 0.25% shall be reported.

Table 1 – A694F65(MOD) chemical composition

Elements	Min.	Max.
C	-	0.12
Si	0.2	0.35
Mn	1.1	1.4
S	-	0.01
P	-	0.015
Cr	0.15	0.25
Mo	0.15	0.3
Ni	0.55	0.7
V	0.02	0.05

Nb	-	0.02
Ti	-	0.01
Al	0.02	0.04
Cu	-	0.3
N	-	0.0005
B	-	0.0005
Pcm	-	0.28
CE	0.4	0.43
H	-	2.0 ppm

Notes:

- *The CE shall be determined using the following formula:*

CE = %C + %Mn/6 + (%Cr+%Mo+%V)/5 + (%Ni+%Cu)/15

- *The Pcm shall be determined using the following formula:*

Pcm = %C + (%Mn+%Cu+%Cr)/20 + %Si/30 + %Ni/60 + %Mo/15 + %V/10+ 5(%B)

- *Titanium +Vanadium +Columbium (Niobium) = max. 0.15%*

2.3. Forging Procedure

Starting size of raw material: A694F65(MOD) Ingot Type: A900 (9500 kg); (D bottom =780 mm) – BREITENFELD Edeltahl, Mitendorf, Austria. Polygonal Ingot, or from European Steel Mill on Forja Rotec AVL, that has previous history of supplying to VGS specifications.
For this Purchase Order we will be using:

- Press Forging: **1600 tf-Hydraulic forging press** used for open die forging process with hydraulic manipulator 15 tf.
- Open Die: **3000 Kgf-Electro-Hydraulic forging hammer** with electrical manipulator 1 tf.
- Gas furnaces for preheating;

Preheating of the ingot

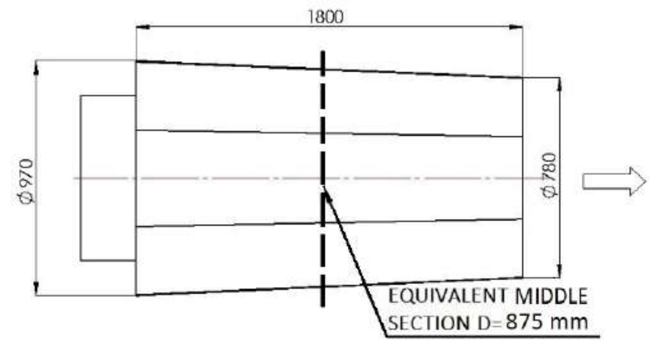
All furnace equipment used for forging heating are calibrated. A loading surface/volume, and on each load heat number and bar number are kept in the Forging Division Records (manufacturer sheets and heating charts). Working Instruction for Forging POSC- 7.5.1-11.

Heating paramaters are controlled by electronic devices (thermocouples, recorders with chart, programmer and safety devices, etc.) and a pyrometer is used in order to check the temperature before / in time of / and after the last forging operation.

The forging process starts after reaching suitable temperature

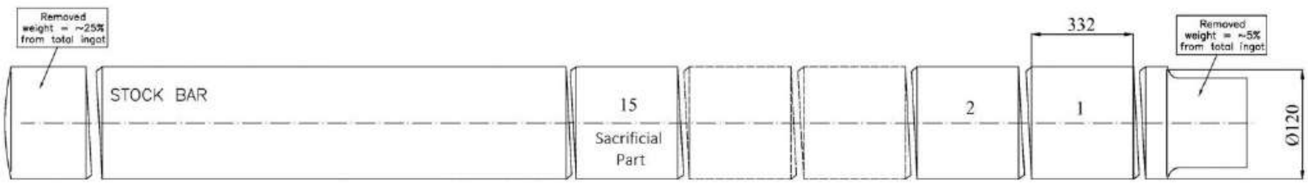
- Initial temperature (in Degree Celsius): 1110÷1150℃
- Final temperature (in Degree Celsius): 890÷850℃

- **Stretching process: polygonal equivalent middle section 875 → Ø120mm x L**



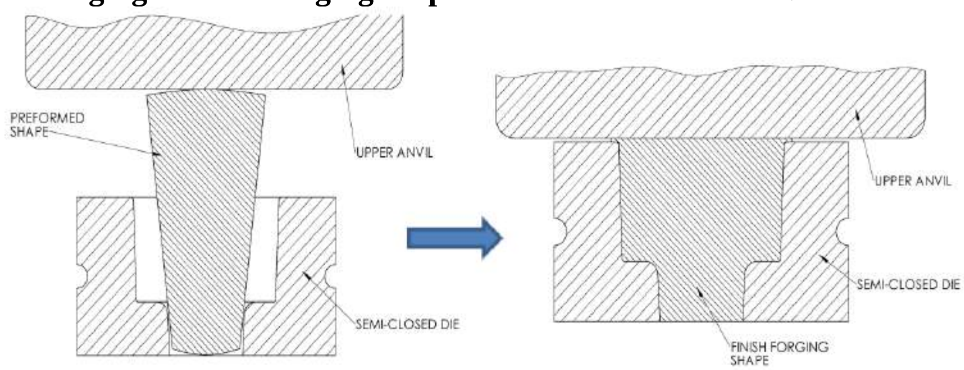
Hot work ratio: **53.2 : 1**

- **Cutting of the segment accordingly to the forged weight of the product to Ø120 X 332**



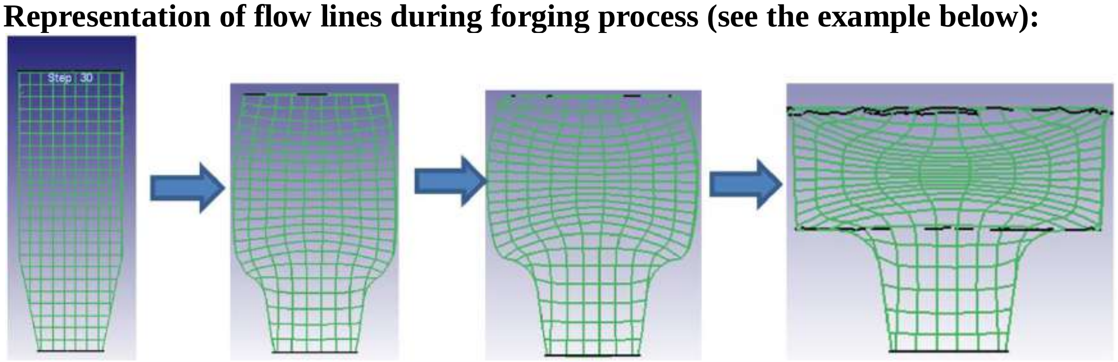
Ingot Discard ~25% TOP and ~5% bottom

- **Forging to finish forging shape in Semi-Closed Die tool.**



Hot work ratio: **1.4:1**

- **Representation of flow lines during forging process (see the example below):**



2.4. Heat Treatment Procedure

The batch should consist of pieces of the same grade of steel. If there are different thickness sizes, the time is calculated considering the maximum section. The procedure shall be in accordance to API RP 6HT.

All furnaces are electrical, computer controlled, qualification of Heat Treat Equipment is acc. requirements of API 6A- annex M and include the temperature uniformity requirements of AMS 2750.The temperature uniformity survey was carried out at 960°C with a load of 600 kg. Size of working zone: *CVE3- R 800 X 1500(mm) for N&Q&T -vertical furnace.*

Heating parameters are controlled by thermocouples and are recorded with chart and programmer.

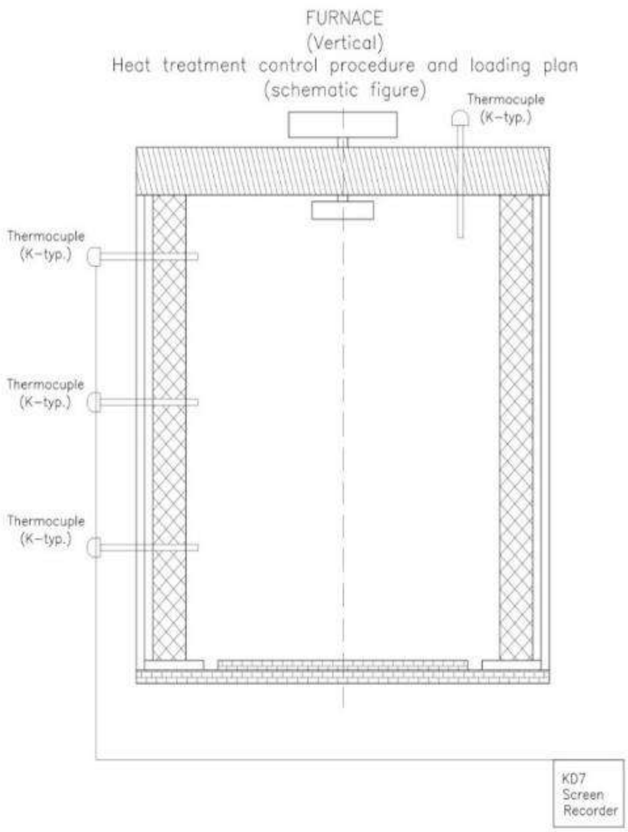


Figura 3. Electrical heat treatment furnace

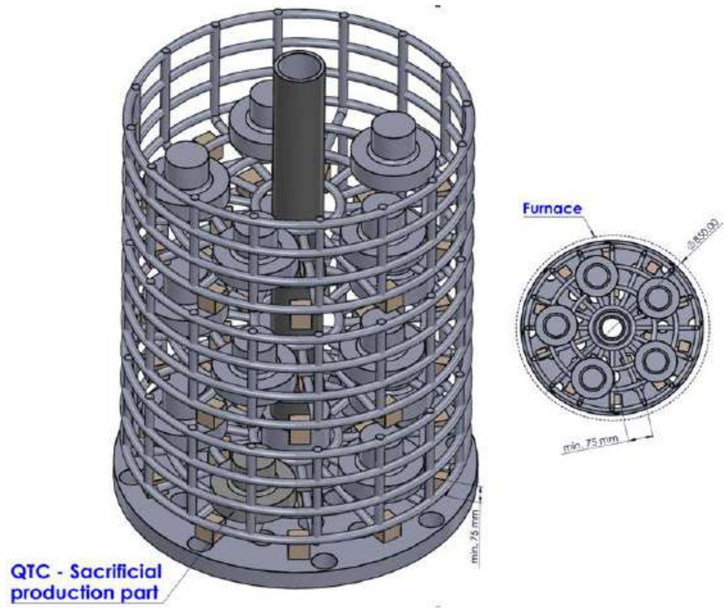


Figure 4 Example of loading senario above. Orientation of the furnace: vertical

The material shall be loaded into the heat treatment equipment such that the presence of one part does not adversely affect the heat treatment response of any other part in the same heat treatment load. Parts shall be separated for quenching so that there is sufficient space between each part to provide adequate quench media coverage during to quenching operation. Minimum spacing distance 75 mm / 3 inch.

QTC-SACRIFICIAL PART in accordance with A694/A694M. Maximum ruling section at heat treatment will be ***102.00 mm***, maximum load mass will be up to ***600 Kg.***

Table 2 - Heat treatment parameters

Item	Number of heat-treat. batchs.	Normalizing		Austenization		Tempering	
		Temp. °C	Holding time	Temp. °C	Holding time	Temp. °C	Holding time
1	1	913÷968°C	min. 2 hrs.	885÷ 941°C	min. 2 hrs.	540÷665°C	min. 4 hrs.

For every quenching tanks there are: the temperature control, agitators, time controls and recirculation system for cooling liquid. Quench tank 3# have a capacity of 7 000 l (load 600 kg). Maximum cross section: 102.00 mm. The transfer of material from heat treatment furnace into the quenching tank is performed within maximum 90 seconds from the time the austenitizing furnace door opens until the forgings have been completely immersed in the quench media.

Heat Treatment Process Parameters applicable for this Purchase order:

Normalising:

- Loading temperature: ≤ 750°C
- Rate of Heating: max. 150°C per hour
- Holding temperature: 913÷968°C
- Holding time: minimum 2 h after the furnace thermocouple reaches temperature.
- Cooling: In air

Austenitising:

- Loading temperature: ≤ 750°C
- Rate of Heating: max.150°C per hour
- Holding temperature: 885÷ 941°C
- Holding time: minimum 2 h after the furnace thermocouple reaches temperature.
- Cooling in water
- Water Temparature maximum 40°C at the start of the quench.
- Max temperature after quenching: 50°C

Tempering;

- Loading temperature: ≤ 400°C
- Rate of Heating: max.150°C per hour
- Holding temperature: 540÷665°C
- Holding time: minimum 4 h after the furnace thermocouple reaches temperature
- Cooling: In air or faster.

The target heat treatment temperature will be established depending on the A694/A694M, and the heat of material (cast chemistry) and it will have the following tolerance: for normalizing and austenitising ±10°C and for tempering ±15°C.

No. of thermocouples: 4 furnace thermocouples

2.5. Mechanical Testing Methods and Properties

Test specimens shall be removed as per A694/A694M. Mechanical test to be performed according to ASTM A370.

Test Laboratory is accredited and calibrated by National Standards and by ISO9001 management system:

- two tensile specimens taken at T/2, one in axial and one in tangential direction.
- two sets of Charpy V-notch specimens taken at T/2, one in axial and one in tangential direction.

Tabel 3 **Mechanical Test Requirements**

Mechanical Test Requirements	
Yield strength MPa (ksi) min	450 (65ksi)
Tensile strength Mpa (ksi) min	530 (77 ksi)
Elongation (%) min	20
Charpy V Notch at -46 ºC *Longitudinal and Transverse*	34J min. Single / 42J min Average
Hardness HBW 3000/10 / (Hv10)	174-237 HBW

Mechanical trial results:

MECHANICAL PROPERTIES PER QTC
with standard tensile and Charpy specimen used

Material	Heat	QTC Dimmensio[mm]	QTC Type	Heat treat . QTC Lot
A694F65MOD			SAC.PART	

T1 (°C) / T2 (°C)	Tensile Strength [Mpa]	Yeld Strength 0.2% Offset [Mpa]	Elongation A [%]	Reduction of area Z [%]	Temp. Impact Test [°C] / Charpy impact values / Average [J]	Hardness / Hardness UM
Lateral Expansion [mm] / average						
Sampling Mode QTC: 1/2T			ORIENTATION: L			
20	574	457	24.00	75.00	-46	192
			/		166.00 189.00 202.00 / 185.67	hbw
Sampling Mode QTC: 1/2T			ORIENTATION: T			
20	580	464	27.00	70.00	-46	192
			/		118.00 154.00 146.00 / 139.33	hbw

Methods for determining the size of the grain

The size of the grain is expressed by a figure called score. Between the score (N) and the number of grains (n) included in an area of one mm^2 at the increase of 100X there is the relation: $n = 8\ 2^N$.

The method of counting the grains

In the corresponding image of a circle with a diameter of 79,8 mm for the increase of 100X (an effective actual area of 0,5 mm^2) the grains that are entirely contained inside the circle (m) and the grains intersected by circumference (m1) are counted. The total number of grains for an area of 0,5 mm^2 at the increase of 100X is calculated with the relation: $m_{100} = m + 0{,}5\ m_1$. For one mm^2, a double number of $M=2\ m_{100}$ grains will be found. For increases other than 100X, M will be located with the formula: $M=2\ (\ g\ /\ 100\ )^{2}\ mg$, in which : g-is the increase used under the microscope, m_g-the number of grains at size g. After examining at least three visual fields of the sample (of the most characteristic) the average number of grains (Mmed) will be determined. The average area (S_{med}) and the average diameter (d_{med}) of the grain is calculated with relations: $S_{med} = 1\ /\ M_{med}$; $d_{med} = 1\ /\ (\ M_{med}\ )^{1/2}$.

By comparing the values obtained for M_{med}, S_{med} and d_{med} with those in Table 14. 1., the N score is determined.

Depending on the dimensions of austenitic grain, the usual steels are classified in: thick granulation steels (N=1-3); medium granulation steels (N=4... 6); fine granulation steels (N=7... 10).

Austenitic grain analysis results:

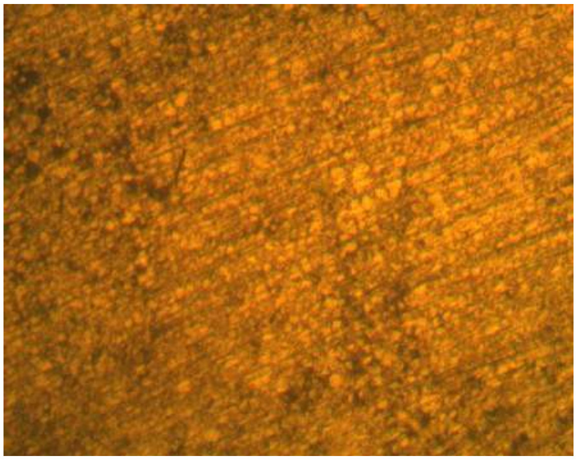
Method: ASTM E 112-2013

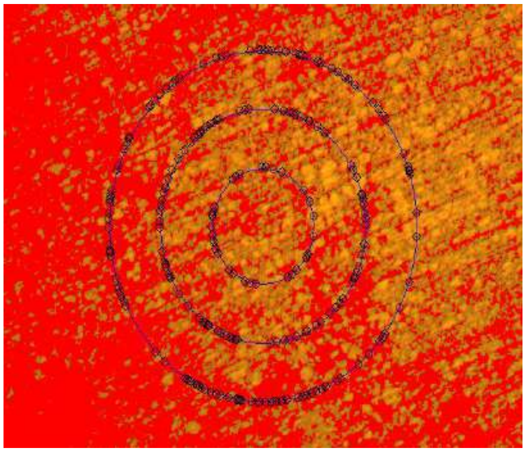
Echipment:

Objectiv: X100

Heat Number:88882

Heat Treatment:10592





Sr No	No of Intercepts	Grain Number (G)
1	169	7
2	192	8
3	163	7
4	187	8

Austenitic grain = 7-8

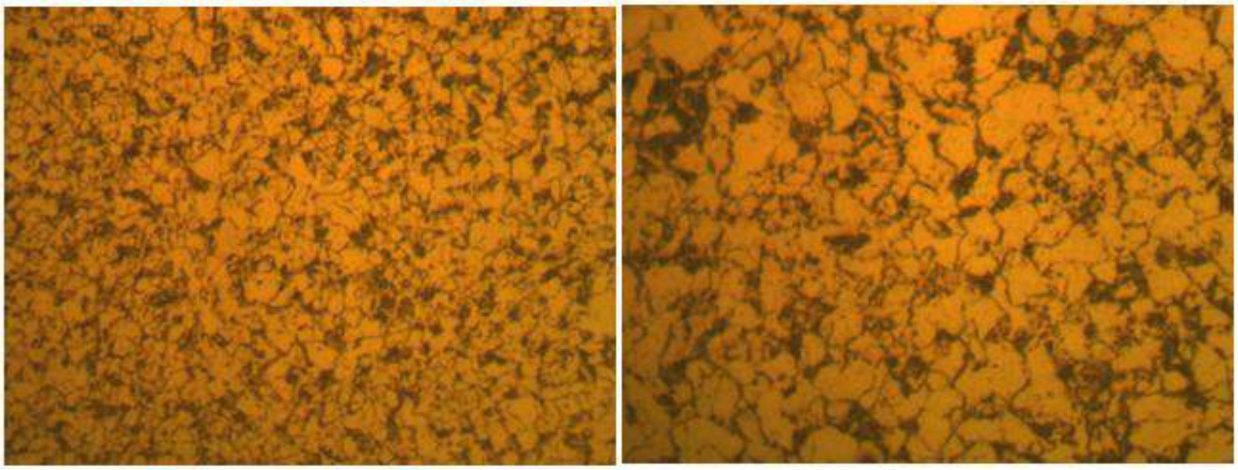


Figura 5 Microstructure on longitudinal direction at 1/2T area (A694F65 Mod- Microscop XJP-6A // MAGNIFICATION: 200 // MAGNIFICATION: 500 X - XETCHANT: Nital Reagent).

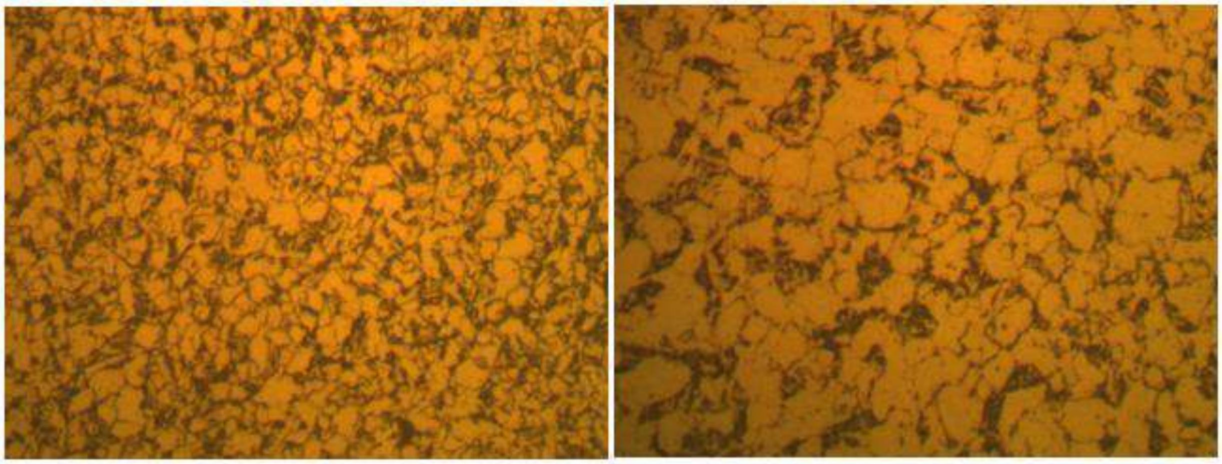


Figura 6 Microstructure on transverse direction at 1/2T area (A694F65 Mod- Microscop XJP-6A // MAGNIFICATION: 200 // MAGNIFICATION: 500 X - XETCHANT: Nital Reagent).

In Figure 5 and 6, we can see the microstructure of the semi-finished product in A694F65(MOD). An increase in the size of grain is observed with the increase in temperature (or the variation of the alloying elements). Rapid and uneven cooling leads to the appearance of a relatively coarse structure.

CHAPTER 3

THE INFLUENCE OF HEATING TEMPERATURE AND COOLING RATE AFTER FREE FORGING ON THE MICROSTRUCTURE AND THE MECHANICAL PROPERTIES OF AISI4140 STEEL

The modern technique of free forging imposes new requirements for the obtained semi-finished forgings, each operation requiring time, energy, human and financial resources. In order to evaluate these processes, it was indicated the importance of the application of primary heat treatments after free forging a part on a 1200 tf hydraulic press, the studied material being an AISI4140 steel heated at an austenitic temperature of 1150°C.

Both types of (normalized) applied treatments, following experimental tests, have determined hardness values and mechanical characteristics according to ASTM A29/A29M standard, which requires their application after the free forging or molding operations.

Keywords: AISI4140, free forging, microstructure, mechanical properties, temperature

3.1. Introduction

Forging is a technological process for processing metals and alloys (in a plastic state) under the action of dynamic (shock) or quasi-static external forces. Forging operations may be classified as follows:

1. free forging, in which the material flow is guided by the deformation anvils and some simple tools,
2. forging in a mold (molding), in which the material deformation takes place in some tool cavities called molds, so that in the end the part will take the shape of the cavity (which is the negative of the part)[1-5].

Generally, after forging, the parts are subjected to processing by chipping and heat treatments in order to bring them to the required shape, precision and characteristics [6-7]. There are also parts that do not undergo any processing after forging, this being actually the current trend [8].

The material capacity for being processed by forging is appraised by forgeability, which implies deformation resistance and deformability [9-11]. The level of forgeability is higher as the deformation resistance is lower and deformability higher. Forged metallic materials are those materials that can be deformed plastically by any of the two forging processes. The main forging materials include ferrous and non-ferrous alloys [12-15]. Ferrous alloys can also be divided in carbon steels and alloyed steels, and non-ferrous materials, in heavy metals and alloys and light metals and alloys. From a practical application point of view, the most widespread ferrous alloys are carbon steels [16].

In order to make them easily deformable, the most frequently used materials are steels with a carbon content up to 1.4%. Above this limit, they become difficult to forge. In the category of forgeable carbon steel are included OL ordinary carbon steels and OLC quality carbon steels. Another class of forging materials is that of alloy steels for machine building, refractory steels for springs, steels for bearings and other special steels [17-18].

The presence of some chemical elements in the steel composition influences their deformation behavior [19].

The standard that regulates the mechanical characteristics and the chemical composition of AISI4140 steel is ASTM A29/A29M. The chemical composition of the steel is given in **Table 1**. This may vary depending on the obtaining method, but without exceeding the ranges provided by the standard.

The samples for mechanical tests were sectioned according to the following considerations:

1. Small pieces not exceeding 500 mm in section, taken longitudinally along the forging axis;

2. For sections smaller than 100 cm^2, the samples can be taken from the entire surface of the part, starting from axis"0" of the forjed part, up to T/2 distance from the ends (extremities).

3. For part sections having a diameter of more than 100 cm^2, the sample diameter will be maximum Ø25 mm and the sample sectioning method is shown in Figure 1.

4. The number of samples on the drafting / forging batch depends on the shape of the forged parts and the applied heat treatments.

Table 1

The chemical composition of the AISI 4140 steel

STANDARD	RANK	C%	Mn%	P%	S%	Si%	Ni%	Cr%	Mo%
ASTM A29/A29M	4140	0.38-0.43	0.75-1.00	< 0.035	<0.040	0.15-0.35	0.0	0.8-1.10	0.15-0.25
EN 10250-3	42CrMo4	0.38-0.45	0.6-0.9	< 0.035	<0.035	< 0.4	0.0	0.9-1.2	0.15-0.30
JIS G4105	SCM440	0.38-0.43	0.6-0.85	< 0.03	<0.03	0.15-0.35	0.0	0.9-1.2	0.15-0.30

Thus, for parts up to 15 tons, 4-6 samples can be taken for mechanical tests (unless otherwise agreed with the customer).

The austenitic grain size is determined according to the E112 Test Method (if at least 70% of the grain sizes fall within the class requirements of the standard, it can be considered an acceptance basis)[20]. The analysis of the temperature variation regarding heating and cooling can influence the mechanical characteristics and the size of the austenitic grain so that inadequate heating and cooling can lead to austenitic grain growth, inadequate ferrite/perlite ratio, cracks during the use of the work piece or even during mechanical processing.

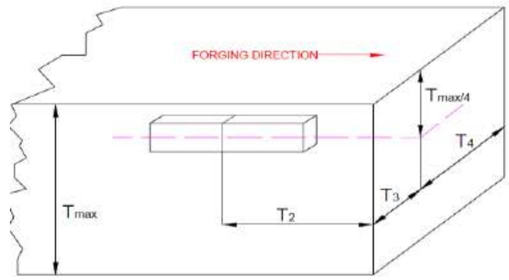


Fig. 1. Sampling method for forging AISI4140 (in this case T_2=T_3=T_4 ≥ T_{max} ; where T_{max} - the maximum section of the piece).

AISI4140 is a chromium-molybdenum alloy steel used in making axes, bolts, gears, shafts, etc. It is to be found under different steel grades, according to region and standard, with small variations of the chemical composition, as shown in Table 2. The forging temperature range is between 1150 - 1200° C and forging below 850°C is forbidden [21-25].

Table 2

AISI 4140 depending on the origin region

ZONE	USA	GERMANY	UK	JAPAN	CHINA	AUSTRALIA
STANDARD	ASTM A29	DIN17200	BS970	JIS G4105	GB/T 3077	AS 1444
RANK	4140	42CrMo4	42CrMo4	SCM440	42CrMo	4140

The primary heat treatments for AISI4140 may be:
- full annealing after forging consisting in heating at 700-820°C followed by furnace cooling;
- normalizing: heating at 820-900° C followed by air cooling;
- normalizing + sub-critical annealing (heating at 550-650° C, holding time 1 hour/25mm section followed by air cooling).

The secondary heat treatment may also be:
- normalization + sub-critical annealing (heating at 450-550° C, holding time 1 hour/25mm section followed by air cooling) or
- quenching (heating at 820-900°C, holding time 10-15 min /25 mm section followed by rapid cooling in water, oil or polymer) + temperature tempering, hardness control being required [26-27].

3.2. Experimental

According to ASTM A29, more samples of 8 mm length and 14 mm diameter were taken (marking and sampling is shown in Figure 2).

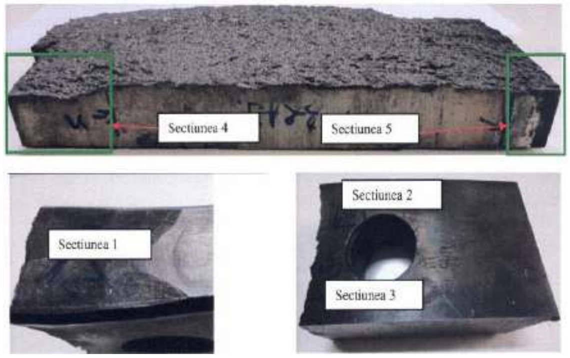


Fig. 2. Sampling of mechanical tests

The samples were used to check the alloy chemical composition and determine the size of the austenitic grain when reheating followed by slow or rapid cooling (the oxidation method was used).

Forging was performed on a 1200 tf press at a maximum deformation rate of 1:2 and a forging temperature of 1100-1150°C.

In order to investigate the effect of reheating followed by the two cooling methods, samples of 45 mm diameter and 250 mm length were cut, being subjected to annealing heat treatment for 5 minutes, followed by furnace or air cooling.

The mechanical characteristics assayed at room temperature as well as the chemical composition of the samples submitted to ~~the~~ tests are given in Table 3. The chemical composition analysis was carried out on alloy batch no. 83377 and heat treatment lot, the used equipment being Spectrolab M10 / 76004135 in accordance with LAR-PL-01 Rev.03 ASTM E415-14.

Table 2

Chemical composition of analyzed samples

Mark	C%	Si%	Mn%	P%	S%	Cu%	Ni%	Cr%	Mo%	V%	Al%	Ti%
Samples AISI4140	0.44	0.31	0.96	0.009	0.002	0.16	0.16	1.04	0.17	0.03	0.025	0.003

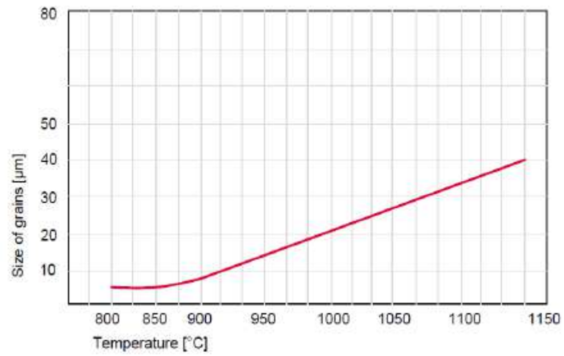


Fig. 3. Grain size variation with temperature

3.3 The effect of different heating and cooling parameters on the alloy grain size

The relationship between the heating temperature and the grain size is shown in Figure 3.An increase in the grain size is found when increasing the temperature or when varying the chemical composition.Rapid and non-uniform cooling leads to the appearance of a relatively rough structure, as shown in Figure 4.

In this case, the grain size varies from 0 to 5, according to SR EN ISO 643-2013. The grain size was determined by the oxidation method, and the obtained score was 7.

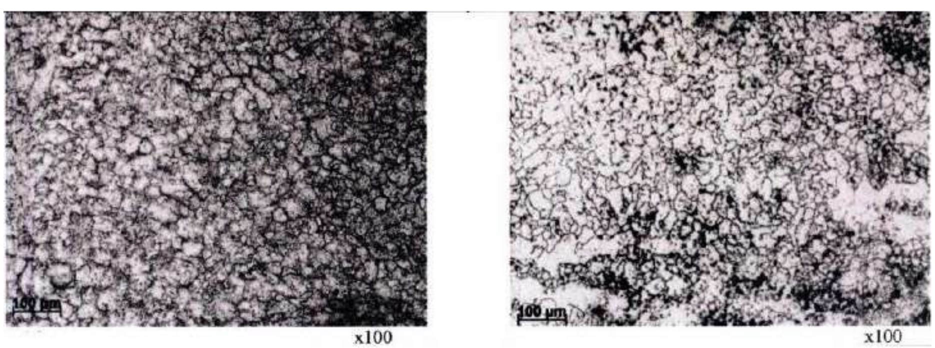


Fig. 4. Size of hereditary grain by the oxidation method

3.4 The effect of different heat treatment heating and cooling parameters on the alloy microstructure and mechanical characteristics

Figure 5 shows the macroscopic features under two states: a- forged semi-finished product and b - annealed semi-finished product, previously forged . It is to be noticed that after forging, the grain size distribution is relatively rough and inhomogeneous at the base, as compared to the sample submitted to primary heat treatment.

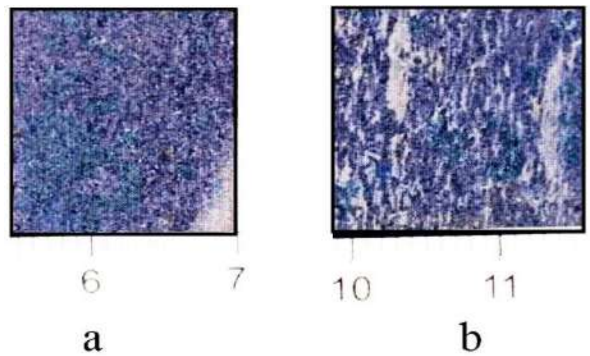


Fig. 5. The macroscopic aspect of the analyzed section

The microscopic analysis performed in the first raw forged section (shown in Figure 6) reveals the existence of isolated non-metallic inclusions such as sulphides and oxides of small size. These may have a negative effect along the mechanical processing (causing micro-fissures, deformations of the workpiece under mechanical force or processing temperature etc.) and by decreasing the working life of the finished product as a result of premature wear, superficial corrosion of the parts, a.s.o.

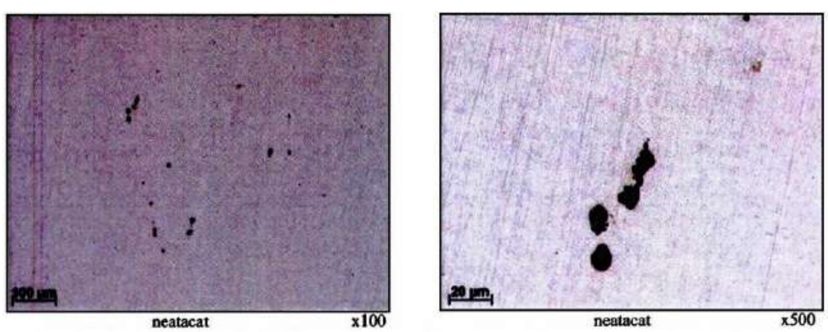


Fig. 6. Isolated metallic inclusions

Several microscopic analyses were performed on the above-mentioned samples, highlighting the following conclusions:

In Figure 7 the microstructure is made of lamellar and spheroidized perlite, a small proportion of ferrite and bainite. In some areas grains having grain size 1 have been identified. The grain size distribution is relatively inhomogeneous, ranging from 1 to 5, according to SR EN ISO 643-2013. Heat treatment applied after forging: normalization + sub-critical annealing (heating at 450-550° C, holding time 1 hour/25mm section followed by air cooling)

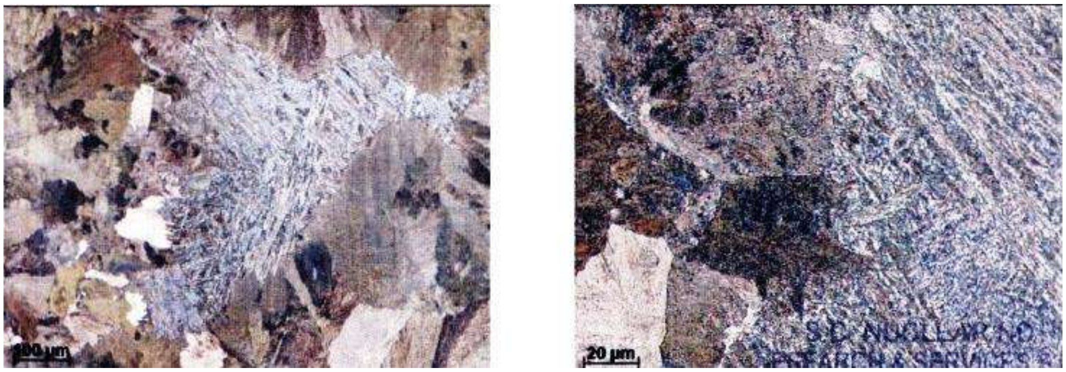


Fig. 7. Microstructure of the analyzed samples - sample Nr. 1 (ATAC - REAGENT, ×100 and ×500). Heat treatment applied after forging: normalization + sub-critical annealing (heating at 450-550° C, holding time 1 hour/25mm section followed by air cooling)

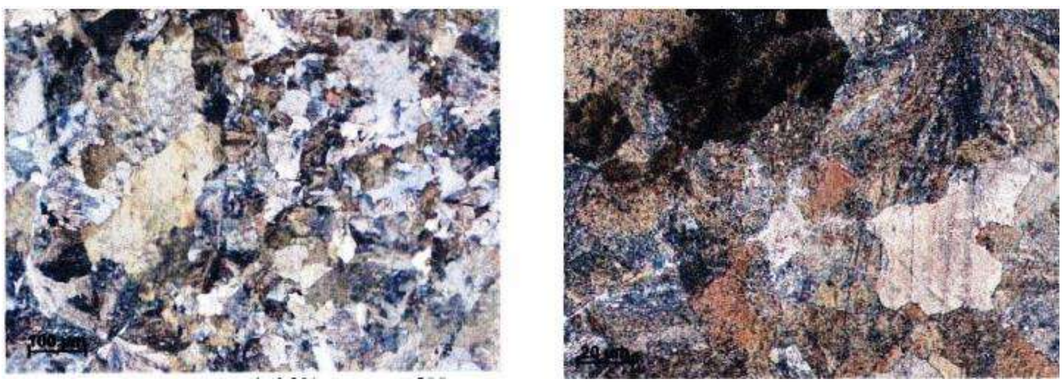


Fig. 8. Microstructure of the analyzed samples - sample Nr. 2 (ATAC - REAGENT, ×500). Heat treatment applied after forging: normalization + sub-critical annealing (heating at 550-650° C, holding time 1 hour/25mm section followed by air cooling)

In Figure 8 the microstructure exhibits lamellar and spheroidized perlite, a small proportion of ferrite and bainite. The granulation is slightly inhomogeneous, ranging from 1 to 5, according to SR EN ISO 643-2013. On the inner surface of the forged piece there is a slight decarburization. Heat treatment applied after forging: normalizing + sub-critical annealing (heating at 550-650° C, holding time 1 hour/25mm section followed by air cooling).

Figure 9 shows an inhomogeneous microstructure made of lamellar and spheroidized perlite, a small proportion of ferrite and bainite. The structure is characteristic of a raw forging followed by air cooling structure. In some areas of segregation, large pearlite and bainite grains (grain size 0) were found. The grain size distribution is inhomogeneous, ranging from 0 to 5, according to SR EN ISO 643-2013. Heat treatment applied after forging: normalization (heating at 820-900° C followed by air cooling)

Fig. 9. Microstructure of the analyzed samples - sample Nr. 3 (ATAC - REAGENT, ×50 / ×100 / ×500). Heat treatment applied after forging: normalization (heating at 820-900° C followed by air cooling)

Pearlite is a mechanical mixture of ferrite and cementite. The two phases are simultaneously separated under equilibrium conditions at 727°C from austenite of eutectoid concentration of 0.77% C when slow cooling. According to the microscopic aspect, the lamellar pearlite found in the analyzed samples may be characterized as follows: cementite lamellae are trapped in a mass of [restrans] ferrite, appearing when slow cooling from temperatures higher than the eutectoid transformation of cementite critical point temperature (at the end of the transformation it exhibits a digital fingerprint feature).

On some surfaces spheroidized perlite in the form of globules in a metallic mass is found. This constituent resulted from spheroidizing annealing (soaking) of the lamellar perlite gives the best machinability. The bainite resulting from carbon supersaturated ferrite "α" and fine globular carbides Fe_xC is formed not only at temperatures close to the kinetic maximum in the alloy TTT diagram (minimum stability of austenite), but also at low temperatures above the M_s point (of the Fe-C diagram), having a relatively acicular structure similar to martensite. The relatively high resistance of bainite is due to the ferrite crystals small sizes, to the carbide precipitates dispersion, to the ferrite network distortions due to carbon supersaturation.

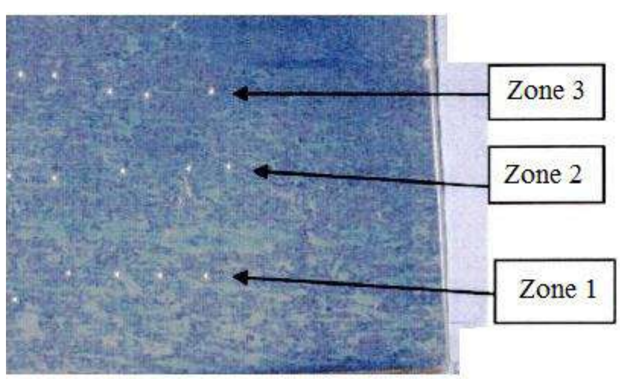


Fig. 10. Hardness test in 3 zone distincte dupa aplicarea tratamentului termic de: normalizing + sub-critical annealing (heating at 550-650° C, holding time 1 hour/25mm section followed by air cooling).

Table 4

Test for VICKERS hardness in three distinct zones

Nr.	Zone Test	VICKERS hardness results					HV10	Conversion according to SR EN ISO 18265-2014 HRC
		1	2	3	4	5		
1	Zone 1	262	264	262	260	254	**260**	**24**
2	Zone 2	260	254	262	258	262	**259**	**24**
3	Zone 3	256	249	256	256	254	**254**	**23**

Figure 10 presents the locations where the VICKERS hardness test was performed on the sample, according to SR EN ISO 6507-1 / 2006. The HV10 hardness test was performed on three axial lines of the sample and the obtained values are shown in Table 4.

Concerning the traction test, a 20 tf - 1033/91 universal mechanical testing machine was used, and the tests were carried out after performing the primary heat treatment on the semi-finished products, according to SR EN ISO 6892-1/2010 _ (method B). The results are shown in Table 5.

Table 5

Tensile testing

Marking	**Direction of sampling**	**Type of specimen**	**$R_{p0,2}$** YS (PS) [MPa]	**R_m** UTS [MPa]	**A** EL [%]	**Z** RA [%]
731 T	Long.	Ø10 mm	512	843	15.4	31.1
Imposed values			≥ 500	≥ 750	≥ 14	

CHAPTER 4

EXPERIMENTAL RESEARCH ON THE PROCEDURE FOR THE PREPARATION OF STANDARDIZED FORGED SEMI-FINISHED PRODUCTS IN USE IN UNDERWATER SYSTEMS

The paper systematically presents the forging process for a MPS certified piece in accordance with the DNVGL-RP-34 standard. The DNV-GL-ST-F101 standard is important and widely used in the metallurgical field, especially in the field of forged semi-finished products for the extractive, natural gas, oil, maritime industry, etc.

API RP 6HT (Normalization, Austenitization, Recovery) thermal treatments were applied to the analyzed piece, and the sampling method and direction was performed according to DNV-GL-ST-F101. All mechanical features, including those obtained from SPWHT, are indicated on the finished product quality certificate.

Keywords: Free forging; deformation degree, MPS, DNVGL-RP-0034; API RP 6HT

4.1. Introduction

Forging means the procedure of processing a metallic semi-finished product by hot plastic deformation, without cracking, by means of static or dynamic forces exerted by presses or hammers [1], [3], [5] Forging has the following advantages: rapid processing, low cost and simple workmanship. Disadvantages include: low dimensional accuracy, poor surface quality and the need for large deformation forces. [2], [6], [9]. The main factor characterizing forging is the forging degree (corroyage).

Forging is classified according to the following criteria:

- by the degree of freedom of the material during deformation: free forging; profiling forging on machines for limited use; forging in die;
- by the working temperature: cold; hot;
- by the deformation speed: low speed; high speed;
- by the application of the deformation force: manual and mechanical.

Free forging is the forging in which plastic deformation is made unlimitedly and can be done manually or mechanically. Mechanical free forging is applied in most of the small or single series forging sections. [8], [10], [19]

The standard regulating the forging process of special purpose metallic materials for underwater systems is DNV-GL-ST-F101.

The purpose of applying this standard is to guide the requirements of design and development concepts, the competent use of metallic materials and the effect of corrosion, coatings and protection in operation, mechanical operations and tests of the materials used, welding assembling, NDT control.

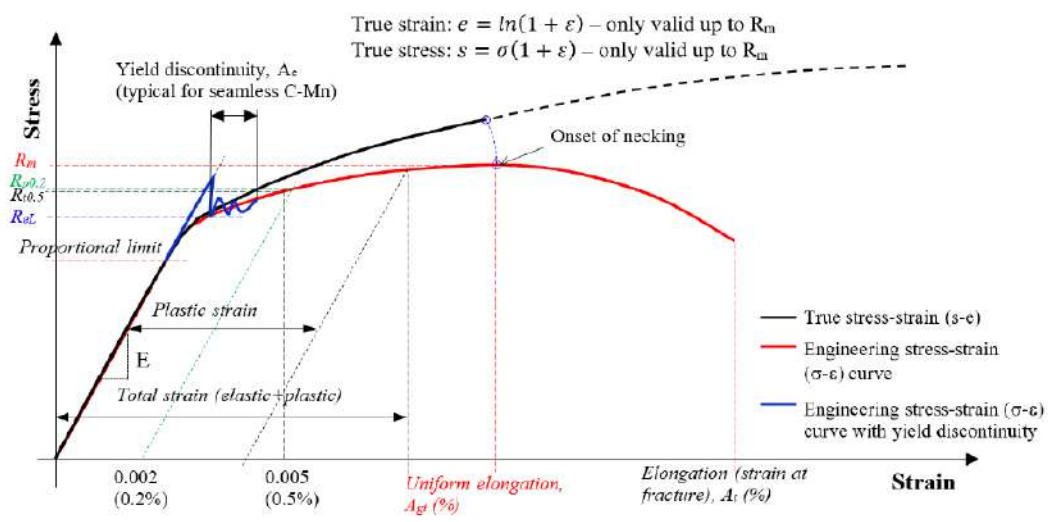


Figure 1 Dependence of mechanical characteristics according to DNV-GL-ST-F101

The main objectives of the DNV-GL-ST-F101 are: to ensure the development of the concept and the operations necessary to obtain the finished product in a regime of public safety and protection of the environment; [11], [16], [20] to ensure international recognition of the use of special purpose materials by defining minimum requirements for product realization; to offer high tradability to both manufacturers and buyers or final contractors.

The applications of the DNV-GL-ST-F101 forged semi-finished products are multiple and vary depending on the industry in which the finished product operates (oil, natural gas industry [11], [21], [22] etc.), the forged material used, the characteristics, the shape and dimensions of the forged semi-finished product, the mechanical characteristics necessary for the exploitation. In this respect, a particular importance is given to the relationship between stress and deformation shown in Figure 1.

It can be observed the dependence of the mechanical characteristics: mechanical resistance and elongation, flow and plastic deformation of the material.

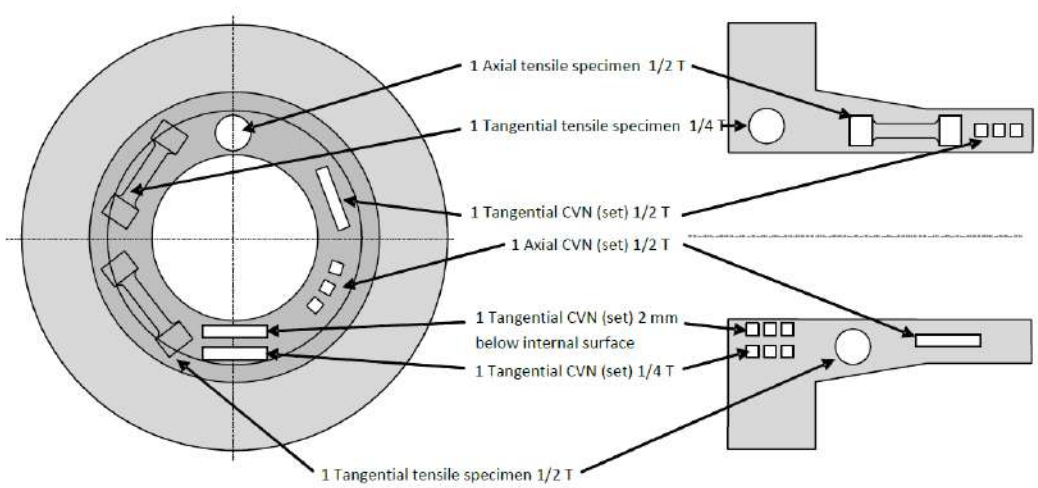


Figure 2. Sampling method and direction according to DNV-GL-ST-F101.

The sampling method and direction according to DNV-GL-ST-F101 is shown in Figure 2 (longitudinal and tangential to 1/2T and 1/4T tangential to mechanical resistance specimens, longitudinal and tangential to 1/2T and 1/4T with a 2-mm distance from the surface for KV).

4.2. Experimental methodology

The objective of the procedure for obtaining special purpose forged semi-finished products is to ensure high transparency in the certification of the finished product and throughout the elaboration process and to ensure the superior certification of a third party at the certificate level 3.2.

The need to implement the Preparation Procedure (MPS) is joining higher 3.2 standardized certifications on certain fields of exploitation of forged semi-finished products.

For the application of the requirements of the DNV-GL-ST-F101 for the experimental part, the forging of a piece of the shape and dimensions shown in Figure 3 (drawing no. A452392-18 Rev D) was made.

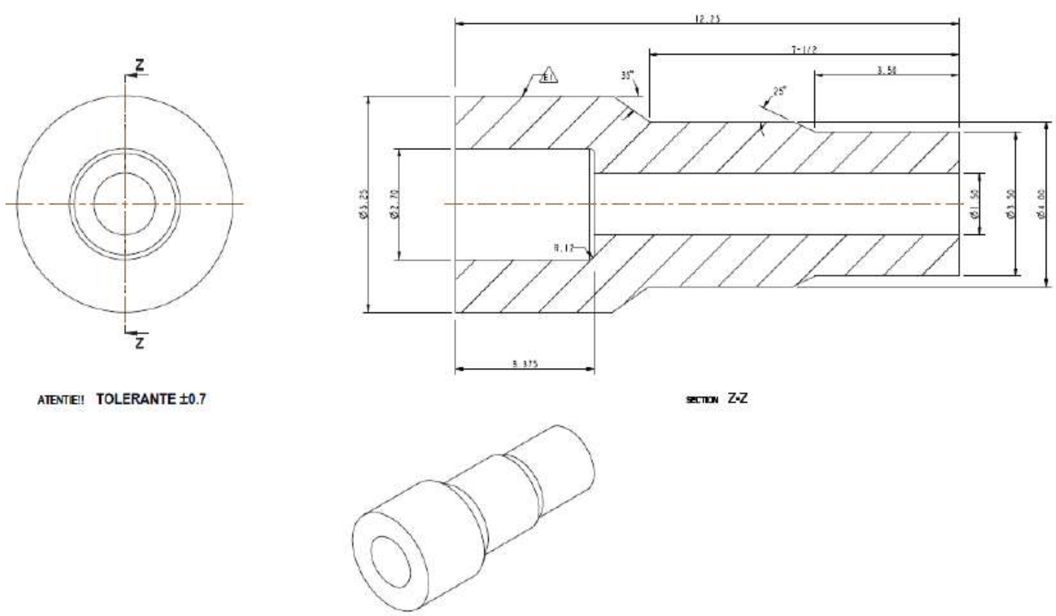


Figure 3. The piece subject to MPS preparation in accordance with DNV-GL-ST-F101 (the dimensions of the drawing are in inches)

Starting materials (ingots, rolled bars etc.) are ordered in accordance with the specifications of material standards. The material is melted and prepared in an electric furnace followed by deoxidation (AOD) or vacuum (VAR) [14], [15], [18]. The base material is AISI4340 (the number of pieces and sacrificing for mechanical testing are shown in Table 1).

Table 1. The piece subject to MPS preparation in accordance with DNV-GL-ST-F101

Line item	Part number	Dimensions	Quantity	Material
1	A111827-14 rev A RAW	A452392-18 Rev D	58	AISI 4130
2	A111827-14 rev A RAW*	As RAW	2(SAC COMP)	AISI 4130

The chemical composition of the material used (AISI4130) is presented in the Table.

Table 3. Chemical composition of the studied material (%)

where:

Elements	Min.	Max.			
C	0.27	0.33	B	-	0.0010
Mn	0.30	0.70	Ti	-	0.025
Si	0.15	0.35	Cu	-	0.35
P	-	0.015	V	-	0.06
S	-	0.010	Nb	-	0.02
Cr	0.75	1.20	N	-	0.012
Ni	-	0.25	Pb	-	0.010
Mo	0.15	0.25	Bi	-	0.010
Al	-	0.055	H	-	2ppm
sn	-	0.020	O	-	25ppm
Sb	-	0.03	J(1)	-	180
As	-	0.030	X(2)	-	20
			Ce(3)	(4)	0.68(5)

where:

1) J- Factor: (%P +%Sn) x (%Mn + %Si) x 10^4
2) X-Factor: (10 P[%] + 5 Sb [%] + 4 Sn [%] + As [%]) x 10000/100
3) Ce (Carbon Equivalent): C (%Mn/6) + (%Cr + %M + %V)/5 + (%Ni + %Cu)/15

The forging procedure involves the starting material: AISI4130 ingot type A900 (9500 kg), diameter = 780 mm - polygonal ingot. For this forging procedure we will use:

- hydraulic press of 1600 tf with 15 tF/KJ manipulator
- 3000Kgf (Electro-Hydraulic) forging hammer

- gas required to heat the ingot

The entire equipment for the preparation of the forged semi-finished product is calibrated. Each forging batch will be taken into account, the heating parameters are electronically controlled (contact thermocouple), a pyrometer is used to check the temperature before, during and after the last forging operation.

The forging process starts after reaching the optimal temperature):

- the initial temperature 1,110-1,150 ^{o}C
- the final temperature 890-850 ^{o}C

Stage 1 - Start from a middle section 875 mm of ingot in Ø180 mm x L (according to Figure 4). Minimum deformation: 23.6:1.

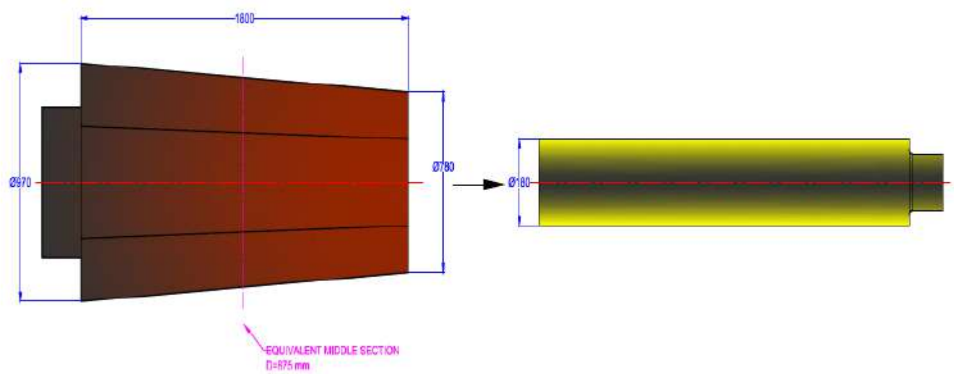


Figure 4. Stage 1 of the forging process according to DNV-GL-ST-F101
(Hot work ratio: 23.6:1)

Stage 2 - Cutting the segments according to the weight of the forged semi-finished product at Ø180 mm x 170 mm (according to Figure 5).

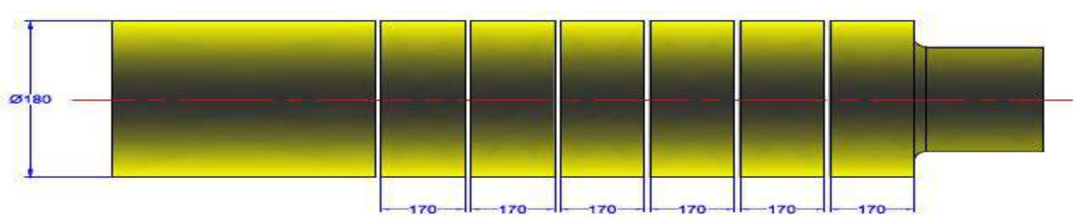


Figure 5. Stage 2 of the forging process according DNV-GL-ST-F101

Stage 3 - Reduction of the section from Ø180 mm x 170 mm to Ø143 x 270 (shown in Figure 6.)

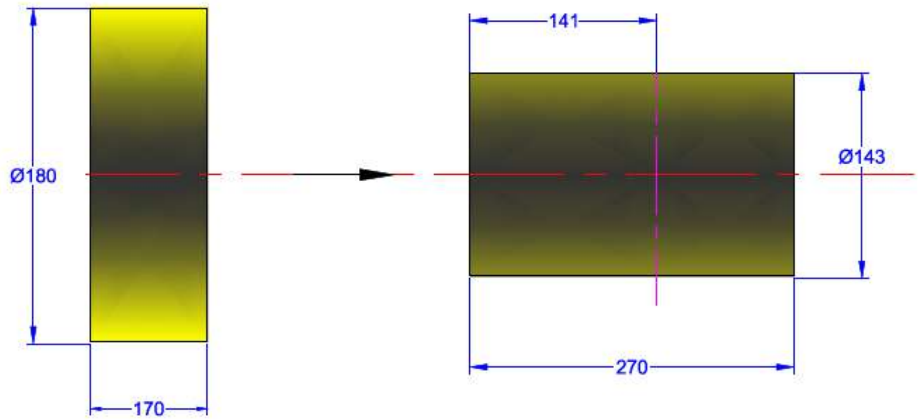


Figure 6. Stage 3 of the forging process according DNV-GL-ST-F101

Stage 4 - Setup from Ø143 x 270 in order to obtain the final forging dimensions (Ø143 x141)x(Ø112 x 191) shown in Figure 7. Total degree of deformation 23.6 x 1.6 = 37.7:1

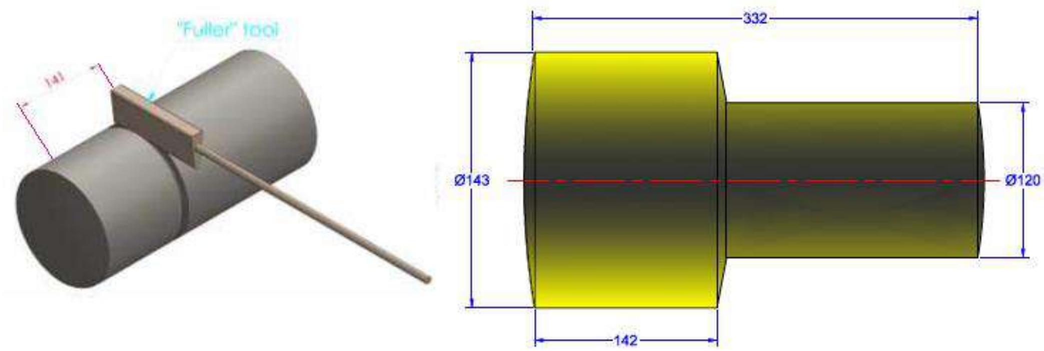


Figure 7. Stage 4 of the forging process according DNV-GL-ST-F101

The forged semi-finished products are cut to the dimensions of the client's execution drawing, on the appropriate machines to ensure the dimensional accuracy and surface quality required in the DNV-GL-ST-F101.

The secondary thermal treatment procedure is performed on a batch of parts of the same type (material quality). [13], [17] The procedure is in accordance with API RP 6HT (DNV-GL-RP-0034 Section 3.1.4.2). All heat treatment furnaces are computer controlled and calibrated according to API 6A Annex M/ASTM A991.

Furnace dimensions 4m x 2m x 1.2m (LxlxÎ). All furnaces are equipped with contact thermocouples connected to the computer with associated thermal treatment graphs. The control system is in line with DNVGL-RP-0034/API RP 6HT.

Table 4 - Thermal treatment parameters

Item	Number of heat-treat. batches.	Normalizing		Austenization		Tempering	
		Temp. °C	Holding time Hours (hrs.)	Temp. °C	Holding time Hours (hrs.)	Temp. °C	Holding time Hours (hrs.)
1	1	890°C	min. 1hrs.	860°C	min. 1 hrs.	685-690°C	min. 2 hrs.
2	1	890°C	min. 1 hrs.	860°C	min. 1 hrs.	685-690°C	min. 2 hrs.

All parts will be loaded in a single row with about 50 mm between them. Furnace orientation: horizontal. The furnace loading method is in accordance with DNV-GL specifications and the sacrificial sample will be placed in the center of the furnace. Thermal treatment parameters are highlighted in Table 4.

Thermal treatment will begin when all contact thermocouples reach the set point with tolerance ±14°C and minimum ±8°C. In the thermal treatment furnace there are: temperature monitoring systems, ventilation systems (turbines), time control and a cooling liquid recirculation system. [13], [17]

Transferring the parts from the thermal treatment furnace to the cooling trough is done within 60 seconds.

Thermal treatment parameters:

1. Normalization:

- loading temperature: <750^{o}C
- heating speed: 100-200 ^{o}C/ hour
- maintaining at austenitization temperature 890 ^{o}C min. 1 hour / contact thermocouple
- cooling in the air

2. Austenitization

- loading temperature: <750^{o}C
- heating speed: 100-200 ^{o}C / hour
- maintaining at austenitization temperature 860 ^{o}C min. 1 hour / contact thermocouple
- cooling in water
- water temperature is max. 20 ^{o}C
- the surface temperature of the parts at the completion of the treatment approx. 50 ^{o}C

3. Recovery

- loading temperature: <550^{o}C
- heating speed: 100-200 ^{o}C/ hour
- maintaining at austenitization temperature 685-690 ^{o} C min. 2 hours / contact thermocouple
- cooling in the air.

The entire heat treatment cycle according to API RP 6HT by using contact thermocouples, at the critical treatment points, will be at least 60 minutes and at least 120 minutes on the recovery treatment.

A special simulation treatment under certain extreme conditions sometimes of use of the piece or to highlight certain mechanical features in our case SPWHT will be:

- heating at 650 °C maintaining 2.5 hours minimum and maximum 5 hours (at a heating temperature of approx. 150 °C / hour).

Final conclusions, original contributions and future research directions

This doctoral thesis presents the stages of making a forged semi-finished product and the required conditions and technologies that the forged semi-finished product must meet in order to meet the requirements of the DNV-GL-ST-F101 standard.

Both the forging technique and the types of heat treatment applied are important to meet the requirements of the assessment standard and to offer the possibility of issuing an internationally recognized quality certificate.

The main conclusions can be summarized as follows:

1. The DNV-GL-ST-F101 standard is important and widely used in the field of metallurgy, especially of forged semi-finished products in the field of extraction, natural gas, oil, marine, etc. The procedure for issuing a material certificate with international recognition DNV-GL type 3.2 involves the fulfillment of some essential criteria both in the field of forging and heat treatment;

2. Forging materials involves obtaining forged semi-finished products in several successive stages and with a certain deformation accepted. A technological forging scheme was created with the optimal parameters for a 12CrMo9-10 semi-finished product as follows:

 steels with a high content of alloying elements that make them harder to forge (in our example 12CrMo9-10), [3] following the tests performed, a high forgability can be attached to it for a maintenance interval of 5-16 hours, ingot 18-25 tons (range in which all critical parameters are favorable in ensuring a good forging technique: forging temperature at 1092-1170 ° C, final forging temperature> 850);

 following the forging process it was also observed that the deformation resistance decreases with the increase of the deformation temperature and decreases with the decrease of the deformation speed. [3] [12] [21]

 according to the presented technology, the experimental data obtained are confirmed graphically (the intersection on the Fe-C diagram with the vertical afferent to a chemical composition of 0.11-0.15% C), revealing the critical points established in the forging technology;

 the application of this forging technology for ingots of 18-25 tons of 12CrMo9-10 led to the reduction of energy consumption (with subsequent reheating processes), the semi-finished product cooling more slowly and reaching a temperature below 850 ° C.

3. The primary heat treatment used in our manufacturing technology is normalization to 941-968 ° C, monitoring with contact thermocouples, maintenance for at least 1 hour after reaching the equalization temperature and cooling to room temperature in the air. The subsequent behavior of the semi-finished product was superior due to the machinability by cutting at subsequent operations, observing the partial elimination of stresses in the part after forging and mechanical properties of plasticity / toughness superior to those after forging.

4. As a secondary heat treatment for this processing technology, a hardening at 940 ° C with 120 minutes holding and cooling in water was applied to the 12CRMo9-10 material, followed by a return to 655 ° C holding for 180 minutes and cooling in air having the following experimental

results: Rm = 697 N / mm2, Rp0.2 = 577 N / mm2, A = 24%, Z = 76%, KV (-46 ° C) = 240 J, HBW = 217. As a fulfillment of the objective of the doctoral thesis (in establishing a superior technology for the elaboration of semi-finished products from 12CrMo9-10), it can be recommended to use on a large scale the parameters obtained in different sectors of the metallurgical industry.

5. In order to highlight the need to use new, superior technologies in the field of forging materials, a series of additional tests were performed on other steels from 42CrMO4 and 25CrMo4 in order to obtain superior mechanical characteristics in operation.

- For the material 42CrMo4 (steel alloyed with Cr and Mo) we used the same parameters as in 12CrMo9-10: forging temperature 1150 ° C (forging end temperature above 850 ° C), ingot 25 tons with the application of the primary heat treatment of normalization. The mechanical characteristics obtained are: Rm = 843 N / mm2, Rp0.2 = 512 N / mm2, A = 15%, Z = 31%, KV (+ 25 ° C) = 210 J, HBW = 255.
- Following the application of the secondary heat treatment, an improved structure favorable to mechanical processing appears, it is no longer prone to cracks or the appearance of intergranular inclusions, but harder but a little more fragile than that obtained from 12CrMo9-10 (following the application of the same MPS technology).
- The same technology was applied to a semi-finished product made of 25CRMO4: 1150 ° C as forging temperature and finally, secondary heat treatment at 860 ° C with maintenance after equalization for 60 minutes and cooling in water followed by a return to 550 ° C with 120 minutes maintenance and cooling in air.
- The variation of the austenitic grain size depends on the concentration of the alloying elements and temperature, in our case for the forged part at 1150 oC of 42CrMo4 a coarse structure was found consisting of lamellar perlite, bainite and ferrite in small proportions;

6. The modeling of the mechanical characteristics depends on the type of operations in the technological flow: free forging, primary TT, secondary TT but also on the chemical composition, the alloying elements of the steels used. Compared to the 2 analyzed technologies, the semi-finished product from 42CrMo4 ensures a higher hardness compared to the one from 12CrMo9-10 which has a higher plasticity.

6.2 Personal and original contributions

In order to fulfill the committed objective of the doctoral thesis, during the doctoral internship we performed scientific documentation activities, laboratory experiments and experiments on pilot installations.

The researches undertaken within the doctoral thesis aimed at elaborating complex specifications regarding the obtaining of forged semi-finished products with special destinations in accordance with the regulated standards.

The topic of the doctoral thesis was proposed and accepted in order to identify the main stages in the analysis of the quality of forged semi-finished products for this purpose, analyzing step by step the characteristics and compliance with standards for different materials of 12CrMo9-10, 42CrMO4 and 25CrMo4.

The original own contributions brought within the doctoral thesis are supported by the following activities:

1. We conducted a documentary study based on the literature on the current state of processing by hot plastic deformation - forging of steel blanks including methods and parameters of the plastic deformation process.
2. We performed a documentary study on the elements of the process of free mechanical forging of steel semi-finished products, the basic operations of the forging process but also the elimination of imperfections, defects and stresses arising from the process.
3. We performed our own research on the process of plastic deformation by forging the different semi-finished products from 12CrMo9-10, 42CrMo4 and 25CrMO4.
4. Following the identification, as a special requirement from most internal and external economic agents, due to the complexity of the manufacturing stages of forged semi-finished products, we have developed a special elaboration procedure (MPS) that brings together all stages and all characteristics required for each type. of material.
Experimental research has focused on several types of materials in order to develop related MPS as part of the concept of quality assurance in this sector.
5. We analyzed in detail all the stages attached to the elaboration procedure for a 12CrMo9-10 semi-finished product: hot plastic deformation, 12CrMo9-10 ingot forging, description of the forging process phases, application of the primary normalization heat treatment, roughing of the analyzed semi-finished product, application secondary heat treatment (hardening + tempering), elaboration of the batch drawing in the oven for secondary treatment.
6. We developed a laboratory study in order to analyze the mechanical characteristics for the analyzed semi-finished product taking into account the analysis of the chemical composition, the description of the mechanical tests and the values obtained, an analysis of the austenitic grain size determination, structural determinations - metallographic analyzes. presentation of all equipment used in this experimental study.
7. We performed the same study in order to approve the elaboration procedure on other materials from 12CrMo9-10, 42CrMo4 and 25CrMo4, highlighting the importance of describing each process but also the transparency in execution.

The main objectives of the doctoral thesis were reflected in national and international articles and papers published in specialized journals. Research results and the importance of developing the execution procedure in quality assurance and increasing credibility at the small and macroeconomic level.

Future directions for the development of research in the doctoral thesis

In the continuation of the researches from the present doctoral thesis, the following can be considered:

-experimental research on the implications of the chemical composition and forging parameters on the final qualitative properties of the product obtained by forging.
-extension of research on other types of semi-finished products processed at the economic agent and with high demand on the market.
-design of some types of ecological heating furnaces for environmental protection.
-designing economical technologies for stamping forged semi-finished products.

-experimental research on the influence of alloying elements on the chemical composition and mechanical characteristics of steels.
- simulative experimental analyzes of the material flow process after stamping with the help of different computer programs such as SolidWorks, Autocad Mechanical, Autodesc Simulation CFD etc.
- analysis of the quality of semi-finished products by design and execution stages;
- simulative studies on the variation of mechanical characteristics with the chemical composition of steels;
- the influence of alloying elements on the chemical composition and mechanical characteristics of steels;
- studies regarding the analysis of fuel consumption assimilated to free forging processes;
- laboratory research on the analysis of the metallographic structure on forged and heat treated semi-finished products;

DISSEMINATION OF THE RESULTS FROM THE DOCTORAL THESIS

Papers published in journals in the field of the thesis:

1. **Caloian, V;** Constantin, N; Vlad,M ,The influence of heating temperature and cooling rate after free forging on the microstructure and the mechanical properties of AISI4140 STEEL, UNIVERSITY POLITEHNICA OF BUCHAREST SCIENTIFIC BULLETIN SERIES B-CHEMISTRY AND MATERIALS SCIENCE Volume: 81 Issue: 1 Pages: 173-182 Published: 2019- **indexata ISI**, https://www.scientificbulletin.upb.ro/SeriaB_-Chimie_si_Stiinta_Materialelor.php?page=indexareWOS:000444602300041
2. **Valentina CALOIAN**, Experimental research on the procedure for the preparation of standardized forged, U.P.B. Sci. Bull., Series B, Vol. 81, Iss. 4, 2019 ISSN 1454-2331, pag253-262-230 , **indexata ISI**, https://www.scientificbulletin.upb.ro/SeriaB_-_Chimie_si_Stiinta_Materialelor.php?page=indexare
3. Costica Mustata, Elena Madalina Vlad, **Valentina Caloian**, Cristian Pandelescu, Research on the loading with pollutants and dust of flue gases resulting from the process of steel production in the LD-type converter; U.P.B. Sci. Bull., Series B, Vol. 82, Iss. 4, 2020 ISSN 1454-2331, pag 261-270, **indexata ISI,** https://www.scientificbulletin.upb.ro/SeriaB_-_Chimie_si_Stiinta_Materialelor.php?page=indexare

4. Cristian PANDELESCU, **Elisa-Florina PLOPEANU**, Nicolae CONSTANTIN, Elena Mădălina VLAD; Analysis of the current situation concerning the duration of use and the main defections of the pumping aggregates, ACTA TECHNICA CORVINIENSIS – Bulletin of Engineering [e–ISSN: 2067–3809], TOME XIII [2020] | FASCICULE 2 [April – June], pag 147-152, indexata B+, BDI , http://acta.fih.upt.ro/index.html

Papers presented at International Conferences, indexatae ISI, in the field of thesis:

1. E M Vlad, **V E Caloian**, C Pandelescu, E F Plopeanu, V Oancea, V Rucai, N Constantin and M Hritac, Experimental research on the effect of additives on the sintering process of alumina-based refractory materials, Conferinta Internationala, International Conference on Applied Sciences – ICAS 2020, May 22, 2020, Hunedoara, Romania, indexata ISI, http://icas.science/forms/Program_ICAS2020.pdf

2. **V E Caloian**, E M Vlad, C Pandelescu, E F Plopeanu, V Oancea, V Rucai, N Constantin and M Hritac, Experimental research with the help of thermal - derivatographic analysis on coal powder that can be blowed in the blast furnace, Conferinta Internationala, International Conference on Applied Sciences – ICAS 2020, May 22, 2020, Hunedoara , Romania, **indexata ISI**, http://icas.science/forms/Program_ICAS2020.pdf

3. **V E Caloian (Necula),** The procedure for drawing up specifications for flanged forged semi-finished products of A182F22 with special purpose for naval systems, Conferinta Internationala, 8th International Conference on Materials Science and Technologies – RoMat 2020 November 26-27, 2020 Bucharest, Romania, http://www.sim.pub.ro/index.php/47-noutati/anunturi/227-romat2020

4. **V E Caloian (Necula), Nicolae Constantin, Adrian Ioana, M E Vlad** The procedure for drawing up specifications for flanged forged semi-finished products of A694F65 with special purpose for naval systems, 14 th International research conference, International conference on applied mechanics and materials engineering, IRC 2020, April 16-17, 2020 Lisbon, Portugal, https://waset.org/applied-mechanics-and-materials-engineering-conference-in-april-2020-in-lisbon

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