ICEC/ICMC

29th International Cryogenic Engineering Conference
International Cryogenic Material Conference 2024
July 22-26, 2024, Geneva, Switzerland

Ignacio Avilés Santillana
CERN EN / MME – MM
Email: iaviles@cern.ch

(metallic) Materials selection for cryogenic applications

CERN

Outline

- Introduction to cryogenic materials
- General rules for materials selection at cryogenic temperature
- Families of cryogenic materials:
  - Stainless steel
  - Aluminum
  - Copper
  - Titanium
  - Niobium
  - Ni alloys
- Conclusions

Introduction

- When materials are cooled to cryogenic temperatures, their properties can change significantly. Properties like thermal and electrical conductivity might increase, as well as yield strength and ultimate tensile strength, but ductility and toughness typically decrease, and for some materials, there is even a transition from ductile to brittle.
- To ensure the safe design of cryogenic equipment, engineers depend on the scarce existing data or create new design data. Performing standard tests like tensile and fatigue tests at cryogenic temperatures, or measuring thermal or electrical conductivity necessitates specialized tools, specimens, sensors, and testing methods.

Introduction

- The historical drivers for development and characterization of cryogenic materials are superconducting magnets (including RMIs) and gas liquefaction, but currently aerospace, energy and transportation, due to the potential of hydrogen as an energy vector, are pushing for the development of cryogenic materials.
- Since cryogenic applications are a very small share of the market, the main users of these materials typically produce their properties at in – house laboratories or use commercially available minimum guaranteed properties.
- The golden rule: selection of a material for cryogenic application is not a mere specification of composition or alloy designation, often strict specifications (including manufacturing controls) and QC tests are required that will have an impact on the price.
- This class (partially) covers the selection of materials for cryogenic temperatures.

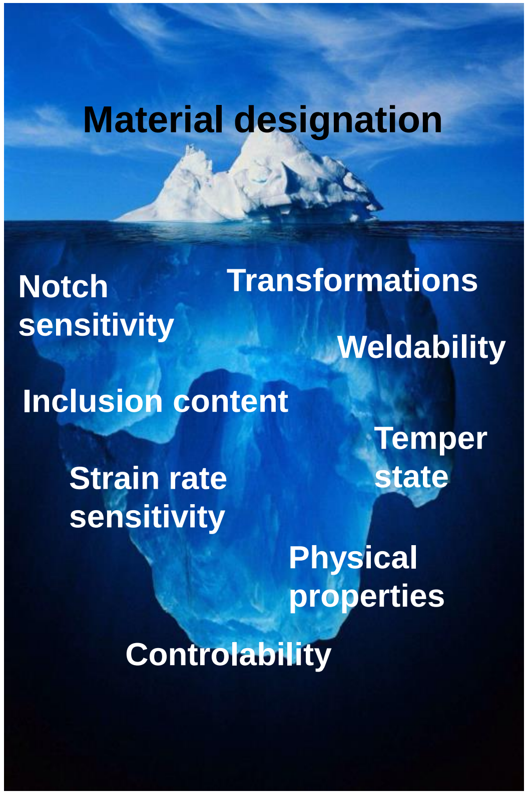
Some of the Important Materials Commonly Used for Cryogenic Applications

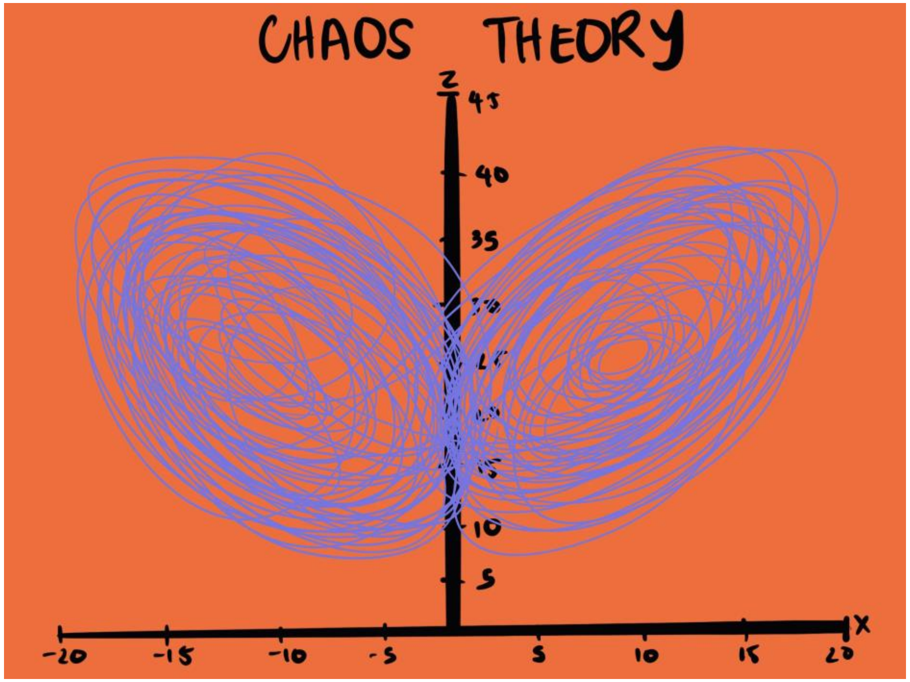
Class	Material	Information
Metals	Aluminum	1100, 2219, 2024, 6061, 7075
	Copper	OFHC CU, C104, 110, CuBe 172
	Titanium	Grade 5 Ti, 6Al-4V-Ti, 5Al-2.5Sn-Ti
	Austenitic Steels	304, 316, 316L, 316LN, JK2LB, Nitronic 40, Nitronic 50 (XM-19)
	Nickel Alloys	Inconel 718, Hastelloy C276, Haynes 242
	SuperAlloys	Maraging, MP35N (Magnetic)
Fiber-reinforced Polymer Composites	High Pressure Laminates	G-10, G11,FR4, Micarta, Bakelite, Cotton/Phenolic, Paper/Phenolic- woven fiber cloth is laminated/impregnated with thermoset epoxy/pressure molded into; Plates, Rods or Tubes
	High Performance Filament Wound Tubes, Struts, Plates	FRP's with fibers that are filament wound or layed up and impregnated with thermoset epoxy matrix - fibers such as E-glass, S2-glass, Carbon, Kevlar, Xylon are used
Polymers	PTFE	Synthetic flouropolymer, low coefficient of friction, good low temp dimensional stability
	Nylon	Polyamide synthetic polymer with high stiffness and low friction coefficient
	Delrin	Acetal resin with high stiffness, low friction coefficient, good dimensional stability
	Torlon	Polyamide-imide, bearing grades are glass or carbon filled, high stifness and strength,
	PEEK	Polyaryletherketone thermoplastic
	Epoxies for Impregnation	2-part thermoset epoxies used for coil impregnation usually requiring low viscosity, long pot life
	Adhesive Epoxies	2-part thermoset epoxies for bond strength, Stycast, M-Bond, EPO-TEK, Cryobond
Superconductors	LTS	Nb3Sn and NbTi - Technical superconductors used for the majority of Superconducting Magnet Applications
	HTS	REBCO,YBCO,BiSSCO - Next generation Superconducting Magnets and Current Leads

Courtesy: Bob Walsh

General rules for materials' selection for cryogenic application

- The golden rule (from S. Sgobba):
  - "A material for critical applications (cryogenic) is not a mere chemical composition or designation"
  - Safety and reliability at very low temperature require special care.



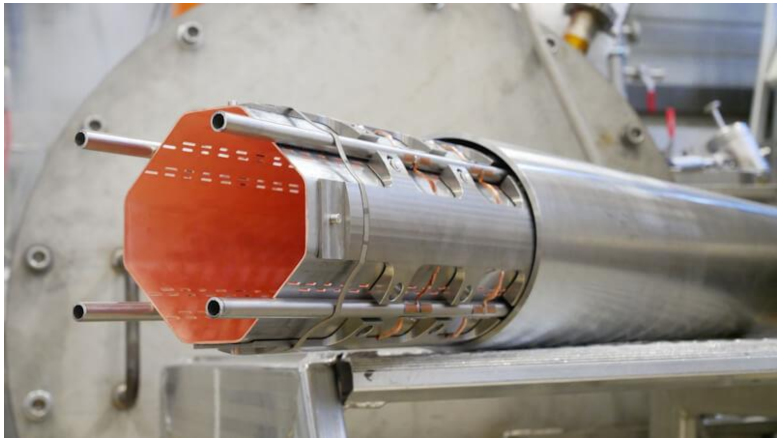


Stainless steel

Stainless steel for cryogenic application

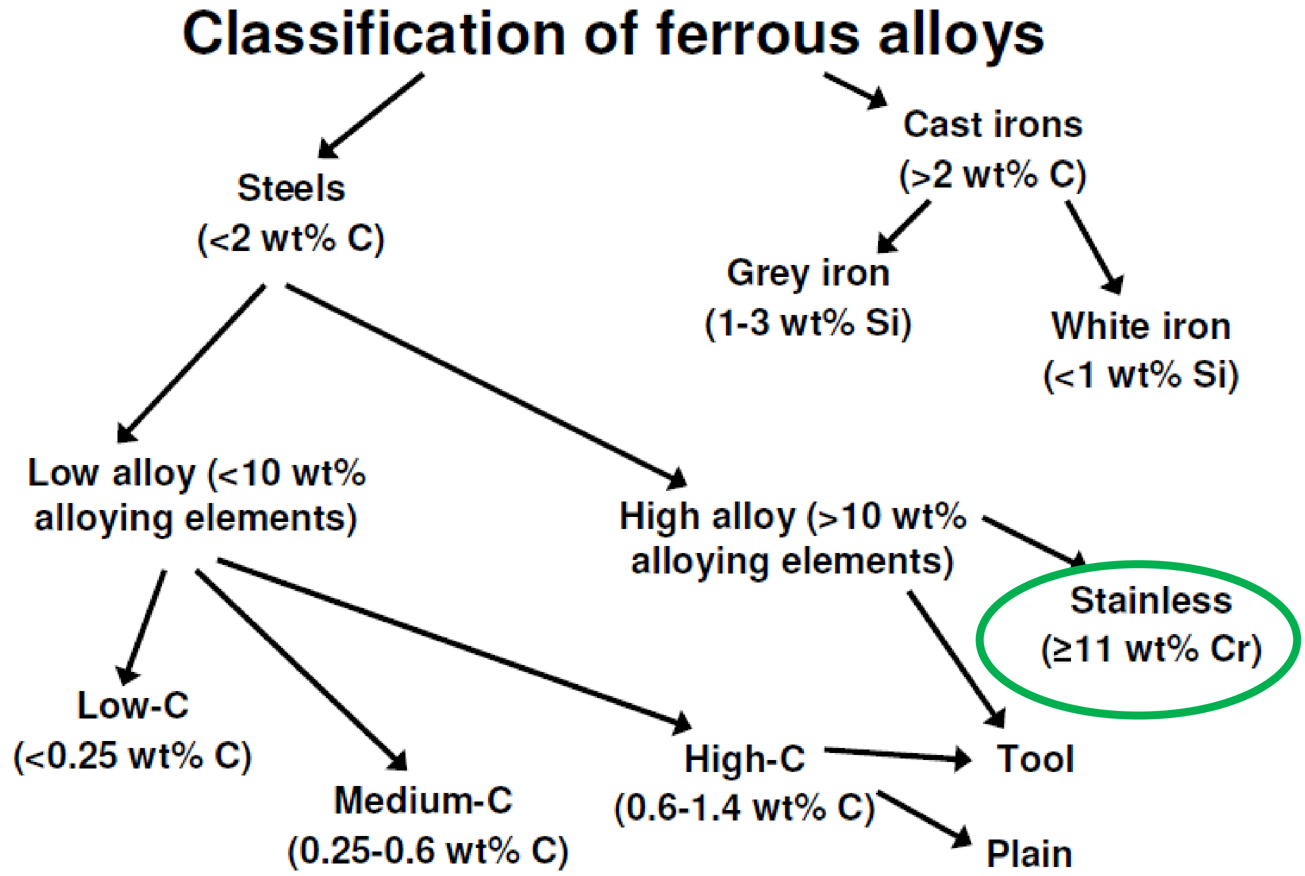
- Ductile and tough at any temperature (no DTBT).
- High strength at cryogenic temperature
- Available, known, weldable, workable…

15 m long ITER central solenoid tie plate made in **FXM-19**.

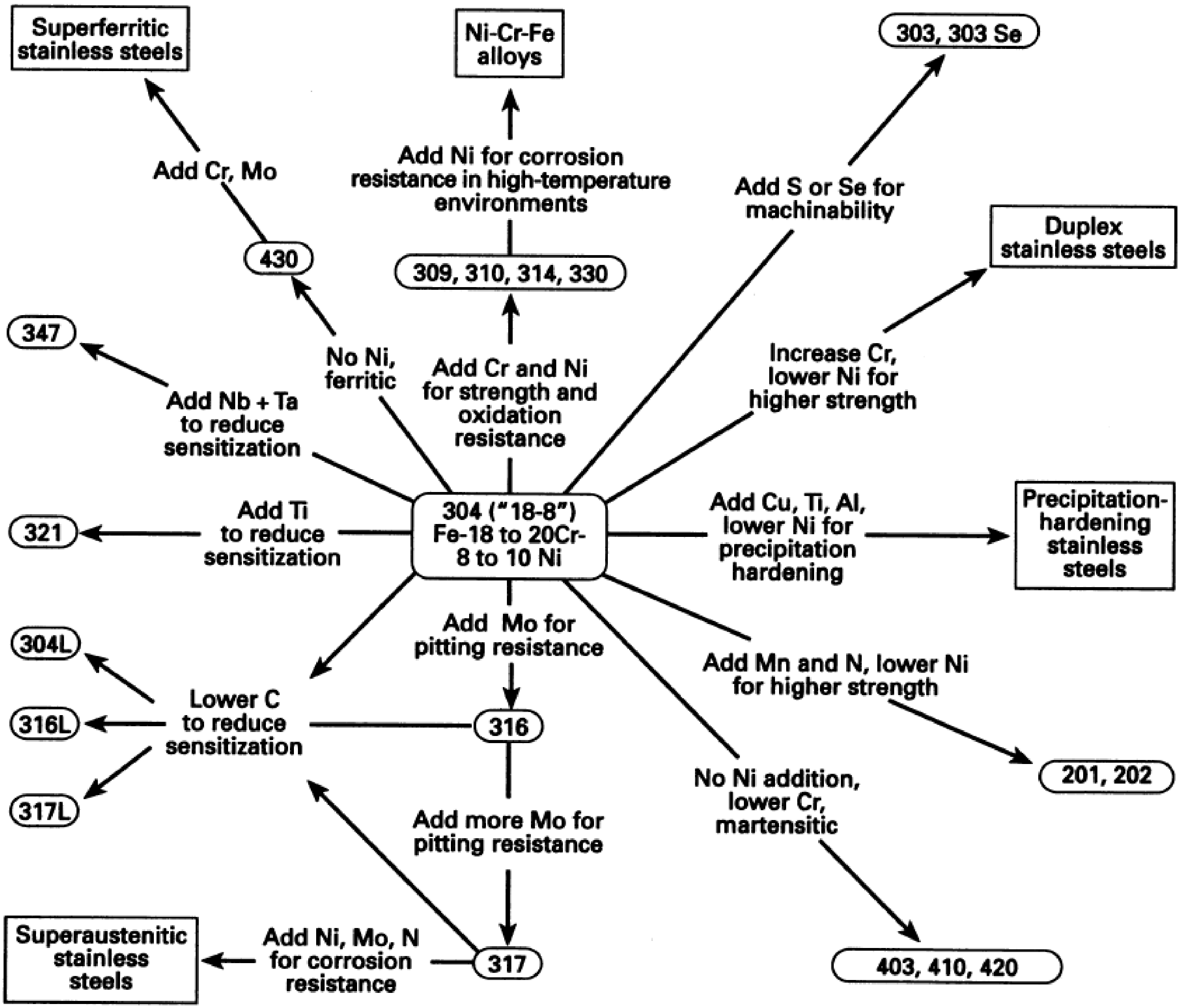


Beam screens for the HL-LHC triplet magnets in a **high Mn – high N grade**, equipped of four seamless cold-drawn cooling tubes in the same grade and inserted into their **316LN** cold bores.

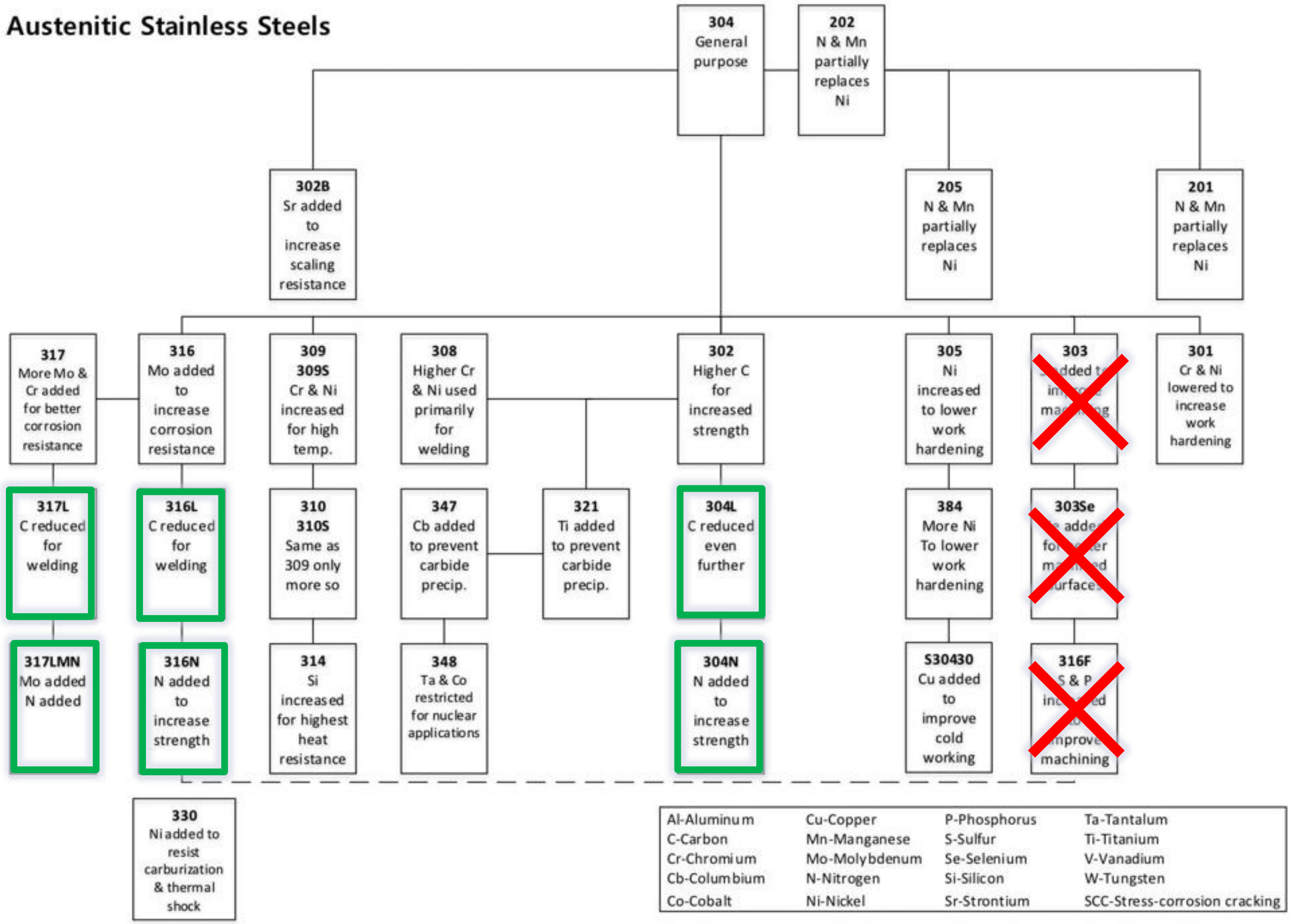
Stainless steel



Stainless steel grades

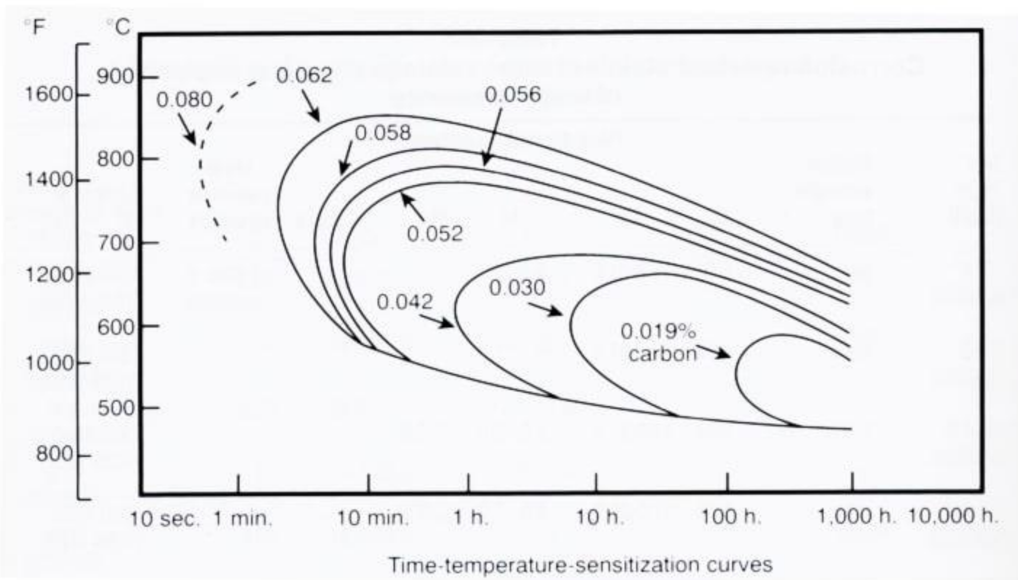


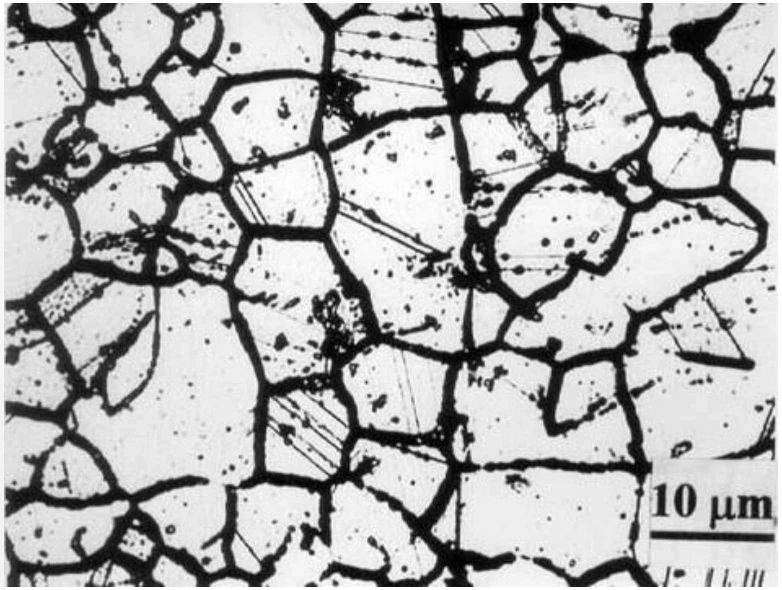
Austenitic stainless steel grades



Austenitic stainless steel grades

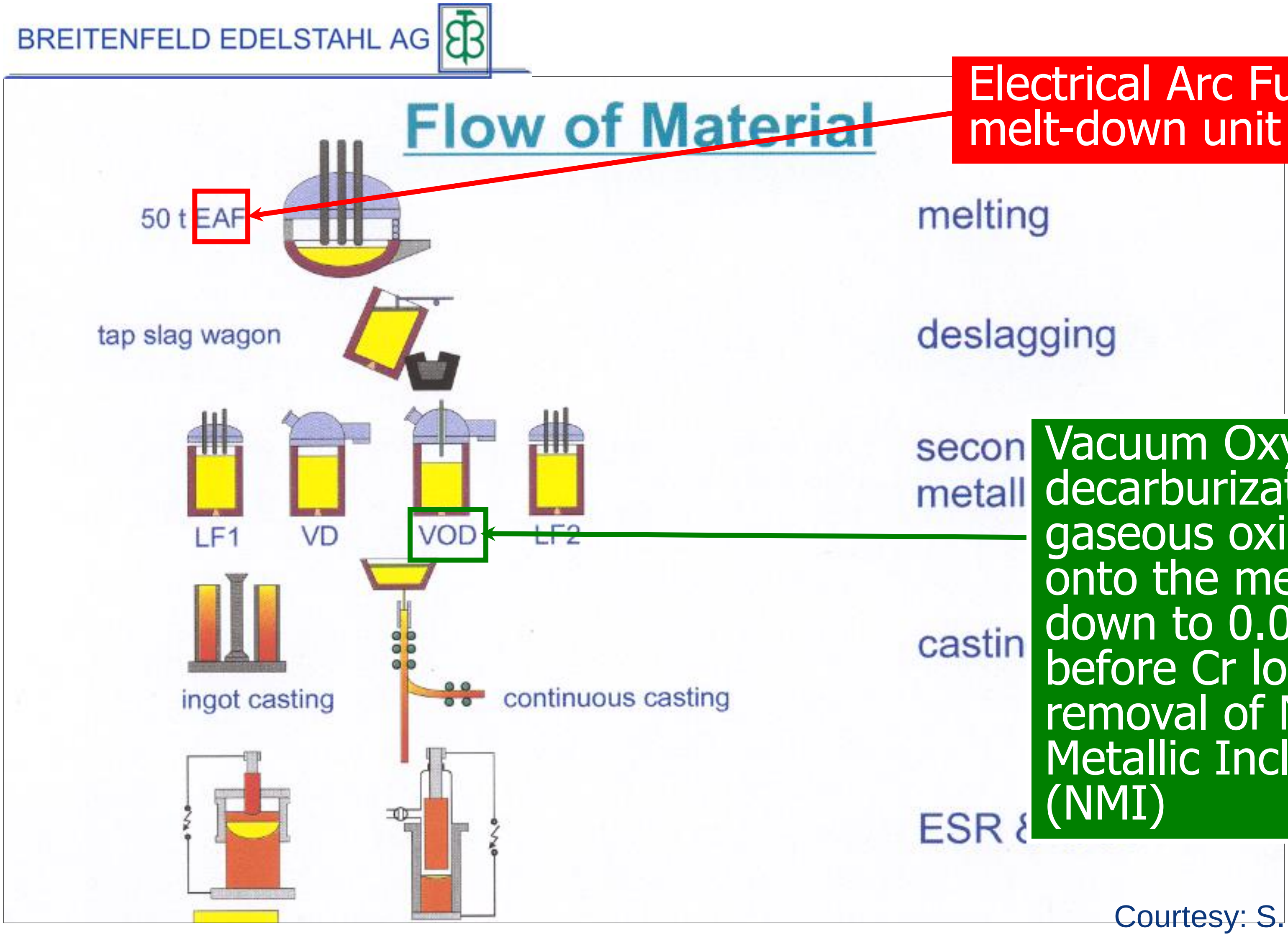
- Why ‘L’ grades?
  - ‘L’ is for low carbon (C ≤ 0.03% in wt%)
  - Reduces the risk of sensitization, specially in welds and HAZs





Sensitized structure of 304 SS

Austenitic stainless steel : steelmaking

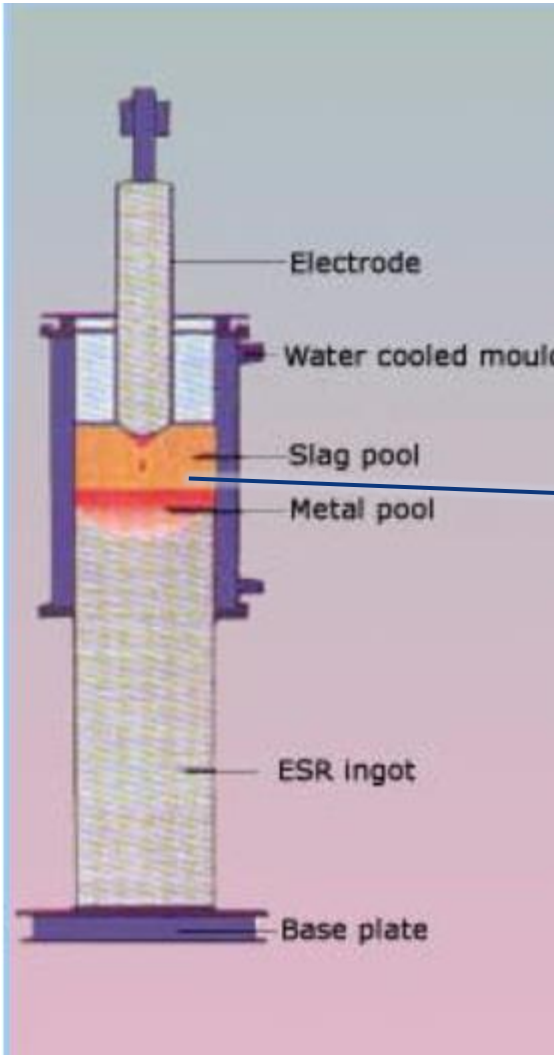


Electrical Arc Furnace: melt-down unit

Vacuum Oxygen decarburization: Pure gaseous oxigen blown onto the metal; C down to 0.015 % before Cr losses begin; removal of Non-Metallic Inclusions (NMI)

Courtesy: S. Sgobba

Austenitic stainless steel : steelmaking



Electroslag remelting (ESR) →process used for remelting and refining of steels and special alloys which are used for critical applications thanks to its capability to control both solidification structure and chemical homogeneity simultaneously.

As the developing droplets pass through the slag, the **metal is cleaned of non-metallic impurities** which are removed by chemical reaction with the slag or by physical flotation to the top of the molten pool. Due to presence of an active slag which is essentially a mixture of CaF_2, CaO and Al_2O_3, **sulphur removal** from the liquid metal takes place rapidly

Austenitic stainless steel : inclusions

Type A: sulfide
Type B: aluminates
Type C: silicates
Type D: globular oxides

Designation: E45 – 13

Standard Test Methods for
Determining the Inclusion Content of Steel[1]

This standard is issued under the fixed designation E45; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

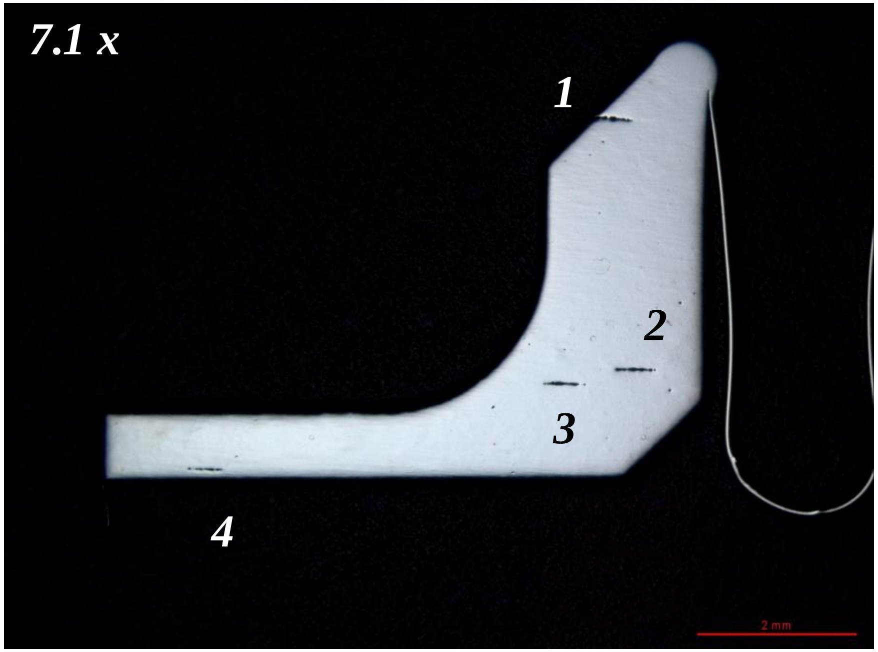
TABLE 1 Minimum Values for Severity Level Numbers (Methods A, D, and E)[A,B]

(mm (in.) at 100×, or count)				
Severity	A	B	C	D[C]
0.5	3.7(0.15)	1.7(0.07)	1.8(0.07)	1
1.0	12.7(0.50)	7.7(0.30)	7.6(0.30)	4
1.5	26.1(1.03)	18.4(0.72)	17.6(0.69)	9
2.0	43.6(1.72)	34.3(1.35)	32.0(1.26)	16
2.5	64.9(2.56)	55.5(2.19)	51.0(2.01)	25
3.0	89.8(3.54)	82.2(3.24)	74.6(2.94)	36
3.5	118.1(4.65)	114.7(4.52)	102.9(4.05)	49
4.0	149.8(5.90)	153.0(6.02)	135.9(5.35)	64
4.5	189.8(7.47)	197.3(7.77)	173.7(6.84)	81
5.0	223.0(8.78)	247.6(9.75)	216.3(8.52)	100

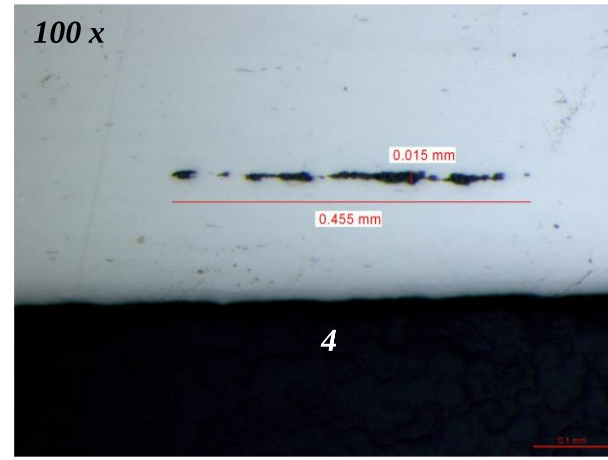
(µm (in.) at 1×, or count)				
Severity	A	B	C	D[C]
0.5	37.0(.002)	17.2(.0007)	17.8(.0007)	1
1.0	127.0(.005)	76.8(.003)	75.6(.003)	4
1.5	261.0(.010)	184.2(.007)	176.0(.007)	9
2.0	436.1(.017)	342.7(.014)	320.5(.013)	16
2.5	649.0(.026)	554.7(.022)	510.3(.020)	25
3.0	898.0(.035)	822.2(.032)	746.1(.029)	36
3.5	1181.0(.047)	1147.0(.045)	1029.0 (.041)	49
4.0	1498.0(.059)	1530.0(.060)	1359.0 (.054)	64
4.5	1898.0(.075)	1973.0(.078)	1737.0 (.068)	81
5.0	2230.0(.088)	2476.0(.098)	2163.0 (.085)	100

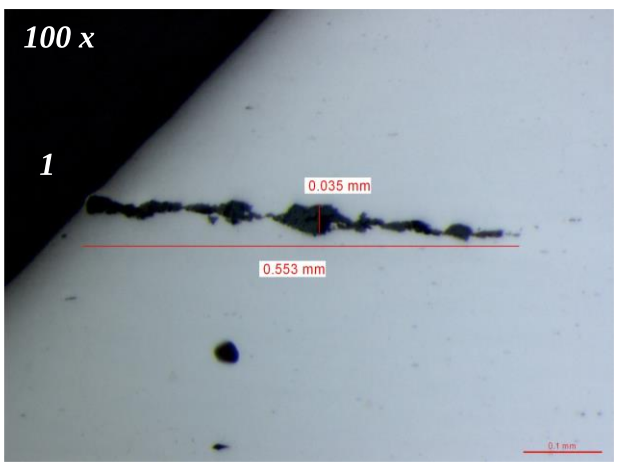
Carefully choose severity depending on the application.

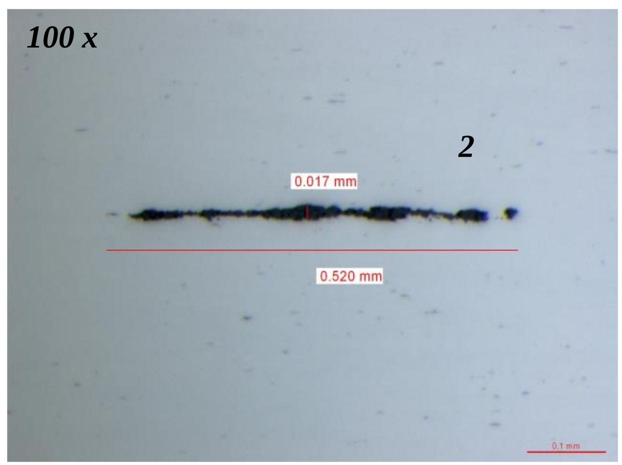
Austenitic stainless steel : inclusions

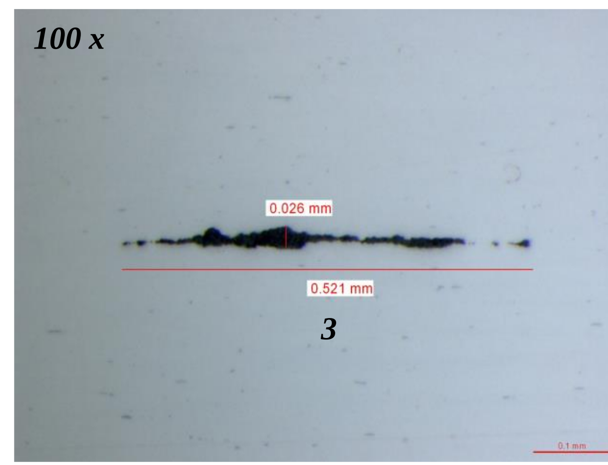


B type inclusions up to class 2





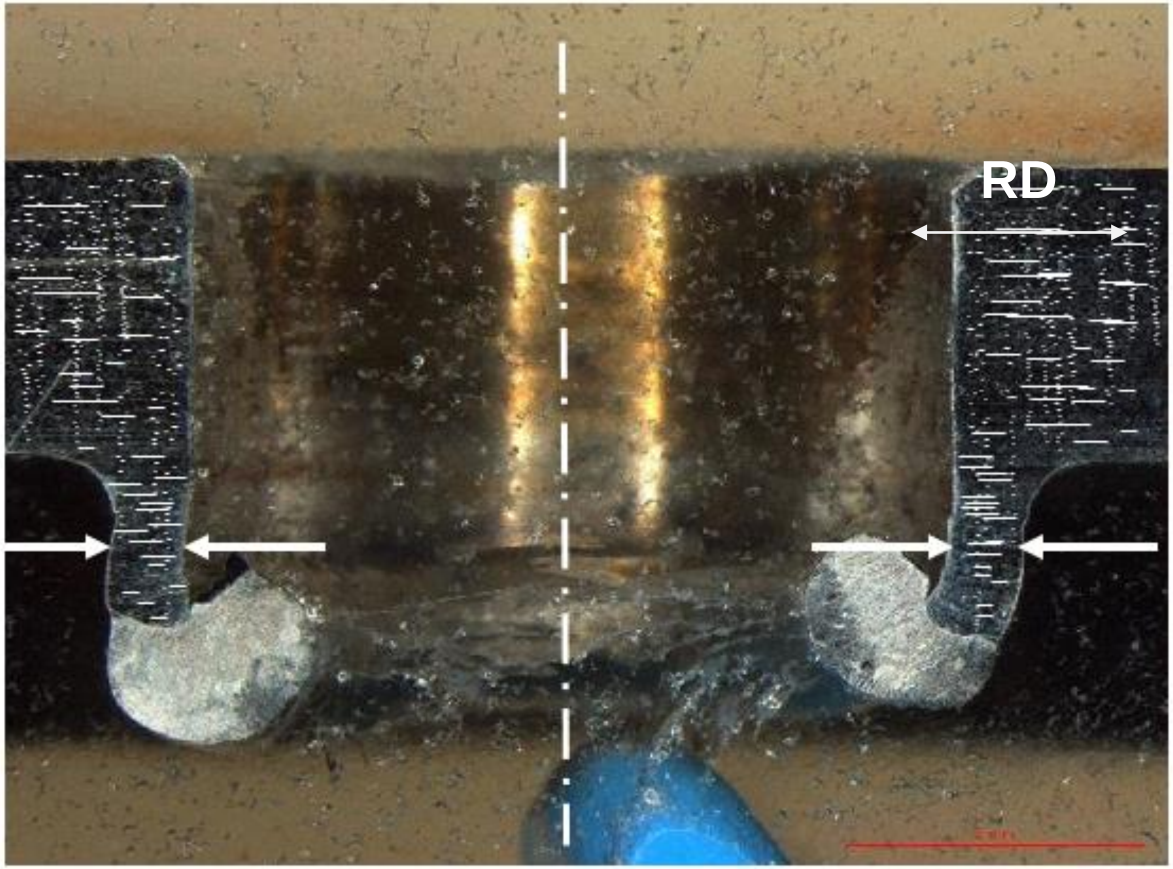




Austenitic stainless steel : inclusions

But not only the severity is important…

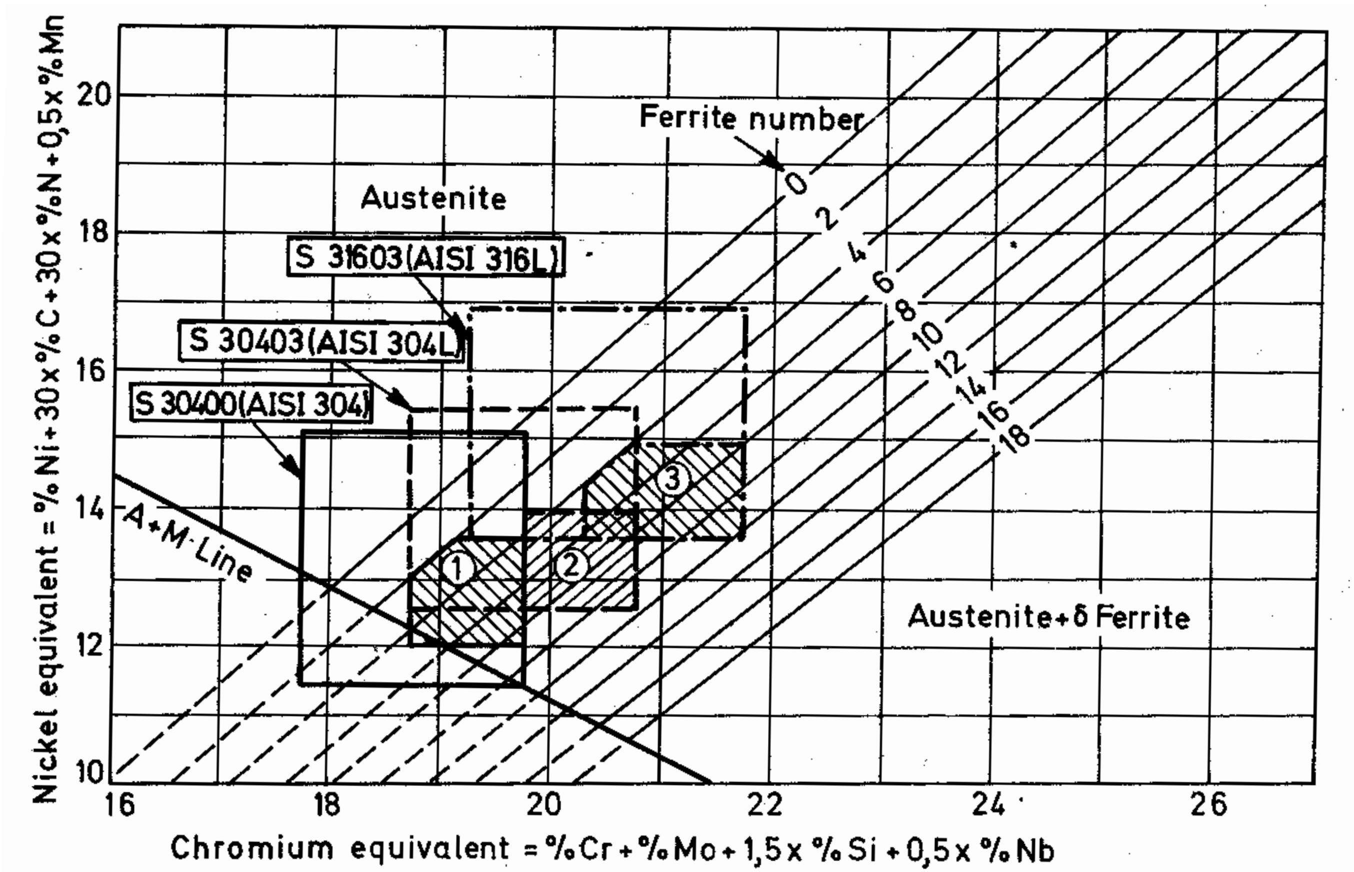
Courtesy: S. Sgobba



For any wrought product (plate, tube, bar), an **unfavourable inclusions alignment** will be anyway present in the rolling or drawing direction → avoid 'cutting the fibre' to minimize the risk of leaks, especially in thin walls.

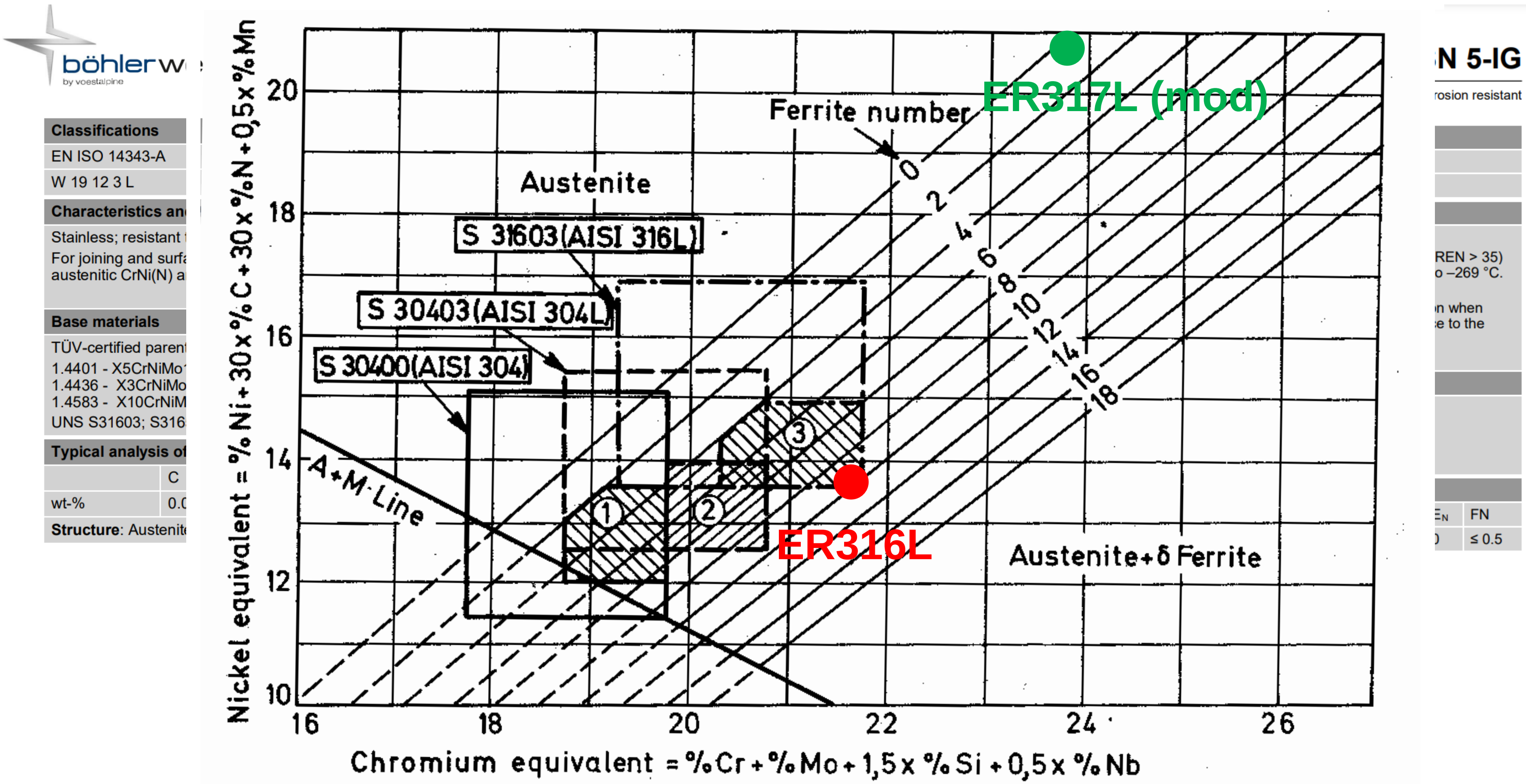
Austenitic stainless steel : welding

- When welding austenitic stainless steel, we aim for a "fully austenitic" weld, for the reasons previously explained:
  - Ferrite and martensite are brittle at cryogenic temperature



Austenitic stainless steel : welding

- When welding austenitic stainless steel, we aim for a "fully austenitic" weld, for the reasons previously explained:
  - Ferrite (and martensite) are brittle at cryogenic temperature



Austenitic stainless steel grades: welding

- When welding austenitic stainless steel, we aim for a "fully austenitic" weld, for the reasons previously explained:
  - Ferrite (and martensite) are brittle at cryogenic temperature

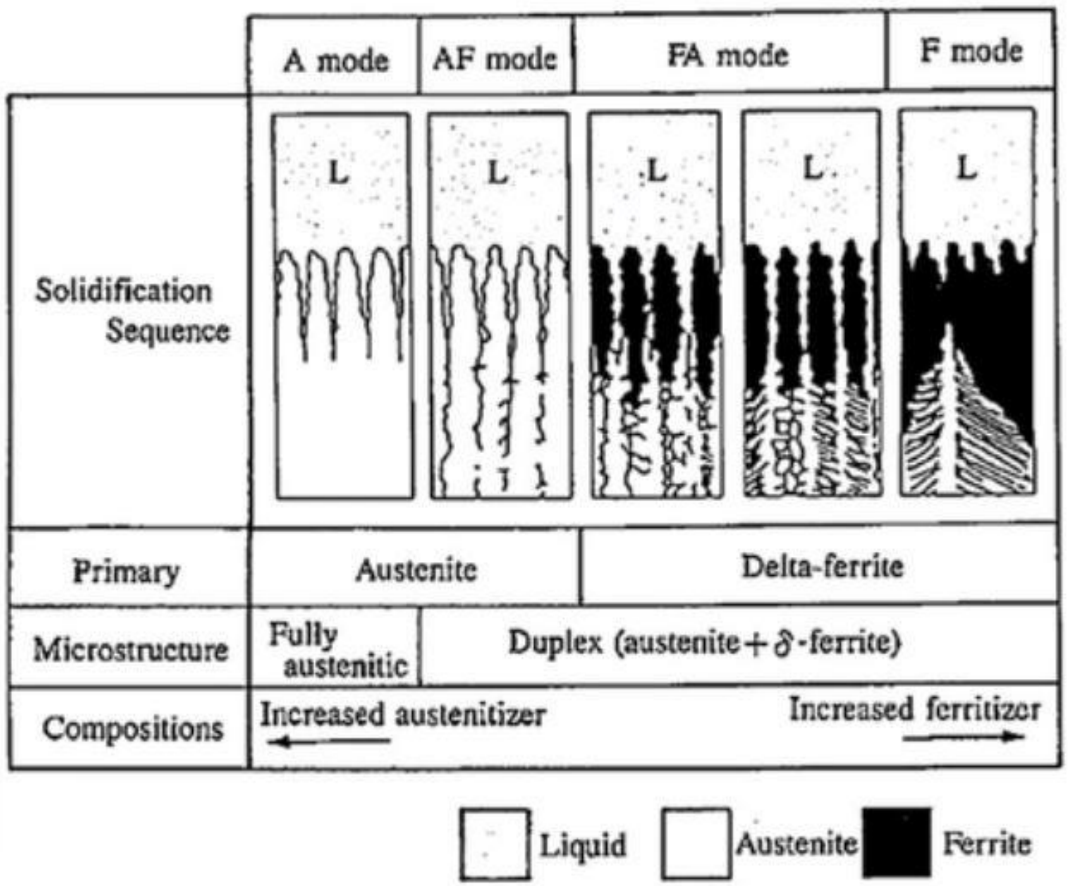
Filler material	K_{JIC} (MPa√m)
ER 316L	105 - 164

Filler material	K_{JIC} (MPa√m)
317L (mod)	226 - 247

For the same base material (316LN), a more ferritic filler material results in a lower fracture toughness of the weld.

Austenitic stainless steel : welding

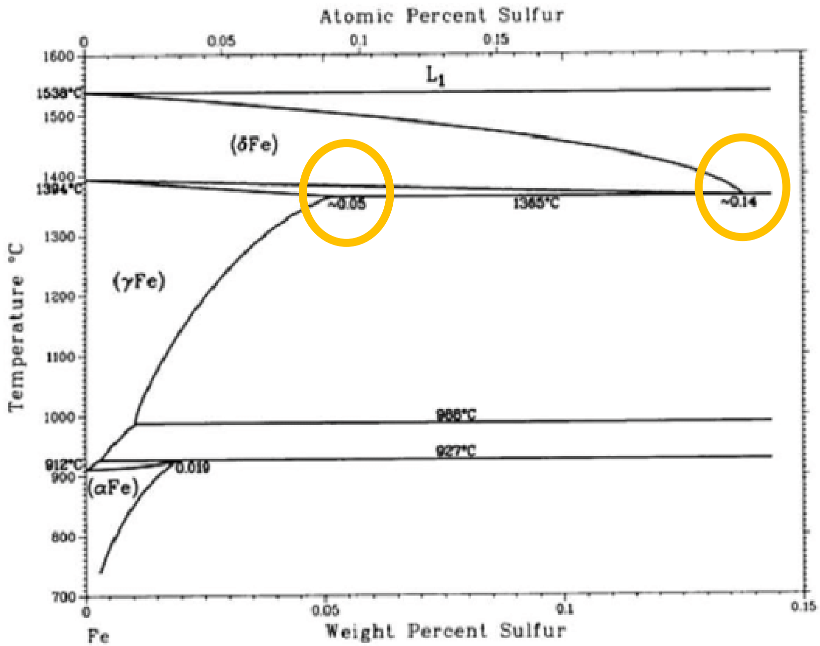
- When welding austenitic stainless steel, we aim for a "fully austenitic" weld, for the reasons previously explained:
  - Ferrite and martensite are brittle at cryogenic temperature

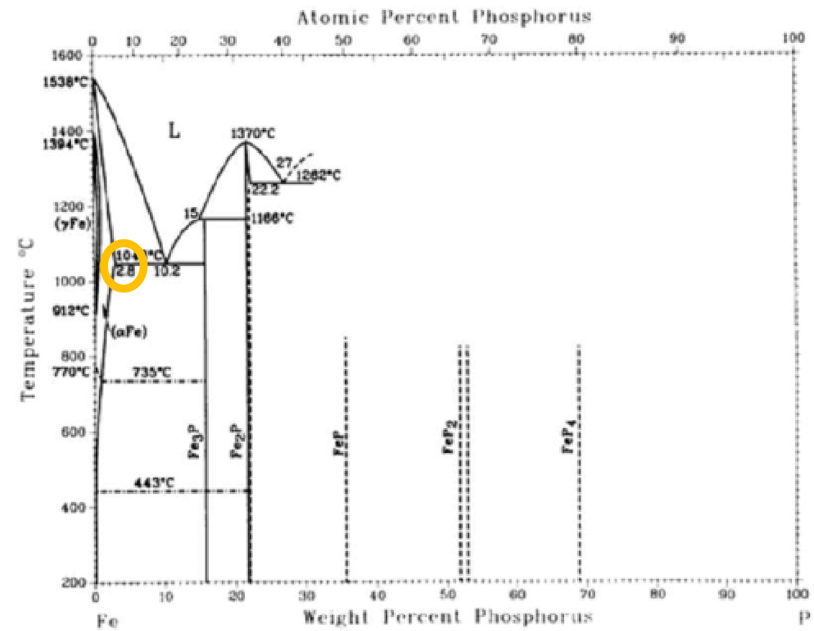


Then why do welders like to have 'a bit' of ferrite in the welds?

Austenitic stainless steel : welding

- The solubility of P & S in ferrite is much higher than in austenite





Solubility of S in δ (ferrite) = 0.14 wt%
Solubility of S in ɣ (austenite) = 0.05 wt%

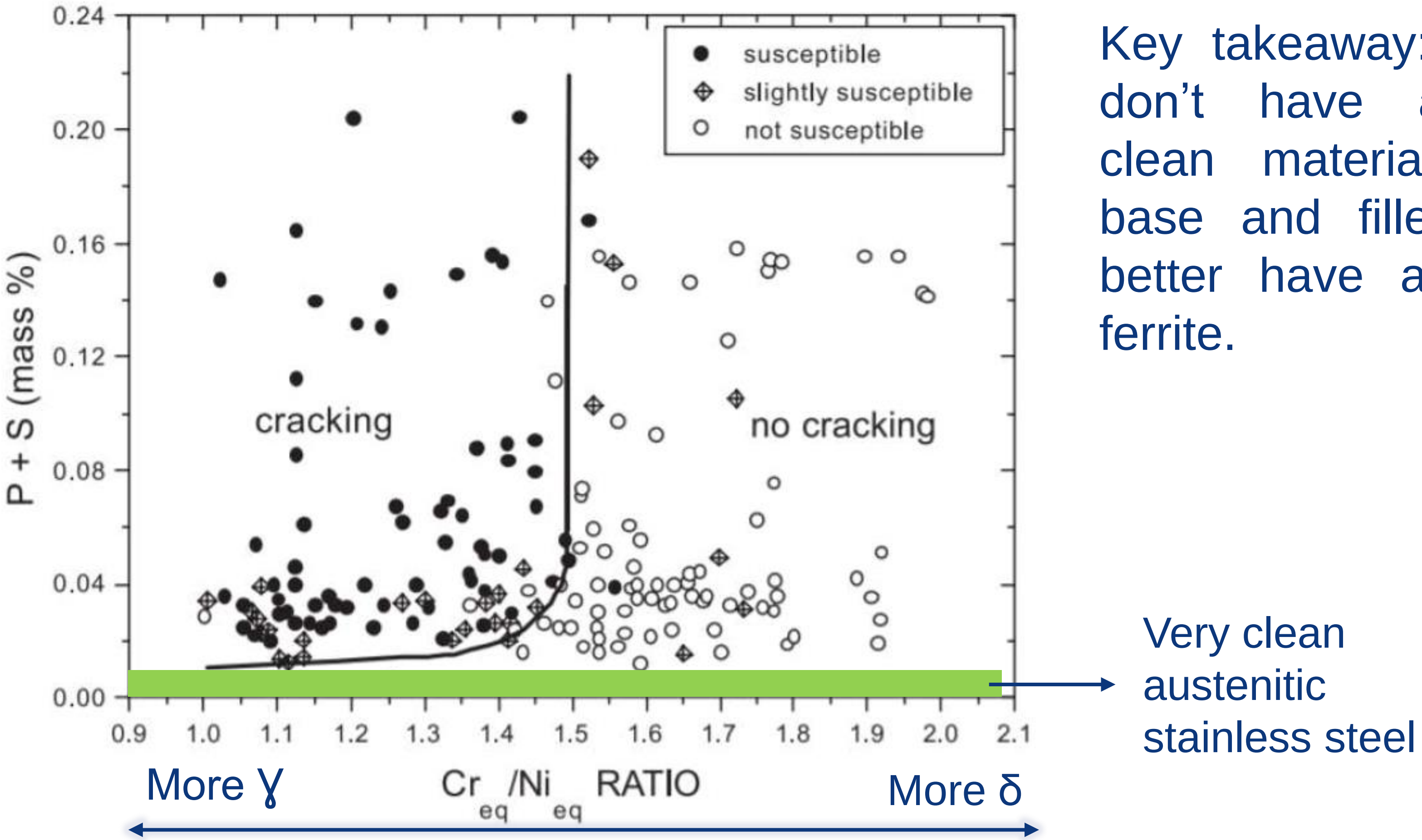
Solubility of P in δ (ferrite) = 2.8 wt%
Solubility of P in ɣ (austenite) = 0.31 wt%

Low-melting phases	
Structure	**Melting point (K)**
Eutectic Fe–FeS	1261
Eutectic Ni–NiS	903
Eutectic Fe–Fe_3P	1321
Eutectic Ni–Ni_3P	1148

P & S are ‘kidnapped’ by the ferrite, and thus they are **not available to form the eutectic phases** which solidify later than the rest of the weld seam, causing **solidification cracking**.

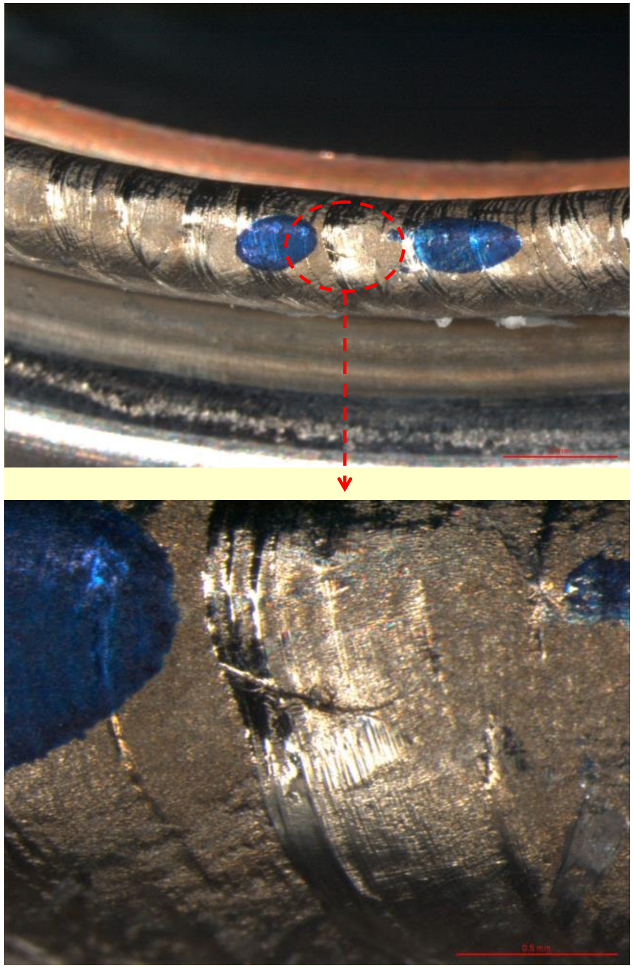
Austenitic stainless steel : welding

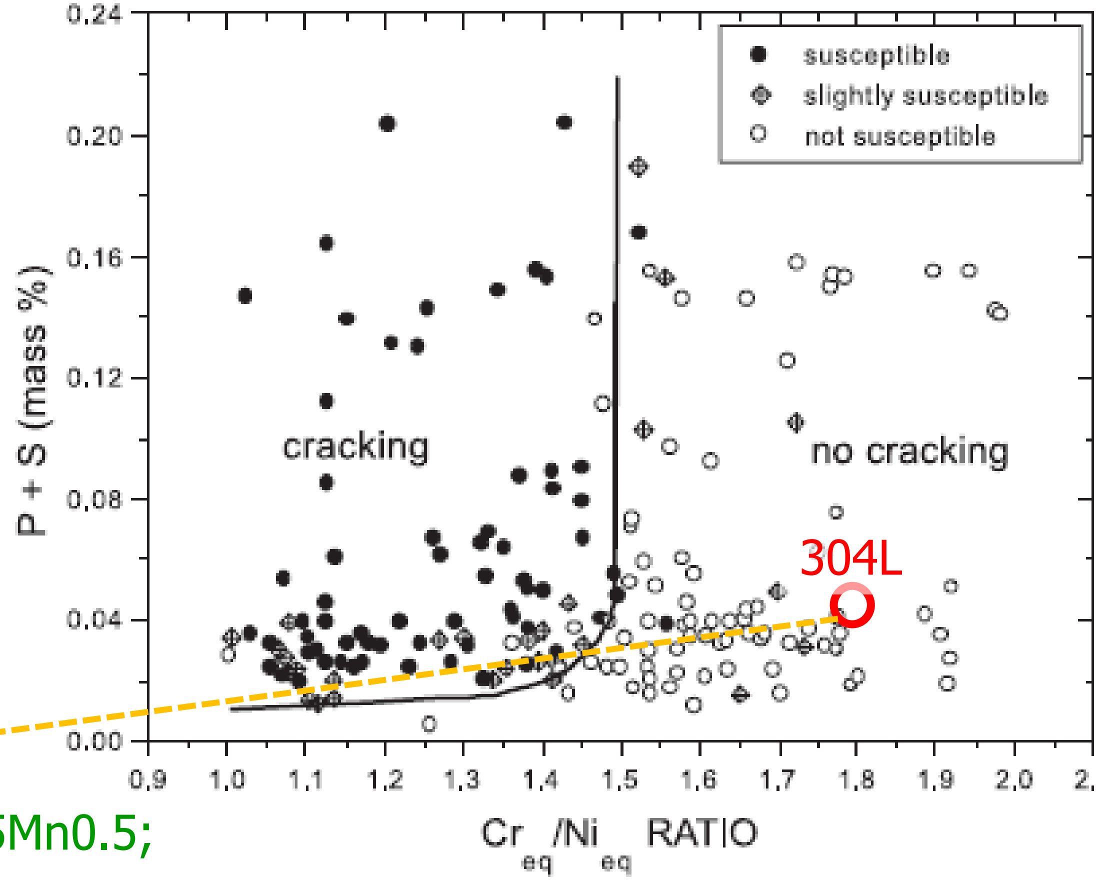
- The solubility of P & S in ferrite is much higher than in austenite



Key takeaway: if you don't have a very clean material (both base and filler), you better have a bit of ferrite.

Austenitic stainless steel : welding





"mumetal", Ni80Mo0.5Mn0.5;
S<0.0005; P=0.003

Courtesy: S. Sgobba

Austenitic stainless steel : martensitic transformations

Table 9.2 Temperature equivalents for calculation of stability parameters of austenitic steels.

Investigator (Year)	Base	Cr	Ni	Mn	Si	C	N	Mo	Other	Comments, Composition Range (wt.%)
f.c.c.→b.c.c. (T_{ms}) cooling [illegible] Eichelman, Hull (1953)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	21 alloys: 10–18Cr, 6–12 Ni, 0.6–5Mn, 0.3–26Si, 0.004–0.12C, 0.01–0.06N
Monkman, Cuff, Grant (1957)	1455	−36.7	−36.7			[illegible]	[illegible]			49 alloys: 11–19Cr, 5–13Ni, 0.035–0.0176(C+N)
Hammond (1963)	1105	−29	−39					−36		16 alloys: 0–12Cr, 4–8Ni, 0.03C, 2–6Mo, 0–15Co, 1–2Ti
Andrews (1965)	273	−12.1	−17.7	−30.4		−423		−7.5		184 alloys from previous studies not in this table. Notice different composition ranges. 0–4.6Cr, 0–5.0Ni, 0.04–4.9Mn, 0.1–1.9Si, 0.11–0.6C, 0–5.4Mo
Hull (1973)	1755	−47	−59	−54	−37	−2390	−3720	−56	−180 (Ti), −14 (Co)	59Ni = average of Eichelmann, Hull (1953) and Monkman et al. (1957), 29 alloys: 12–24Cr, 0–22Ni, 0–20Mn, 0–4Si, 0–0.1C, 0–0.15N, 0–6Mo, Co, 0–2Ti
f.c.c.→b.c.c. (T_{md}), deformation										
Angel (1954)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]0% tension, 50% α′
Hull (1973)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]0% compression, 60 alloys: 12–24Cr, 0–22Ni, 0–20Mn, 0–4Si, 0–0.1C, 0–0.15N, 0–6Mo, Co
Williams, Williams, Capellaro (1976)	686	−6	−25	−16	+21	−222	−222	−11		45% compression, 2.5% α′, 25 alloys: 12–25Cr, 9–20Ni, 1–2Mn, 0.1–0.6Si, 0.04–0.25C, 0.01–0.1N, 0.6–2.8Mo

T_{ms}, temperature of **spontaneous** martensitic transformation

T_{md}, temperature of **strain induced** martensitic transformation

Austenitic stainless steel : martensitic transformations

All coefficients are negative so:

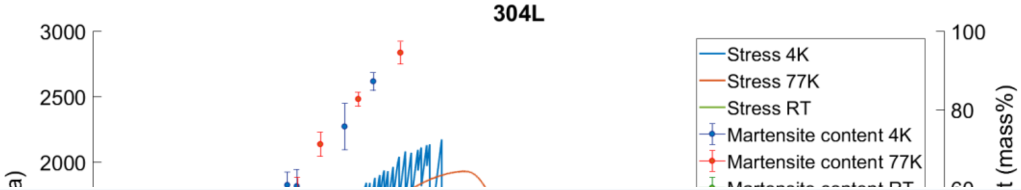
- **Good practice**: "the more alloying elements one uses (and can afford), the more stable the austenite will be"
- Total stability (T_{ms} < 0 K) requires a specific alloy selection or design.

Don't forget that martensite is brittle (specially at cryogenic temperature) and increases magnetic permeability (not good for magnets).

Transformation (**T_{ms}**, **T_{md}**, calculated):

- Basic 304L (1.4307, X2CrNi18-9) ⇒ **T_{ms} = 280 K, T_{md} = 346 K**
- High alloy 304L (1.4306, X2CrNi19-11) ⇒ **T_{ms} = 140 K, T_{md} = 320 K**
- 316LN (1.4429, X2CrNiMoN17-13-3) ⇒ **T_{ms} = n.a, T_{md} = 240 K**
- P506 grade (High Mn, high N) ⇒ **T_{ms} = n.a, T_{md} = 36 K**

Austenitic stainless steel : martensitic transformations



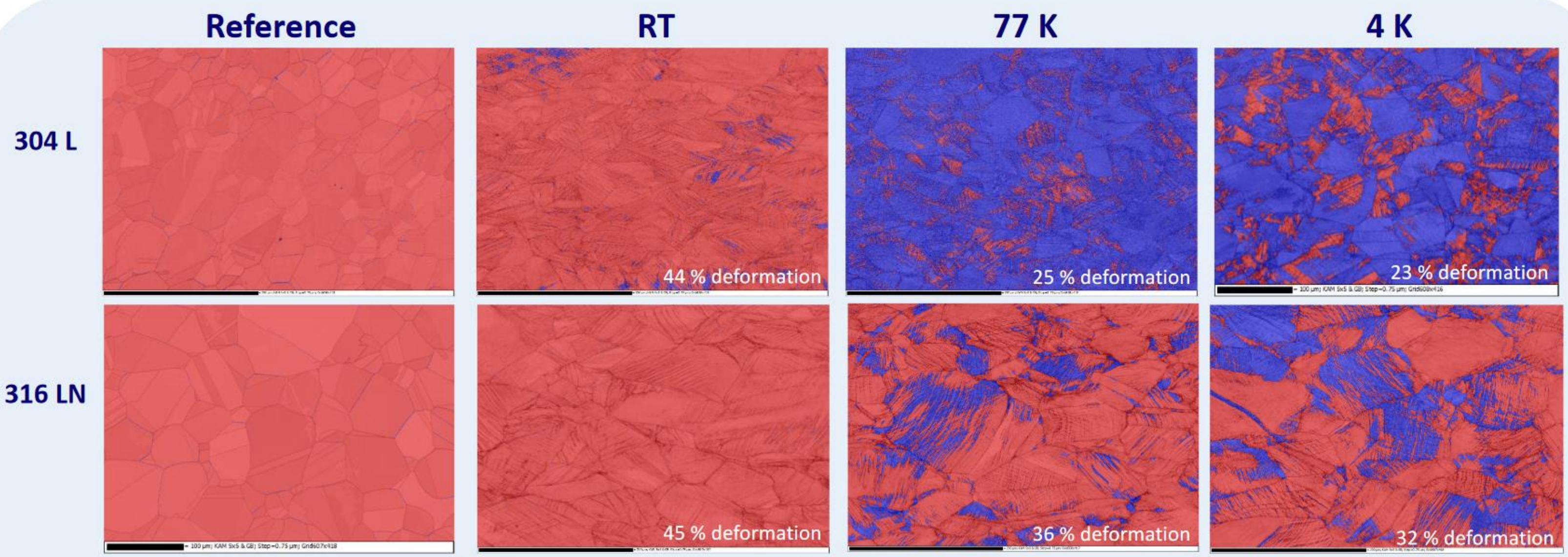
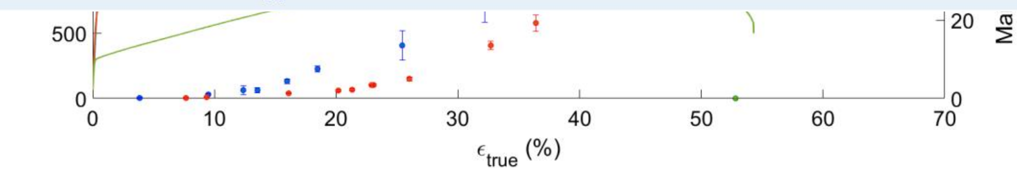


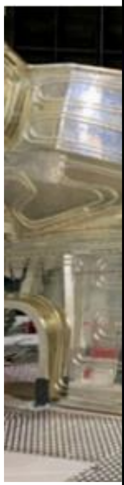
Figure 2 - EBSD phase map and band contrast map on 304 L and 316 LN samples at different temperatures. Colour code: martensite (Fe BCC) appears in blue, while the austenite (Fe FCC) is shown in red



P. Fernández-Pisón, J.a Rodríguez-Martínez, E. García-Tabarés, I. Avilés-Santillana, S. Sgobba, Flow and fracture of austenitic stainless steels at cryogenic temperatures, Eng. Fracture Mechanics, Vol. 258, 2021, 108042, https://doi.org/10.1016/j.engfracmech.2021.108042

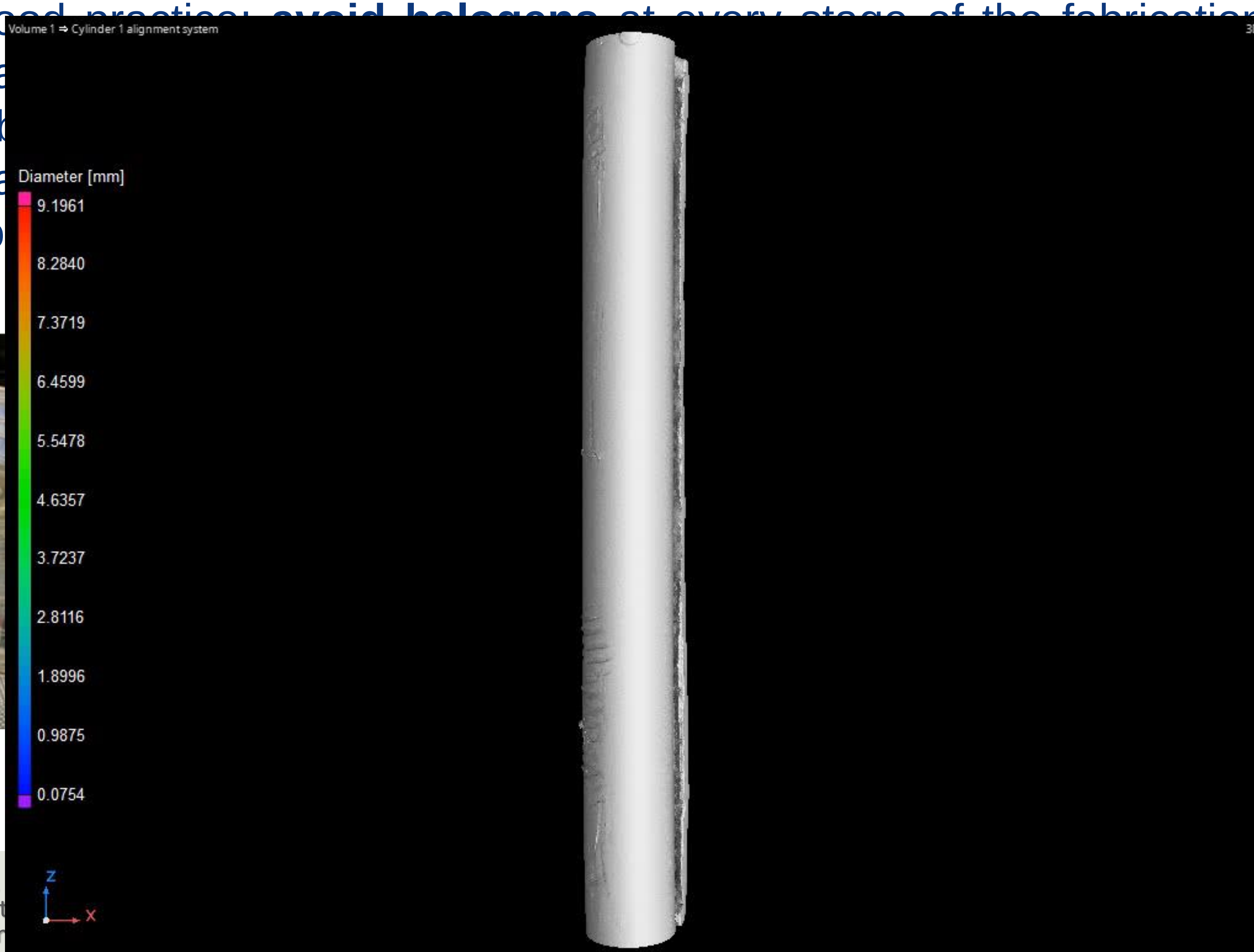
Austenitic stainless steel : corrosion

- Good practice: avoid halogens at every stage of the fabrication of
  sta… all
  fab… ery
  sta… are
  co…



…with Cl if
…ents
…nnealed
…ing

… SCC

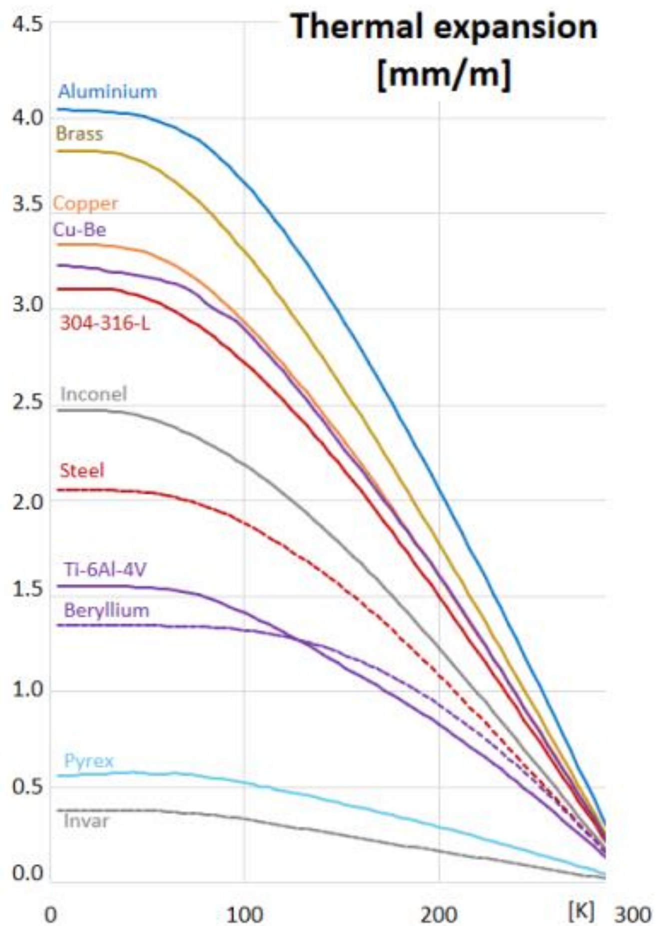


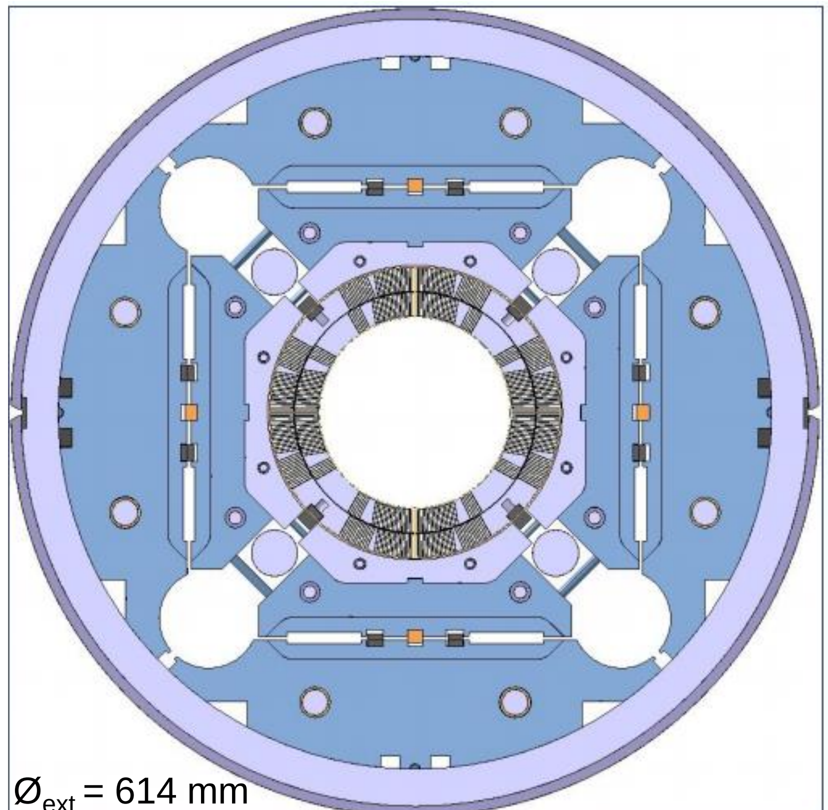
Pearce, … Within t… Cryogen… …s

Aluminium

Aluminium for cryogenic application

- Low modulus of elasticity. Low fracture toughness
- High thermal contraction coefficient





Al shrinking cylinders for MQXF quadrupoles

Source: DUTHIL, Patxi. Material properties at low temperature. arXiv preprint arXiv:1501.07100, 2015.

Aluminium for cryogenic application

- Very low thermal emissivity
- High thermal and electrical conductivity

MLI (Al coated Mylar) HL – LHC's cold box

Al coil for CERN's first particle accelerator: The Synchrocyclotron

Wrought aluminum alloys

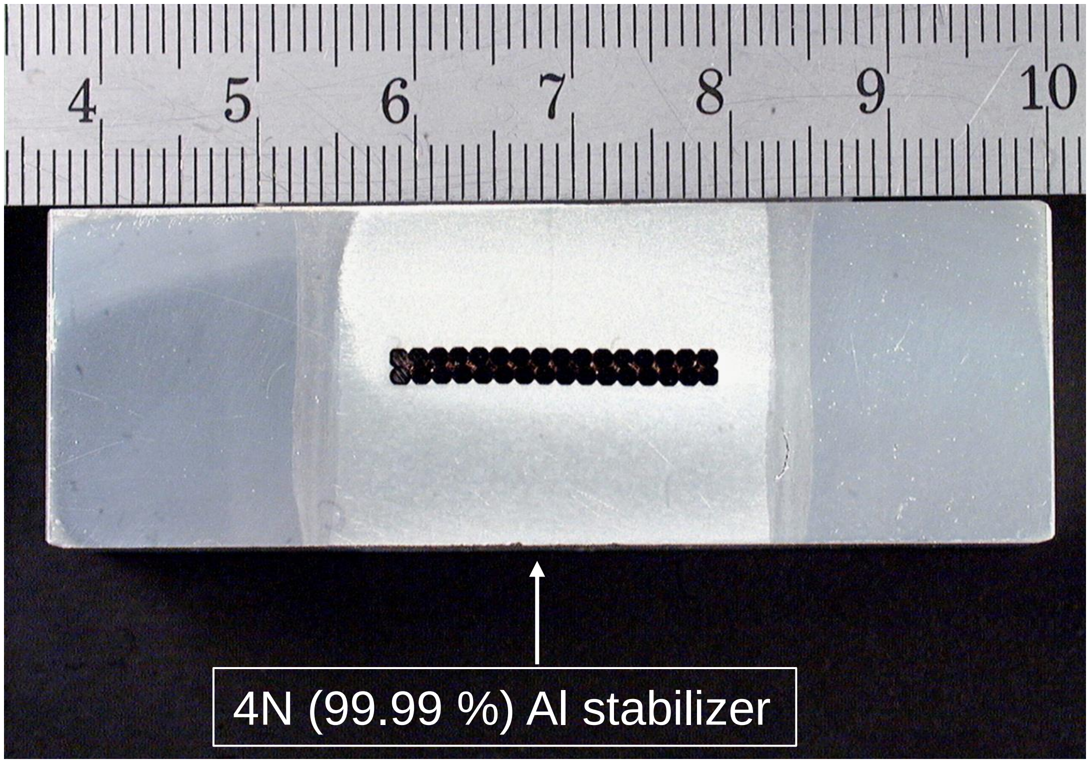
Designation AA	Major alloying elements	Alloy group	Heat treatable ?	Examples
1xxx	**-**	Pure Al	No	
2xxx	**Cu**	Al – Cu	Yes	2219
3xxx	**Mn**	Al – Mn	No	3003
4xxx	**Si**	Al – Si	No	Filler
5xxx	**Mg**	Al – Mg	No	5061
6xxx	**Mg, Si**	Al – Mg - Si	Yes	6082
7xxx	**Zn, Mg**	Al – Zn	Yes	7050
8xxx	**any**		(yes)	8090

(blue) → Can be strengthened by a suitable thermal treatment (heat treatable)

(grey) → Can only be strengthened by hot or cold working (non – heat treatable)

Wrought aluminum alloys designations

- 1xxx series (pure Al) ⟶ Excellent workability & ↑ conductivity

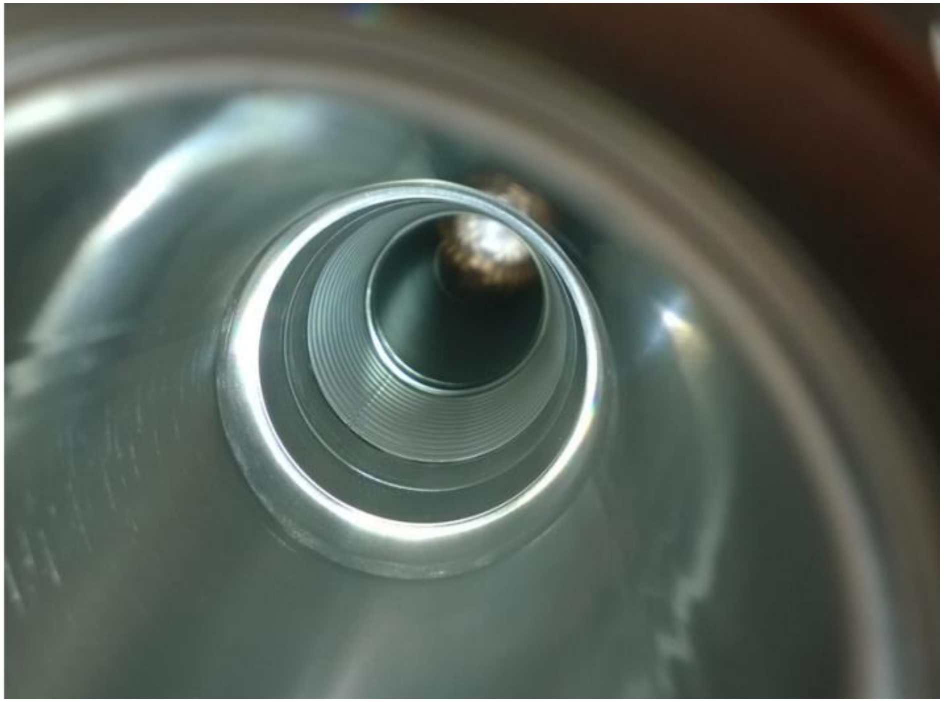


Example: CMS conductor

Wrought aluminum alloys designations

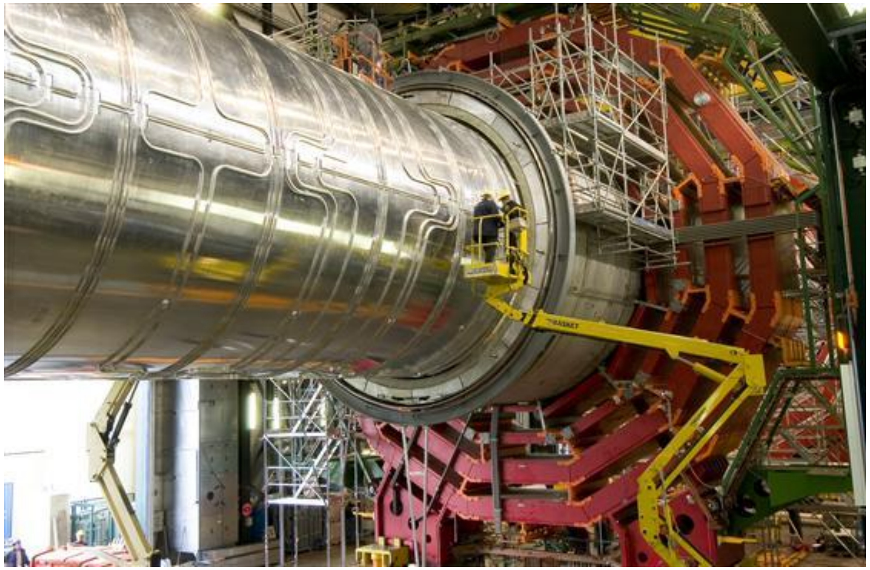
- 2xxx series (Al – Cu) ⟶ ↑ Mechanical properties ↓ corrosion resistance
  - Example: EN AW 2219 T6

Example: vacuum chamber bodies. (NEG coated).



Wrought aluminum alloys designations

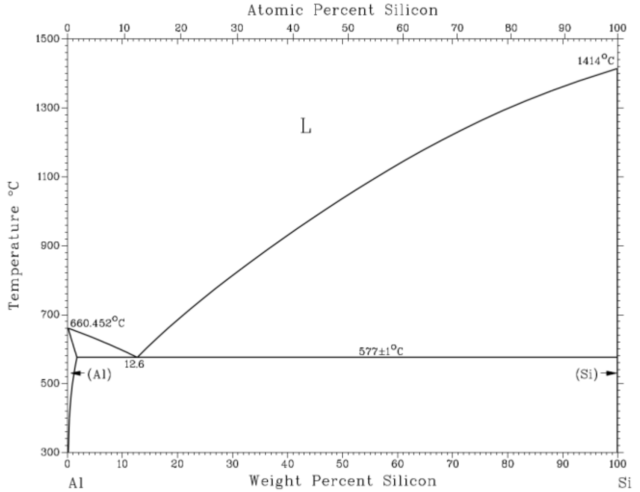
- 3xxx series (Al – Mn) → Moderate mechanical properties, workability, Corrosion resistance and th. conductivity
  - Example: EN AW 3003 H22



Example: CMS Solenoid thermal shield

Wrought aluminum alloys designations

- 4xxx series (Al – Si)
  - Major alloying element of this group is silicon, added in sufficient quantities (around 12%), cause substantial lowering of the melting point without producing brittleness (eutectic point).
  - Al - Si alloys are used in welding wire and as brazing alloys.



Wrought aluminum alloys designations

- 5xxx series (Al – Mg) ⟶ moderately ↑ mechanical properties ↑ ductility, weldability
  - EN AW 5083 H321, H116 and H111

Example: Mandrels for CMS coil

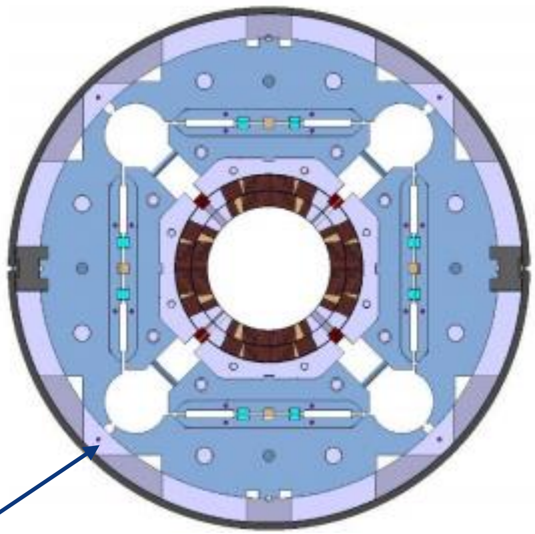
Wrought aluminum alloys designations

- 6xxx series (Al – Mg – Si) ⟶ Combination of formability, mechanical strength, weldability and weight
  - Example: EN AW 6082 T6

Example: ICARUS neutrino detector

Wrought aluminum alloys designations

- 7xxx series (Al – Zn – Mg) ⟶ ↑ Mechanical properties
  - Example: EN AW 7075 T6



Al shell

Example: Al shells MQXFBP1 magnet

Wrought aluminum alloys designations

- 8xxx series
  - Reserved for miscellaneous compositions. Alloying elements include: iron, lithium, copper, zinc, magnesium, silicon, manganese, vanadium, zirconium, titanium, chromium & bismuth.
  - Al – Li alloys, for weight reduction. Al - Li alloys possess increased Modulus of Elasticity, high specific stiffness, increased fatigue strength and cryogenic strength.

Wrought aluminum alloys: temper states

Alphanumeric designations that contain information about the thermomechanical history of the material to achieve the desired properties.

AA 7075	Min.	Max.	Approx
Plates, sheets; Annealed (O); 0.20 <= t <= 0.36 mm; Nominal thickness			
Yield stress, $R_{p0,2}$ (MPa)	-	145	-
Tensile stress, R_m (MPa)	-	276	-
Elongation, A (%)	9	-	-
	L_o = 50.8 mm or 4D		

AA 7075	Min.	Max.	Approx
Plates; Solution heat treated and artificially aged (T651); 0.20 < t <= 0.28 mm; Nominal thickness			
Yield stress, $R_{p0,2}$ (MPa)	434	-	-
Tensile stress, R_m (MPa)	510	-	-
Elongation, A (%)	5	-	-
	L_o = 50.8 mm		

Wrought aluminum alloys: temper states

A capital letter indicating the major class of fabrication treatment(s) used + one (or more) numbers providing more specific information about how the processing was carried out.

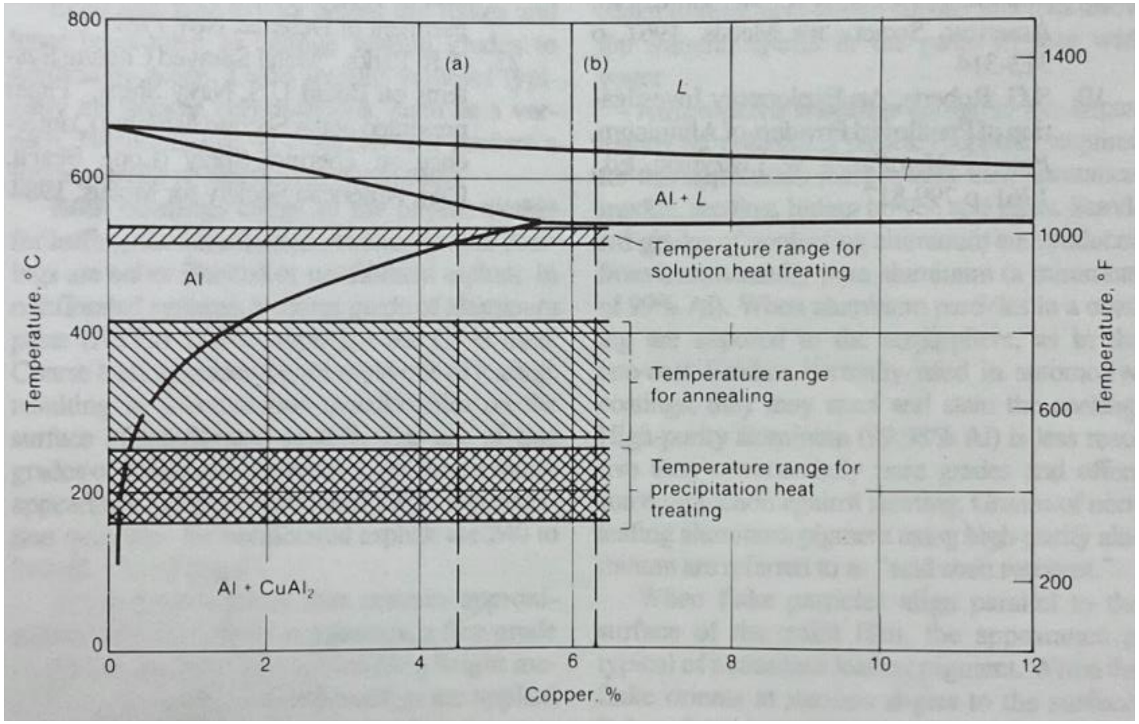
BS EN 515:2017

BSI Standards Publication

Aluminium and aluminium alloys — Wrought products — Temper designations

Wrought aluminum alloys: temper states

- O, annealed: given a high – temperature treatment, sufficient to remove the effects of prior working, usually resulting in complete recrystallization of the material. Lowest strength and maximum ductility and toughness.

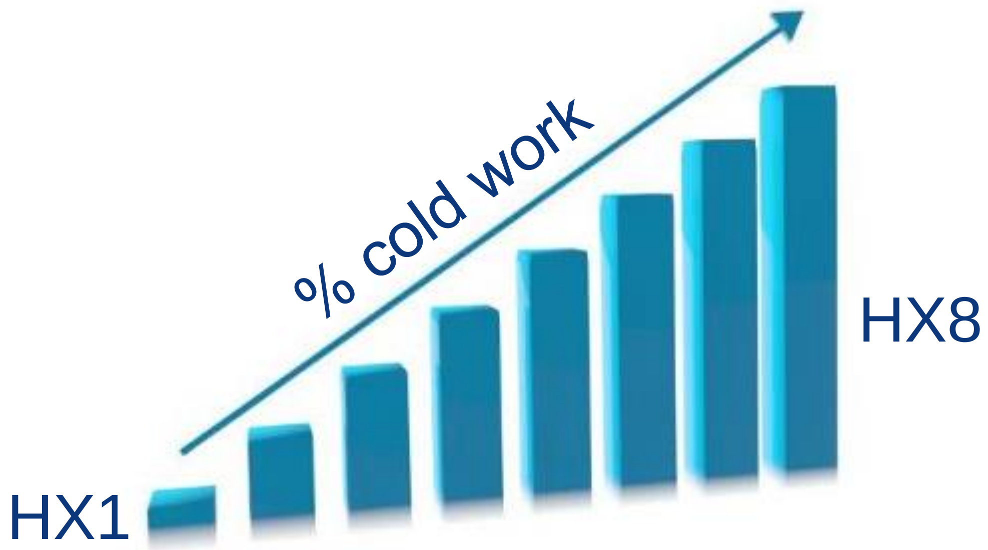


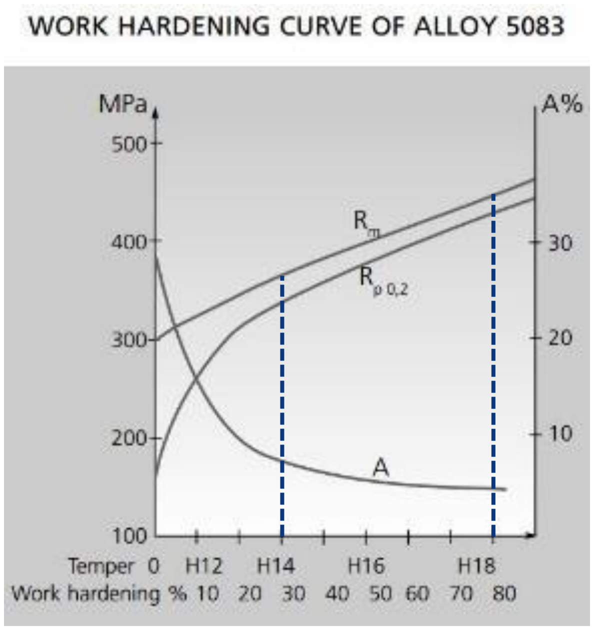
Portion of Al – Cu phase diagram

Source: ASM aluminium and aluminium alloys handbook

Wrought aluminum alloys: temper states

- H, strain hardened: non-heat-treatable wrought alloys that have had their strength increased by strain hardening. H is always followed by two or more digits:
  - The first number after the H tells whether the strain-hardened alloy has been thermally treated
  - The second number indicates the approximate amount of cold work





Source: Aluminium in Commercial Vehicle – European Aluminium Association – 2011

Wrought aluminum alloys: temper states

- H, strain hardened: non-heat-treatable wrought alloys that have had their strength increased by strain hardening. H is always followed by two or more digits:
  - The first number after the H tells whether the strain-hardened alloy has been thermally treated
  - The second number indicates the approximate amount of cold work
  - Any subsequent numbers define special practices, variations of the normal indicated by the first two numbers.

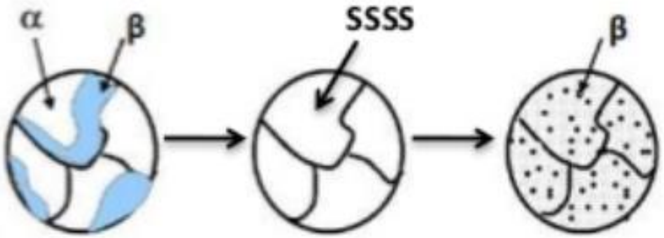
Example:
AA 5083 H116 (marine grade)

Wrought aluminum alloys: temper states

- T, thermally treated to produce stable tempers: heat-treatable wrought alloys that have followed a solution heat treatment followed by a quench and either natural or artificial aging.

Heat treatment to increase the strength of Al alloys is a three step process:

1. Solution heat treatment: dissolution of soluble phases
2. Quenching: development of supersaturation
3. Age hardening: precipitation of finely dispersed precipitates



Wrought aluminum alloys: temper states

- T, thermally treated to produce stable tempers: heat-treatable wrought alloys that have followed a solution heat treatment followed by a quench and either natural or artificial aging. T is always followed by one or more digits:
  - The first digit after the T can be any from 1 to 10. It is a combination of:
    - Cooled from elevated temperature or solution heat treatment
    - Cold worked or not cold worked
    - Naturally aging or artificial ageing

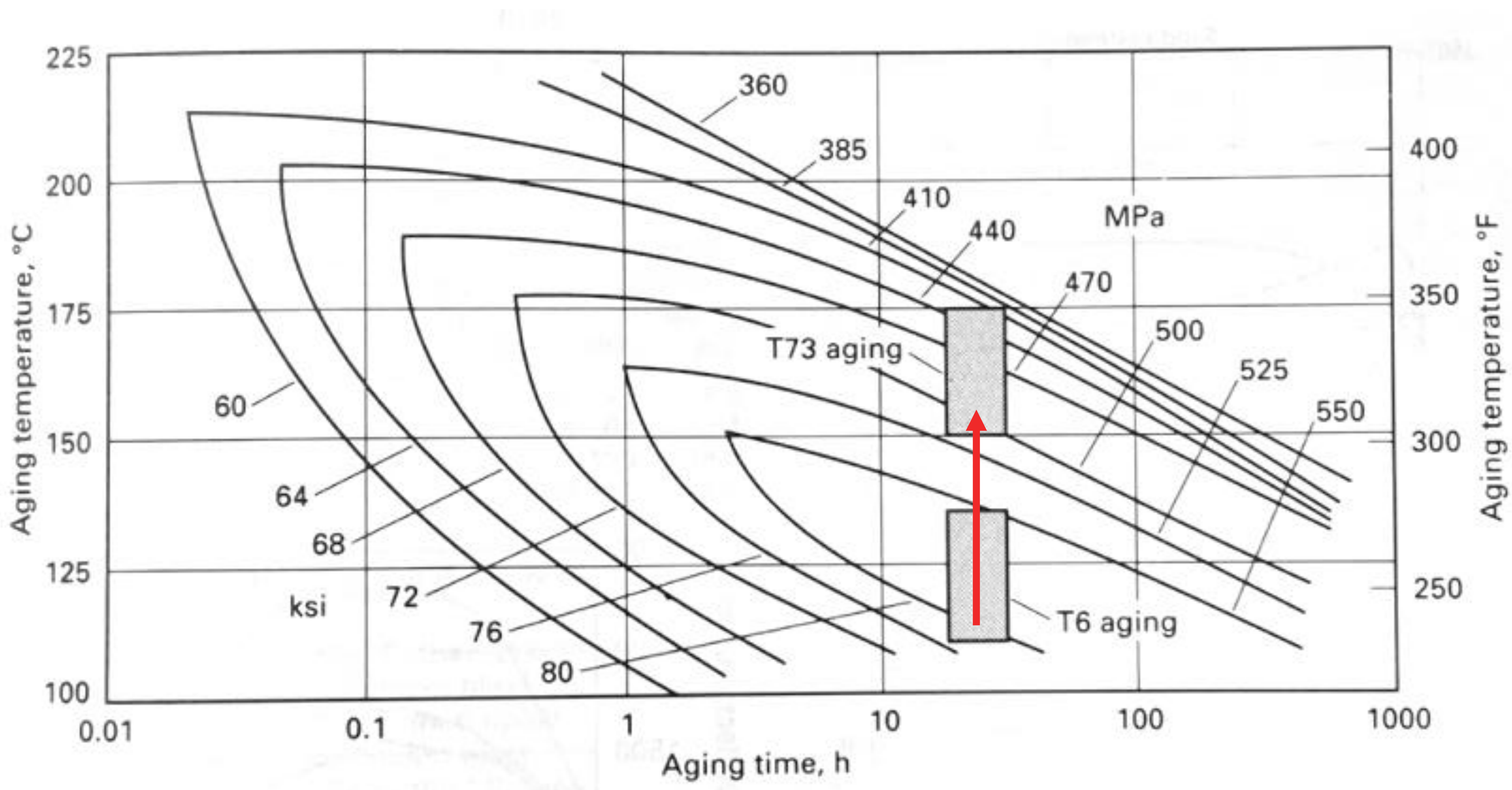
Table 2 — Summary of processing for achieving T tempers

Ageing	Cold worked	Cooled from shaping process	Furnace solution heat-treated[a]
Natural	No	T1	T4
	Yes	T2	T3
artificial	No	T5	T6, T7
	Yes - before ageing	T10	T8
	Yes - after ageing	-	T9

[a] See footnote 4 to text in 8.1

From EN 515:
Al and Al alloys
- Wrought products -
Temper designations

Wrought aluminum alloys: temper states

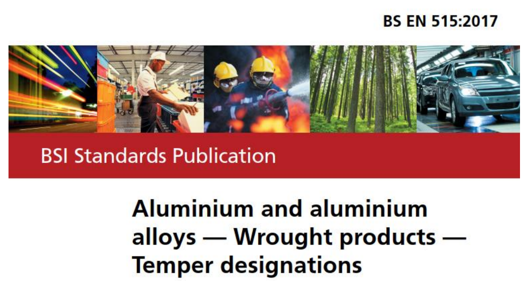


Source: ASM aluminium and aluminium alloys handbook

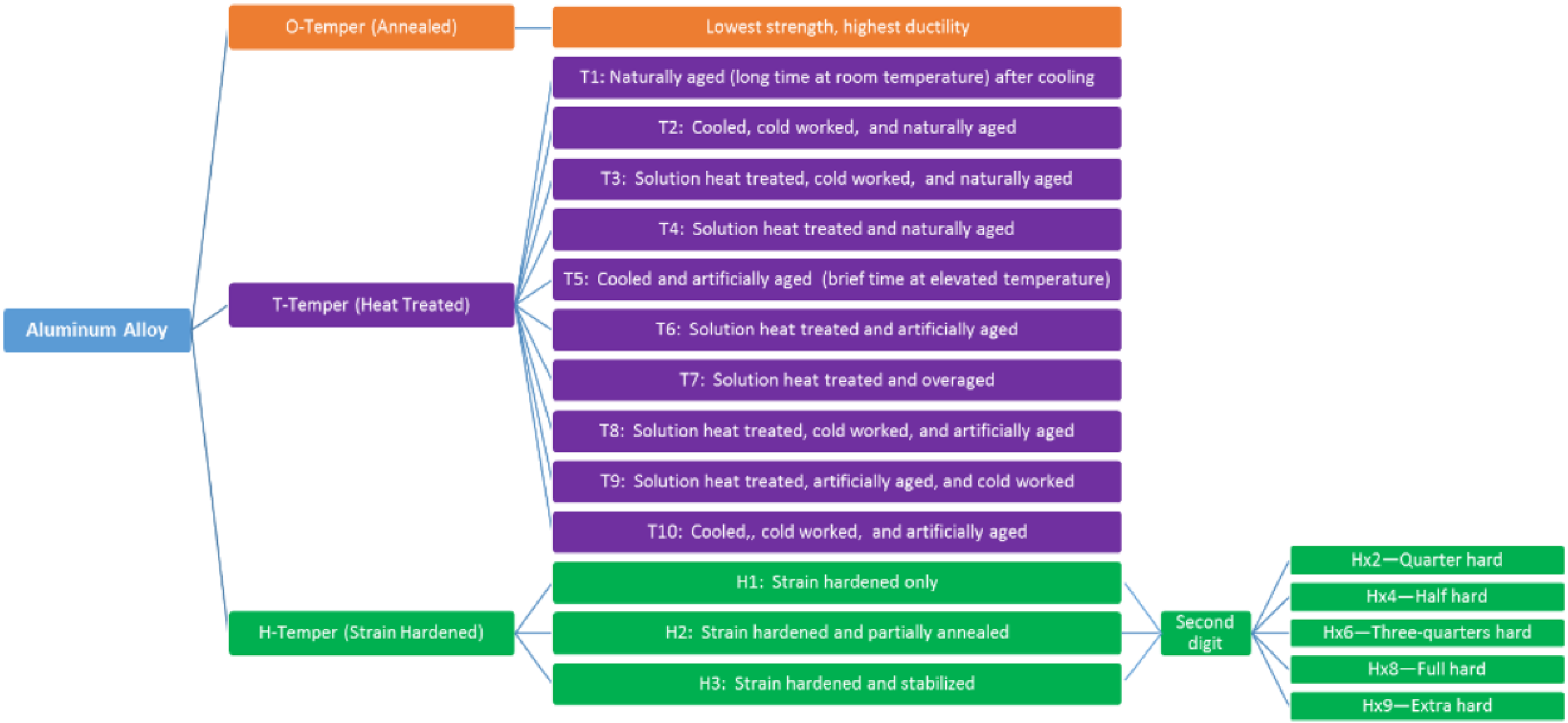
Iso – yield – strength curves for EN AW 7075

Wrought aluminum alloys: temper states

- T, thermally treated to produce stable tempers: heat-treatable wrought alloys that have followed a solution heat treatment followed by a quench and either natural or artificial aging. T is always followed by one or more digits:
  - The first digit after the T can be any from 1 to 10. It is a combination of:
    - Cooled from elevated temperature or solution heat treatment
    - Cold worked or not cold worked
    - Naturally aging or artificial ageing
  - Additional numbers indicate a variation in treatment that significantly alters the product characteristics that are or would be obtained using the basic treatment. There is not a full list of all such possible variations.

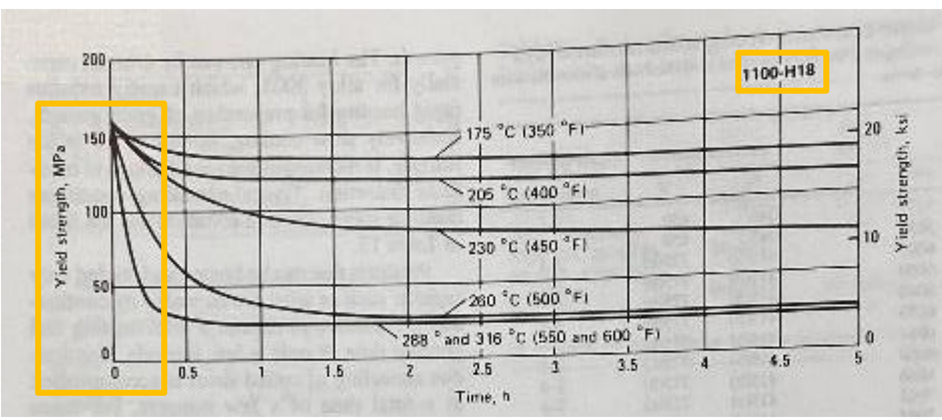


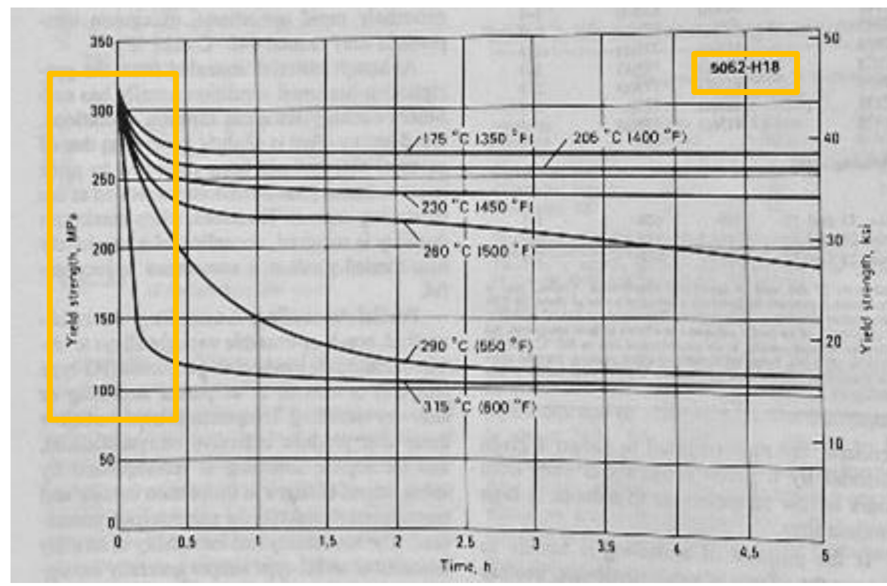
Wrought aluminum alloys: temper states



Wrought aluminum alloys: weldability

- Most non – heat treatable alloys plus series 6xxx can be fusion welded, and precaution should be taken with heat treatable high strength alloys.
  - When non – heat treatable alloys are welded, they loose the effect of an eventual work hardening → softening of HAZ.

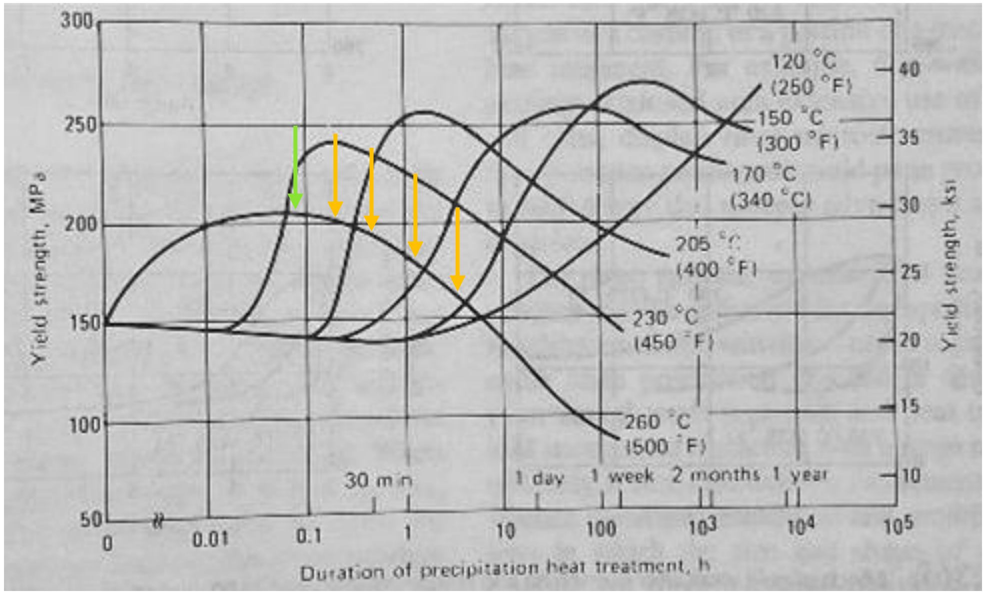




Source: ASM aluminium and aluminium alloys handbook

Wrought aluminum alloys: weldability

- Most non – heat treatable alloys plus series 6xxx can be fusion welded, and precaution should be taken with heat treatable high strength alloys.
  - When welding heat – treatable alloys → redistribution of hardening constituents → softening of HAZ. Attention to liquation cracking.

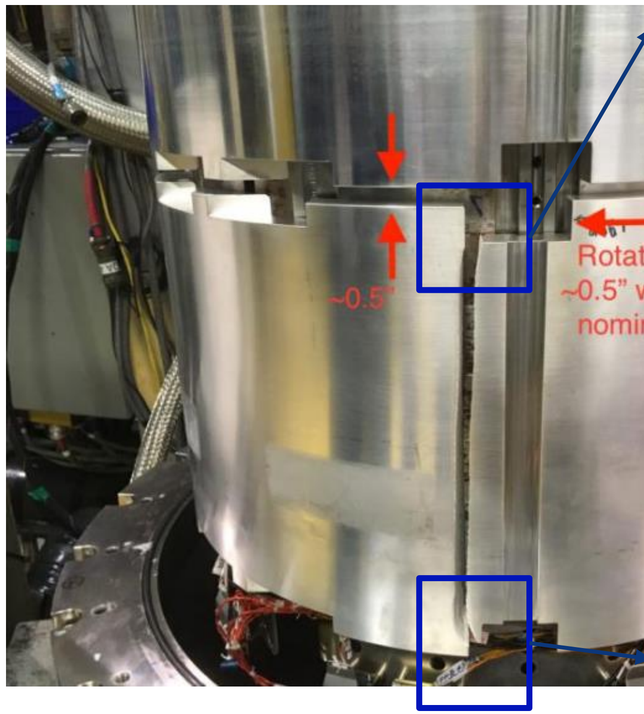


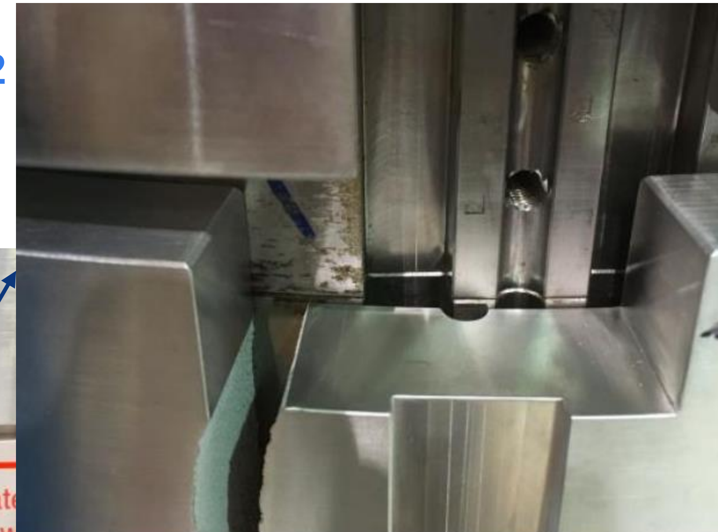
Source: ASM aluminium and aluminium alloys handbook

EN AW 6061

Wrought aluminum alloys: failure analysis

Catastrophic failure of MQXFAP2 Al shell. Failure analysis: EDMS 2088319



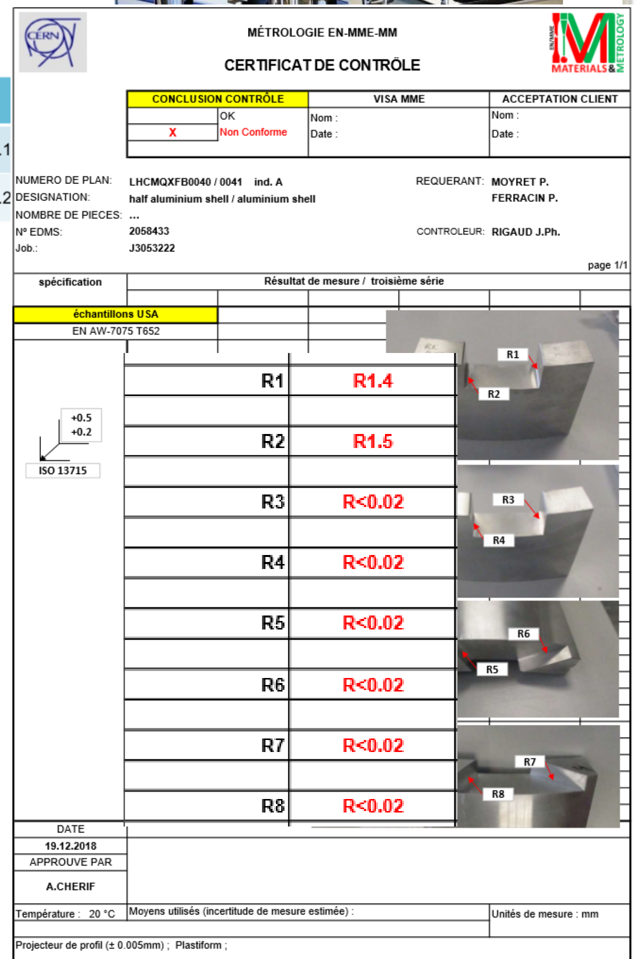


Wrought aluminum alloys: failure analysis

Mechanical testing at cryogenic temperature shows the material choice is correct

Material	Direction	E [GPa]	$Rp_{0.2}$ [MPa]	R_m [MPa]	A [%]	Z [%]
AA 7075 T652	Circumferential	84.0 ± 1.4	634.1 ± 11.5	750.6 ± 9.8	4.1 ± 0.2	12.2 ± 1.1
AA 7075 T652	Axial	85.2 ± 1.3	539.6 ± 5.2	659.7 ± 5.8	4.5 ± 0.1	12.0 ± 0.2

Material	Direction	K_Q [MPa√m]
AA 7075 T652	R - C	15.8 – 16.5
AA 7075 T652	C - R	24.0 – 27.2
AA 7075 T6	R - C	13.6 – 16.3



MÉTROLOGIE EN-MME-MM

CERTIFICAT DE CONTRÔLE

CONCLUSION CONTRÔLE		VISA MME	ACCEPTATION CLIENT
	OK	Nom :	Nom :
X	Non Conforme	Date :	Date :

NUMERO DE PLAN: LHCMQXFB0040 / 0041 ind. A — REQUERANT: MOYRET P., FERRACIN P.
DESIGNATION: half aluminium shell / aluminium shell
NOMBRE DE PIECES: ...
Nº EDMS: 2058433 — CONTROLEUR: RIGAUD J.Ph.
Job.: J3053222

page 1/1

spécification		Résultat de mesure / troisième série
échantillons USA EN AW-7075 T652		
+0.5 +0.2 ISO 13715	R1	R1.4
	R2	R1.5
	R3	R<0.02
	R4	R<0.02
	R5	R<0.02
	R6	R<0.02
	R7	R<0.02
	R8	R<0.02

DATE 19.12.2018
APPROUVE PAR A.CHERIF

Température : 20 °C — Moyens utilisés (incertitude de mesure estimée) : — Unités de mesure : mm
Projecteur de profil (± 0.005mm) ; Plastiform ;

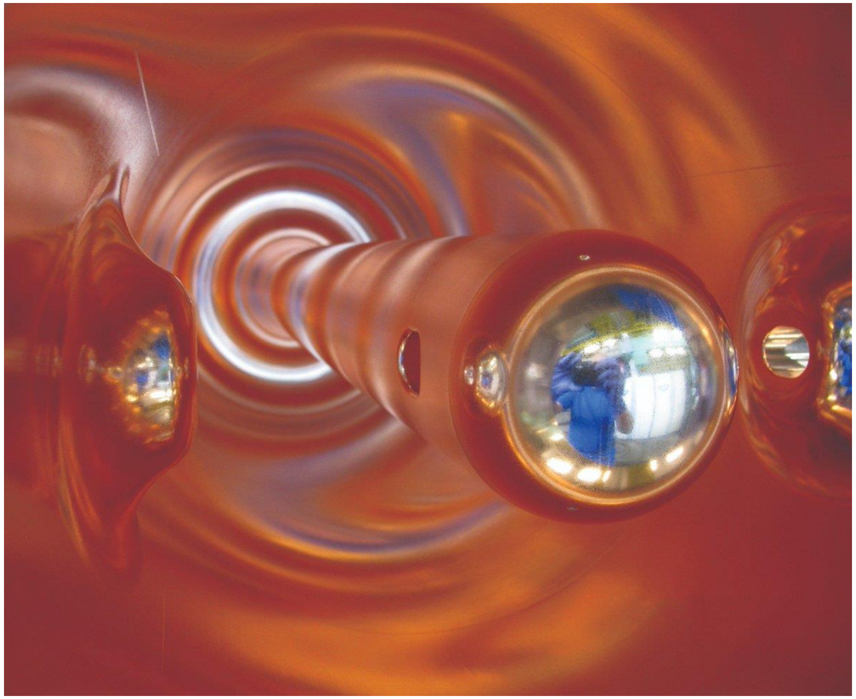
However, EN AW 7075 T651 is sensitive to the presence of sharp notches at 4 K.

Aluminium alloy shell:
failure analysis and material properties at cryogenic temperature.
EDMS 2088319

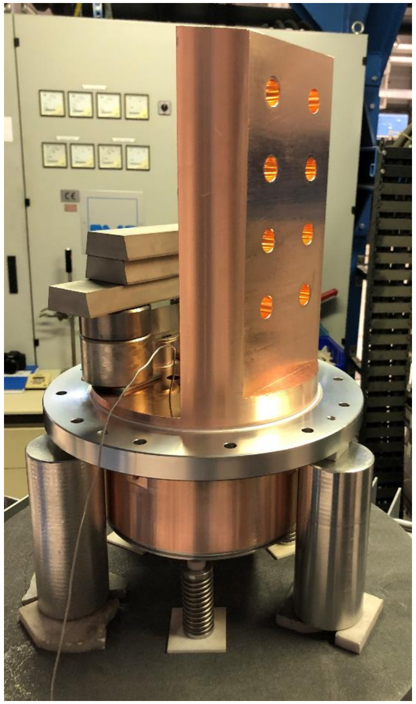
Copper

Copper for cryogenic application

- Extremely high thermal and electrical conductivity



HIE ISOLDE quarter wave resonator substrate

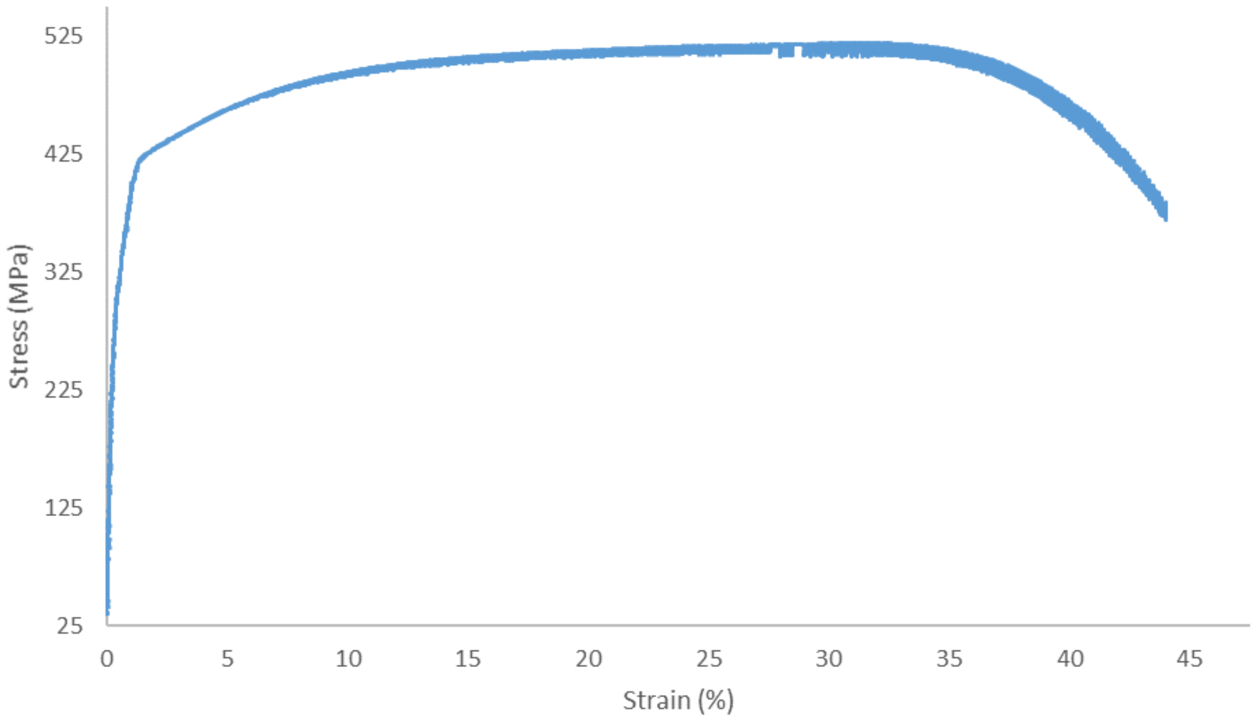


Courtesy: P. Moyret

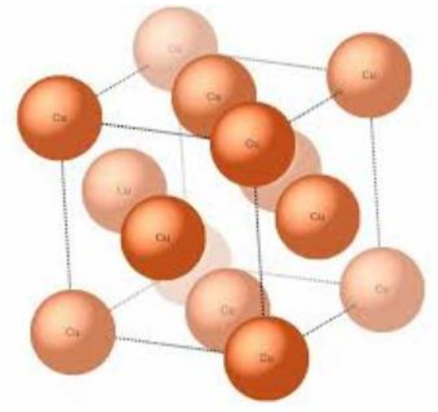
18 kA HL-LHC current leads

Copper for cryogenic application

- Ductile and tough down to 4 K



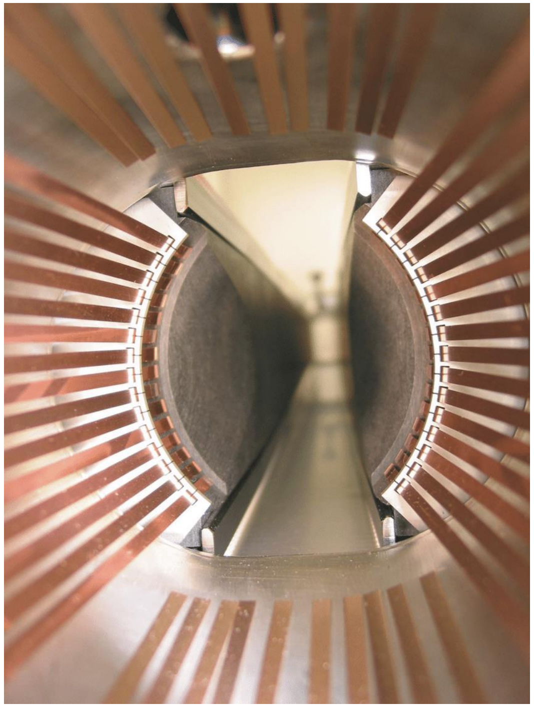
Stress vs strain curve OFE – copper. ¼ hard (H01) temper state.



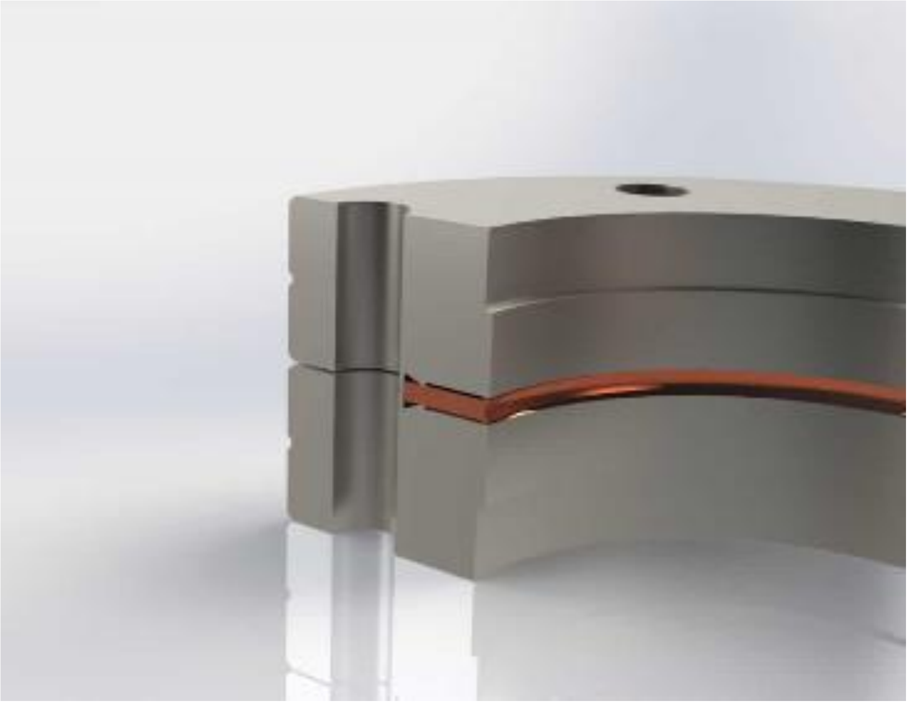
Face centered cubic (FCC) crystal structure

Copper for cryogenic application

- High availability, moderate price, formability, machinability.



RF fingers



Cu gasket Conflat flange UHV

Wrought copper alloys: temper states

Annealed tempers

Temper Codes	Temper Names
O10	Cast and Annealed (Homogenized)
O11	As Cast and Precipitation Heat Treated
O20	Hot Forged and Annealed
O25	Hot Rolled and Annealed
O26	Hot Rolled and Temper Annealed
O30	Hot Extruded and Annealed
O31	Hot Extruded and Precipitation Heat Treated
O32	Hot Extruded and Temper Annealed
O40	Hot Pierced and Annealed
O50	Light Anneal
O60	Soft Anneal
O61	Annealed
O65	Drawing Anneal
O68	Deep Drawing Anneal
O70	Dead Soft Anneal

Cold worked tempers

Temper Codes	Temper Names
H00	⅛ Hard
H01	¼ Hard
H02	½ Hard
H03	¾ Hard
H04	Hard
H06	Extra Hard
H08	Spring
H10	Extra Spring
H12	Special Spring
H13	Ultra Spring
H14	Super Spring

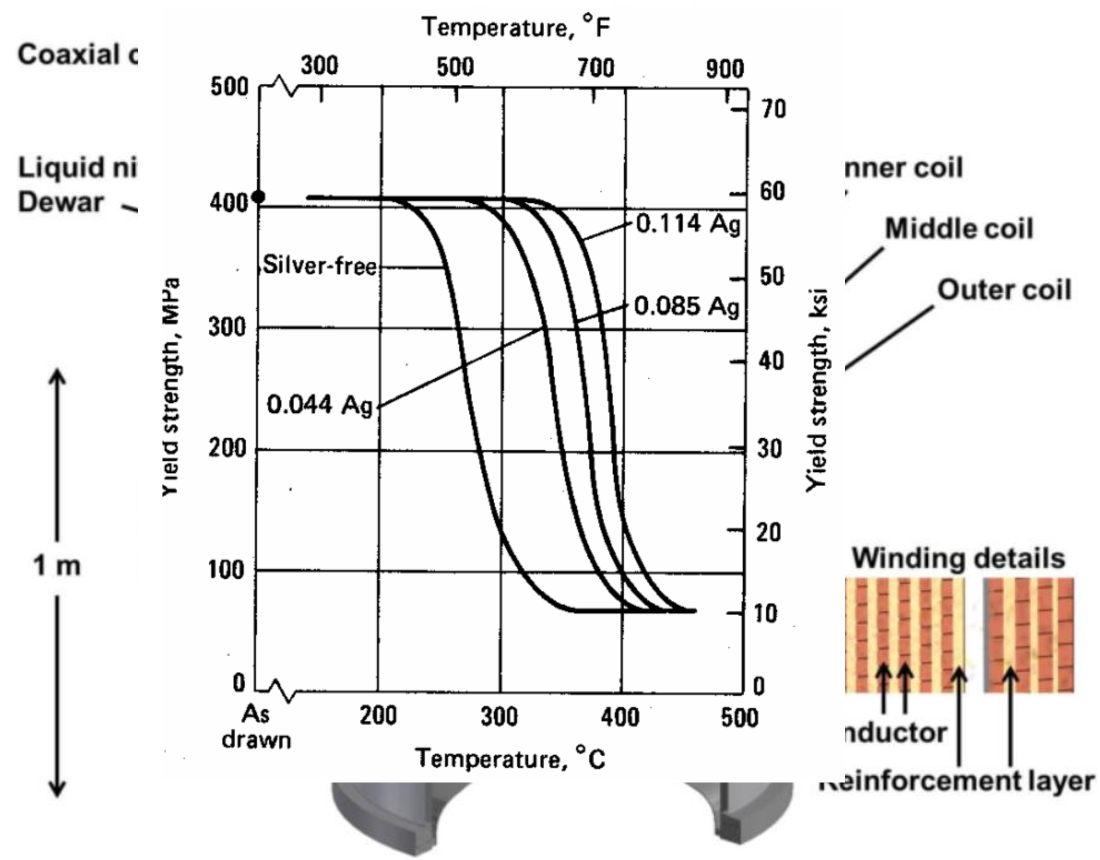
Temper Codes	Temper Names
H50	Hot Extruded and Drawn
H52	Hot Pierced and Drawn
H55	Light Drawn, Light Cold-Worked
H58	Drawn General Purpose
H60	Cold Heading, Forming
H63	Rivet
H64	Screw
H66	Bolt
H70	Bending
H80	Hard Drawn

Copper and copper alloys

Generic name	UNS No.	Composition
Wrought alloys		
Coppers(a)	C10100–C15815	>99% Cu
High-copper alloys(b)	C16200–C19900	>96% Cu
Brasses		
Leaded brasses		
Tin brasses		

"electrical coppers"
Cu OFE, C10100
Cu OF, C10200
Cu OFS, C10700

99.8Cu-0.15Al_2O_3, C15715



LNCMI 100 magnet (GLIDCOP AL-15 reinforced with Zylon fibers)

Copper and copper alloys

Generic name	UNS No.	Composition
Wrought alloys		
Coppers(a)	C10100–C15815	>99% Cu
High-copper alloys(b)	C16200–C19900	>96% Cu
Brasses	C20100–C28000	Cu-Zn
Leaded brasses	C31200–C38500	Cu-Zn-Pb
Tin brasses		Cu-Zn-Sn-Pb
Phosphor bronzes		Cu-Sn-P
Leaded phosphor bronzes		Cu-Sn-Pb-P
Copper-phosphorus and copper-silver-phosphorus alloys(c)		Cu-P-Ag
Aluminum bronzes		Cu-Al-Ni-Fe-Si-Sn
Silicon bronzes		Cu-Si-Sn
		Cu-Zn-Mn-Fe-Sn-Al-Si-Co
		Cu-Ni-Fe
		Cu-Ni-Zn

High strength copper alloys
Cu-2%Be, C17200
Cu-0.3%Be-0.5%Co, C17410

C17410 RF fingers of a secondary LHC collimator

Titanium

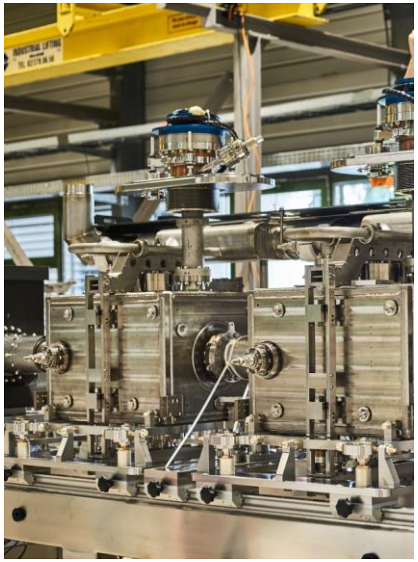
Titanium for cryogenic application

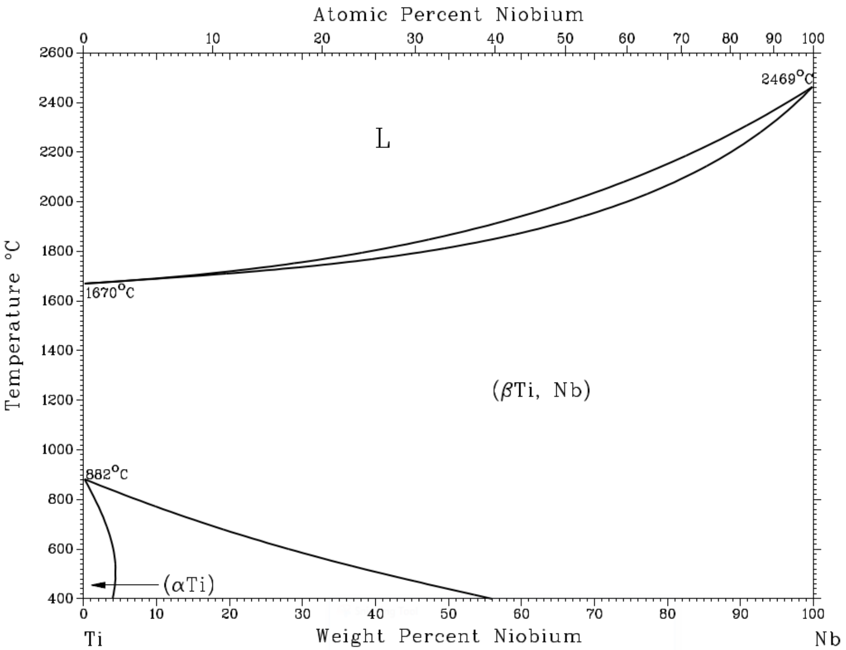
- High specific strength.
- Paramagnetic
- Lower thermal expansion / contraction than stainless steel

Axial force tie rods of the ATLAS barrel toroid magnet

Titanium for cryogenic aplication

- Certain grades are ductile and moderately tough at cryogenic temperature.
- Thermal contraction closer to Nb than stainless steel.
- Weldable with Nb (total solubility)

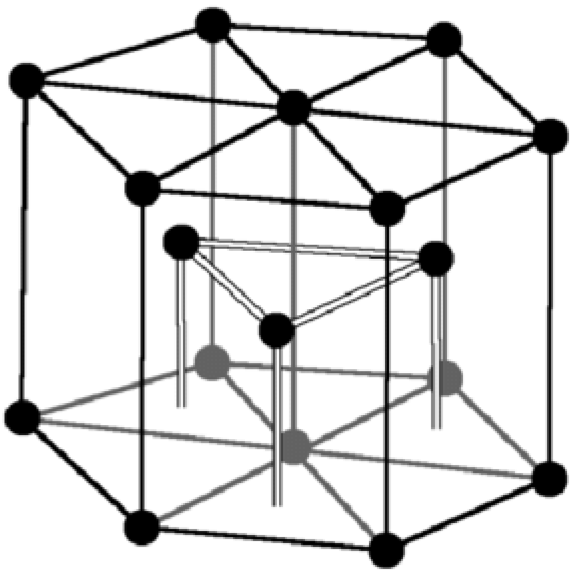




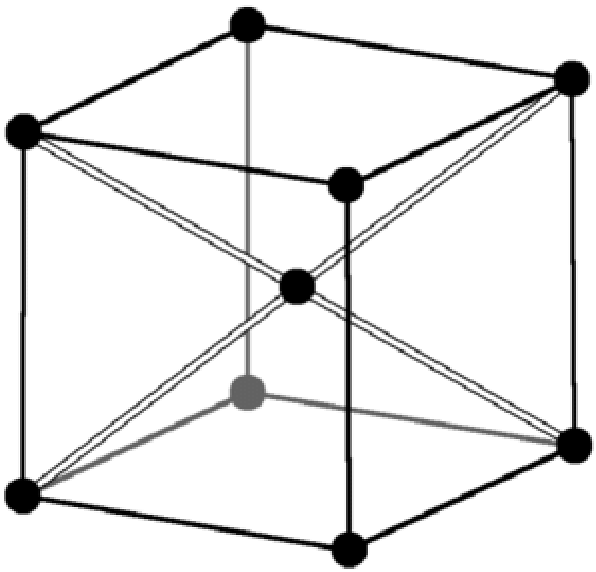
Titanium (II) He tanks of the crab cavities for HL - LHC

Titanium grades

- Microstructures of Ti



α Ti – hexagonal closed packed (HCP)



β Ti – body centred cubic (BCC)

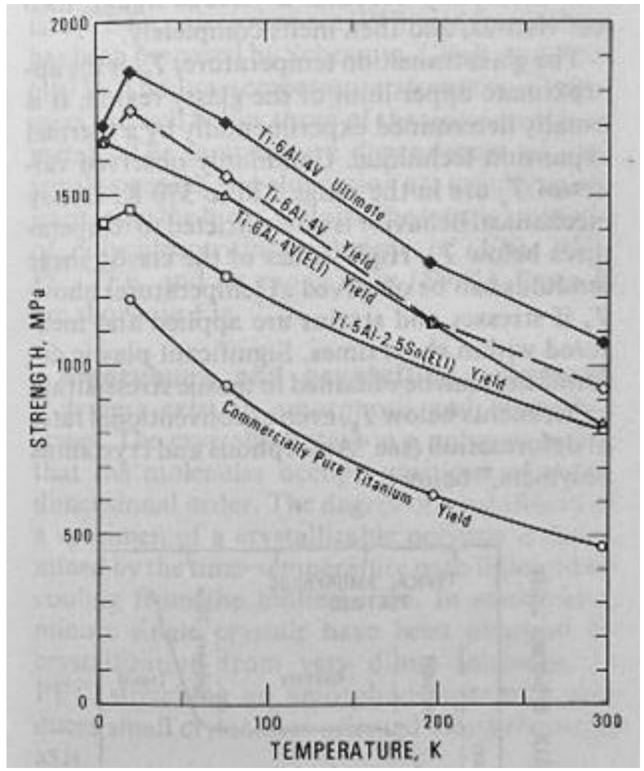
We privilege compact structures (**α – Ti**) for cryogenic application

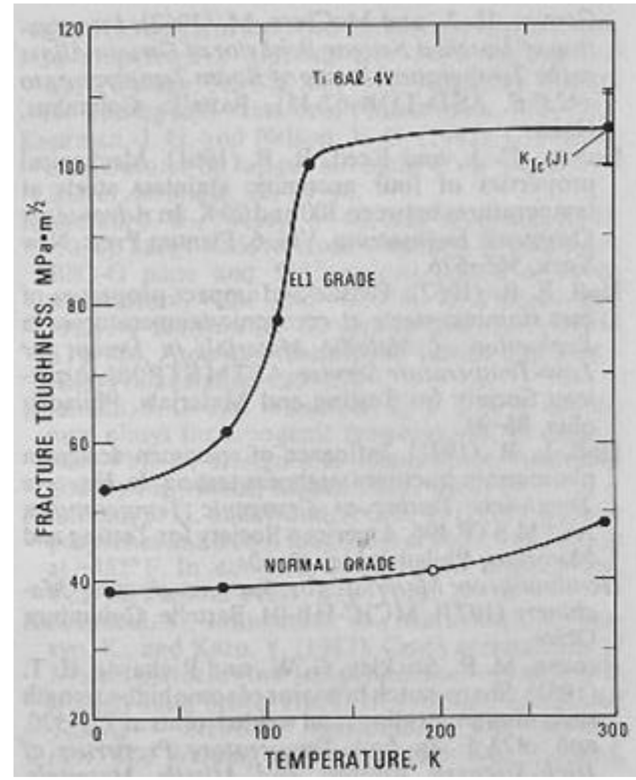
Titanium grades

- Extra – low interstitials (ELI)

At any rate pure titanium was finally produced. But it was only by stretching the point considerably that this metal could be accepted as pure, for it still contained several tenths of a per cent of impurities. Only several tenths, but they were like a fly in the ointment. The impurities made titanium fragile and brittle and unsuitable for machining. It earned a bad fame for being a useless, good-for-nothing metal.

In 1925, the Dutch scientists van Arkel and de Boer decomposed titanium tetrachloride on a heated tungsten wire and obtained high-purity titanium. And then it became clear that Hunter's assertion concerning the brittleness of titanium could not stand up to criticism: the metal produced by van Arkel and de Boer was highly plastic, could be forged like iron and rolled into sheets, strip, wire and even the thinnest foil.





We privilege ELI grades for cryogenic application

Titanium grades

Designation	Tensile strength (min) MPa	Tensile strength (min) ksi	0.2% yield strength (min) MPa	0.2% yield strength (min) ksi	Impurity limits, wt% (max) N	C	H	Fe	O	Nominal composition, wt% Al	Sn	Zr	Mo	Others
Unalloyed grades														
ASTM grade 1														
ASTM grade 2														
ASTM grade 3														
ASTM grade 4														
ASTM grade 7														
ASTM grade 11														
α and near-α alloys														
Ti-0.3Mo-0.8Ni														
Ti-5Al-2.5Sn														
Ti-5Al-2.5Sn-ELI														
Ti-8Al-1Mo-1V														
Ti-6Al-2Sn-4Zr-2Mo														
Ti-6Al-2Nb-1Ta-0.8Mo														
Ti-2.25Al-11Sn-5Zr-1Mo														
Ti-5.8Al-4Sn-3.5Zr-0.7Nb-0.5Mo-0.35Si														
α-β alloys														
Ti-6Al-4V(a)														
Ti-6Al-4V-ELI(a)														
Ti-6Al-6V-2Sn(a)														
Ti-8Mn(a)														
Ti-7Al-4Mo(a)														
Ti-6Al-2Sn-4Zr-6Mo(b)														
Ti-5Al-2Sn-2Zr-4Mo-4Cr(b)(c)														
Ti-6Al-2Sn-2Zr-2Mo-2Cr(c)														
Ti-3Al-2.5V(d)														
Ti-4Al-4Mo-2Sn-0.5Si														
β alloys														
Ti-10V-2Fe-3Al(a)(c)														
Ti-13V-11Cr-3Al(b)														
Ti-8Mo-8V-2Fe-3Al(b)(c)														
Ti-3Al-8V-6Cr-4Mo-4Zr(a)(c)														
Ti-11.5Mo-6Zr-4.5Sn(a)														
Ti-15V-3Cr-3Al-3Sn														
Ti-15Mo-3Al-2.7Nb-0.2Si														

Pure Ti

He tanks surrounding Crab cavities. Ti grade 2

(a) Mechanical properties given for the annealed condition; may be solution treated and aged to increase strength. (b) Mechanical properties given for the solution-treated-and-aged condition; alloy not normally applied in annealed condition. (c) Semicommercial alloy; mechanical properties and composition limits subject to negotiation with suppliers. (d) Primarily a tubing alloy; may be cold drawn to increase strength. (e) Combined $O_2 + 2N_2 = 0.27\%$. (f) Also solution treated and aged using an alternative aging temperature (480 °C, or 900 °F)

Titanium grades

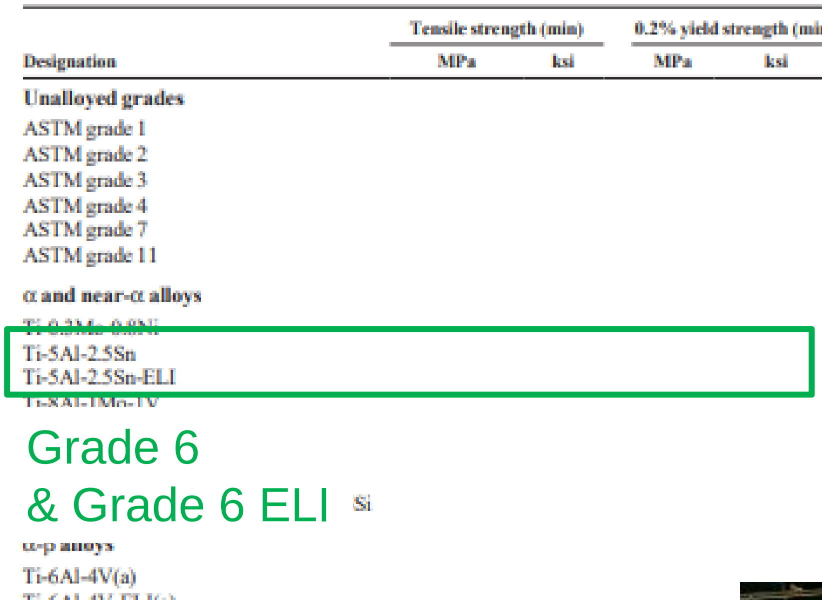
Designation	Tensile strength (min) MPa	Tensile strength (min) ksi	0.2% yield strength (min) MPa	0.2% yield strength (min) ksi	Impurity limits, wt% (max) N	C	H	Fe	O	Nominal composition, wt% Al	Sn	Zr	Mo	Others
Unalloyed grades														
ASTM grade 1														
ASTM grade 2														
ASTM grade 3														
ASTM grade 4														
ASTM grade 7														
ASTM grade 11														
α and near-α alloys														
Ti-0.3Mo-0.8Ni														
Ti-5Al-2.5Sn														
Ti-5Al-2.5Sn-ELI														
Ti-8Al-1Mo-1V														
Ti-6Al-2Sn-4Zr-2Mo														
Ti-6Al-2Nb-1Ta-0.8Mo														
Ti-2.25Al-11Sn-5Zr-1Mo														
Ti-5.8Al-4Sn-3.5Zr-0.7Nb-0.5Mo-0.35Si														
α-β alloys														
Ti-6Al-4V(a)														
Ti-6Al-4V-ELI(a)														
Ti-6Al-6V-2Sn(a)														
β alloys														
Ti-10V-2Fe-3Al(a)(c)														
Ti-13V-11Cr-3Al(b)														
Ti-8Mo-8V-2Fe-3Al(b)(c)														
Ti-3Al-8V-6Cr-4Mo-4Zr(a)(c)														
Ti-11.5Mo-6Zr-4.5Sn(a)														
Ti-15V-3Cr-3Al-3Sn														
Ti-15Mo-3Al-2.7Nb-0.2Si														

Grade 5
& Grade 5 ELI
(grade 23)

(a) Mechanical properties given for the annealed condition; may be solution treated and aged to increase strength. (b) Mechanical properties given for the solution-treated-and-aged condition; alloy not normally applied in annealed condition. (c) Semicommercial alloy; mechanical properties and composition limits subject to negotiation with suppliers. (d) Primarily a tubing alloy; may be cold drawn to increase strength. (e) Combined $O_2 + 2N_2 = 0.27\%$. (f) Also solution treated and aged using an alternative aging temperature (480 °C, or 900 °F)

RF feedthrough flanges
of the crab cavities.
Ti grade 23

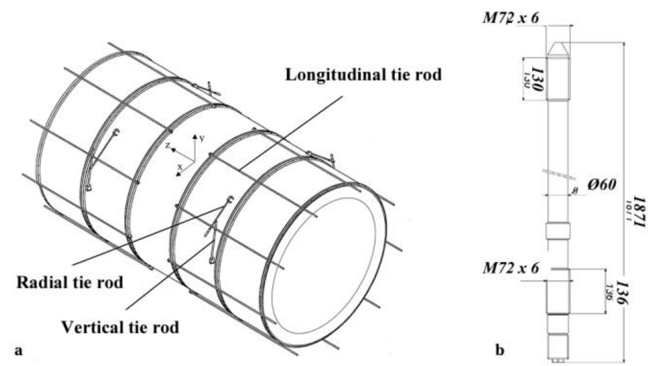
Titanium grades



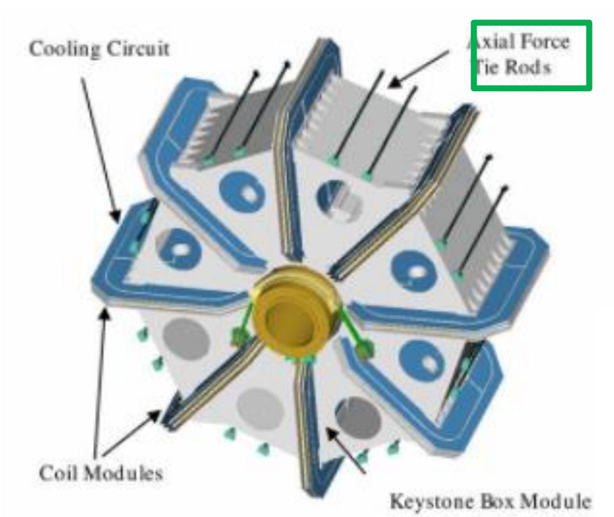
Designation	Tensile strength (min)		0.2% yield strength (min)	
	MPa	ksi	MPa	ksi
Unalloyed grades				
ASTM grade 1				
ASTM grade 2				
ASTM grade 3				
ASTM grade 4				
ASTM grade 7				
ASTM grade 11				
α and near-α alloys				
Ti-0.3Mo-0.8Ni				
Ti-5Al-2.5Sn				
Ti-5Al-2.5Sn-ELI				
Ti-8Al-1Mo-1V				
... Si				
α-β alloys				
Ti-6Al-4V(a)				

Grade 6
& Grade 6 ELI

Source: S. Sgobba *et al*. (2003, July). Manufacturing, quality control and assessment of the cryogenic properties of a titanium alloy for application to the coil suspension system of the Compact Muon Solenoid (CMS). In *Proc. 10th World Conference on Titanium* (pp. 13-18).



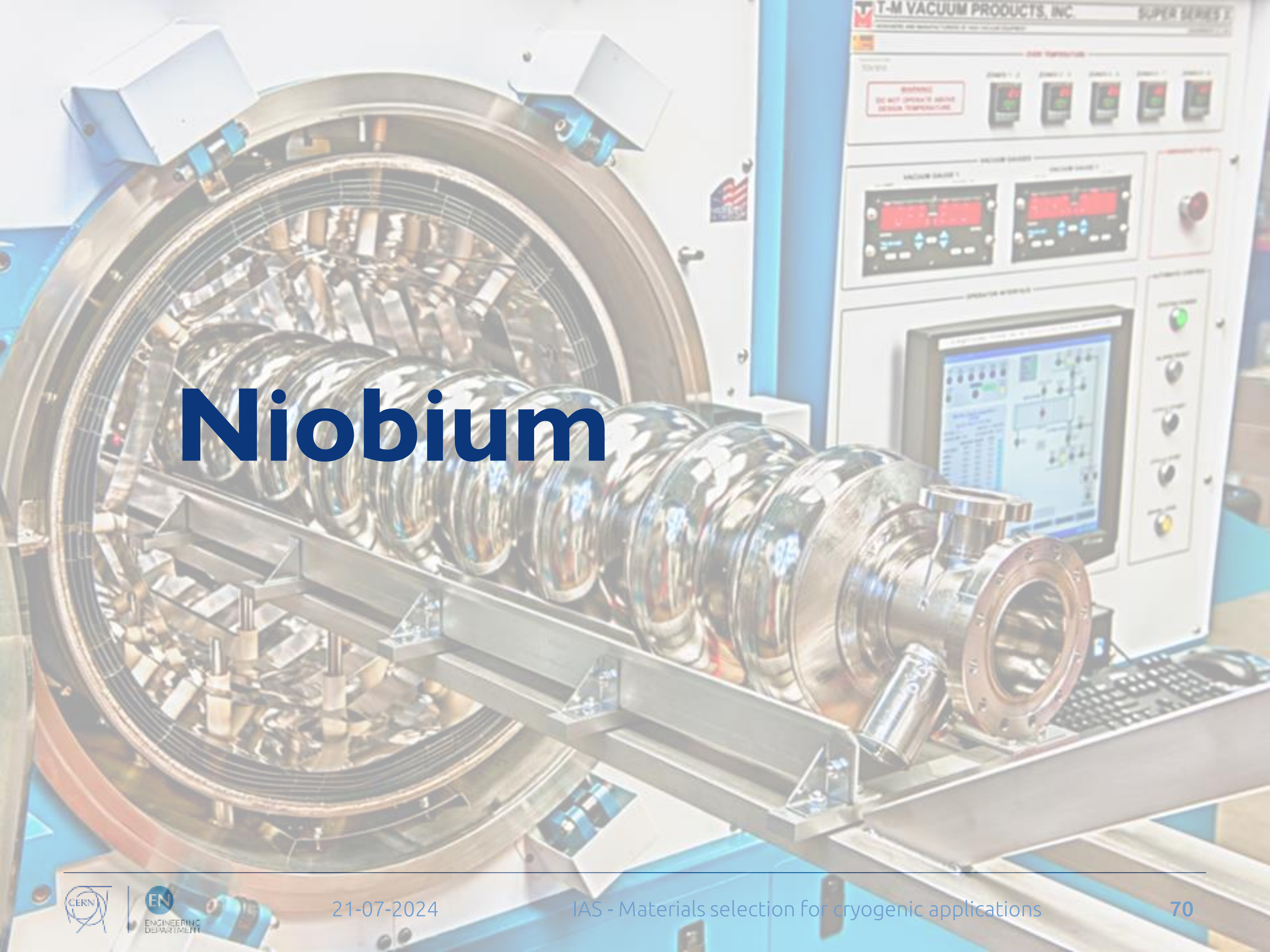
CMS longitudinal tie rods



Axial force tie rods of the ATLAS barrel toroid magnet

Source: P. Jenni (CERN), ATLAS overview, status and plans. Workshop on cooperation in HEP between CERN and China. Beijing, 14 – 15 May 2005.

Niobium



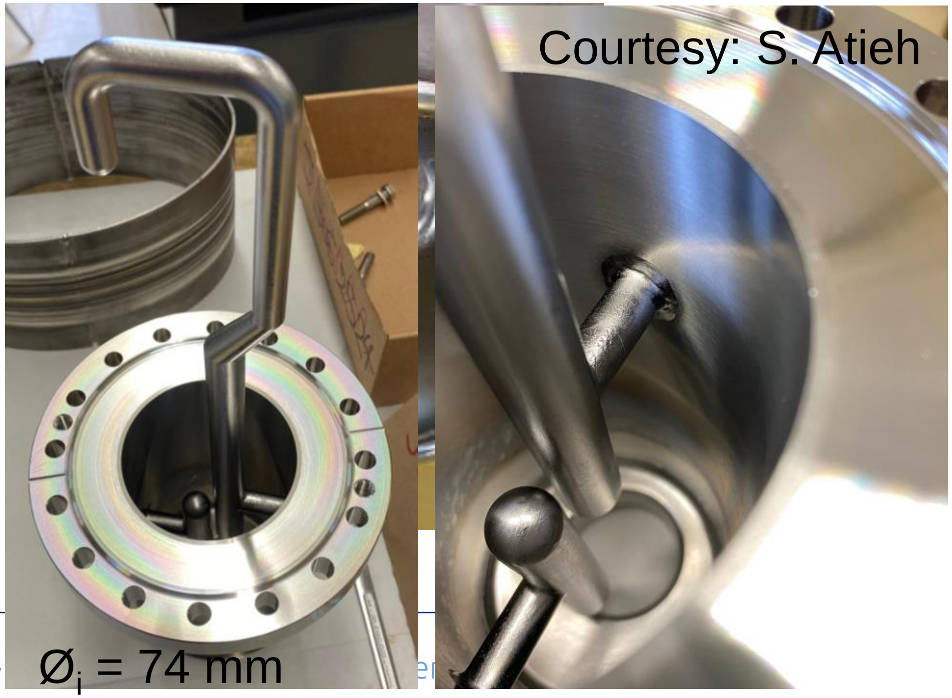
Niobium for cryogenic application: SCRF cavities

- High critical temperature (9.2 K)
- High critical magnetic field
- High formability, ‘easy’ to machine and weldable.
- Available in practically any size

Spoke cavity

Source: Shepard, K. W et al, (1999). Prototype 350 MHz niobium spoke-loaded cavities.

High order mode (HOM) antenna



Niobium grades

- ASTM B392 & B393

Designation: B393 – 18

Standard Specification for
Niobium and Niobium Alloy Strip, Sheet, and Plate[1]

Designation: B392 – 18

Standard Specification for
Niobium and Niobium Alloy Bar, Rod, and Wire[1]

1. Scope

1.1 This specification covers five grades of wrought niobium and niobium alloy strip, sheet, and plate as follows:

1.1.1 *R04200-Type 1*—Reactor grade unalloyed niobium,

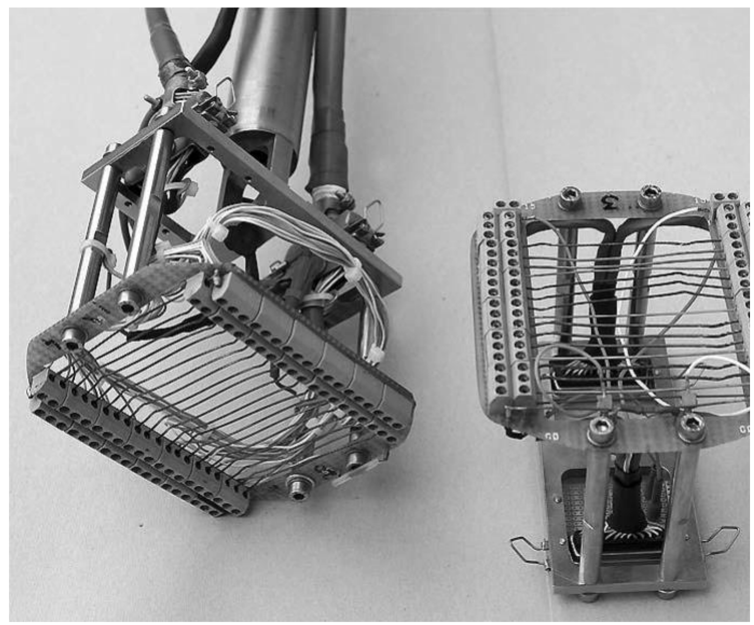
1.1.2 *R04210-Type 2*—Commercial grade unalloyed niobium,

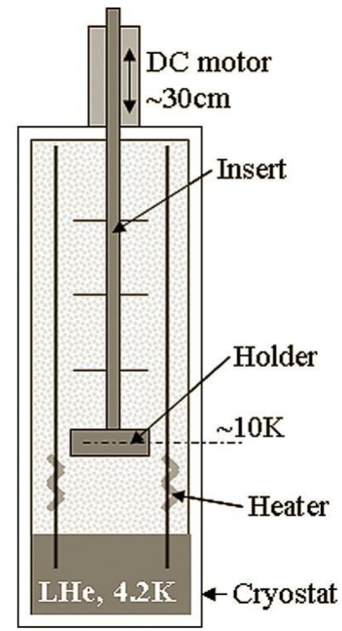
1.1.3 *R04251-Type 3*—Reactor grade niobium alloy containing 1 % zirconium,

1.1.4 *R04261-Type 4*—Commercial grade niobium alloy containing 1 % zirconium, and.

1.1.5 *R04220-Type 5*—RRR grade pure niobium.

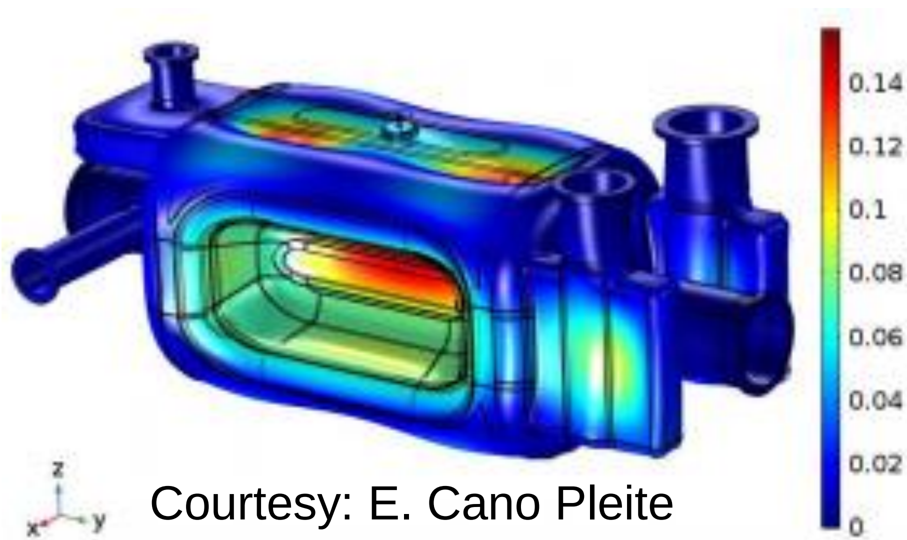
RRR = residual resistivity ratio
An accurate measurement of the purity above 99.999%





Niobium grades & SCRF

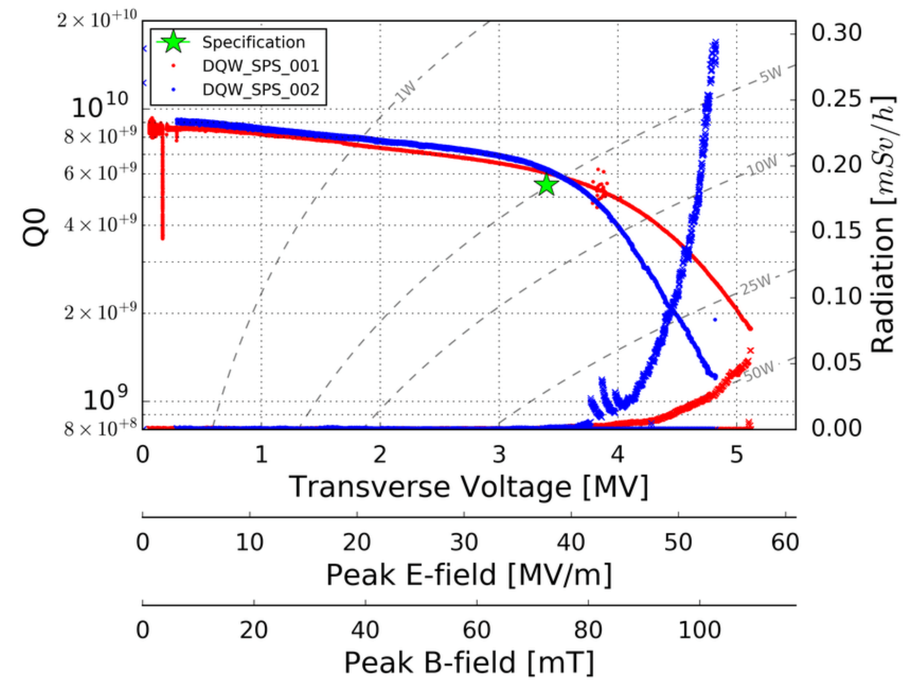
- CERN technical specification
  - Equilibrium between:
    - RF performance
    - Mechanical robustness
    - Material soundness



Courtesy: E. Cano Pleite

Displacements (in mm) of RFD cavity.
1 bar external pressure

RF performance DQW cavities

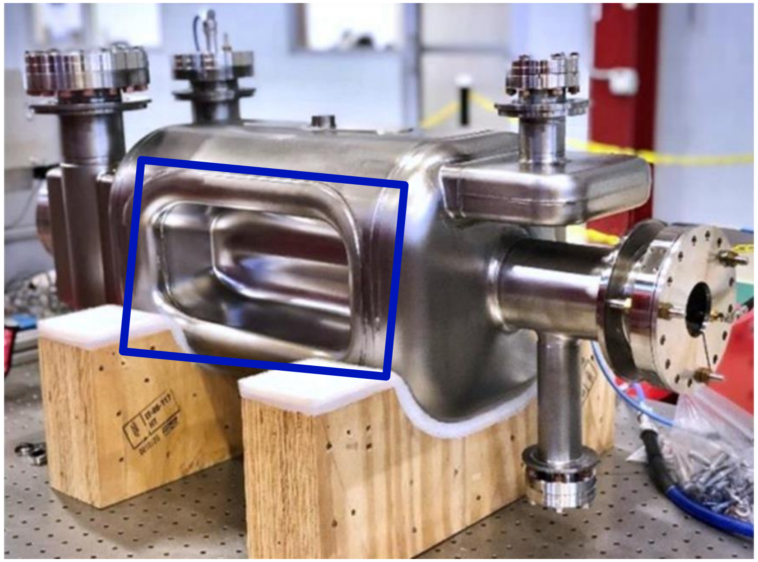


NDT of Nb technical specification

Visual inspection	EN 13018	On final product after all metallurgical processing for every item, 100%
Dye penetrant (optional)	Written procedure, based on EN 10228-2, following approval by CERN	Upon demand by CERN; on final product after all metallurgical processing for every item, 100%
Macro-/ micro-optical	Specimen preparation: ASTM E3 Macro-etching: ASTM E340 Micro-etching: ASTM E407	Upon demand by CERN; on witness sample of final product after all metallurgical processing for every item.
Ultrasonic	Written procedure based on: -EN 10228-4 for the method -EN 4050-4 for acceptance criteria Following approval by CERN	On final product after all metallurgical processing for every item, 100%

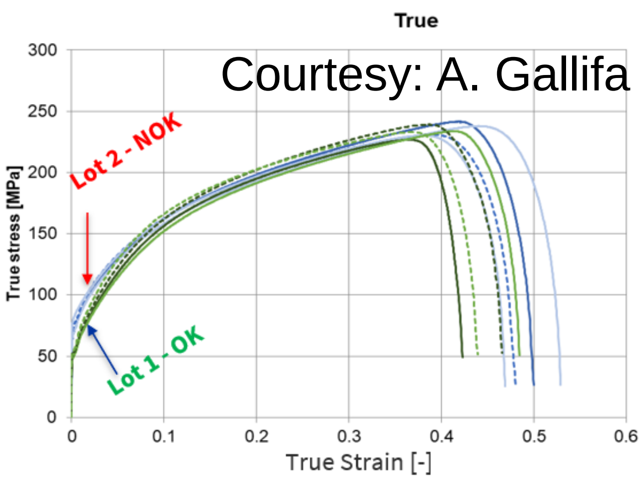
SCRF Nb: failure analysis

RFD Pole forming trials held at CERN Main Workshop (May 2022).



- Same material specification
- Same material supplier
- **2 different material lots**
- Same tooling
- Same operators
- Same forming procedure
- Same press machine

Material that shows bad formability complies with CERN Nb spec. & DESY Nb Spec.!



Material	$n_{0.02-0.20}$	$R_{p0.2}/R_m$
Lot OK	0.35 – 0.38	0.31 – 0.33
Lot NOK	0.28 – 0.30	0.42 – 0.50

A. Gallifa Terricabras, et al Optimizing the Manufacture of High-Purity Niobium SRF Cavities Using the Forming Limit Diagram: A Case Study of the HL-LHC Crab Cavities RFD Pole.

Strain hardening coefficient ‘n’ value seems to be significantly different, as well as the ratio $R_{p0.2}/R_m$

Ni - alloys

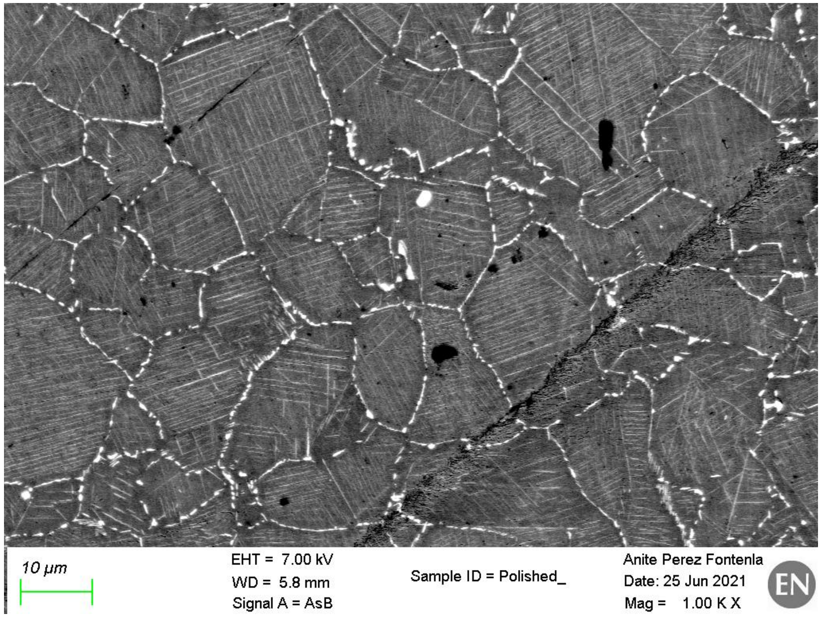
Ni alloys for cryogenic application

- High specific strength
- Austenitic
- High E modulus

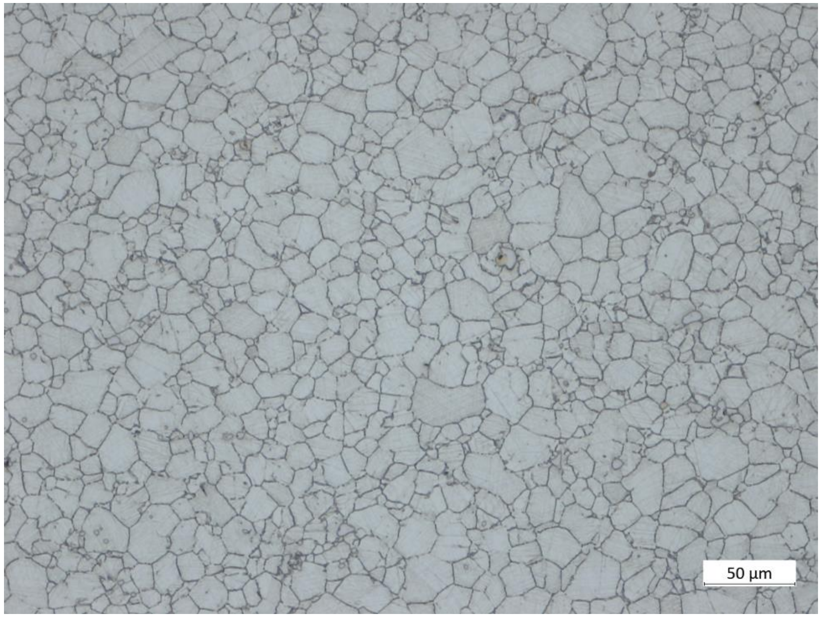
Inconel 718 for ITER magnet supports

Ni alloys for cryogenic application

- Complex material (as stainless steel)
  - Careful control all fabrication steps to avoid undesired secondary phases.
  - Aim for small and homogeneous grain size to enhance UT controllability.



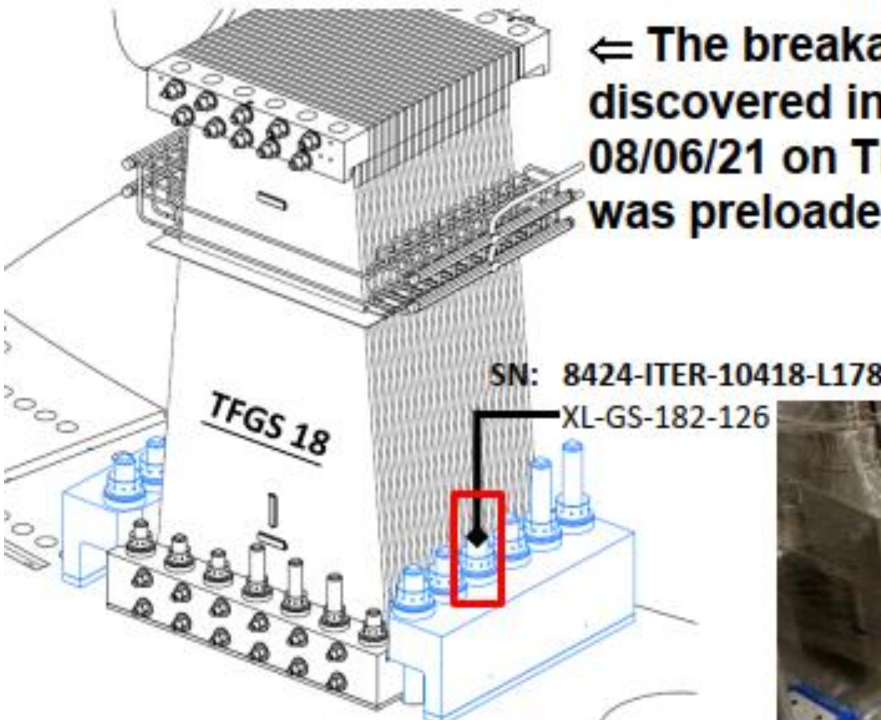
Continuous network of secondary phases at GB

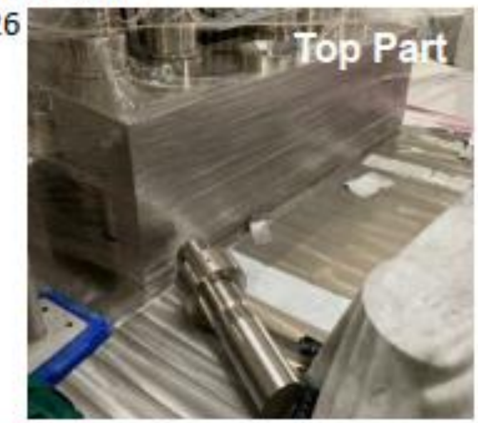


Homogeneous equiaxial grains (G = 9)

Ni alloys: failure analysis

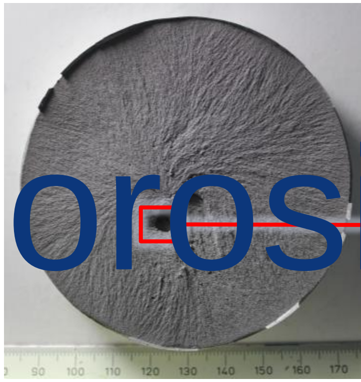
⇐ **The breakage was discovered in the morning of 08/06/21 on TFGS 18, which was preloaded on 27/04/21**





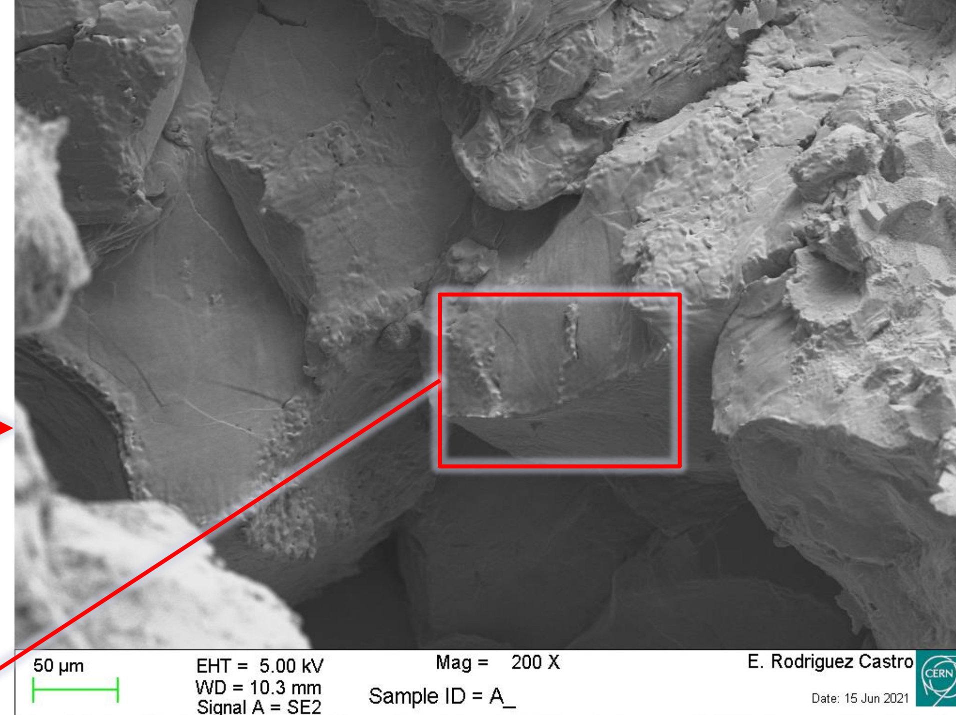
A complete break of one M85 stud was discovered, after its installation and preloading up to 1.41 MN (60 % of Rp0.2). The upper half of the part blew out from the bolthole and was found lying horizontally on the Cryostat Base near the TFGS while the lower half part shot downward and lied in the confined space of Cryostat Base.

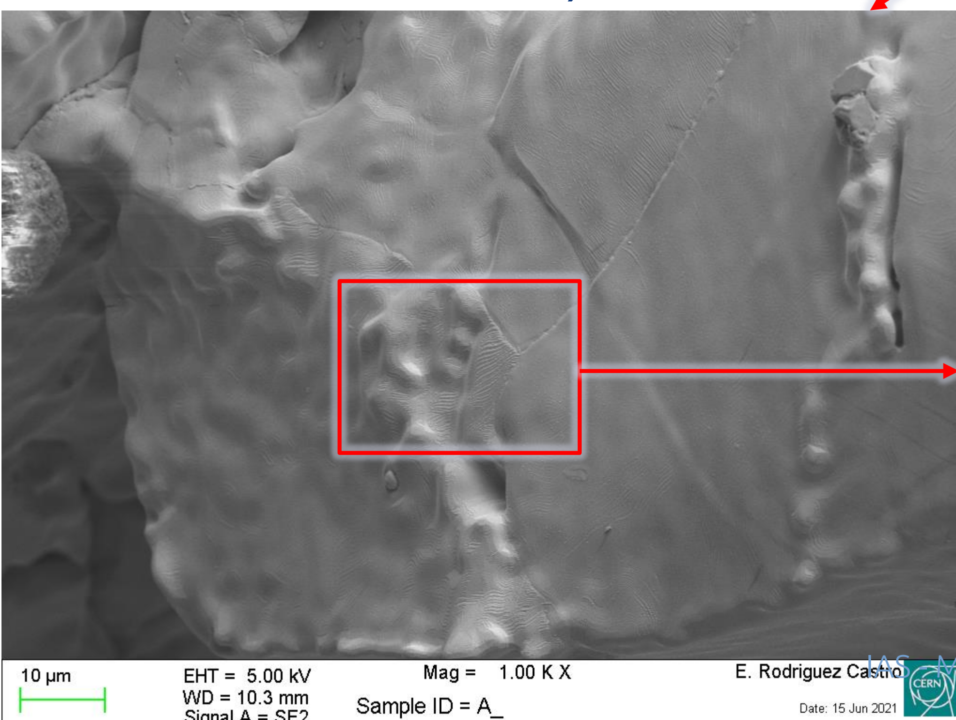
Ni alloys: failure analysis

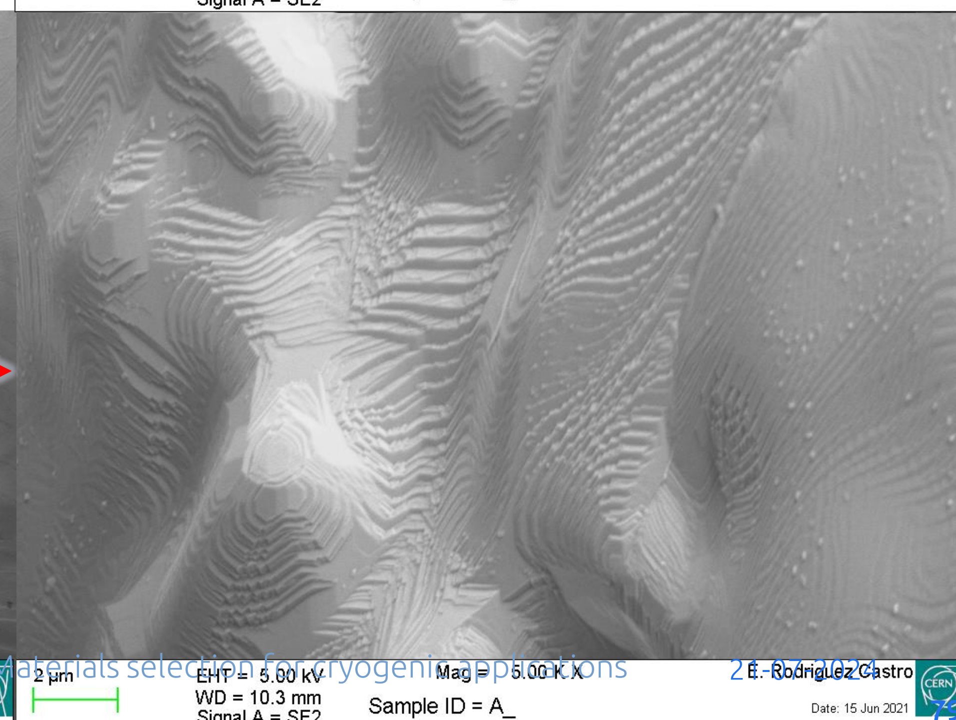


Porosities

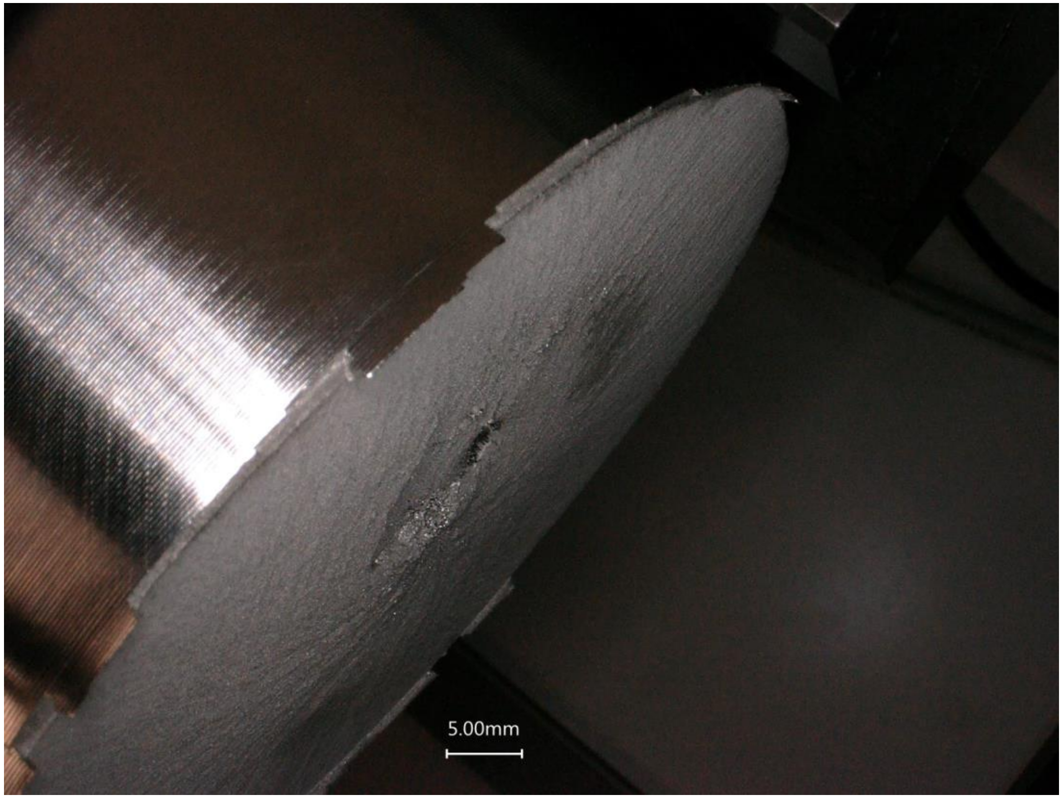
Rippled surface (presence of surface terraces)

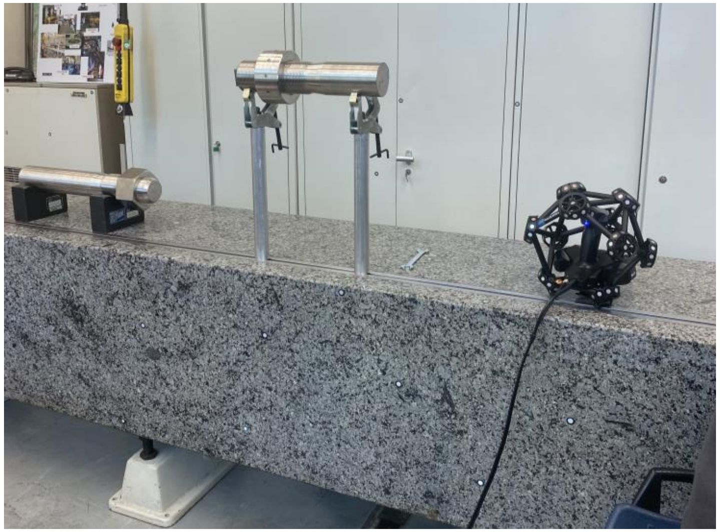


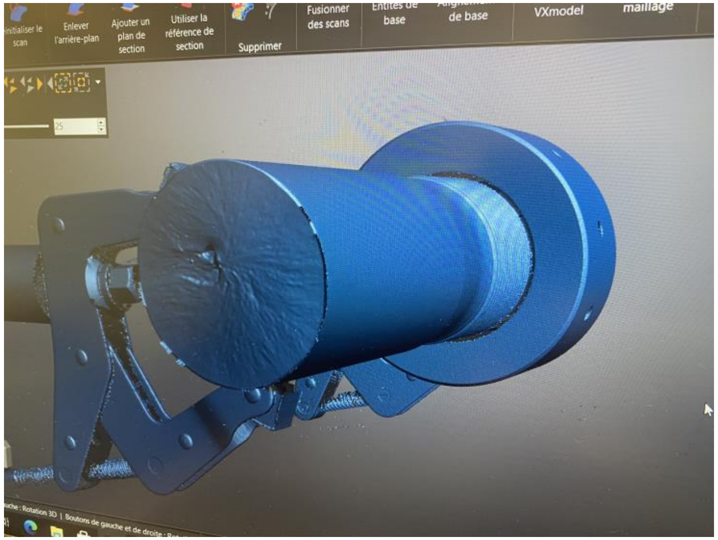




Ni alloys: failure analysis

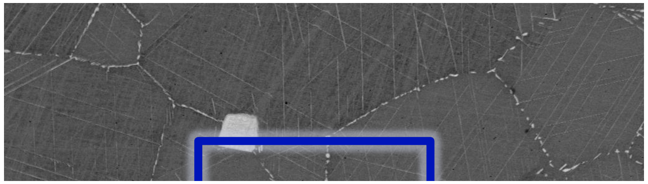


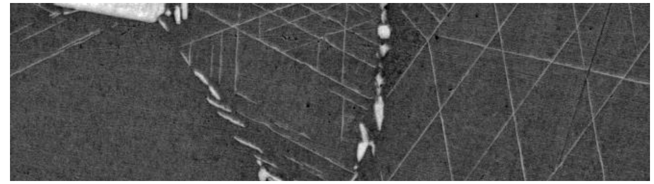




No necking → macroscopically brittle

Ni alloys: failure analysis





A non – forgiving material (low toughness) containing porosities failed at 60% of its $Rp_{0.2}$)

Pq	11.7	12.86	11.54	13.2
Pmax	13.6	15.23	12.01	15.2
Pmax/Pq	1.16	1.18	1.04	1.15
Kq, MPa*m^0.5	**62.8**	**68.0**	**62.5**	**71.2**

Very low impact and fracture toughness → brittleness

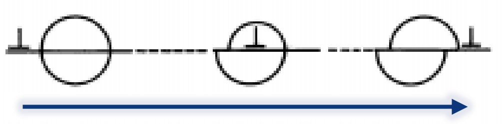
Conclusions

- Key takeaways:
  - "A material for a critical application is not a mere chemical composition or designation":
    - Specification. Fabrication route. Temper state
    - Controls
    - Price
  - Attention to "the hidden iceberg properties" (e.g. notch sensitivity, strain hardening) and to the butterfly effect.
- Whenever possible, try to use FCC materials (no DTBT).
- Use more advanced properties (fatigue, toughness) and not 2/3 $Rp_{0.2}$ or ½ UTS). Avoid stress concentrators.
- Time (and problems) are saved if material selection is integrated from the beginning of the conceptions.
  - Advanced non conventional materials require extensive prior R&D

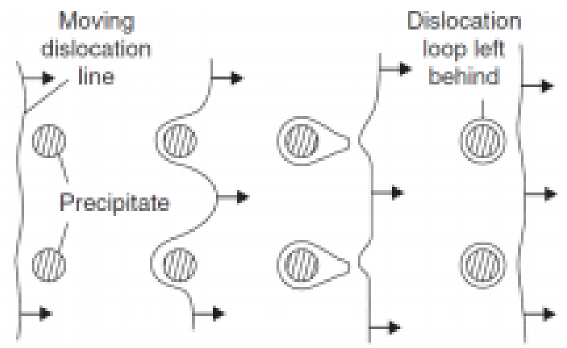
Additional slides

Precipitation strengthening

- Dislocations are the carriers of plastic deformation
- Precipitates hinder dislocation motion. There are 2 types: coherent and incoherent



A dislocation cuts through a coherent particle, i.e. it passes through the precipitate on the same slip plane as in the matrix



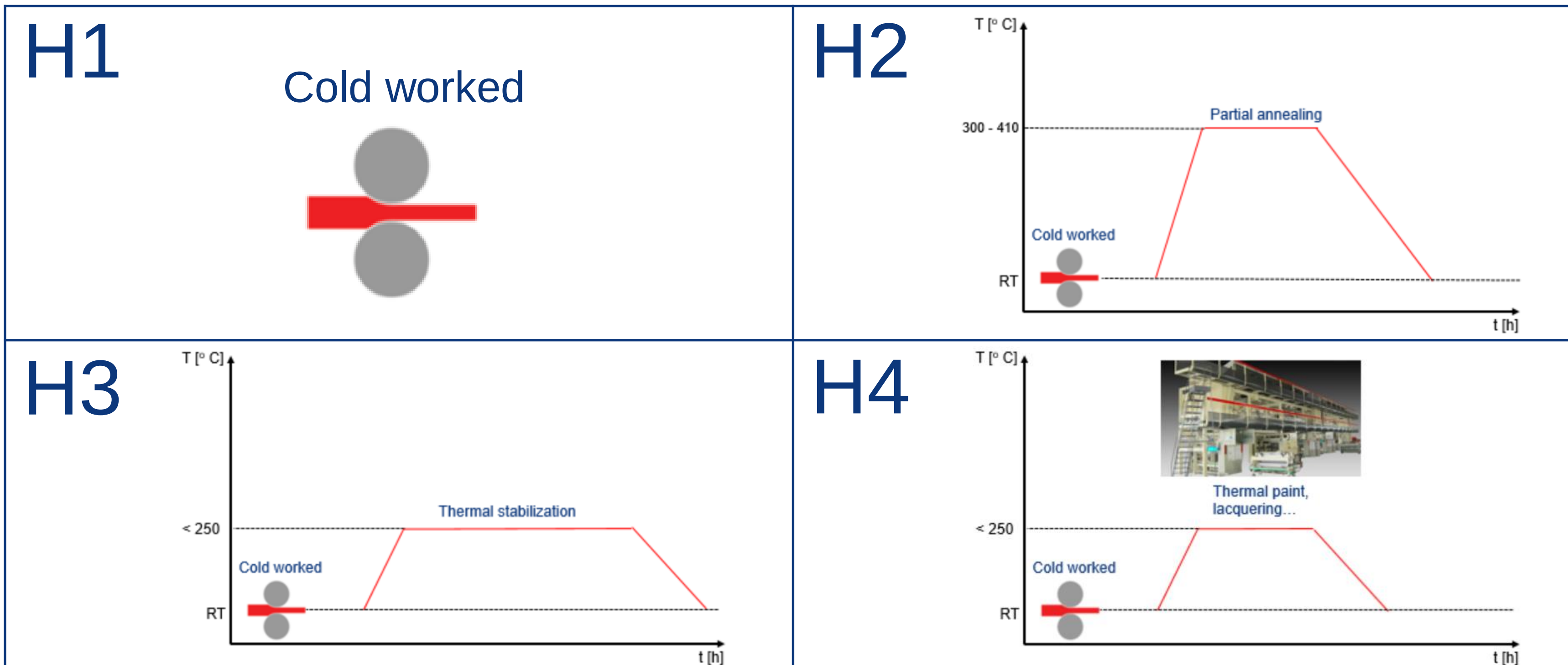
Dislocation bows out between incoherent precipitates

A loop is left around each particle

From R. E. Smallman and A. H. W. Ngan. Physical Metallurgy and Advanced Materials. Elsevier, 2007

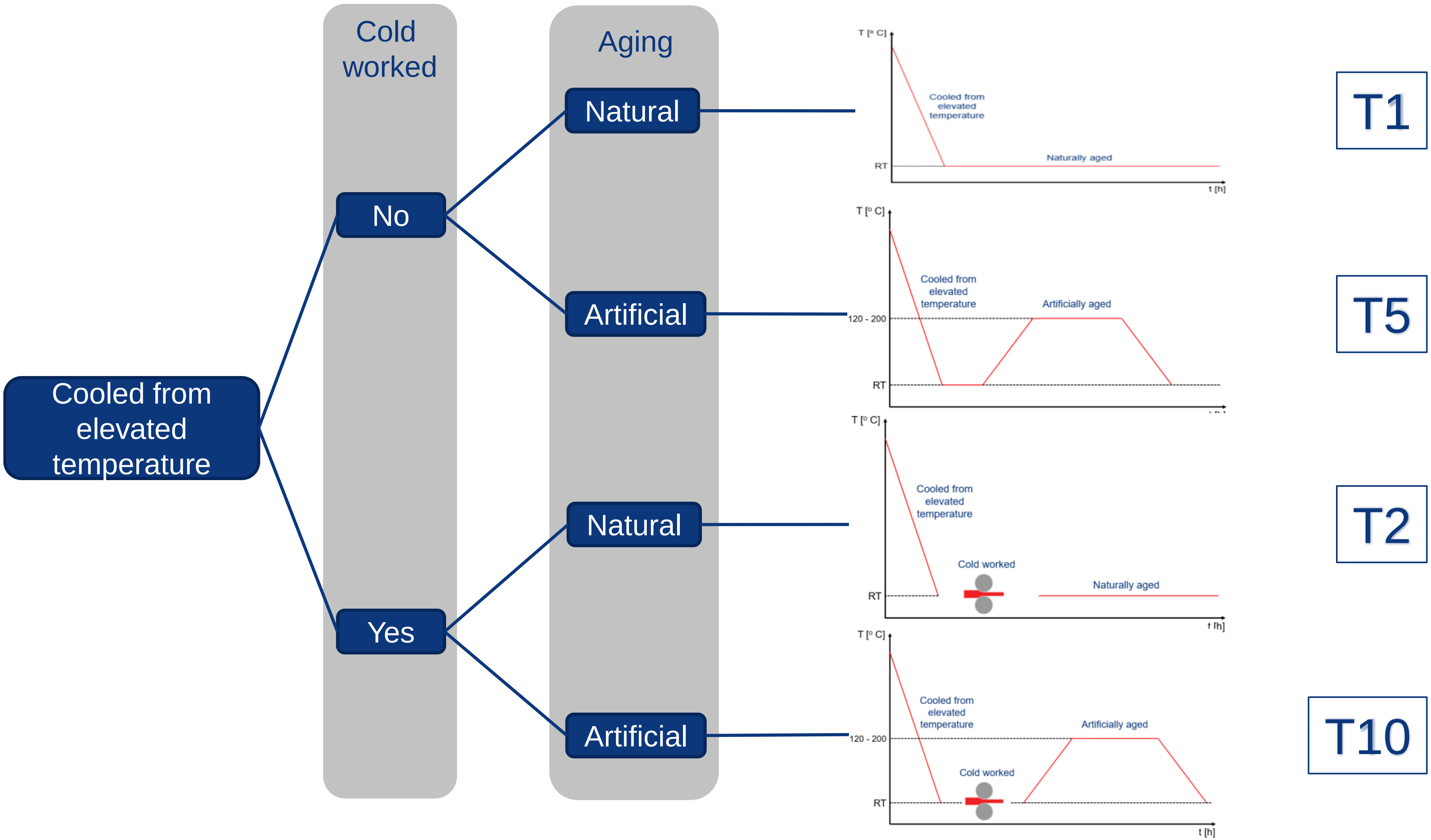
Wrought aluminum alloys: temper states

- **H, strain hardened:** non-heat-treatable wrought alloys that have had their strength increased by strain hardening. H is always followed by two or more digits:
  - The first number after the H tells whether the strain-hardened alloy has been thermally treated:



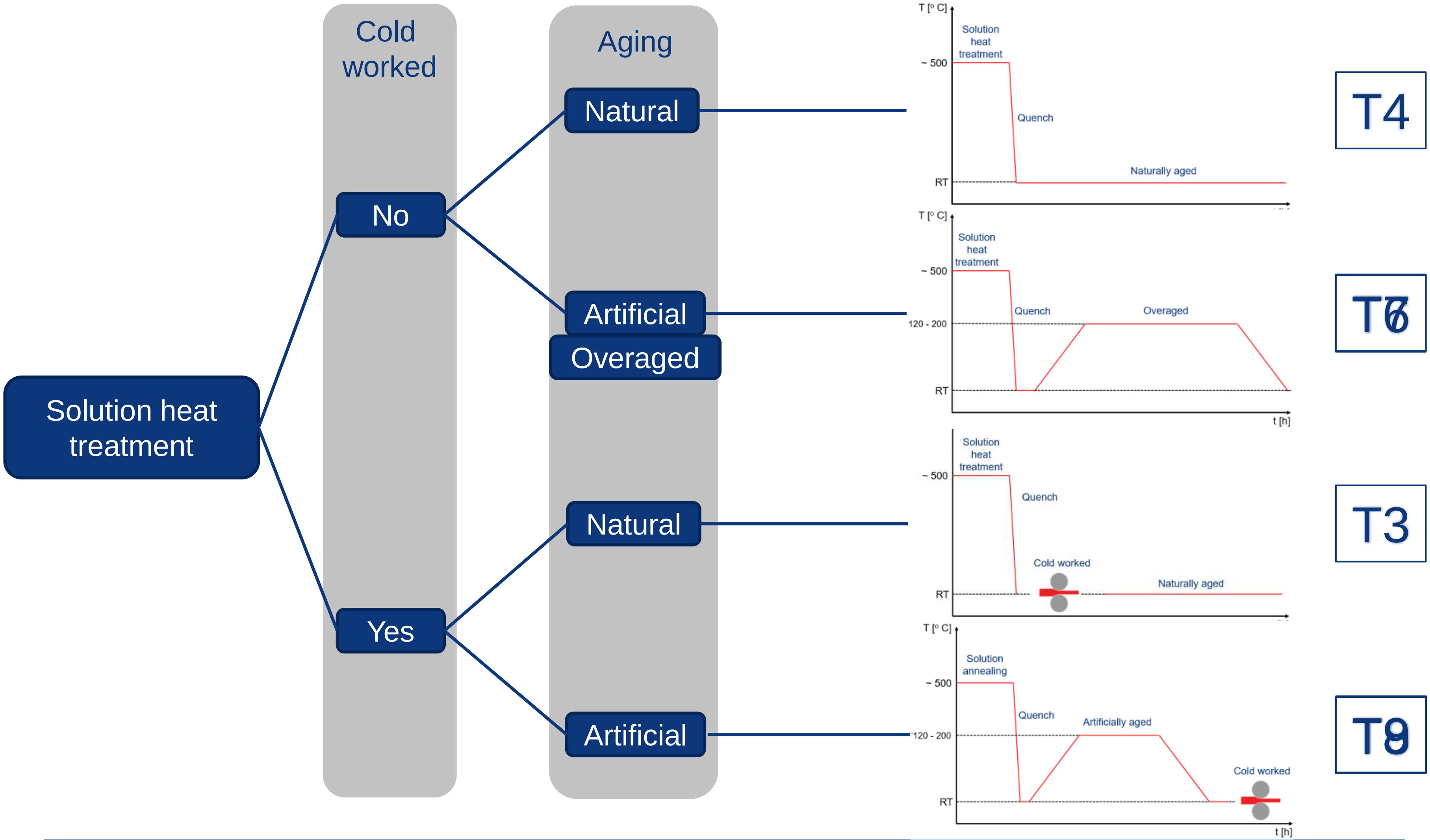
Wrought aluminum alloys: temper states

- T, thermally treated to produce stable tempers:



Wrought aluminum alloys: temper states

- T, thermally treated to produce stable tempers:



Prices

Aluminium and alloys

- Al and Al alloys general purpose → 5 EUR / Kg
- EN AW 2219 forged blanks → 80 EUR / Kg
- Special forgings, EN AW 6061 → 15 EUR / Kg

Coppers

- OFE Cu → 25 - 40 EUR / Kg (3D forged)
- OF Cu→ 10 EUR / Kg (basis)
- CuBe, high (low) Be → 40 - 90 EUR / Kg (strips)
- Glidcop → 55 EUR / Kg

Titanium

- Grade 2→ 50 EUR / Kg (plates)
- Ti6Al4V (ELI) → 50 (140) EUR / Kg

Niobium

- Nb (RRR 300) → 600 EUR / Kg