Case MC: Material Characterization of stainless-steel flange

PRACTICAL SESSION

Material testing and NDT

CERN

ENGINEERING DEPARTMENT

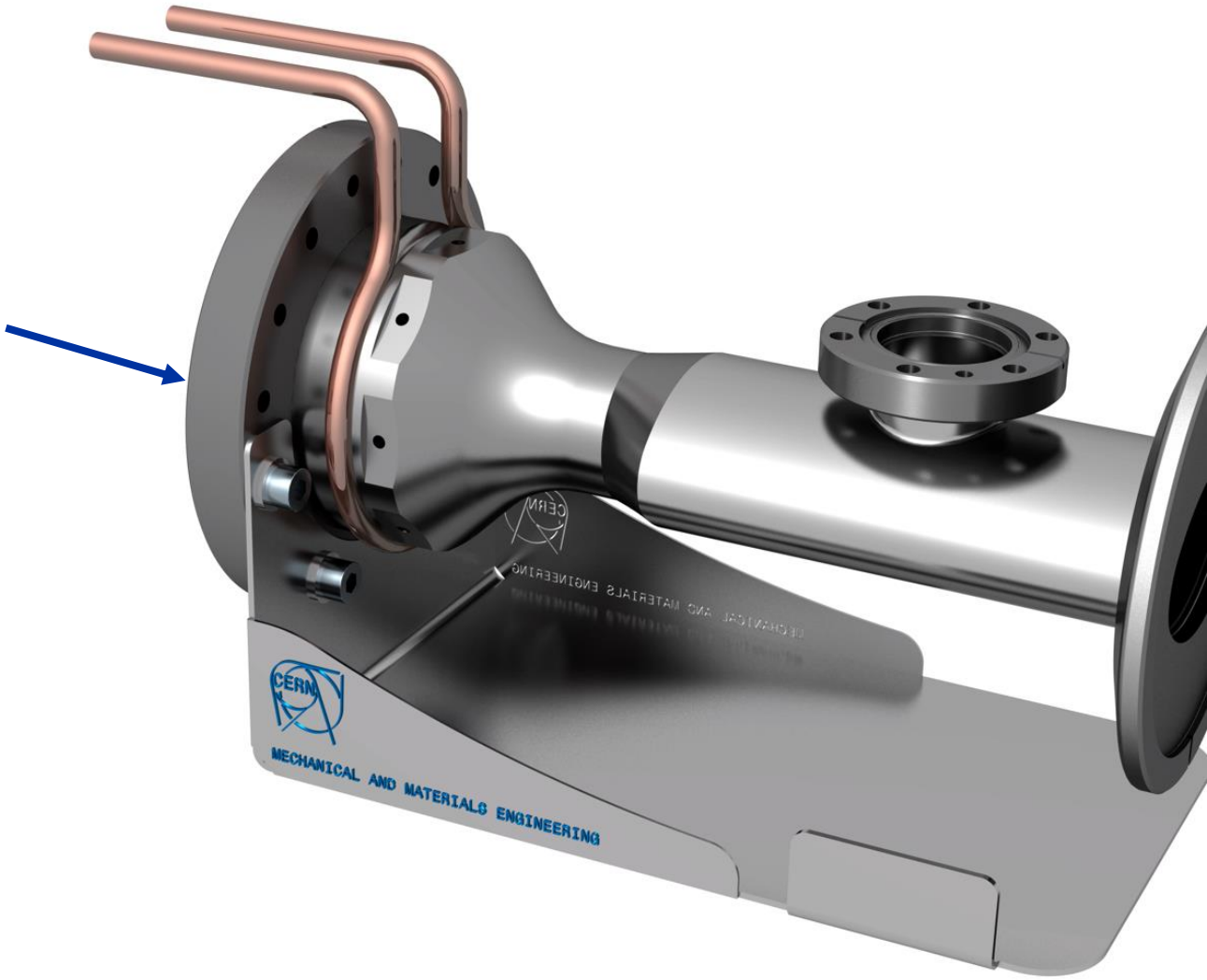
MECHANICAL & MATERIALS ENGINEERING
FOR PARTICLE ACCELERATORS AND DETECTORS

Context

Characterization of the material used for the fabrication of the flange

Timeline :

- 10 min : Introduction
- 40 min : Exercise



Objective

The goal of this hands-on experience is:

***Select** the right technical specification based on the use of the component.*

***Understand** the different sections of a material certificate.*

***Perform** the required **measurements** on material samples to complete some missing information in the certificate: chemical composition, hardness, grain size.*

***Compare** the material certificate with the technical specification to **determine** its **compliance**.*

Available material

This guide: *contains all the practical information to use the instruments you will use for the material characterization. At the end there are some question that (we hope) will help you understand why do we request all these material properties.*

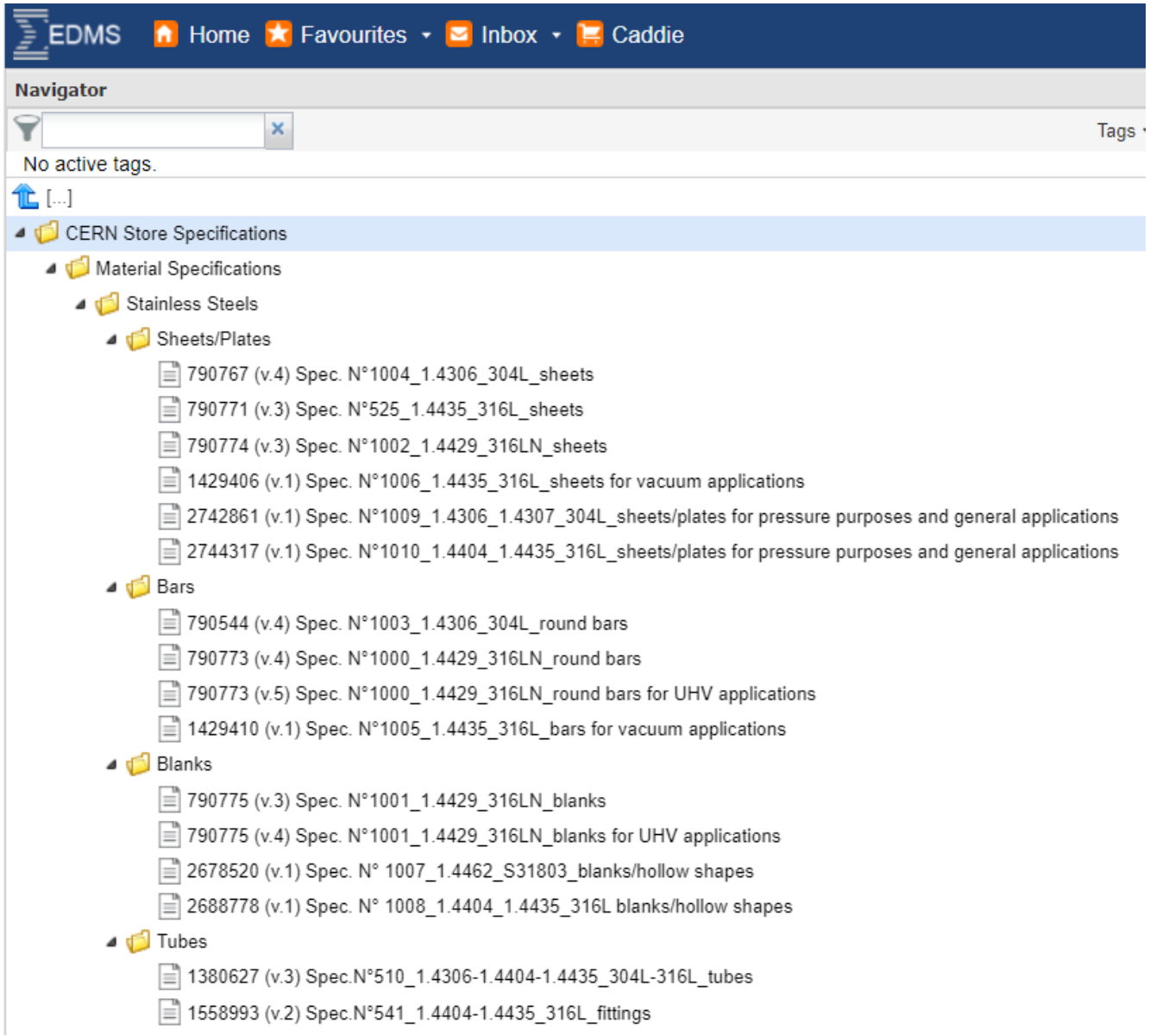
Technical specifications: *curated by the best material experts, they contain all the required properties depending on the use you intend for your material. Which one would you use, and why?*

Material certificate: *a real material certificate you will need to complete and compare to the technical specification you have selected. Is the material compliant, or would you like to do a non – conformity report?*

Helpers: *if there is something unclear or you need help with anything, let us now!*

Compliance with the material certificate

Section	Requirements	Comments	Checked
1	**Normative references**	**Standards mentioned in the document**	
2	**Requirements**	**According to industrial standards and CERN special requirements**	
2.1	*Manufacturing Process*	Mandatory step of electro slag remelting (ESR) + solution annealing + multi-directionally forged	Yes No
2.2	*Chemical Composition*	Co, N, Impurities	
2.3	*Structure*	After solution annealing shall be completely austenitic and homogenous	
2.4	*Inclusions Content*	Macro-inclusions are strictly forbidden, and micro-inclusions estimated	
2.5	*Mechanical Properties*	At room temperature, after solution annealing	
2.6	*Magnetic Properties*	At room temperature, after solution annealing	
2.7	*Surface quality*	All surfaces shall be pre-machined, clean, and free from scale, paint and any other foreign matter that could adversely affect the sensitivity of the non-destructive testing or cause errors in interpretation	
2.8	*Internal Soundness*	UT and PT	
3	**Inspection and testing**		
3.1	*General*	Quality control shall be carried out in accordance with an Inspection and Test Plan (ITP) established between the manufacturer and CERN	
3.2	*Test methods*	Test + applicable standard + extension	
3.3	*Identification*	Marking of the products + traceability number between delivered lot and inspection documents	
3.4	*Inspection certificate*	EN 10204* Type 3.1 is required	
4	**Packing**		


EDMS
Home
Favourites
Inbox
Caddie
Navigator
Tags
No active tags.
[...]
CERN Store Specifications
Material Specifications
Stainless Steels
Sheets/Plates
790767 (v.4) Spec. N°1004_1.4306_304L_sheets
790771 (v.3) Spec. N°525_1.4435_316L_sheets
790774 (v.3) Spec. N°1002_1.4429_316LN_sheets
1429406 (v.1) Spec. N°1006_1.4435_316L_sheets for vacuum applications
2742861 (v.1) Spec. N°1009_1.4306_1.4307_304L_sheets/plates for pressure purposes and general applications
2744317 (v.1) Spec. N°1010_1.4404_1.4435_316L_sheets/plates for pressure purposes and general applications
Bars
790544 (v.4) Spec. N°1003_1.4306_304L_round bars
790773 (v.4) Spec. N°1000_1.4429_316LN_round bars
790773 (v.5) Spec. N°1000_1.4429_316LN_round bars for UHV applications
1429410 (v.1) Spec. N°1005_1.4435_316L_bars for vacuum applications
Blanks
790775 (v.3) Spec. N°1001_1.4429_316LN_blanks
790775 (v.4) Spec. N°1001_1.4429_316LN_blanks for UHV applications
2678520 (v.1) Spec. N° 1007_1.4462_S31803_blanks/hollow shapes
2688778 (v.1) Spec. N° 1008_1.4404_1.4435_316L blanks/hollow shapes
Tubes
1380627 (v.3) Spec.N°510_1.4306-1.4404-1.4435_304L-316L_tubes
1558993 (v.2) Spec.N°541_1.4404-1.4435_316L_fittings

ORGANISATION EUROPÉENNE POUR LA RECHERCHE NUCLÉAIRE
EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

Materials Technical Specification
EN-MME

06.11.2023

Technical Specification

N° 1001 - Ed. 6
EDMS N°: 790775

Stainless steel forged blanks for ultra-high vacuum applications

1.4429

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EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

Materials Technical Specification
EN-MME

06.11.2023

Technical Specification

N° 1000 - Ed. 6
EDMS N°: 790773

Stainless steel round bars for ultra-high vacuum applications

1.4429

Compliance with the material certificate

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TECH SPECIFICATION

2. REQUIREMENTS

The stringent requirements of this material specification for products intended for UHV purposes, impose to apply an adapted steelmaking and manufacturing process, aimed at meeting the structure and inclusion limits outlined in the document.

2.1 STEELMAKING AND MANUFACTURING PROCESS

1.4429 ingots shall be produced including a mandatory step of electro slag remelting (ESR).

The manufacturing process shall be reported in the inspection document and include solution annealing treatment.

For small diameters, the bars may be rolled. The manufacturer is required to specify the production method (forged or rolled) for each diameter.

The bars shall be pickled.

Kunden-Bestell-Nr. **8520**
Customer order no.
Cde. no. du client **Lager Imbach**

BGH-Auftrags-Nr. **816625-003-01**
BGH works no.
BGH référence

BGH
Q 33

Product	Round billet,forged,peeled
Quality	1.4429 X2CrNiMoN17-13-3
Requirements	CERN No. 1000 Edition 5 12/13 AISI 316 LN ESR

Inspection and dimensional control	Meltingprocess/secondary refining	Identification test (spectral-analysis)
without objection	E-AOD-VD/ESR	without objection

Pos. Item Poste	Anzahl Quantity Quantité	Abmessung Dimension Dimensions
3		OD 250 mm x 3141 - mm

Schmelze Heat %	C	Si	Mn	P	S	Cr	Mo	Ni	N	B	Co	Fe
924997	0,014	0,37	1,90	0,017	0,0010				0,1778	0,0014	0,024	63,91

Condition of heat treat: solution annealed

Compliance with the material certificate

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2.2 CHEMICAL COMPOSITION

The cast analysis shall comply with the requirements indicated here below including CERN special requirements.

Element	Chemical composition % by mass
Cr	16.00 – 18.50[1]
Ni	12.00 – 14.00[1]
C	0.030 max.
Si	1.00 max.
Mn	2.00 max.
Mo	2.50 – 3.00[1]
N	0.14 – 0.20[1]
P	0.030 max.[1]
S	0.010 max.[1]
Fe	Remainder

Special requirements:

Cobalt shall be present only as a trace or to a maximum content of 0.10%, including measuring tolerance.

The nitrogen content between 0.14% and 0.20% is imperative.

Content of impurity elements (P, S, B) shall reach the lowest achievable level.

Elements not listed in this table shall not be intentionally added to the steel without the agreement of CERN.

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BGH-Auftrags-Nr. **816625-003-01**
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BGH
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Inspection and dimensional control	Meltingprocess/secondary refining	Identification test (spectral-analysis)
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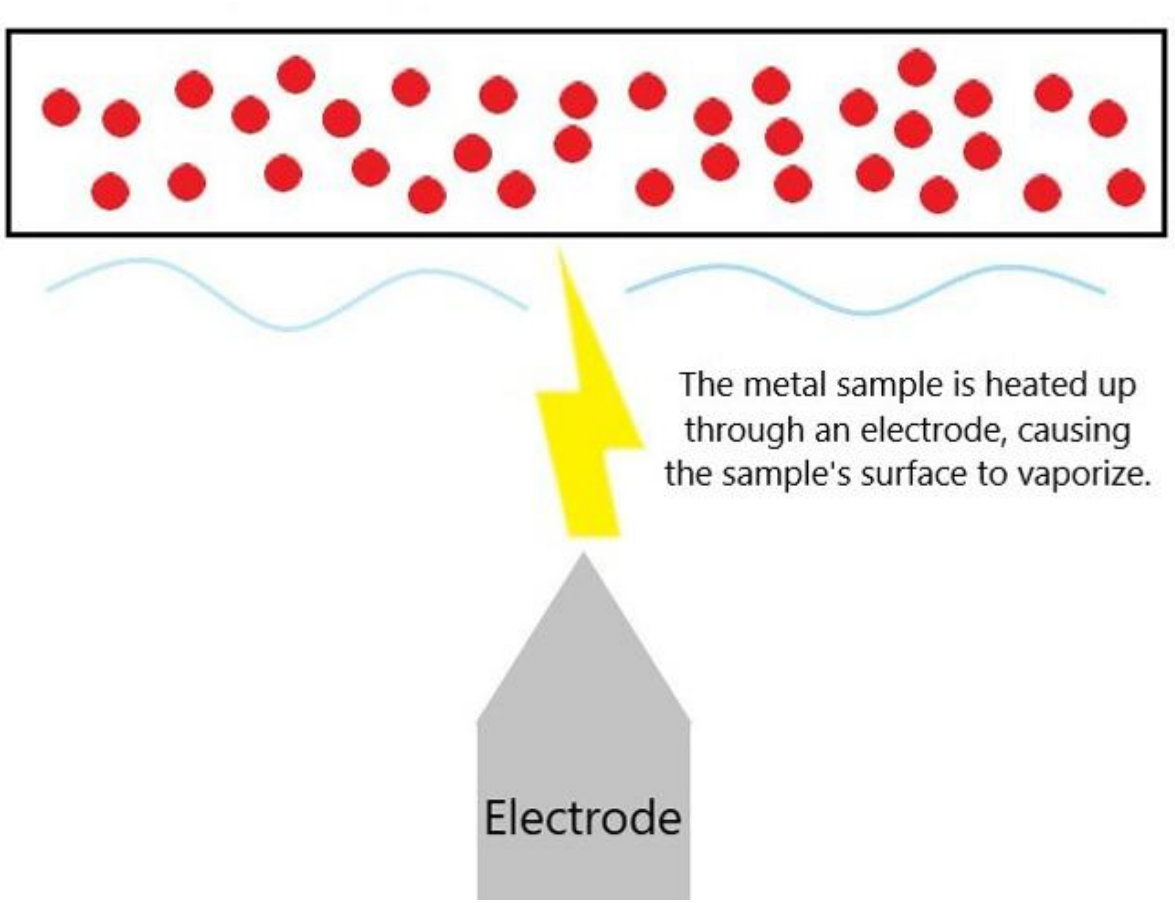
Condition of heat treat: solution annealed

Optical emission spectroscopy (OES)



We need to have a good contact between the electrode head (gun) and the sample surface

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2.3 STRUCTURE

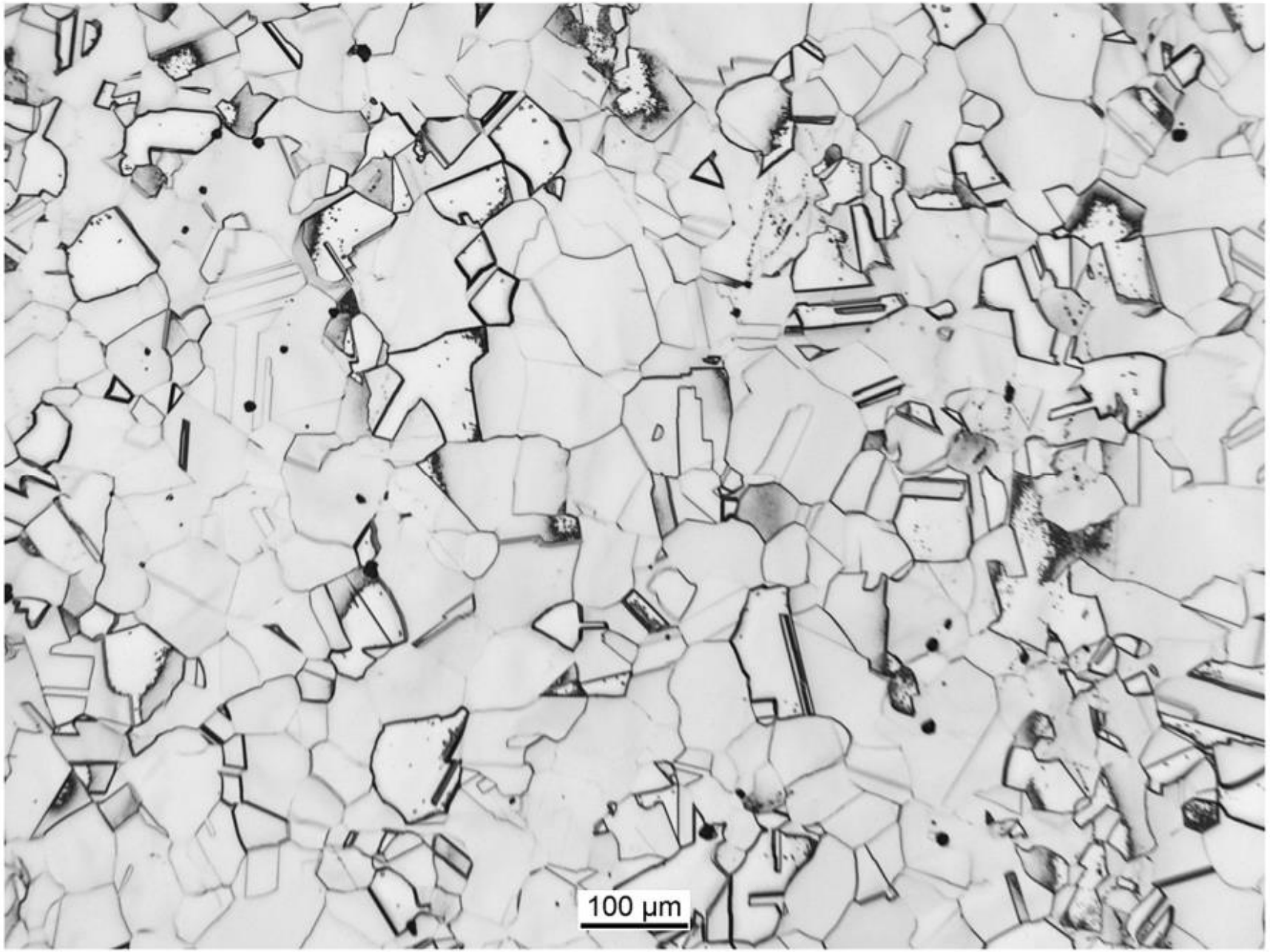
The structure after solution annealing shall be completely austenitic and homogenous. Segregations, as well as presence of intermediate phases, such as Sigma, Chi, Laves…, are not allowed in the final product.

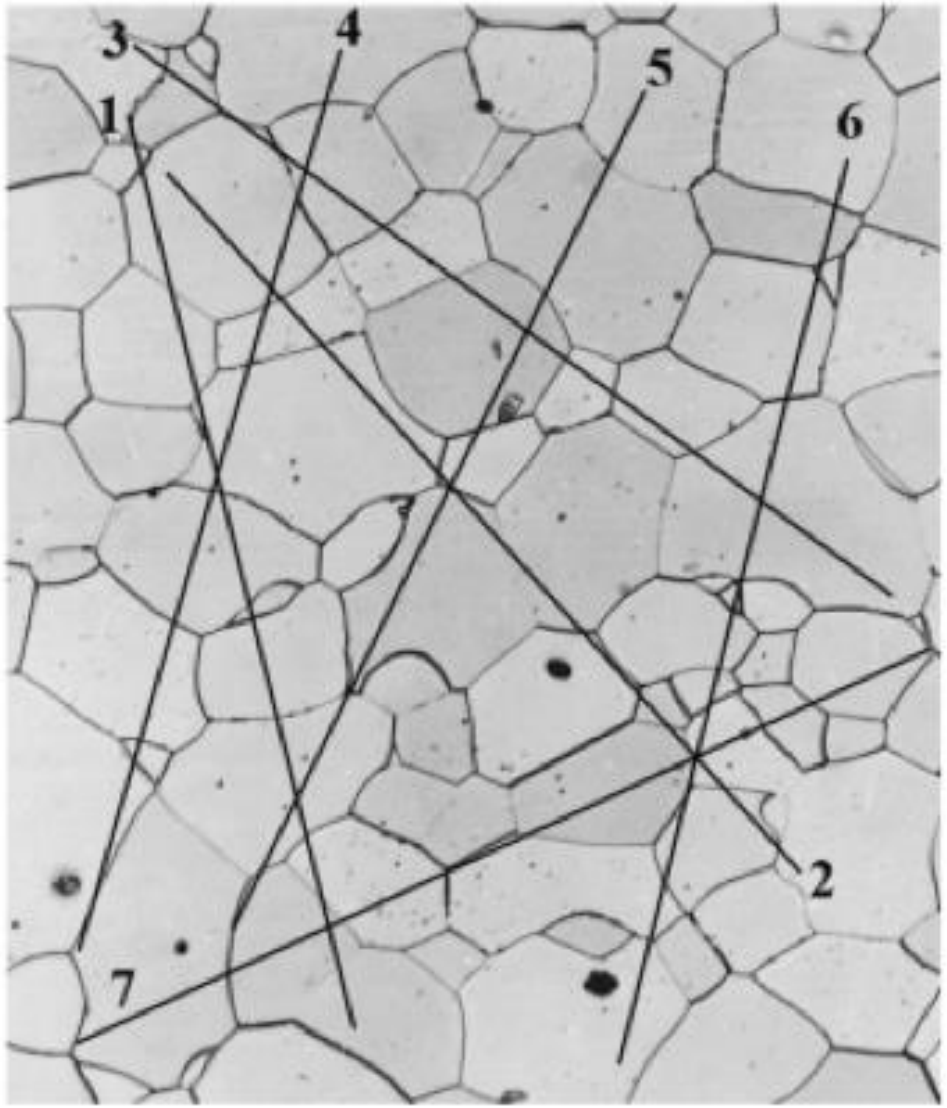
The equivalent grain size number according to ASTM E112 shall be, in average, equal or greater than 3. The grain size shall be homogeneous within the range of ± 0.5 equivalent grain size number around the true average value.

Grain size acc. ASTM E 112:

Microstructure: austenite

The examined samples were free of segregation and deteriorating phases.


100 µm



Line	Number of interceptions
1	
2	
3	
4	
5	
6	
7	
average number of grain boundary intersections	

Average line length intercepted (A) = total length/average
average grain diameter = A/magnification

Grain size

Measuring grain size

- *We will show you here how to measure grain size from a metallographic preparation. It is simple, intuitive and a very useful tool.*
- *When we talk about grain size, in reality, we are talking about the average diameter of of the grains, so it is important that the grains are homogeneous (at least locally) and that they are equiaxial (more or less round, not elongated in any direction).*
- *We need a properly done metallographic preparation, a ruler and a pencil / pen / marker*
- *We will apply an intercept method, this is, counting the number of intersections between a segment of a known length and the grain boundaries.*
- *Tips:*
  - *The longer your segments are, the more precise your estimation will be.*
  - *To reduce systematic errors or biases, draw segments in random directions, not preferential directions.*
  - *The more segments you draw, the smaller your statistical error will be (but do not exaggerate here, remember it is a tool, not a standardized measurement)*
  - *Be careful with twin boundaries: they are not grain boundaries they count 0.5*

TABLE 4 Grain Size Relationships Computed for Uniform, Randomly Oriented, Equiaxed Grains

Grain Size No. G	$\bar{N}_A$ Grains/Unit Area		$\bar{A}$ Average Grain Area		$\bar{d}$ Average Diameter		$\bar{\ell}$ Mean Intercept		$\bar{N}_L$
	No./in.^{2} at 100X	No./mm^2 at 1X	mm^2	μm^2	mm	μm	mm	μm	No./mm
00	0.25	3.88	0.2581	258064	0.5080	508.0	0.4525	452.5	2.21
0	0.50	7.75	0.1290	129032	0.3592	359.2	0.3200	320.0	3.12
0.5	0.71	10.96	0.0912	91239	0.3021	302.1	0.2691	269.1	3.72
1.0	1.00	15.50	0.0645	64516	0.2540	254.0	0.2263	226.3	4.42
1.5	1.41	21.92	0.0456	45620	0.2136	213.6	0.1903	190.3	5.26
2.0	2.00	31.00	0.0323	32258	0.1796	179.6	0.1600	160.0	6.25
2.5	2.83	43.84	0.0228	22810	0.1510	151.0	0.1345	134.5	7.43
3.0	4.00	62.00	0.0161	16129	0.1270	127.0	0.1131	113.1	8.84
3.5	5.66	87.68	0.0114	11405	0.1068	106.8	0.0951	95.1	10.51
4.0	8.00	124.00	0.00806	8065	0.0898	89.8	0.0800	80.0	12.50
4.5	11.31	175.36	0.00570	5703	0.0755	75.5	0.0673	67.3	14.87
5.0	16.00	248.00	0.00403	4032	0.0635	63.5	0.0566	56.6	17.68
5.5	22.63	350.73	0.00285	2851	0.0534	53.4	0.0476	47.6	21.02
6.0	32.00	496.00	0.00202	2016	0.0449	44.9	0.0400	40.0	25.00
6.5	45.25	701.45	0.00143	1426	0.0378	37.8	0.0336	33.6	29.73
7.0	64.00	992.00	0.00101	1008	0.0318	31.8	0.0283	28.3	35.36
7.5	90.51	1402.9	0.00071	713	0.0267	26.7	0.0238	23.8	42.04
8.0	128.00	1984.0	0.00050	504	0.0225	22.5	0.0200	20.0	50.00
8.5	181.02	2805.8	0.00036	356	0.0189	18.9	0.0168	16.8	59.46
9.0	256.00	3968.0	0.00025	252	0.0159	15.9	0.0141	14.1	70.71
9.5	362.04	5611.6	0.00018	178	0.0133	13.3	0.0119	11.9	84.09
10.0	512.00	7936.0	0.00013	126	0.0112	11.2	0.0100	10.0	100.0
10.5	724.08	11223.2	0.000089	89.1	0.0094	9.4	0.0084	8.4	118.9
11.0	1024.00	15872.0	0.000063	63.0	0.0079	7.9	0.0071	7.1	141.4
11.5	1448.15	22446.4	0.000045	44.6	0.0067	6.7	0.0060	5.9	168.2
12.0	2048.00	31744.1	0.000032	31.5	0.0056	5.6	0.0050	5.0	200.0
12.5	2896.31	44892.9	0.000022	22.3	0.0047	4.7	0.0042	4.2	237.8
13.0	4096.00	63488.1	0.000016	15.8	0.0040	4.0	0.0035	3.5	282.8
13.5	5792.62	89785.8	0.000011	11.1	0.0033	3.3	0.0030	3.0	336.4
14.0	8192.00	126976.3	0.000008	7.9	0.0028	2.8	0.0025	2.5	400.0

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2.4 INCLUSIONS CONTENT

Amount and definition shall meet standard ASTM E45.

- Micro-inclusions (indigenous inclusions detectable by microscopical test methods): method D is applicable. Severity level number shall be at most 1 for types A, B and C and at most 1.5 for type D. The tolerance for acceptance may be a half-class above the set limit to the extent of 2% of the fields counted. The table showing field counts shall be attached to the certificate;

- Macro-inclusions (exogenous inclusions from entrapped slag or refractories): they are strictly forbidden and are cause for rejection.

Inclusion content acc. ASTM E 45, method D:

Type	A	B	C	D
thin	0,0	0,4	0,2	0,6
heavy	0,0	0,2	0,1	0,4

Permeability : 1,004 µr

Compliance with the material certificate

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2.5 MECHANICAL PROPERTIES

At room temperature, after solution annealing:

Tensile strength	R_m	min.	600 N/mm²
0,2% proof strength	$R_{p0.2\%}$	min.	300 N/mm²
Elongation at break (long)	A_5	min.	35 %
Brinell hardness	HBW	max.	150-190

Probe-Nr. Test-No.	Lage Loc.	Temp. °C	Rp0,2 N/mm²		Rm N/mm²	A5 %	Z %		Hardness HB
Req.	T	RT	>=300		>=600	>=35			>=160 <=190
540V1 540V2	T	RT	320		632	52,3	78		170-163 172-160

Mechanical values determined on a sample at the bar.

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2.6 MAGNETIC PROPERTIES

The relative magnetic permeability at room temperature after solution annealing shall be lower than or equal to 1.005 for fields of over 80 000 A/m (equivalent to 1000 Oe).

Permeability : 1,004 µr

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2.1	*Manufacturing Process*	Mandatory step of electro slag remelting (ESR) + solution annealing + multi-directionally forged	Yes No
2.2	*Chemical Composition*	Co, N, Impurities	
2.3	*Structure*	After solution annealing shall be completely austenitic and homogenous	
2.4	*Inclusions Content*	Macro-inclusions are strictly forbidden, and micro-inclusions estimated	
2.5	*Mechanical Properties*	At room temperature, after solution annealing	
2.6	*Magnetic Properties*	At room temperature, after solution annealing	
2.7	*Surface quality*	All surfaces shall be pre-machined, clean, and free from scale, paint and any other foreign matter that could adversely affect the sensitivity of the non-destructive testing or cause errors in interpretation	
2.8	*Internal Soundness*	UT and PT	
3	**Inspection and testing**		
3.1	*General*	Quality control shall be carried out in accordance with an Inspection and Test Plan (ITP) established between the manufacturer and CERN	
3.2	*Test methods*	Test + applicable standard + extension	
3.3	*Identification*	Marking of the products + traceability number between delivered lot and inspection documents	
3.4	*Inspection certificate*	EN 10204* Type 3.1 is required	
4	**Packing**		

*EN 10204 Metallic Products—Types of Inspection Documents, with a simplified range of inspection documents (certificate types)

Type	Title	Summary of EN 10204 Requirements
2.1	Declaration of Compliance with the Order	Statement of compliance with the order by the manufacturer.
2.2	Test Report	Statement of compliance with the order by the manufacturer based on non-specific inspections (tests) by the manufacturer
3.1	Inspection Certificate	Statement of compliance with the order by the manufacturer with results of specific inspection
3.2	Inspection Certificate	Statement of compliance with the order with indication of results of specific inspection

The latter two types are the most widely used in steel products. What are their differences?

- The **EN 10204 Type 3.1 Inspection Certificate** are actual test results from the material in the lot from which the steel products have been supplied (formally referred to as “specific testing”). It is a document issued by the manufacturer in which he declares that the products supplied comply with the requirements of the order and in which he supplies test results. The test unit and the tests to be carried out are defined by the product specification.
- Manufacturer may transfer on to the inspection certificate 3.1 relevant test results obtained by specific inspection on primary or incoming products he uses (such as – for example – results of chemical composition analysis or mechanical properties of the strip, provided on the Material Test Reports supplied by the vendor of strip)

- The **EN 10204 Type 3.2 Inspection Certificate** is like the 3.1 but has additionally been countersigned and verified by an independent third-party to validate the material by way of verification test.

Guides to instruments

Portable hardness

Connecting the Probe

Cable Connection: Locate the cable for connecting the probe to the machine (Figure 1). Ensure the cable marking is "35600735 KW 03/21 ST".

Machine (top left), calibration block (top right), probe (bottom left) and connector (bottom right).

Portable hardness

Connecting the Probe

Machine and Probe Connection: Attach the cable to the machine and the probe. Ensure the probe is covered during this process to avoid damage.

Connection of the probe connector to the machine. The arrow indicates the correct port.

Portable hardness

Turning on and setting up the device

Powering On: Activate the machine by pressing and holding the power button for a few seconds.

Adjusting Measurement Settings: Navigate to “Settings” > “Measurement” to adjust the desired force for your measurements (HV5 in our case).

Settings menu

Portable hardness

Turning on and setting up the device

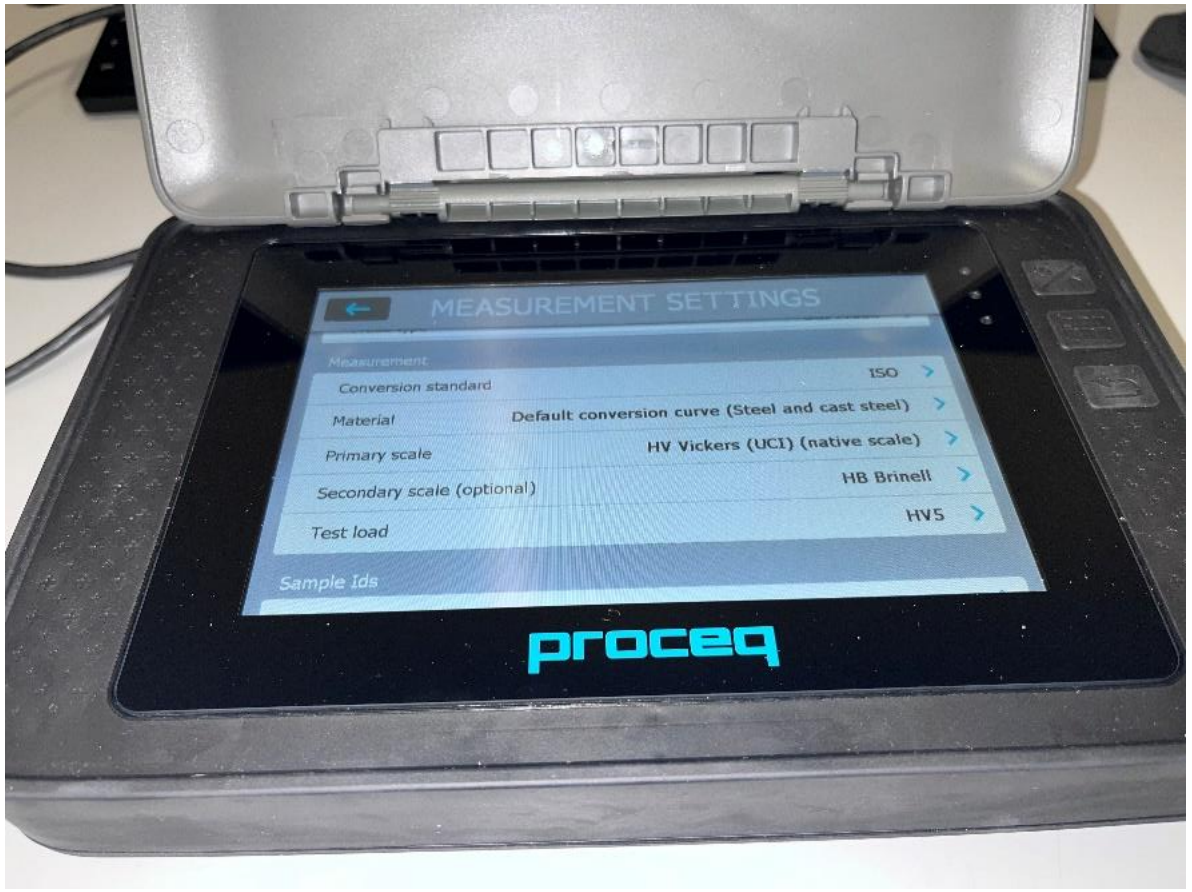
Accessing the Measurement Menu: Return to the main menu by pressing the power button briefly, then select the “Measurement” menu to prepare for testing.

Measurement interface. Drop-down menu at the upper middle and settings at the bottom right (cogwheel).

Portable hardness

Turning on and setting up the device

Go to the measurement settings (cogwheel at the bottom right) to make sure the setup of the measurement is correct: mainly check primary scale is HV and test load is HV5.



Test parameters within the measurement test, including the Test Load configuration.

Portable hardness

Performing Measurements

Remove the cover from the probe.

Place the probe on a flat, stable surface large enough to accommodate it.

Keep the probe perpendicular to the surface.

Apply force until a successful sound is coming from the machine and the value appears.

Indication of ready to position the probe

Portable hardness

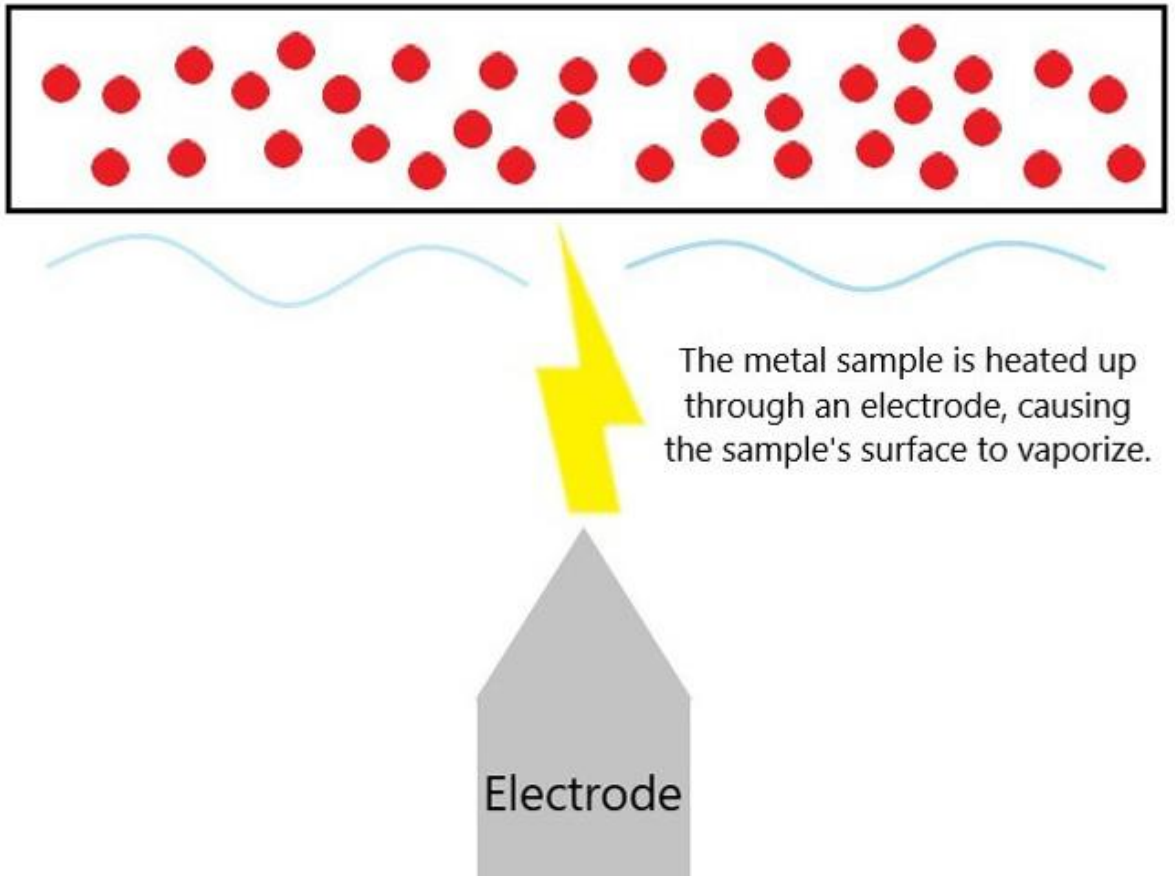
Performing Measurements

If insufficient force is applied, an "Incorrect" message, a cross symbol, along with a warning sound and vibration will alert you.

An over force will trigger a warning message and sound.

Warning that indicates that it is necessary to apply more force.

Optical emission spectroscopy (OES)



We need to have a good contact between the electrode head (gun) and the sample surface

Optical emission spectroscopy (OES)

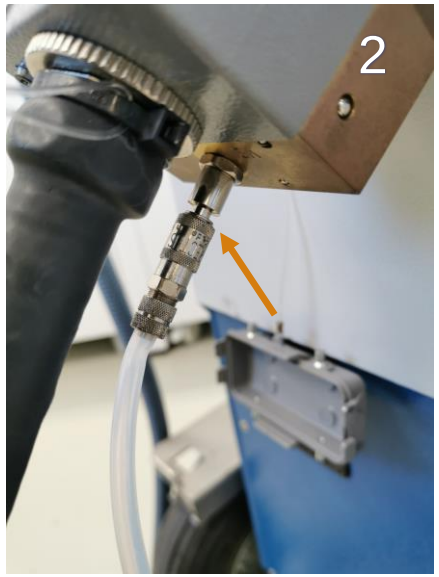
<u>Setting up of the spectrometer</u>

Connect the probe (1),

connect the argon line (2),

open the gas valve and set the delivered pressure around 3,5 bars (3),

switch on the equipment (4).



Optical emission spectroscopy (OES)

Surface preparation

Place a new corundum griding disk onto the adaptor and tighten the screw on it while blocking the axle by pressing the button at the back (1),

Equip the grinding tool with its battery (2),

Tighten your sample, wear gloves and safety glasses and grind the surface to obtain a fresh plane, your sample is ready for measurement (3).

Optical emission spectroscopy (OES)

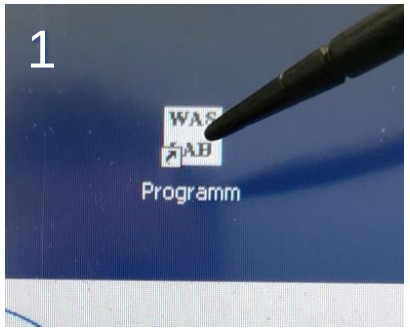
Software initialization

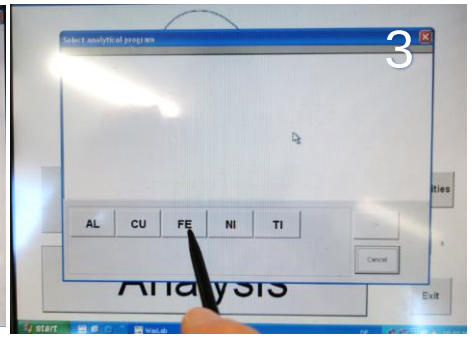
Click on WAS lab icon (1),

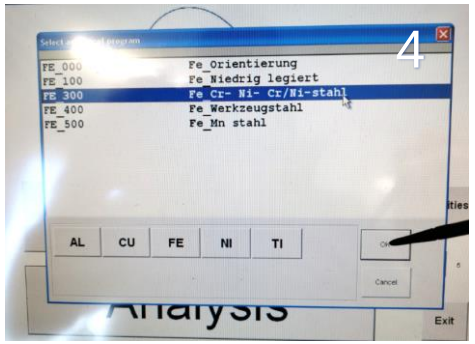
Select “Analysis” then “Spark” (2),

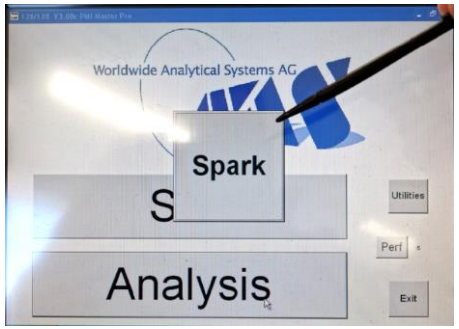
Select the matrix corresponding to your material (3),

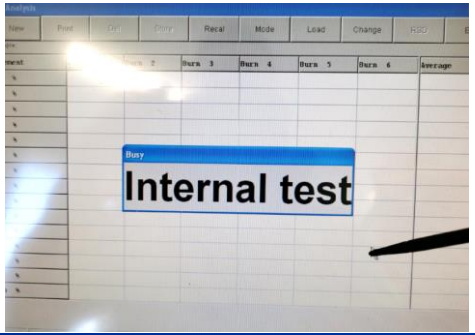
If known, select the alloy grade; if not select the first program “material-orientierung”. Wait few seconds during the internal test (4).











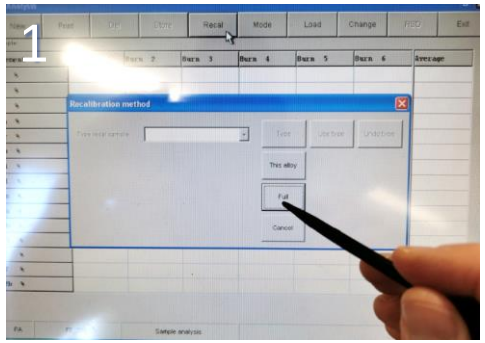
Optical emission spectroscopy (OES)

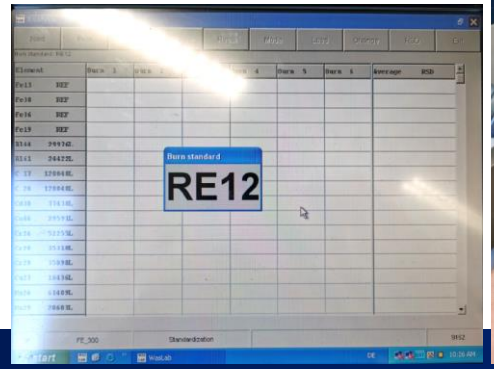
Calibration 1/2

Click on “Recal” to initiate the recalibration mode. Select full to recalibrate the matrix. The first stand will be asked for measurement (1),

Place the standard, previously ground, onto the probe and keep pressing the trigger as illustrated until analysis is complete (2),

Clean gently the tungsten electrode of the probe with the metallic brush after each spark (3).





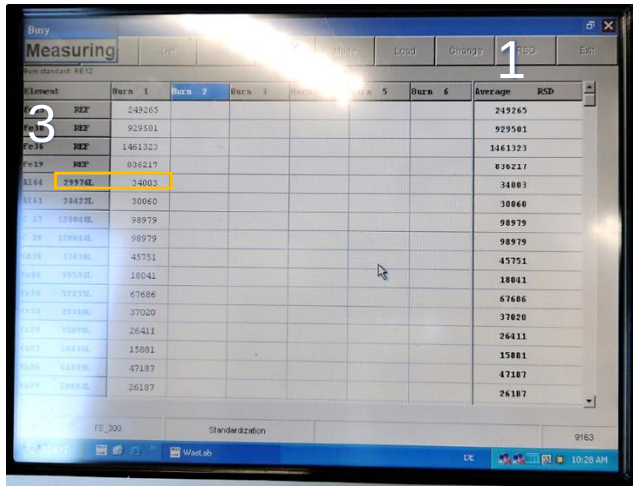
Optical emission spectroscopy (OES)

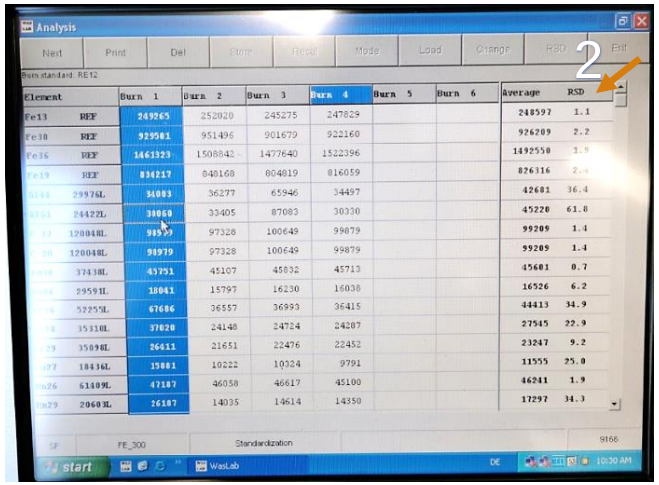
Calibration 2/2

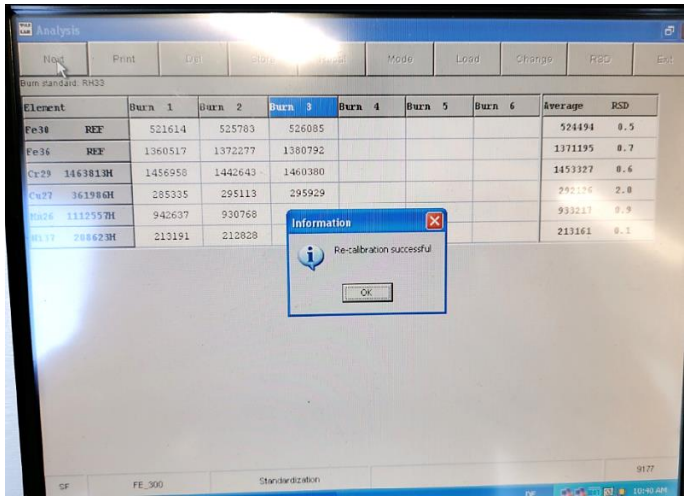
The first measure appears onto the screen, make sure that intensity values are close to expected ones (1),

Repeat the measurements and delete column with erroneous values. In addition, RSD has to be kept under 5%. Keep 3 conform measurements and click on next button to proceed with the next calibration block (2),

After the last calibration block, press "next" button, a message will inform that the calibration is successful (3). The system is ready for analyse.







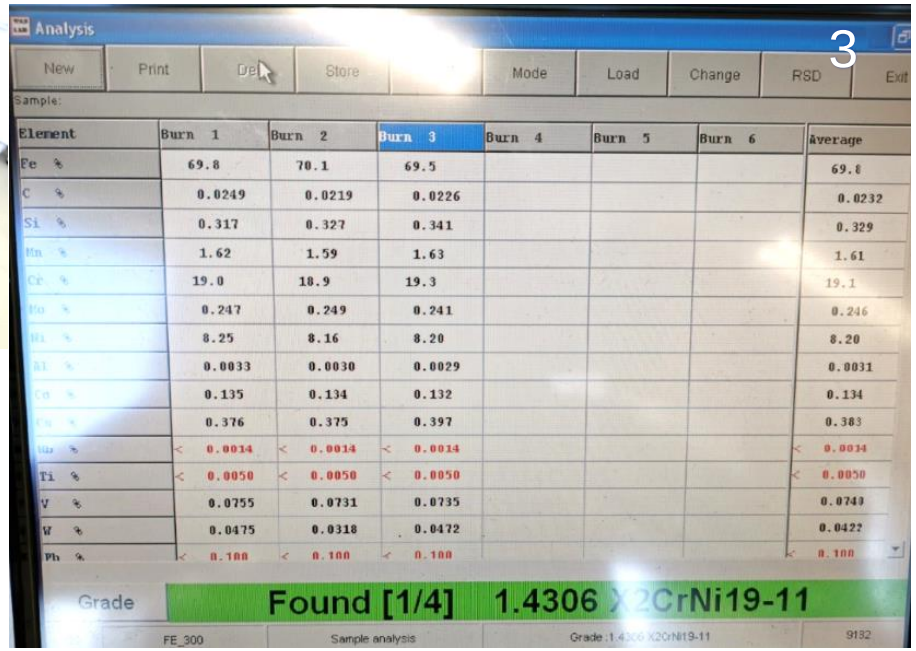
Optical emission spectroscopy (OES)

Measuring

Place the probe in close and flat contact onto your sample previously ground and keep pressing the trigger until analysis is complete (no spark and values displayed onto the screen) (1),

Clean gently the tungsten electrode of the probe with the metallic brush after each spark and repeat the measurement between 3 and 5 times (2),

Double click on column to be deleted (erroneous values, non-conform spark), material identification with statistics are displayed (3),



Questions

Which material specification did you choose? Why? Are several specifications compatible with the final product? (5 minutes)

Hardness:

- Measure the hardness in at least 5 positions and report the average in the material cerficate (10 minutes).
- Is there any problem to measure HV5 if the technical specification requires HV10? Is there any advantage or disadvantage between the different scales (HV1, HV5, HV10…)?

Chemical composition:

- Measure the chemical composition in at 3 positions and report the average of the missing elements in the material certificate (10 minutes).
- Do you know why P + S content is requested to be so low? And the Co?
- Do you know what does the ‘L’ and the ‘N’ mean in 316LN?

Grain size:

- Measure the average grain size and report it in the material certificate (10 minutes).
- Are the material grains equiaxial? Is that important? Why?

Is the material certificate compliant with the technical specification? (5 minutes)

We hope you enjoyed characterizing a material for a particle accelerator

CERN

ENGINEERING DEPARTMENT

MECHANICAL & MATERIALS ENGINEERING
FOR PARTICLE ACCELERATORS AND DETECTORS