Materials for high vacuum applications

Stefano Sgobba

European Organization for Nuclear Research, Geneva, Switzerland

Outline

- Introduction to CERN and the accelerator complex
- Materials engineering at CERN: an integrated tool and know-how for a full life cycle management of materials
- Material selection for ultra-high vacuum and cryogenics, examples through CERN LHC and HL-LHC, ITER and fusion beyond ITER
- Case histories of failures and their elimination
  - LHC vacuum and cryogenic system
  - The ITER vacuum vessel thermal shields
- Lessons learned and conclusions

CERN's mission and the Large Hadron Collider (LHC)

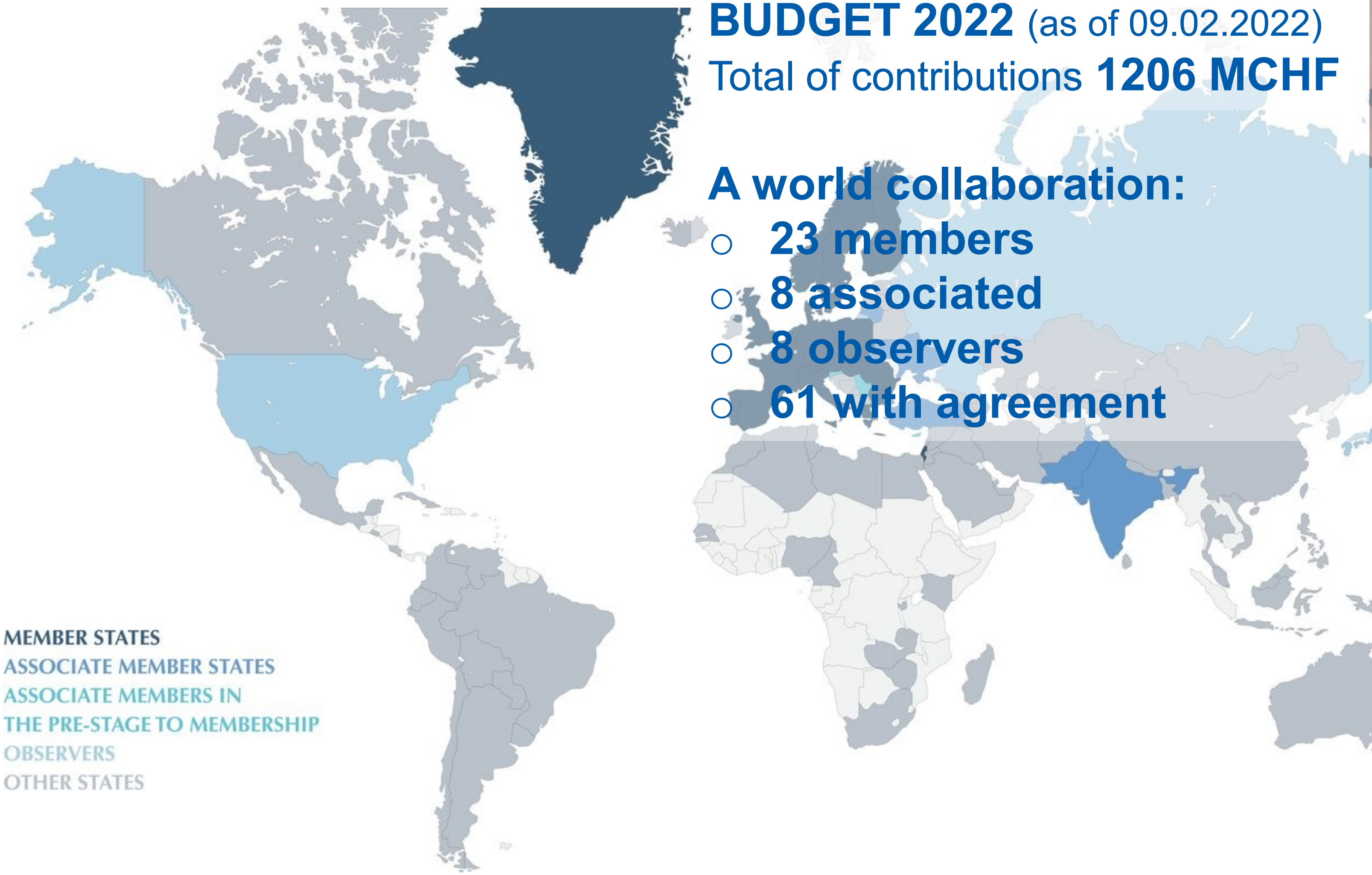
Member States of CERN

Member States (date of accession)

- Austria (1959)
- Belgium (1953)
- Bulgaria (1999)
- Czech Republic (1993)
- Denmark (1953)
- Finland (1991)
- France (1953)
- Germany (1953)
- Greece (1953)
- Hungary (1992)
- Israel (2014)
- Italy (1953)
- Netherlands (1953)
- Norway (1953)
- Poland (1991)
- Portugal (1986)
- Romania (2016)
- Slovakia (1993)
- Spain (1961-1968, 1983-)
- Sweden (1953)
- Switzerland (1953)
- United Kingdom (1953)

States in accession to Membership and Associate Members

- Cyprus (2016)
- India (2017)
- Lithuania (2018)
- Pakistan (2015)
- Serbia (2012)
- Slovenia (2017)
- Turkey (2015)
- Ukraine (2016)

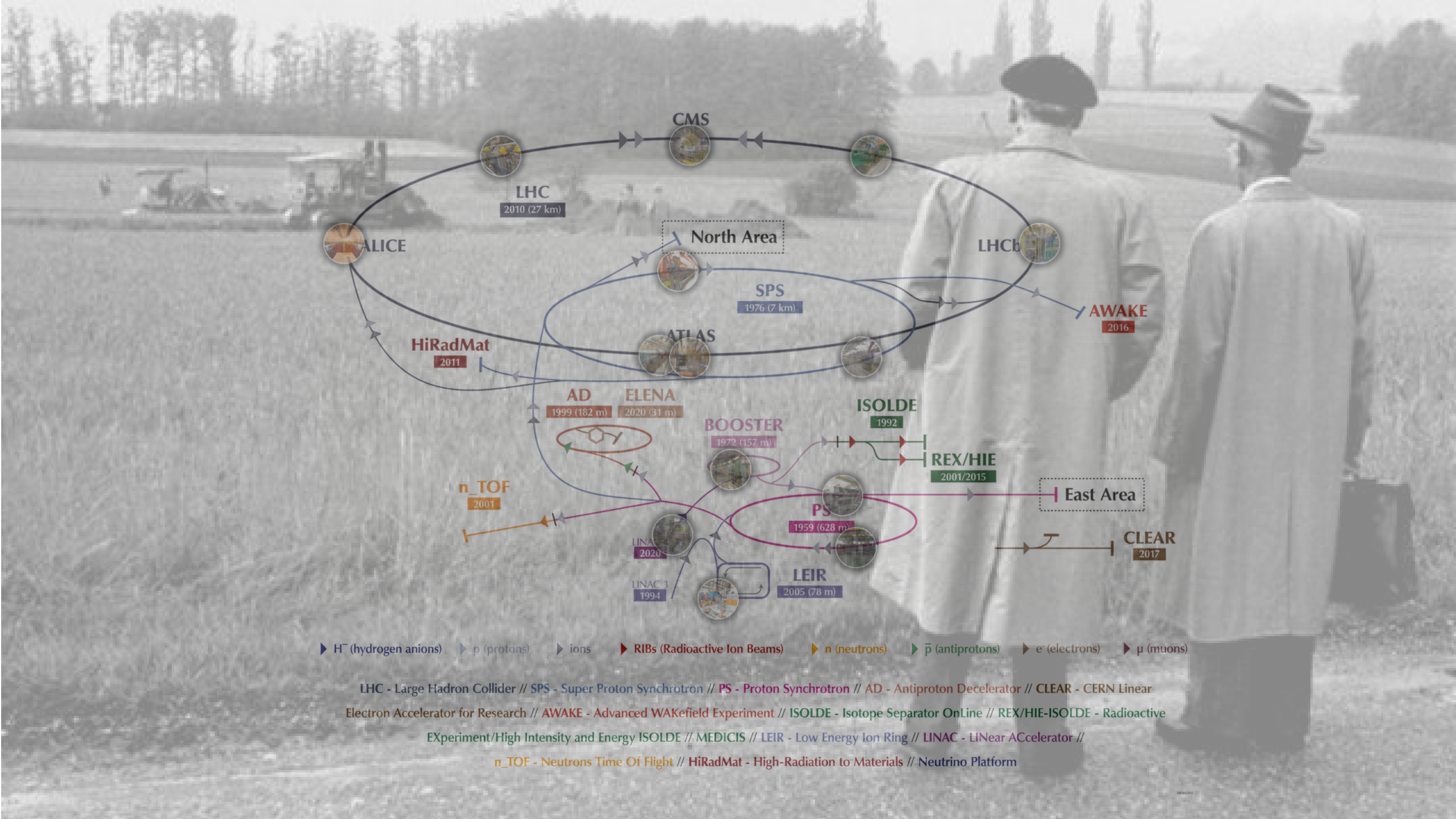


BUDGET 2022 (as of 09.02.2022)

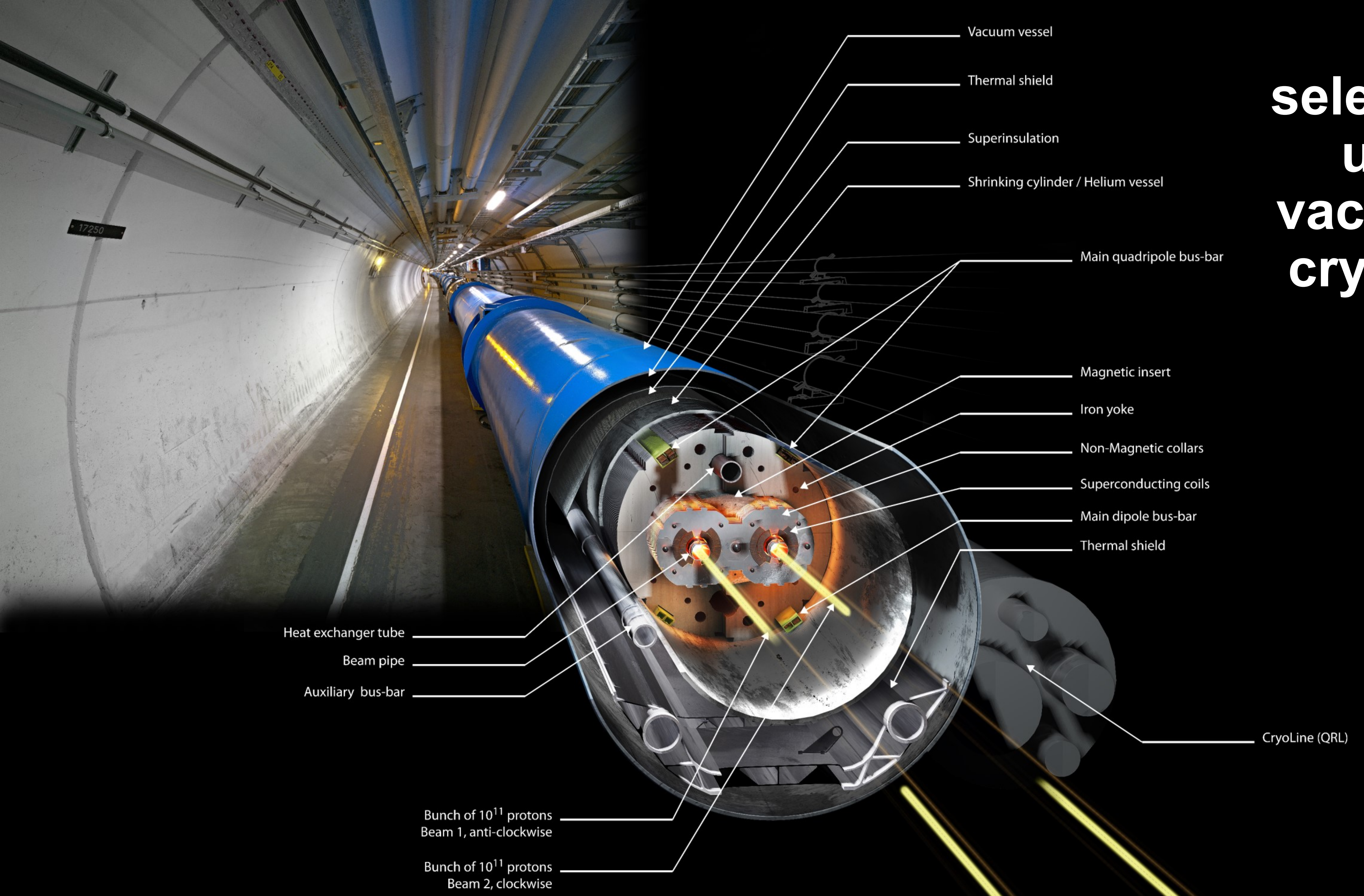
Total of contributions **1206 MCHF**

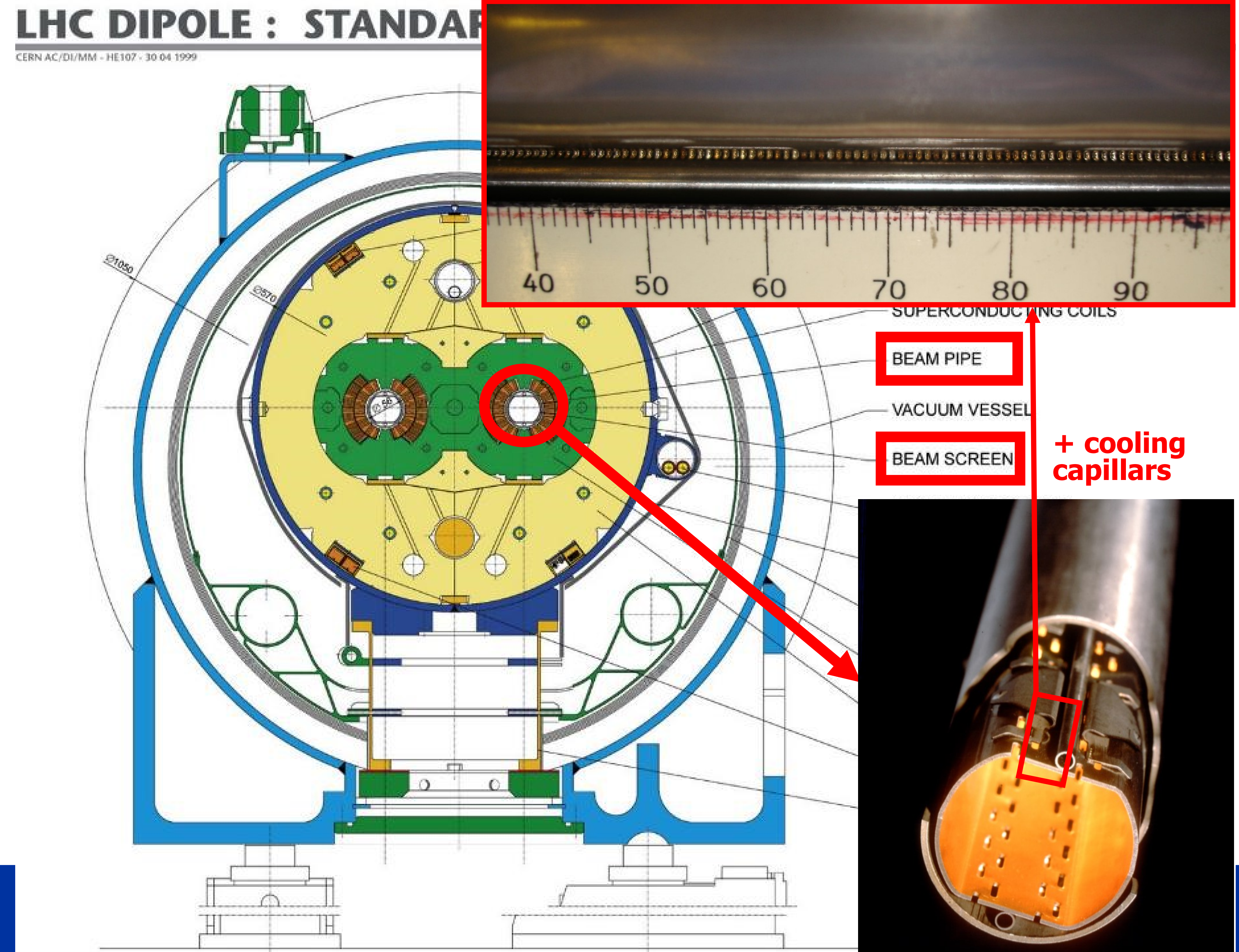
A world collaboration:

- **23 members**
- **8 associated**
- **8 observers**
- **61 with agreement**


CMS
LHC
2010 (27 km)
North Area
ALICE
LHCb
SPS
1976 (7 km)
AWAKE
2016
ATLAS
HiRadMat
2011
AD
1999 (182 m)
ELENA
2020 (31 m)
ISOLDE
1992
BOOSTER
1972 (157 m)
REX/HIE
2001/2015
n_TOF
2001
East Area
PS
1959 (628 m)
CLEAR
2017
LINAC 4
2020
LINAC 3
1994
LEIR
2005 (78 m)
H⁻ (hydrogen anions)
p (protons)
ions
RIBs (Radioactive Ion Beams)
n (neutrons)
p̄ (antiprotons)
e⁻ (electrons)
μ (muons)
LHC - Large Hadron Collider // SPS - Super Proton Synchrotron // PS - Proton Synchrotron // AD - Antiproton Decelerator // CLEAR - CERN Linear Electron Accelerator for Research // AWAKE - Advanced WAKefield Experiment // ISOLDE - Isotope Separator OnLine // REX/HIE-ISOLDE - Radioactive EXperiment/High Intensity and Energy ISOLDE // MEDICIS // LEIR - Low Energy Ion Ring // LINAC - LINear ACcelerator //
n_TOF - Neutrons Time Of Flight // HiRadMat - High-Radiation to Materials // Neutrino Platform

Material selection for ultra-high vacuum and cryogenics: the LHC example

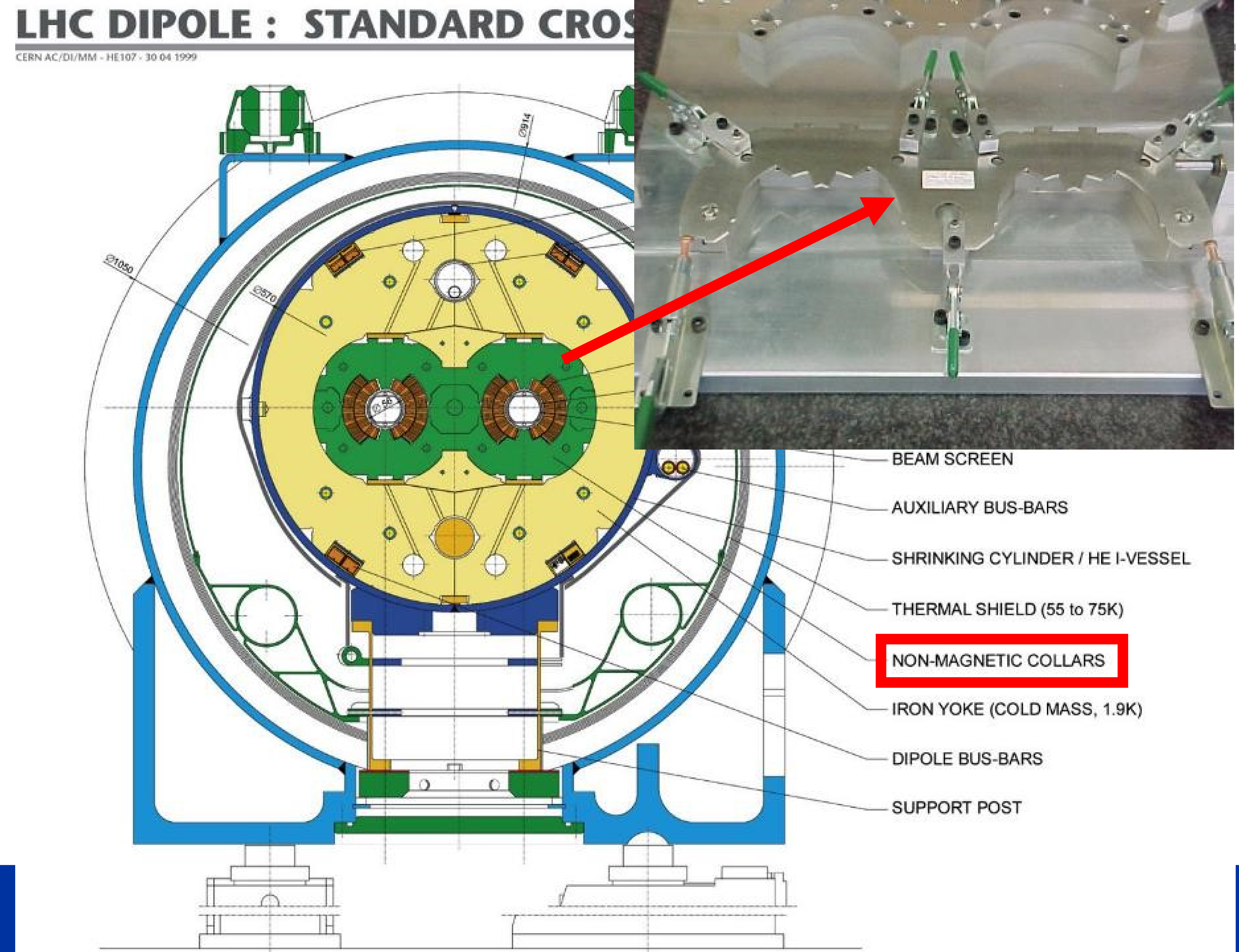




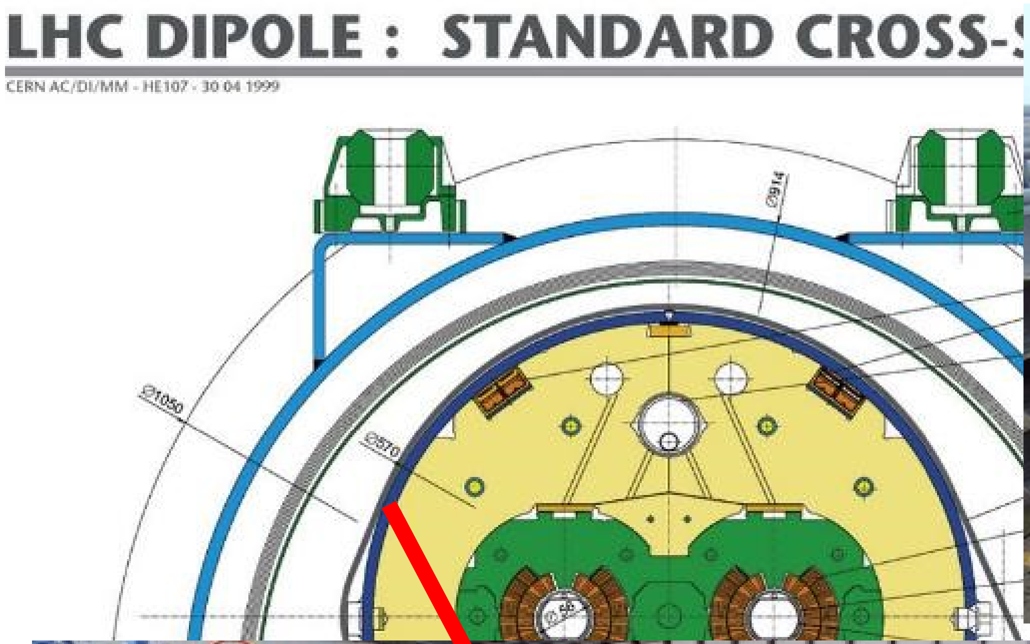
Material selection for ultra-high vacuum

S. Sgobba, G. Hochoertler, A new non-magnetic stainless steel for very low temperature applications, Proc. Int. Congress Stainless Steel '99 : Science and Market, pp.391-401

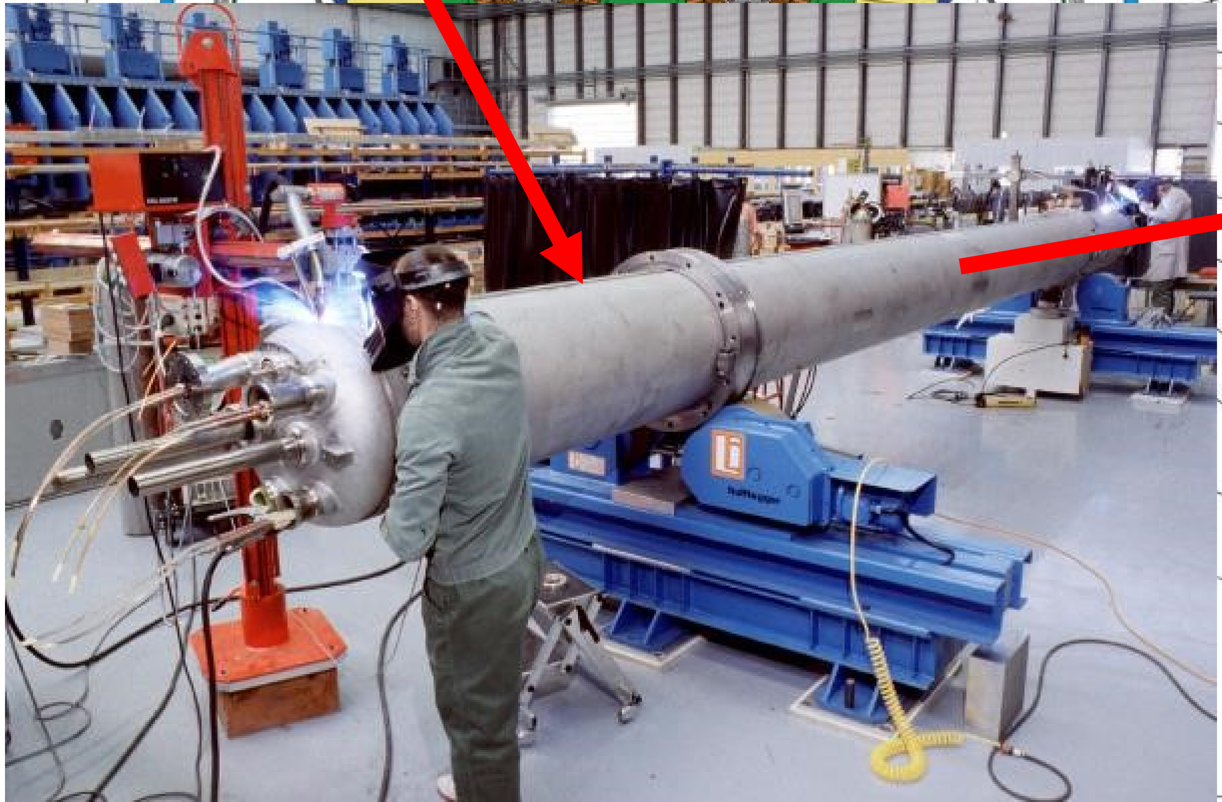
Material selection for ultra-high vacuum



Material selection for ultra-high vacuum



Courtesy Dr. N. Pauze, ArcelorMittal Industeel



- 3000 tonnes of 1.4429 plates delivered by ArcelorMittal Industeel /FR
- 41 km of half-shells, Ø 550 mm, 15 m length, 10.1 mm thick produced by Butting /DE

After capsule removal by pickling and heat treatment, before machining

PM Design Competition Award Winner

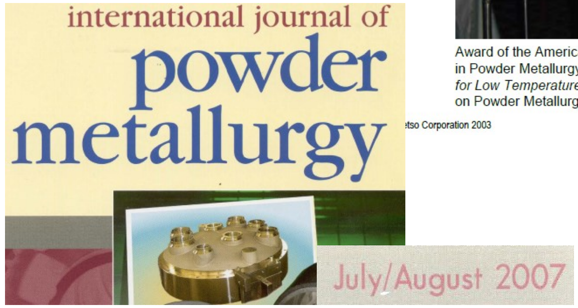
Grand Prize, reception at the PM Part Competition at PowderMet 2007, The International Conference on Powder Metallurgy and Particulate Materials, Denver-CO, May 13-16, 2007

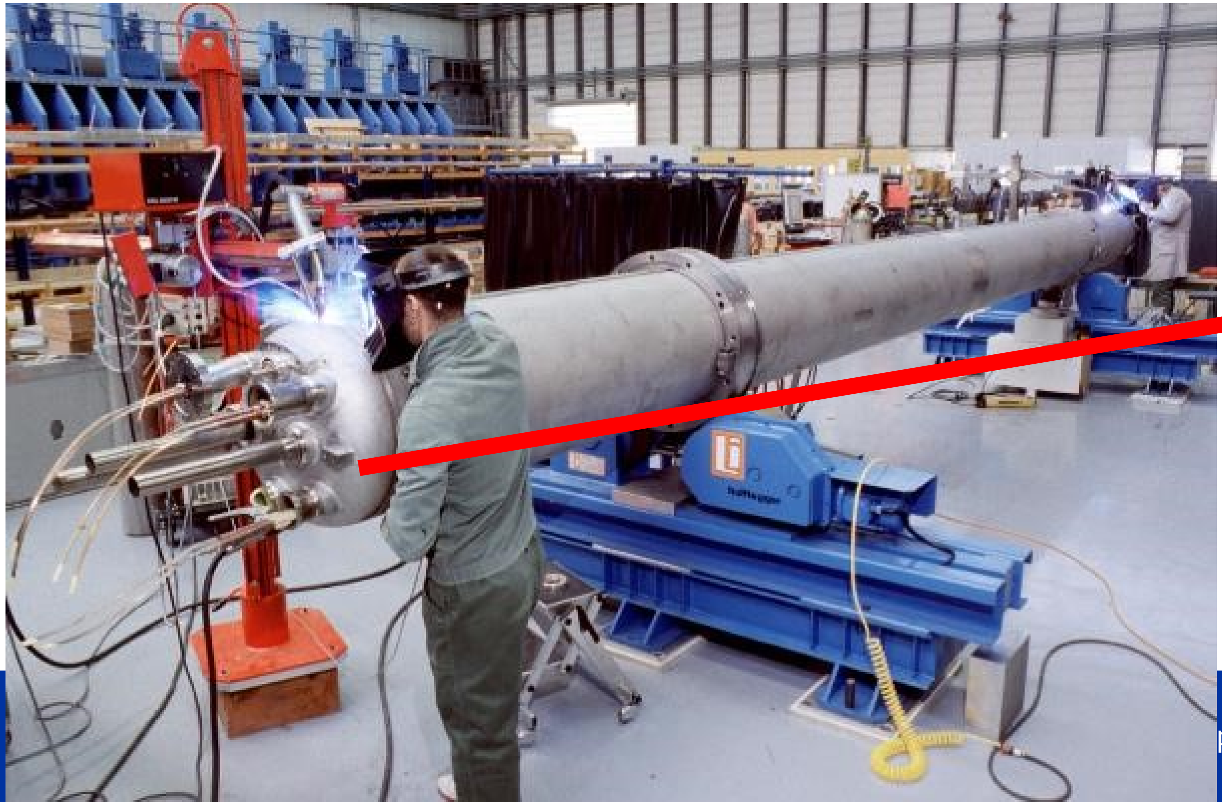
Award: Grand Prize
Year: 2007

Award of the American Society for Metals (ASM), 30/01/2004

Award of the American Society for Metals (ASM International, Finland) for Outstanding Achievements in Powder Metallurgy, to the paper "*Powder HIP End Covers for CERN LHC Project, High Technology for Low Temperatures*", 6th Annual Powder Metallurgy Network Seminar, Engineering Solutions Based on Powder Metallurgy, Tampere 29th - 30th January, 2004

metso





SHRINKING CYLINDER HE I-VESSEL

THERMAL SH... **end covers**

NON-MAGNETIC COLLARS

IRON YOKE (COLD MASS, 1.9K)

DIPOLE BUS-BARS

SUPPORT POST

- Leak tight to gazeous He at 300 K
- Leak tight to superfluid He at 1.9 K
- 25 thermal cycles down to 1.9 K

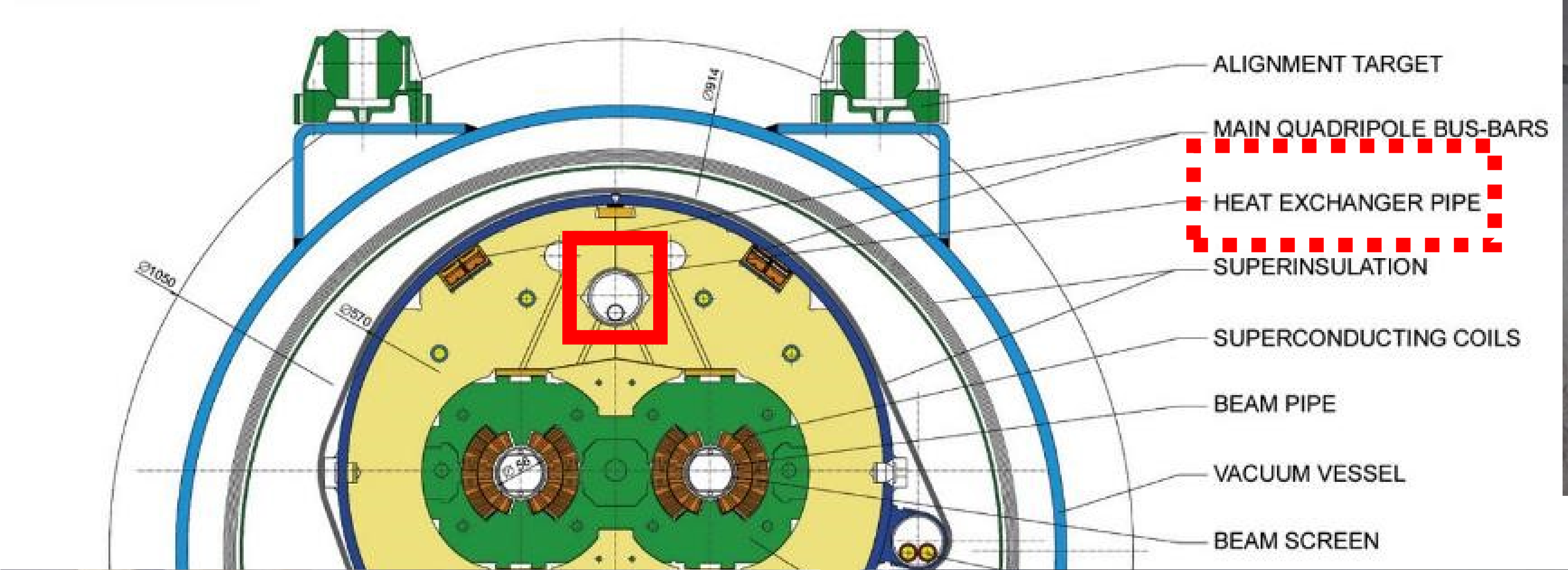
- **High ductility and toughness at low T**
- **Compatible with its environment (316LN)**
- **Cost effective**

S. Sgobba et al., *A Powder Metallurgy Austenitic Stainless Steel for Application at Very Low Temperatures*: Proc. of the 2000 Powder Metallurgy World Congress, Nov. 12-16, 2000, Kyoto, Japan, vol. 2, p. 1002-1005

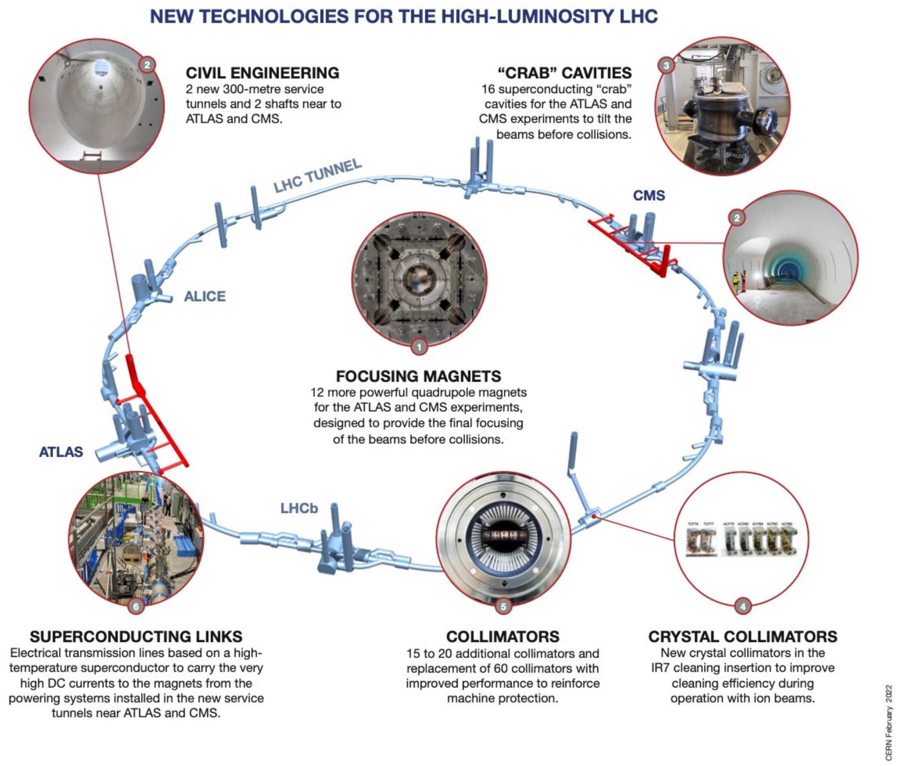
pplica

LHC DIPOLE : STANDARD CROSS-SECTION

CERN AC/DI/MM - HE107 - 30 04 1999



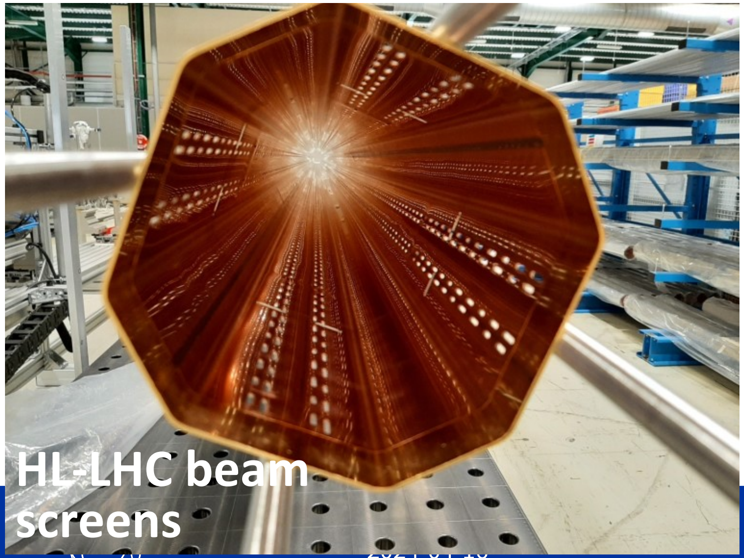
Material selection for ultra-high vacuum



Fine-blanked collars

F712 contract

- More than 450 tonnes of austenitic stainless steel strips
- Same stainless steel specification as for the LHC



Beam screen

HL-LHC implies:

- 3.1 km of finished strip;
- 4600 m of seamless cold-drawn cooling tubes in lengths of up to 14 m
- Same stainless steel as for the LHC

IT-4203/TE/**HL-LHC**

3.1.1 Special austenitic grade stainless steel strip (CERN supply)

The chemical composition of the CERN supplied stainless steel strip is given in table 2.

Table 2 - Typical chemical composition (weight-%) of the CERN supplied stainless steel strip.

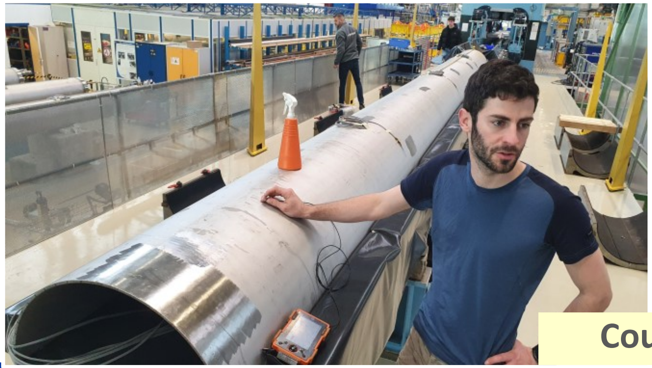
%	C	Cr	Mo	Ni	Mn	Si	N	Cu	S	P	B	Co
Min		19.0	0.8	10.7	11.8		0.30					
Max	0.03	19.5	1.0	11.3	12.4	0.5	0.33	0.15	0.002	0.02	0.002	0.1

Material selection…

HL-LHC further 316LN plate production and forming (2015-16)

- Supplied by Arcelor Mittal Industeel
- 15 mm x 6.5 m (11T project)
- 8 mm x 6.5 m and 10 m for WP3 (Q2, CP et D2)

Courtesy AP TELA OY /FI



Courtesy H. Prin /CERN-TE

Shrinking and inertia cylinders

Case histories of failures and their elimination
LHC vacuum and cryogenic system

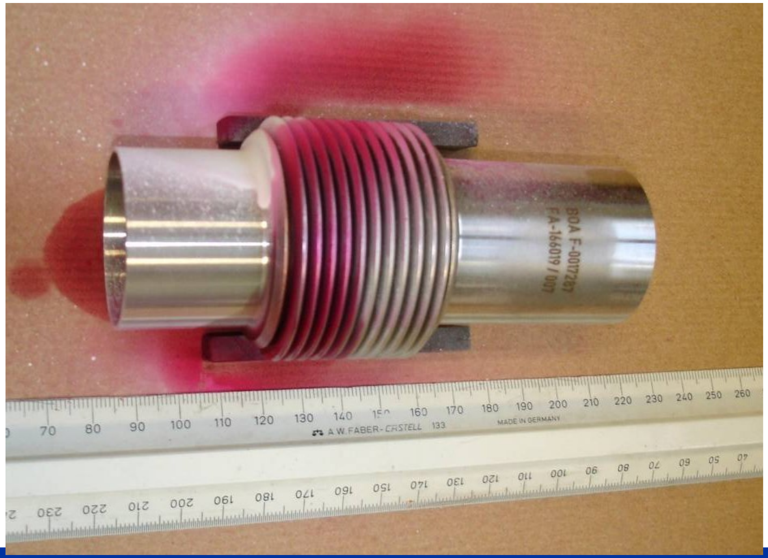
CERN — European Organization for Nuclear Research

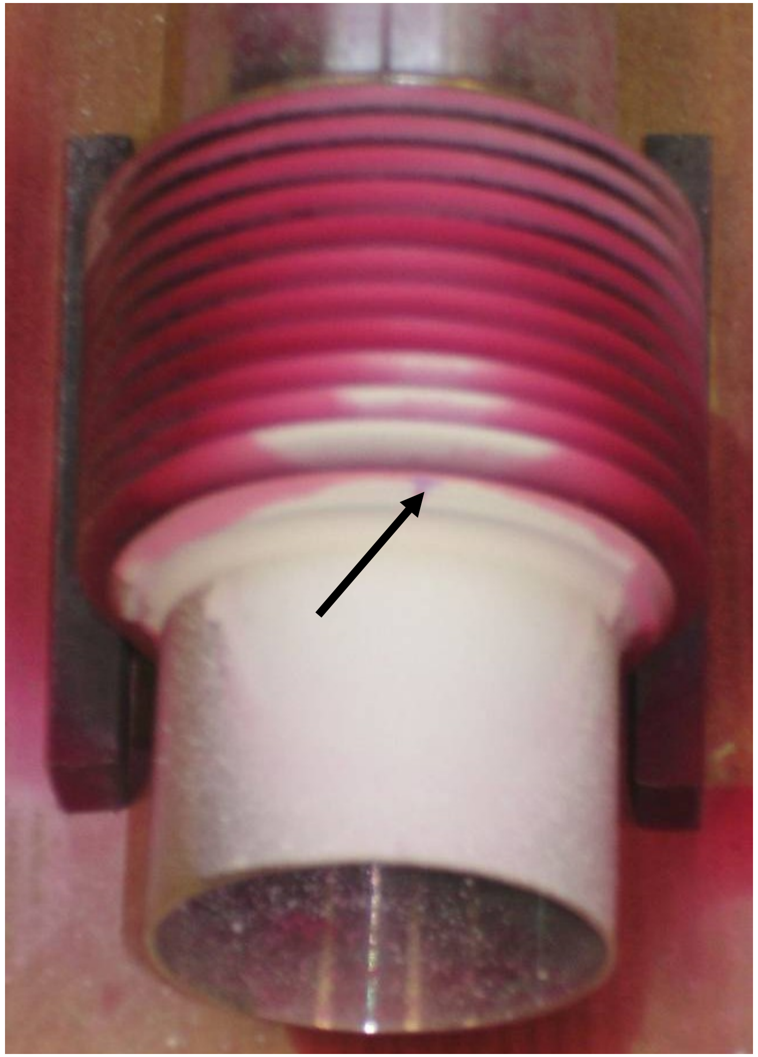
TS/MME-MM
Section de Métallurgie et Métrologie/ *Metallurgy and Metrology section*
Rapport expérimental / Investigation report

TECHNICAL SUPPORT DEPARTMENT

Domaine / Field: *CMS (Ion pump)*	*Date: 10/03/2006*	*N° EDMS / EDMS Nr.: 710706*
Requérant / Customer: *P. Lepeule AT/VAC*	*Liste de distribution / Distribution list:* *G. Faber PH/UCM; A. Hervé PH/CMO; R. Veness AT/VAC* *C. Saint-JAL FI/LS*	

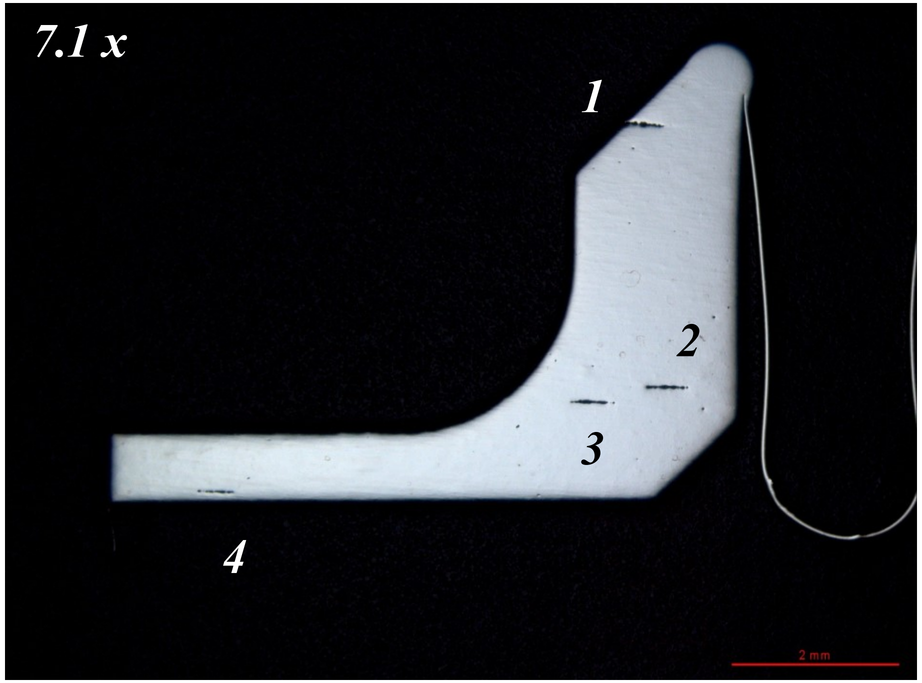
Metallographic observations of 316LN leaking bellow

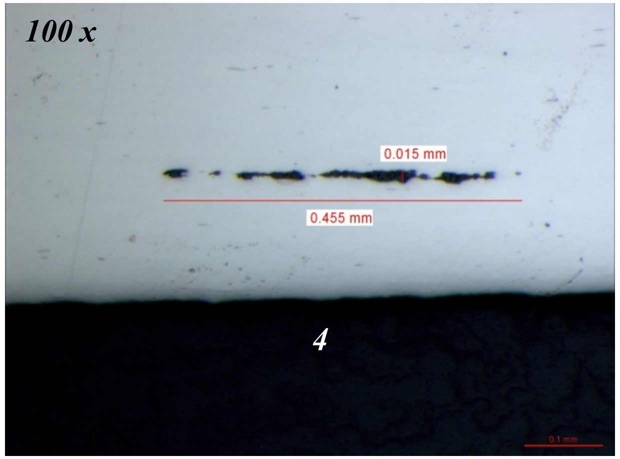


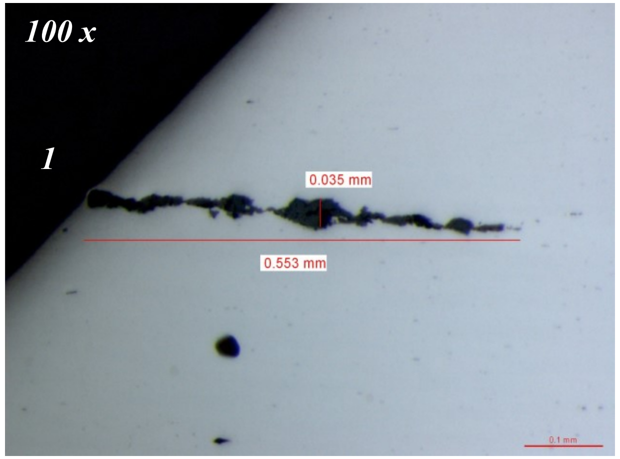


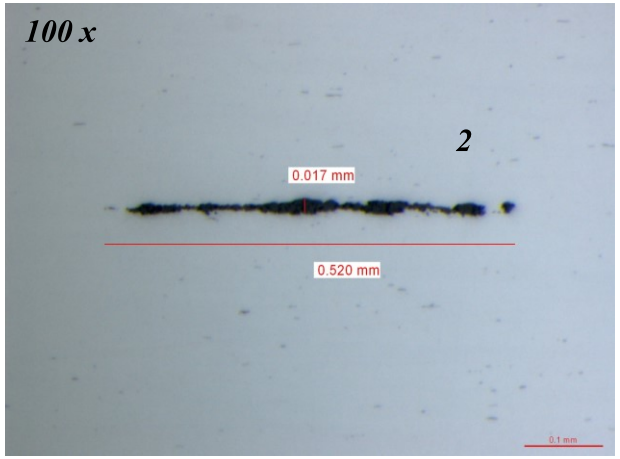
Case histories of failures and their elimination LHC vacuum and cryogenic system

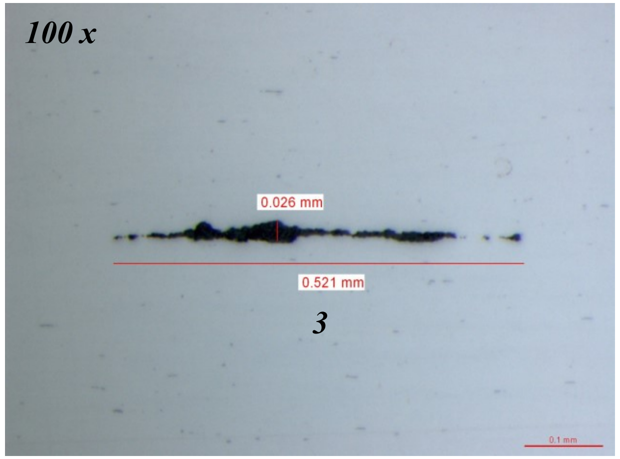
- Oversized (1,2,3) and thick (4) B type inclusions up to class 2.

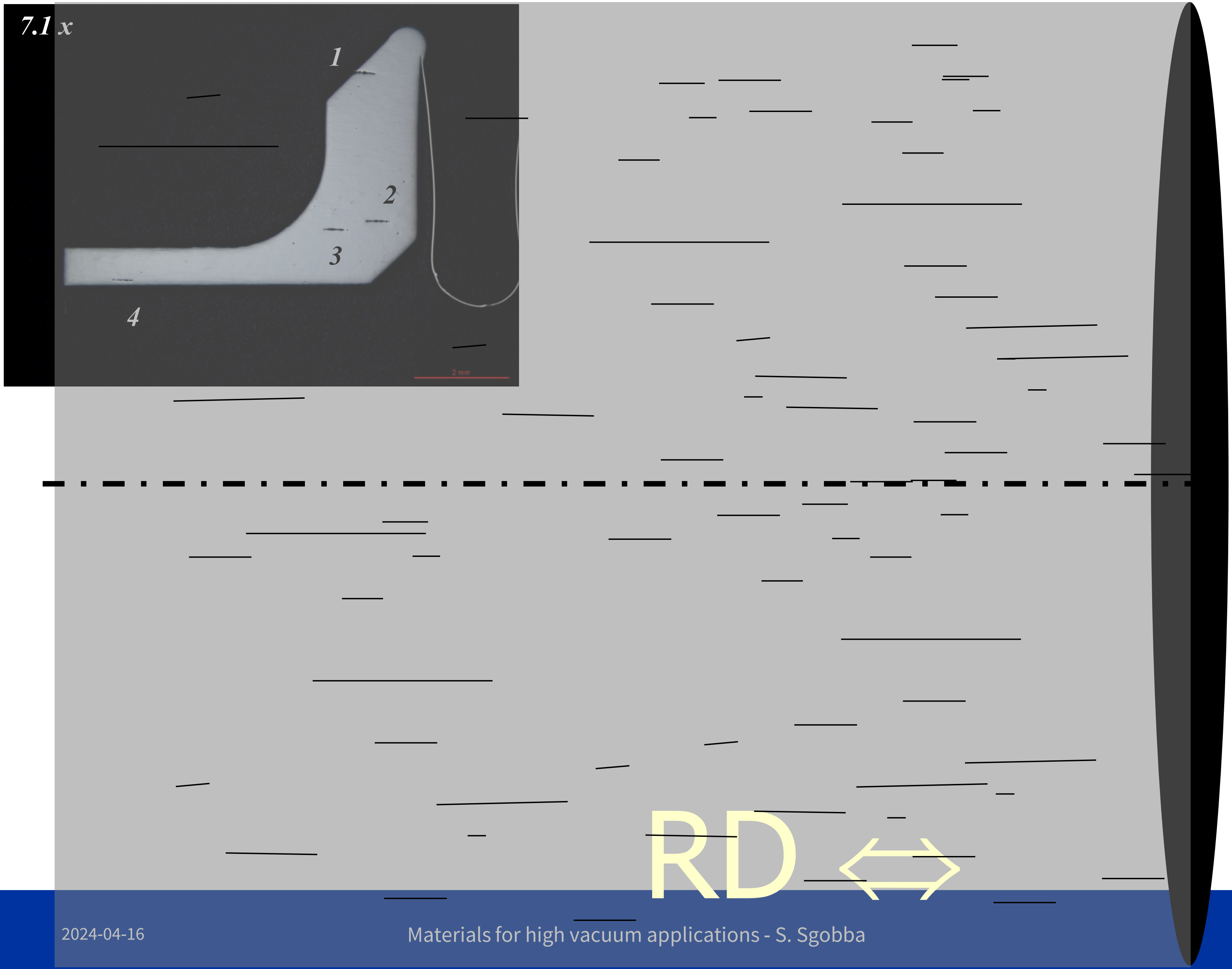






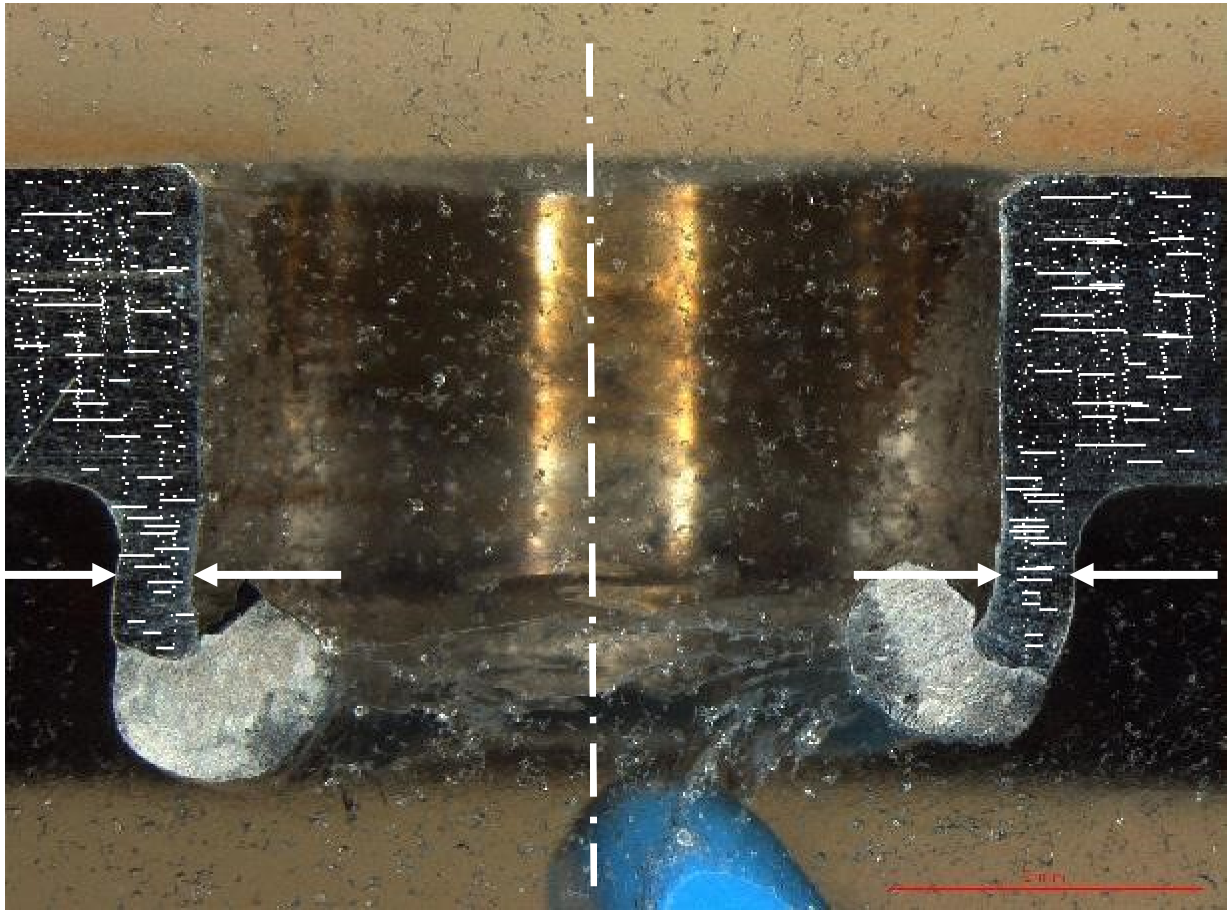






Case histories of failures …

Case histories of failures…



For any wrought product (plate, tube, bar), an unfavourable inclusions alignment will be anyway present in the rolling or drawing direction

Case histories of failures…

Designation: E45 – 13

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

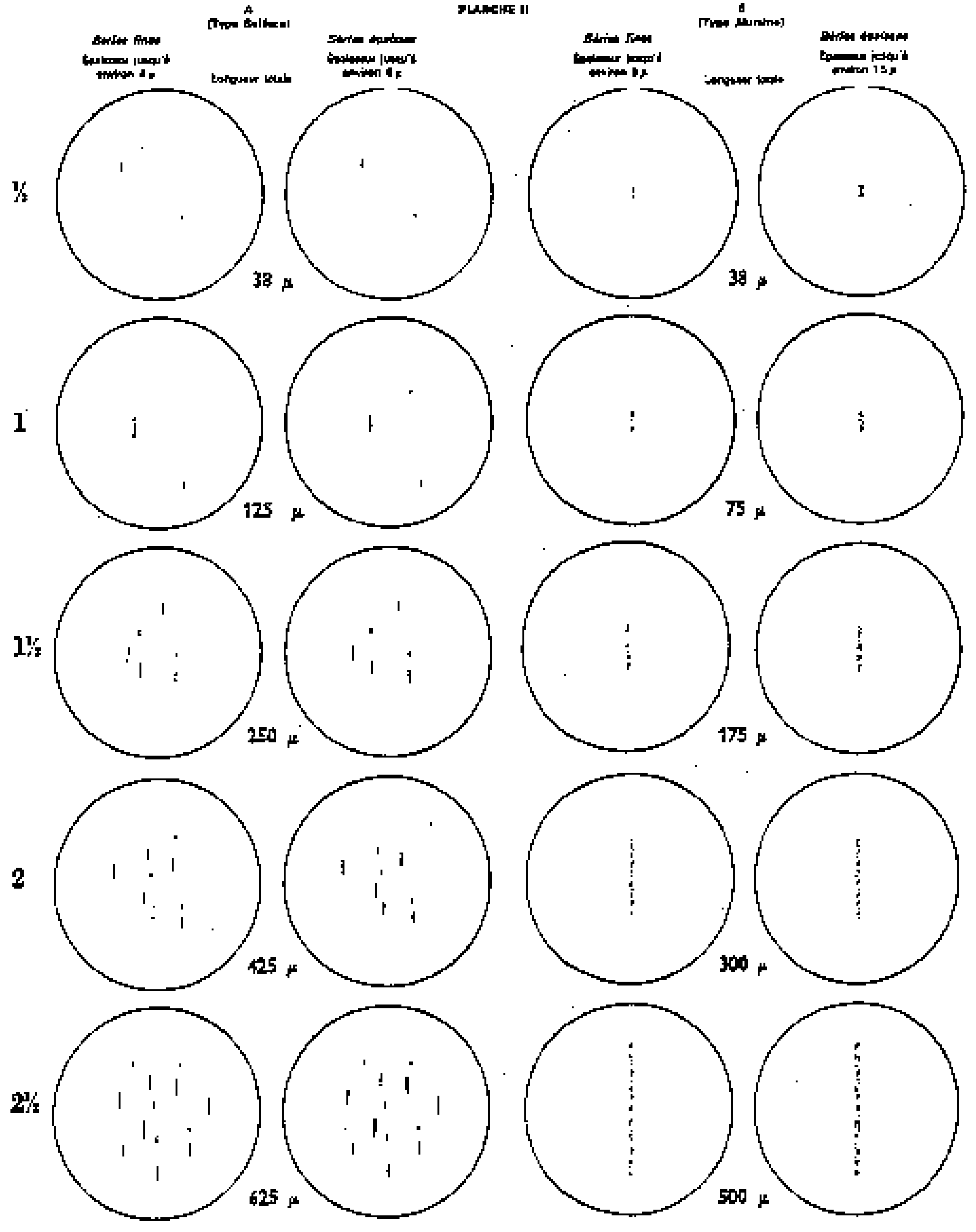
This standard is issued under the fixed designation E45; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates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

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

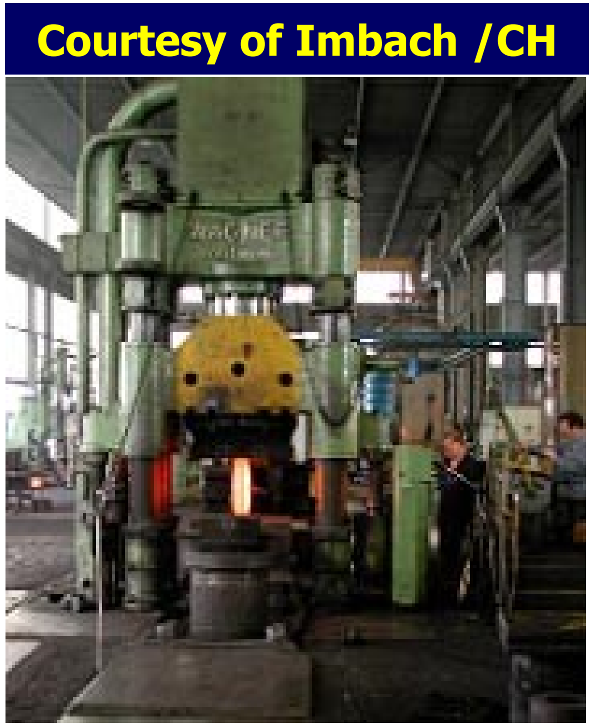
TABLE 1 Minimum Values for Severity Level Numbers (Methods A, D, and E)[A,B]

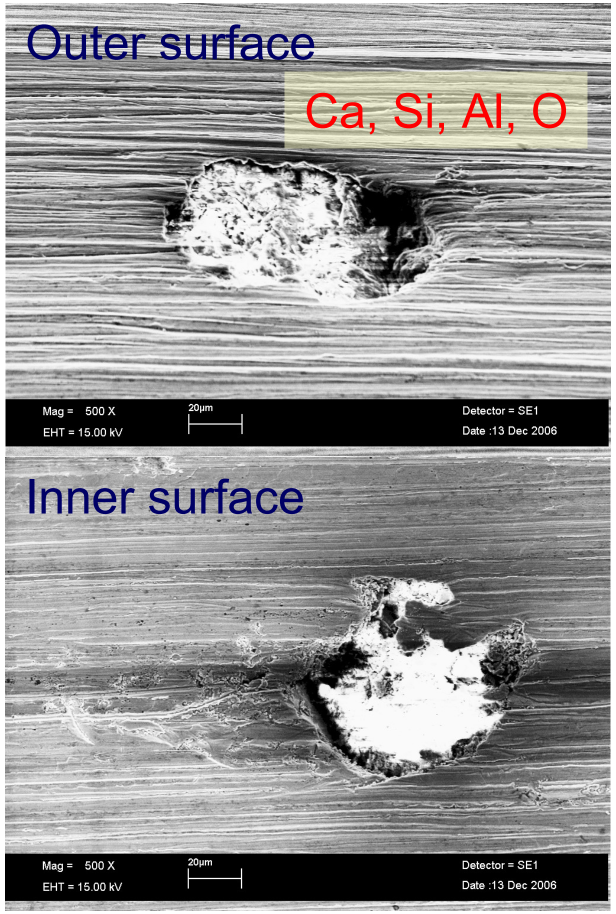
Severity	A	B	C	D[C]
(mm (in.) at 100×, or count)				
0.5	3.7(0.15)	1.7(0.07)	1.8(0.07)	1
1.0	12.7(0.50)	7.7(0.30)	7.6(0.30)	4
1.5	26.1(1.03)	18.4(0.72)	17.6(0.69)	9
2.0	43.6(1.72)	34.3(1.35)	32.0(1.26)	16
2.5	64.9(2.56)	55.5(2.19)	51.0(2.01)	25
3.0	89.8(3.54)	82.2(3.24)	74.6(2.94)	36
3.5	118.1(4.65)	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

QUANTIMÉTRIE



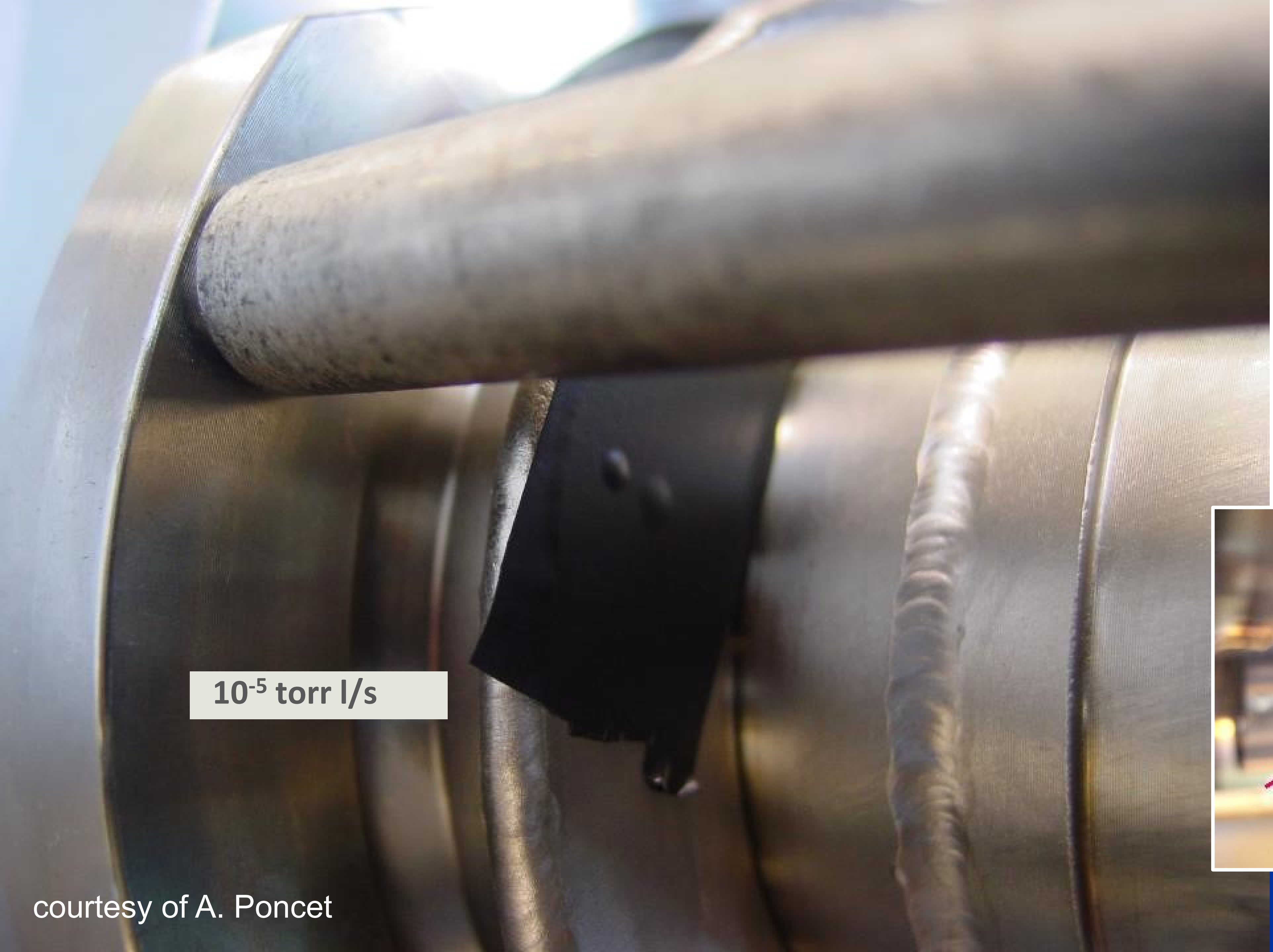
M 220 – 16



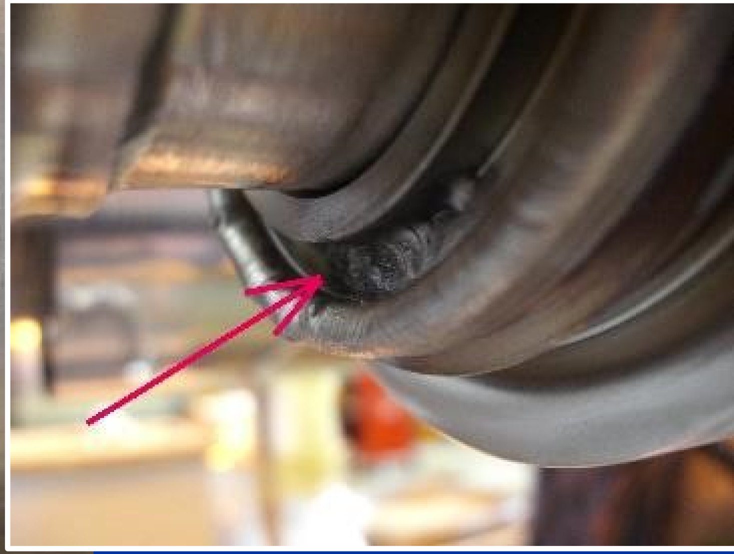


Multidirectional forging alone, even if including upsetting is not enough to avoid the risk of leaks due to macroinclusions

3. Development, requirements and application of grades for accelerator magnets



courtesy of A. Poncet



Case histories of failures…

CERN - CH1211 Geneva 23 - Switzerland

EDMS No.: 790775

2. REQUIREMENTS

2.1. MANUFACTURING PROCESS

The stringent requirements of this material specification for products intended for UHV purposes, impose to apply an adapted metallurgy and manufacturing process, aimed at meeting the structure and inclusion limits specified in this document. The process shall include a mandatory ElectroSlag Remelting (ESR) step.

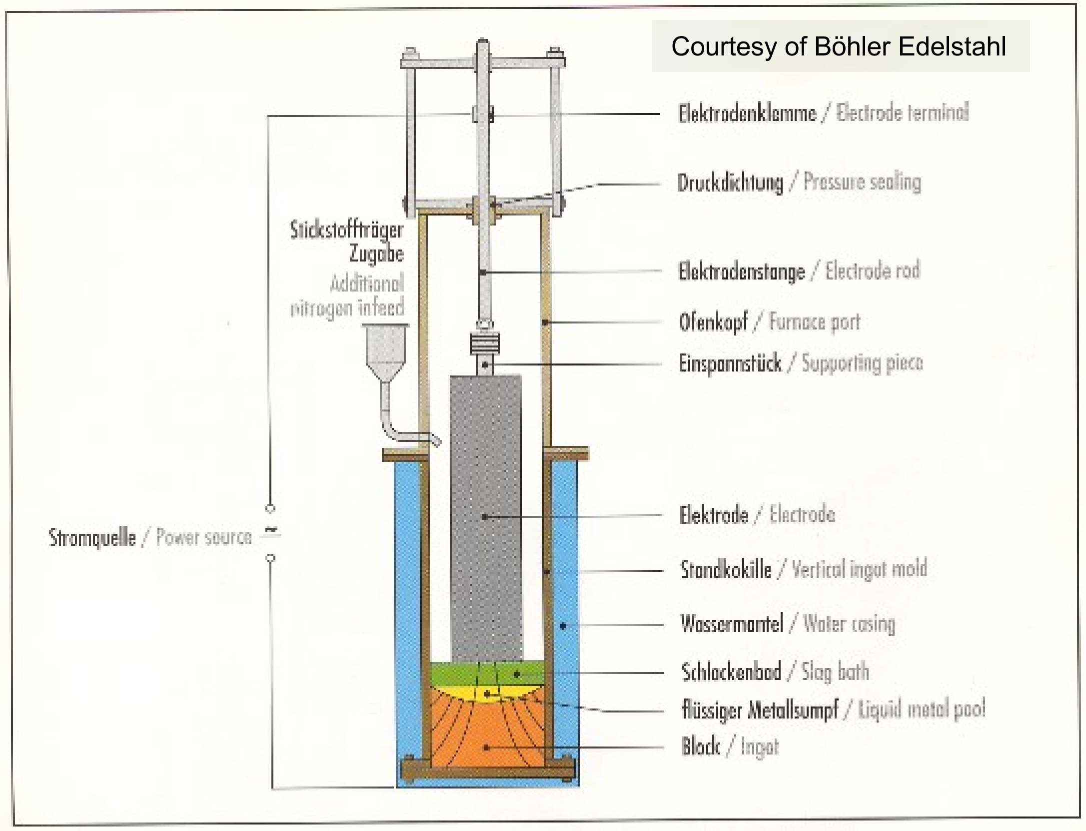
The blanks shall be multi-directionally forged.

Spec. N°1001_1.4429_316LN_blanks

This document specifies the CERN technical requirements for 1.4429 (X2CrNiMoN17-13-3, AISI 316LN) stainless steel blanks for ultra-high vacuum applications (UHV) at CERN requiring vacuum firing at 950°C.

Risk mitigation, enforcing material purity…

Courtesy of Böhler Edelstahl



erial

melting

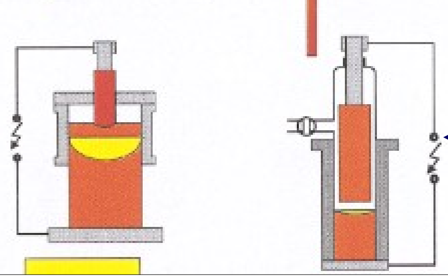
deslagging

secondary-metallurgy

casting

ingot casting

continuous casting



ESR & VAR

A. Choudhury: Vacuum Metallurgy, ASM Int., USA, (1990)

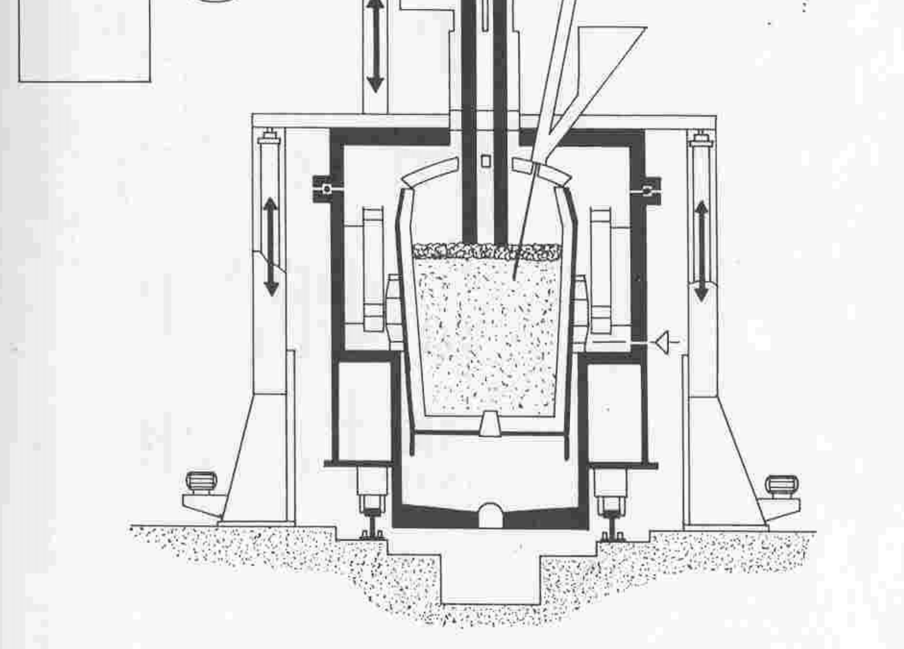


Fig. 23 Layout of a compact ladle furnace

Courtesy of Forgiatura Vienna /IT
Max. ingot weight/capacity: 250 t
Two furnace heads, electrode exchange, protective gas hood, fully coaxial design;
largest ESR plant worldwide in operation

The additional cost of ESR ingots is in the order of 1 EUR/kg (Minutes of the visit to Company A on 27 January 2015, ITER CS Lower Keyblock Material Progress Meeting)

Courtesy of Breitenfeld Edelstahl /AT.
Electrodes of diam. 500 mm, 750 mm, 1000 mm, 1200 mm, respectively, up to a length of 4 m and a weight of 35 t.
Annual capacity is 250 000 t.

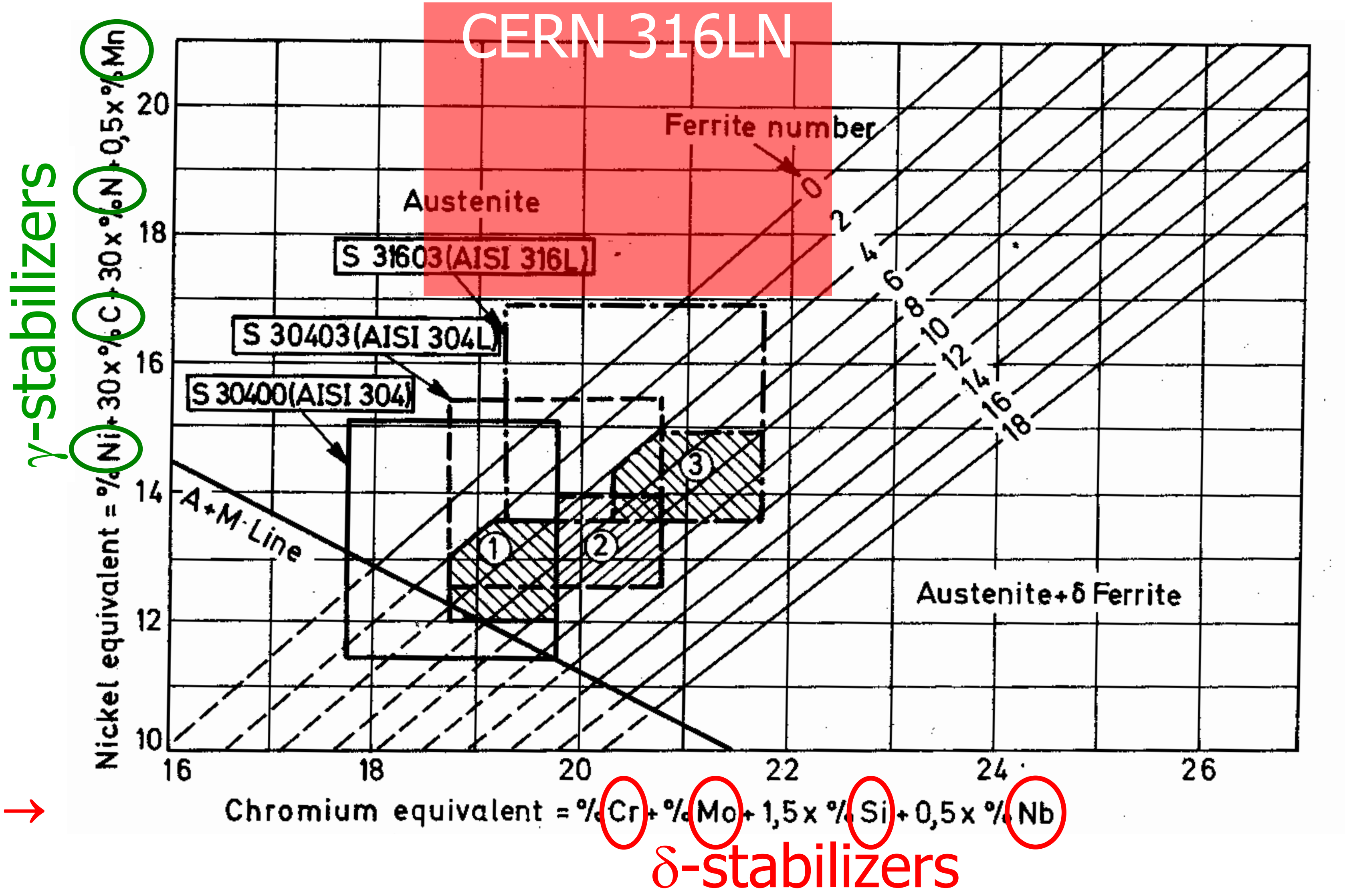
Case histories of failures: welds

CERN 316LN

Element	Chemical composition (product analysis) % by mass
Cr	16.00 – 18.50*
Ni	12.00 – 14.00*
C	0.030 max.
Si	1.00 max.
Mn	2.00 max.
Mo	2.00 – 3.00*
N	0.14 – 0.20*
P	0.030 max.*
S	0.010 max.*
Fe	Remainder

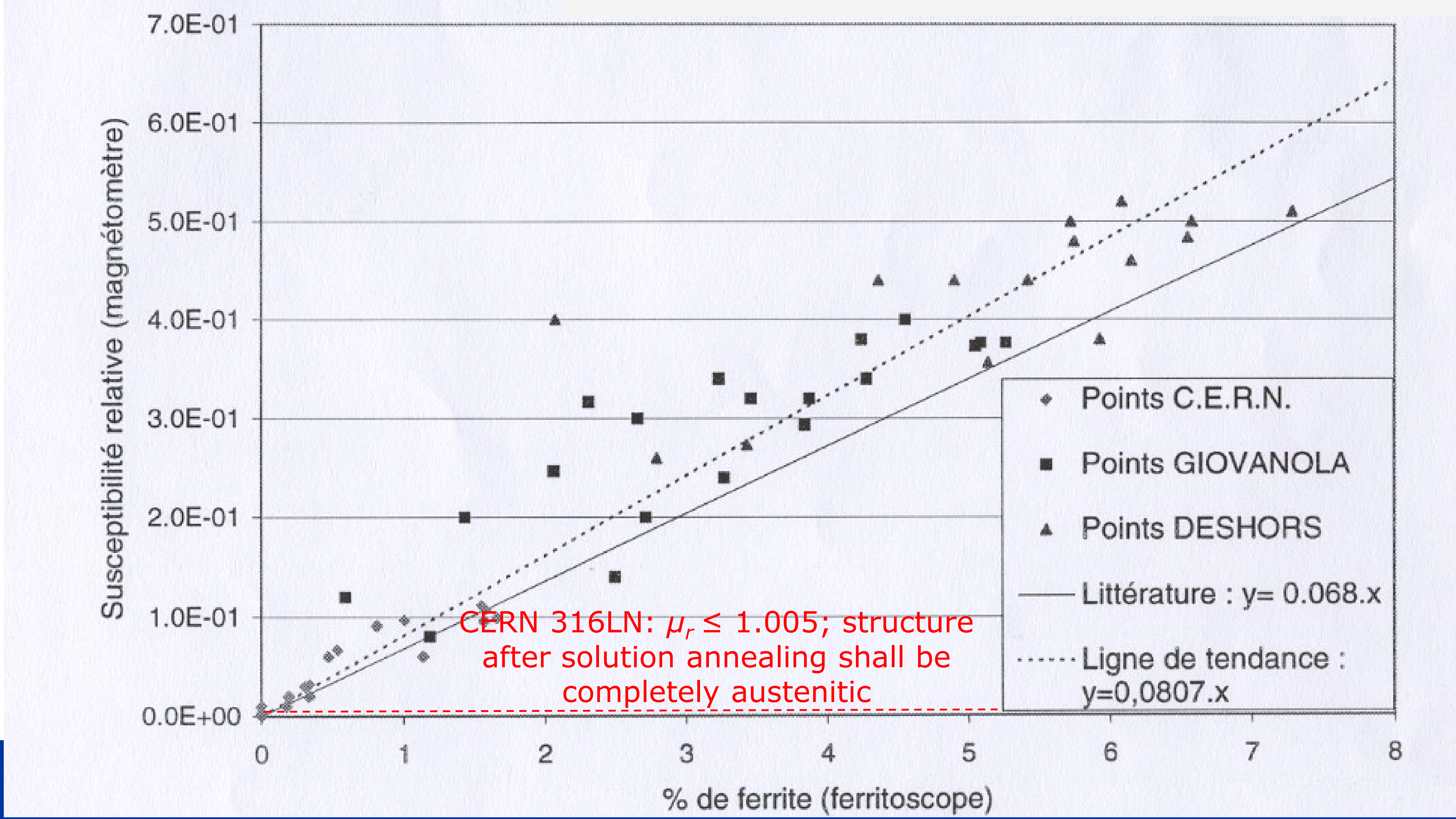
* CERN requirement

Maximum allowed magnetic permeability μ_r = 1.005 at RT → allowed content of δ-ferrite is nil

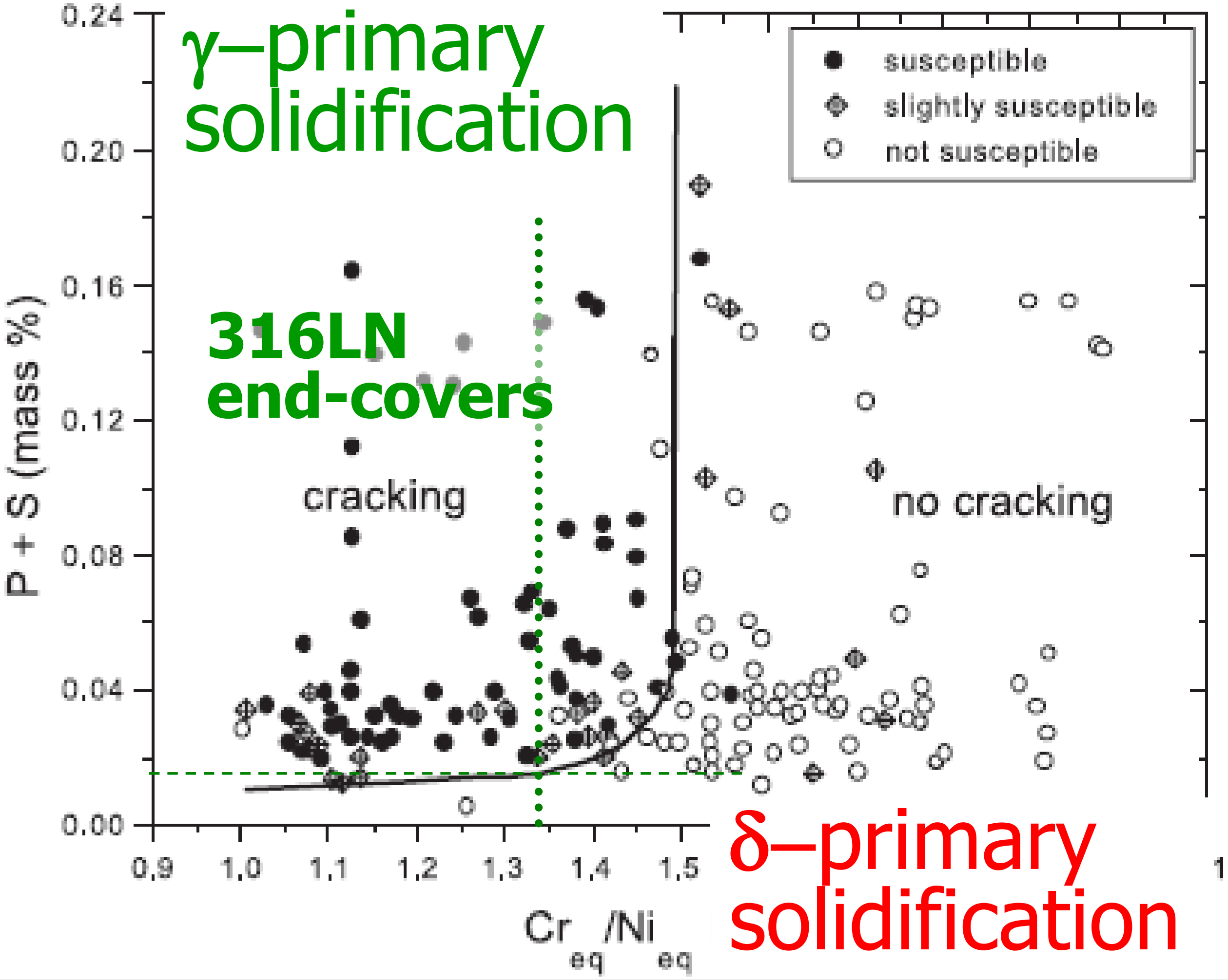


Case histories of failures: welds

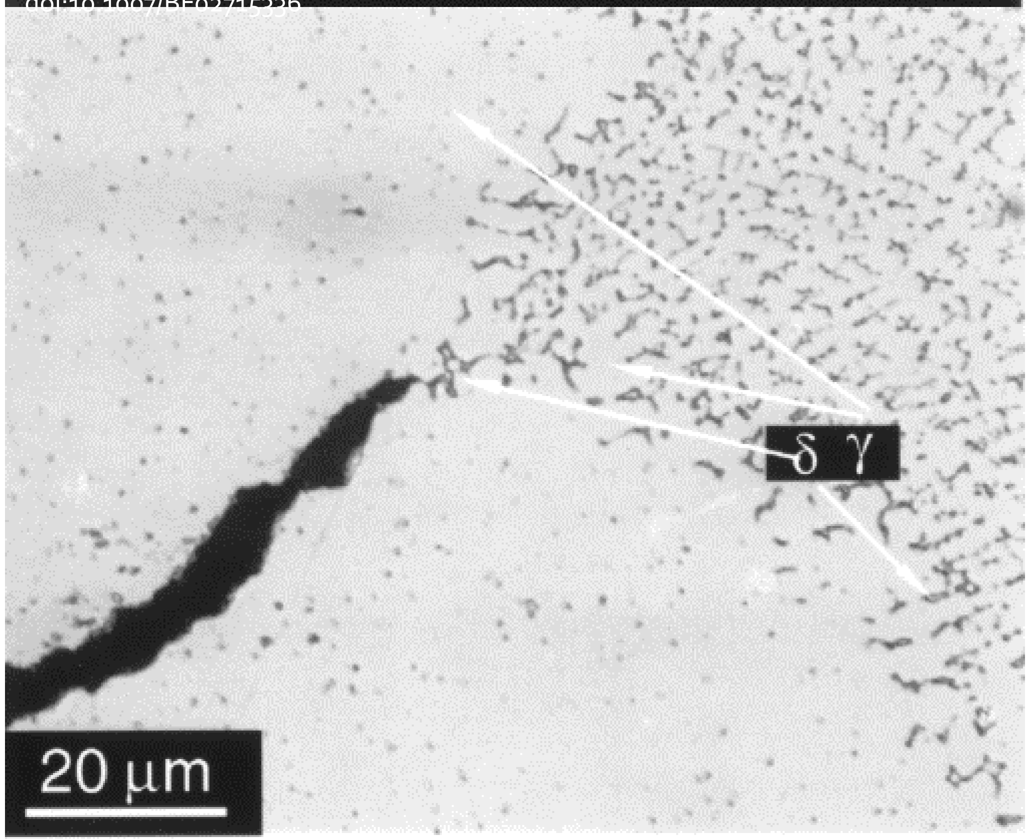
S. Sgobba and C. Boudot, Matériaux et Techniques 95, vol. 11-12, p. 23 (1997)



Case histories of failures: welds



Korinko, P.S. & Malene, S.H. Practical Failure Analysis (2001) 1: 61. doi:10.1007/BF02715336

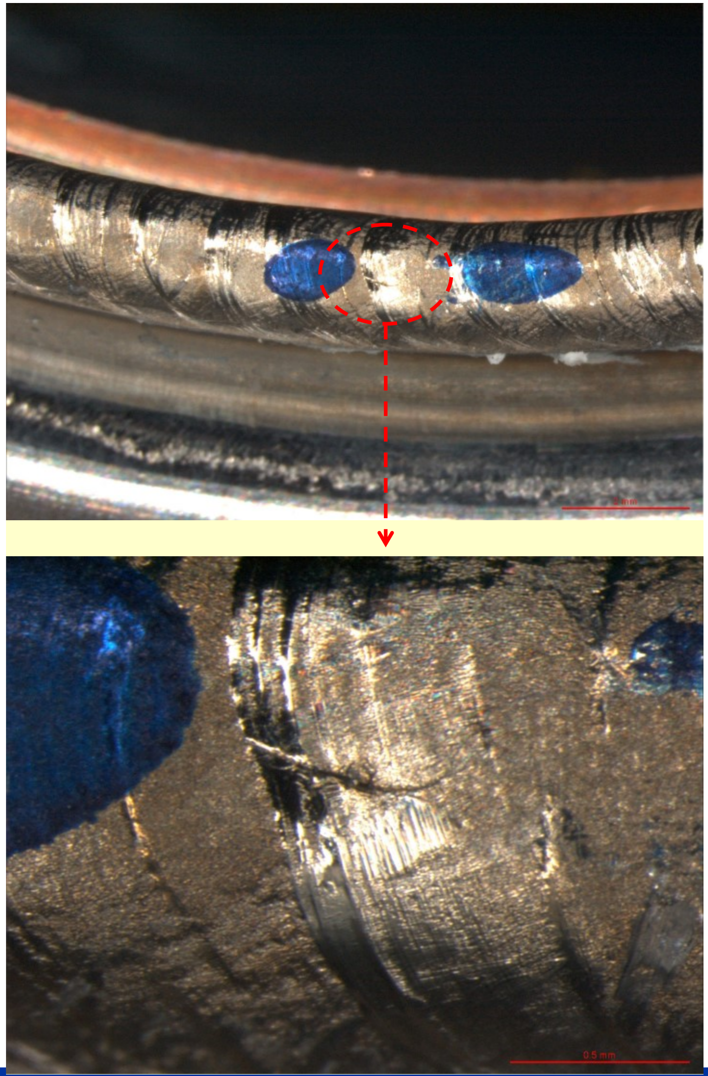


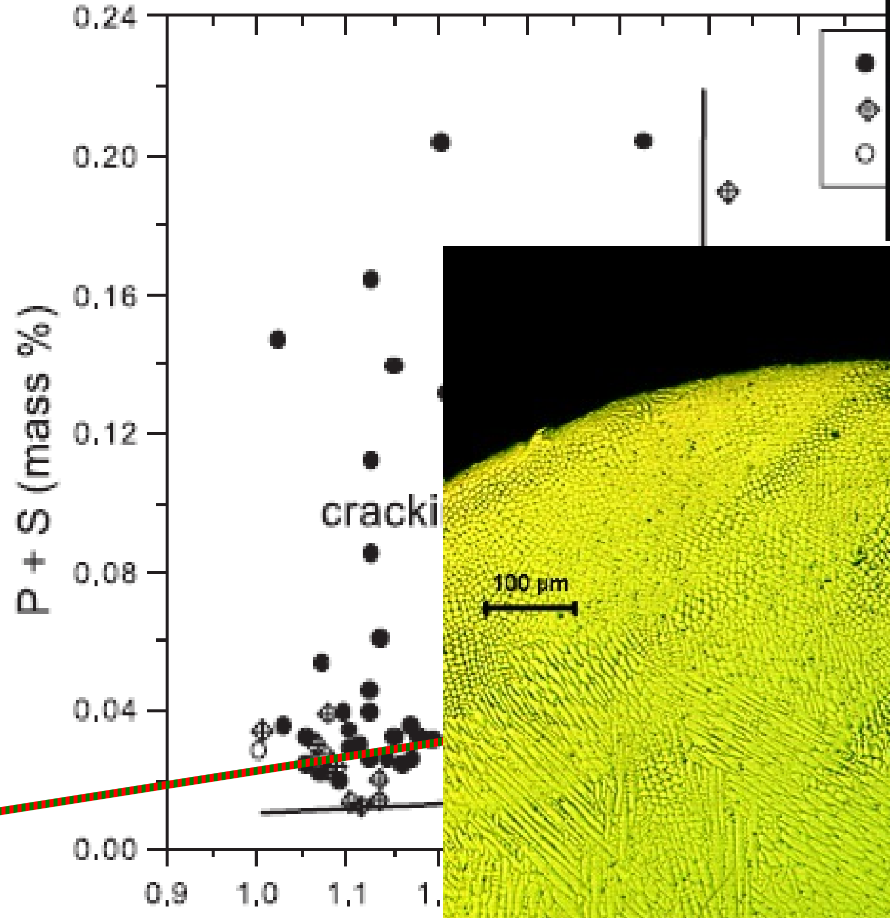
Schaeffler equivalent formulae for Cr_{eq} and Ni_{eq}

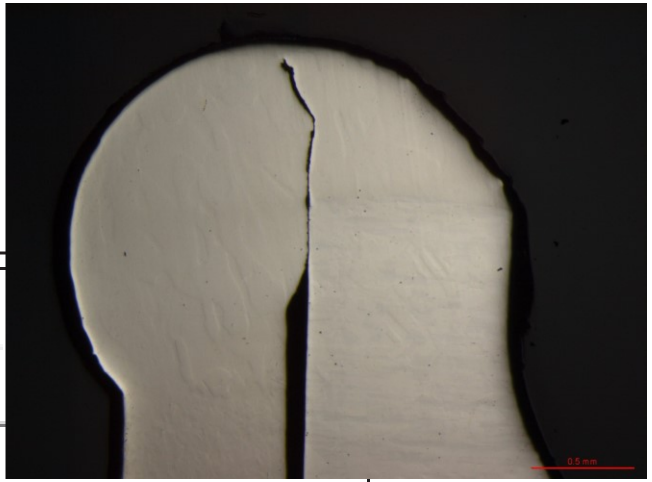
$Cr_{eq} = Cr + 1.5Si + 1.37Mo$

$Ni_{eq} = Ni + 0.31Mn + 22C + 14.2N$

Case histories of failures: welds

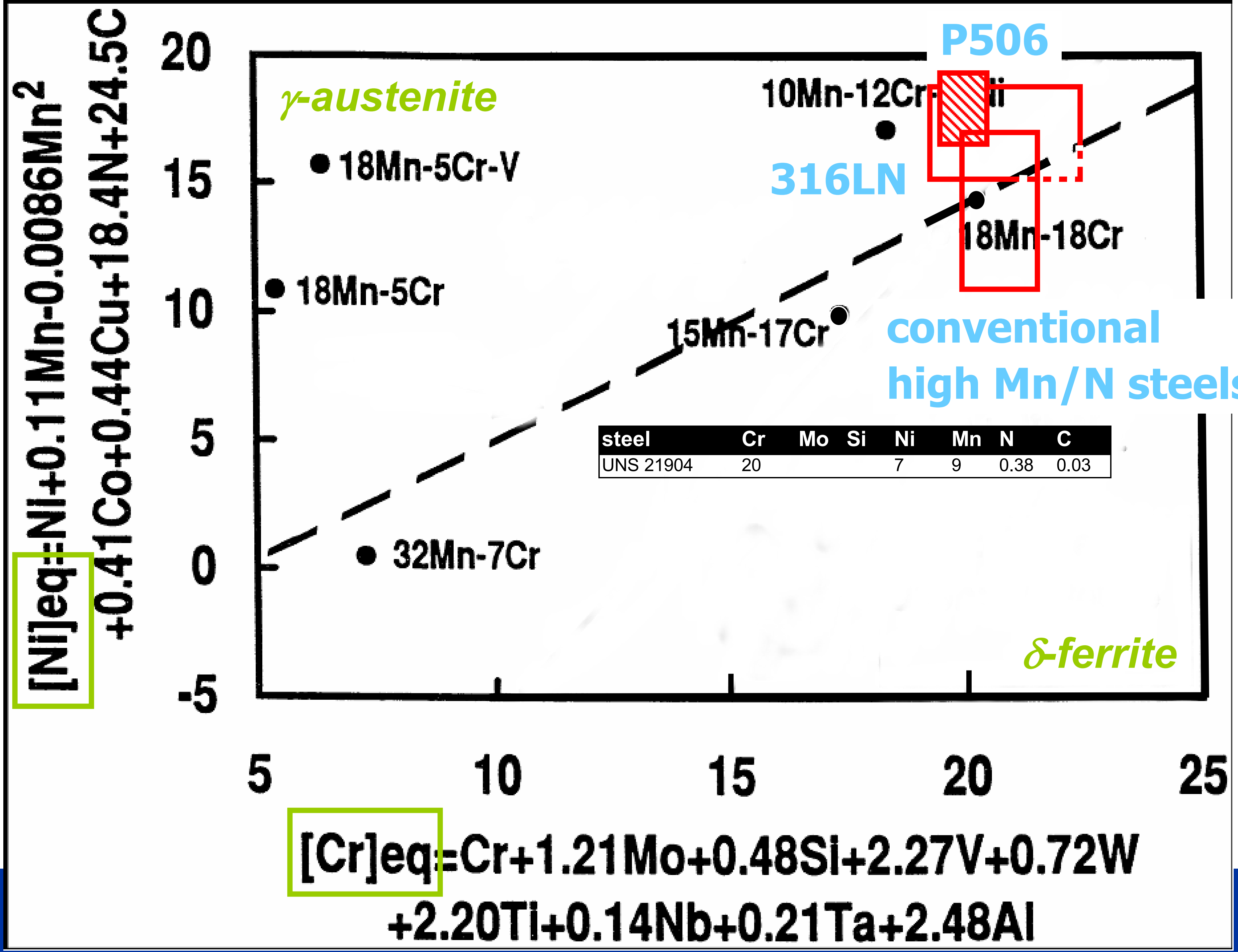






"mumetal", Ni80Mo0.5N
S<0.0005; P=0.003 (!)

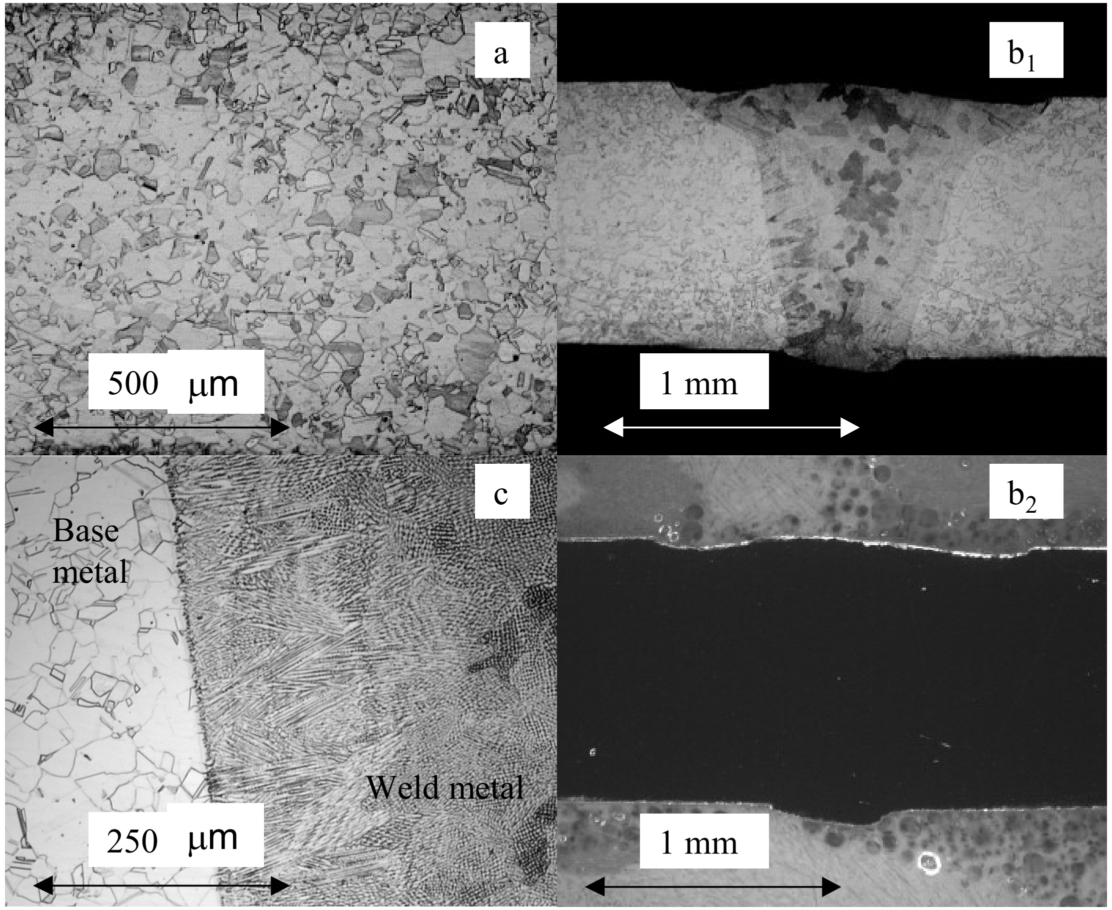
Case histories of failures: welds



steel	Cr	Mo	Si	Ni	Mn	N	C
UNS 21904	20			7	9	0.38	0.03

Case histories of failures: welds

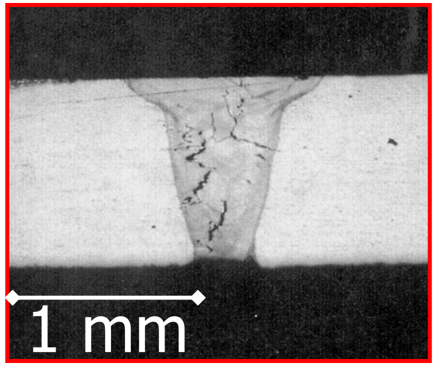
Steels	C	Mn	Ni	Cr	Mo	Si	N	P	S	B
P506	0.012	**12.05**	10.90	19.18	0.86	0.23	**0.33**	0.005	0.001	<0.001



S. Sgobba, C. Boudot, Soudabilité laser d'aciers inoxydables austénitiques, Matériaux et Techniques 95, n°11-12, p. 23 (1997).

J.P. Bacher and S. Sgobba, TIG Weldability of Special Stainless Steels for the Beam Screen of the Large Hadron Collider, Bulletin du Cercle d'Etude des Métaux, XVI, p. 13.1 (1995)

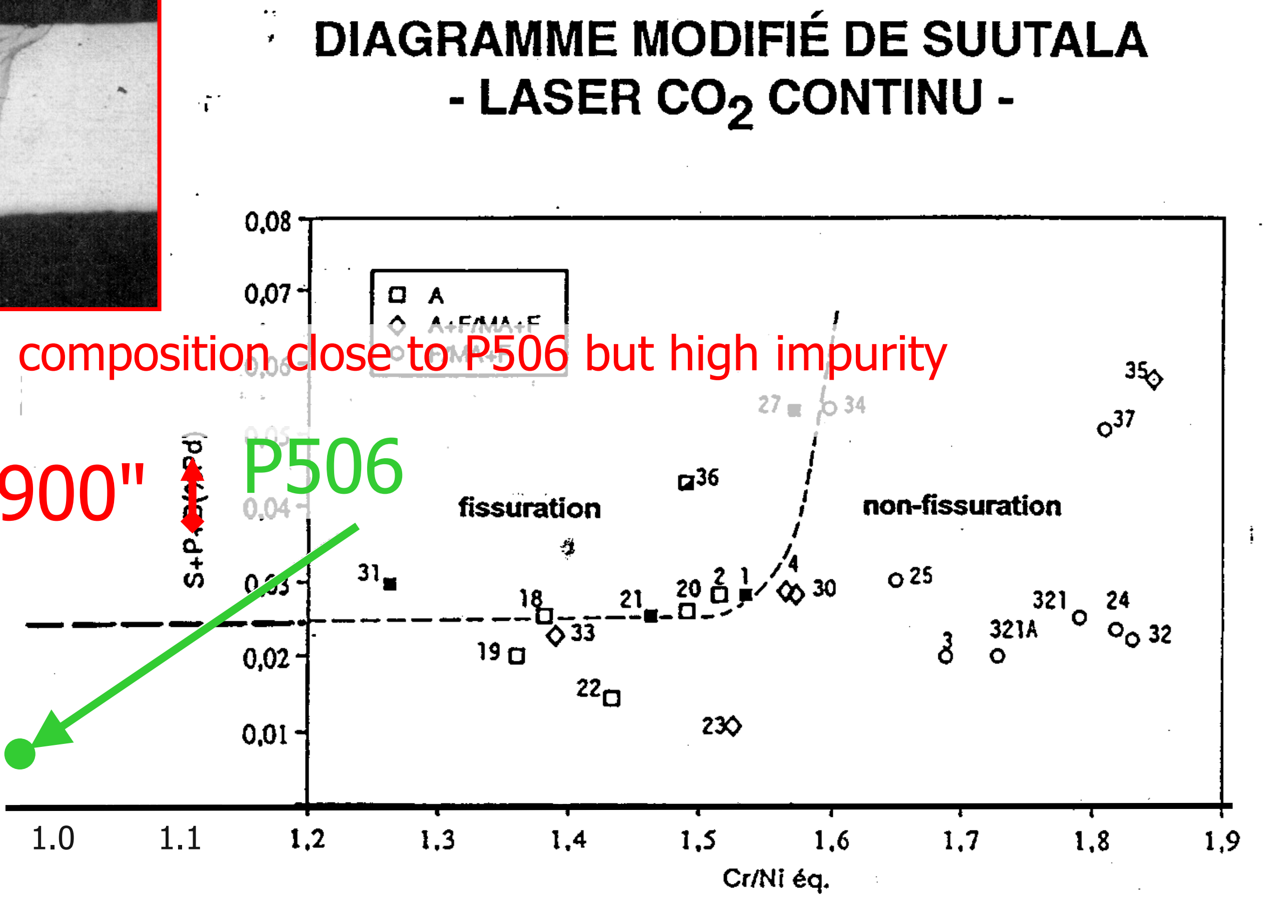
Case histories of failures: welds



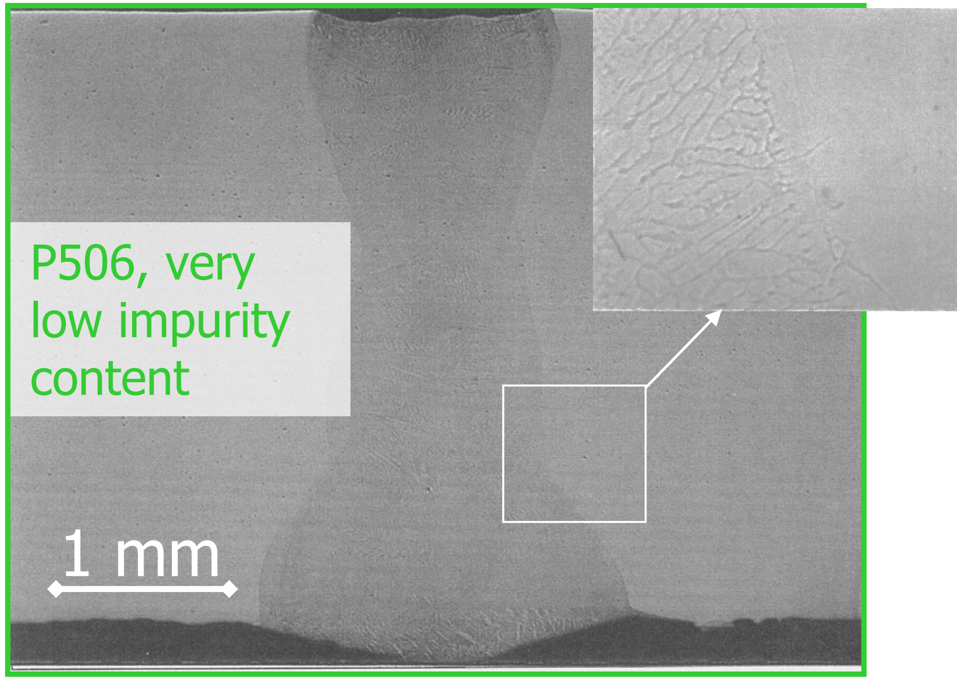
"900" steel: composition close to P506 but high impurity content

steel "900"

P506



$$\frac{\text{Créq.}}{\text{Niéq.}} = \frac{Cr + 1,37Mo + 1,5Si + 2Nb+3Ti}{Ni + 0,31Mn + 22C + 14,2N + Cu}$$



P506, very low impurity content

S. Sgobba: proc. Cycle Métaux et Procédés, CIP - Tramelan /CH, 1996, p. 8/1-10

Courtesy of ITER, @iter.org

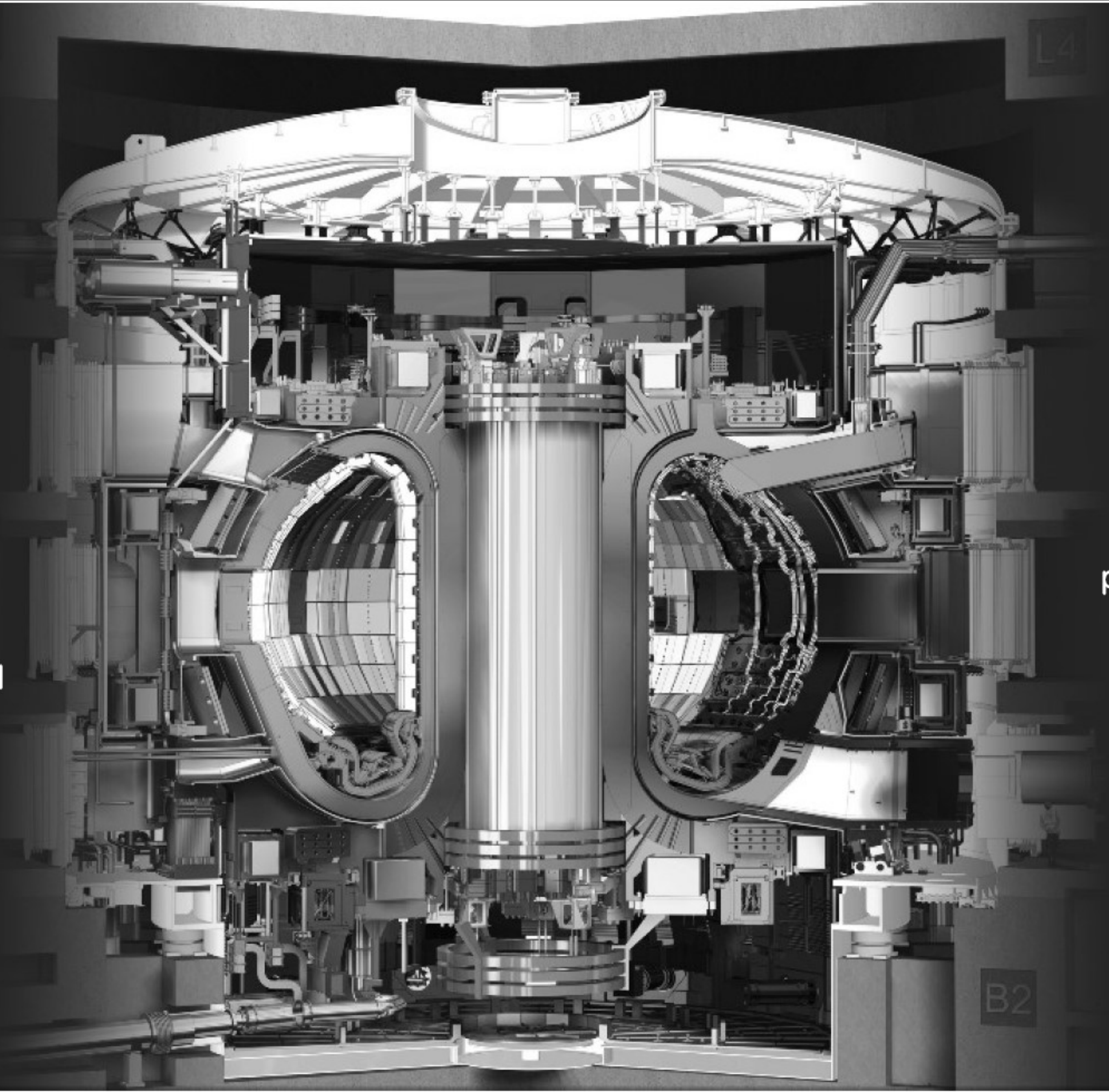
ITER TOKAMAK

AN INTERNATIONAL PROJECT FOR A NEW & CLEAN ENERGY

ITER represents the future of nuclear power where the fission reaction is replaced by a fusion reaction, the nuclear reaction that powers the sun and the stars, a safe, non-carbon emitting and virtually limitless energy.

With its millions of components, ITER will be the largest and most powerful tokamak ever built.

35 countries will collaborate during 35 years

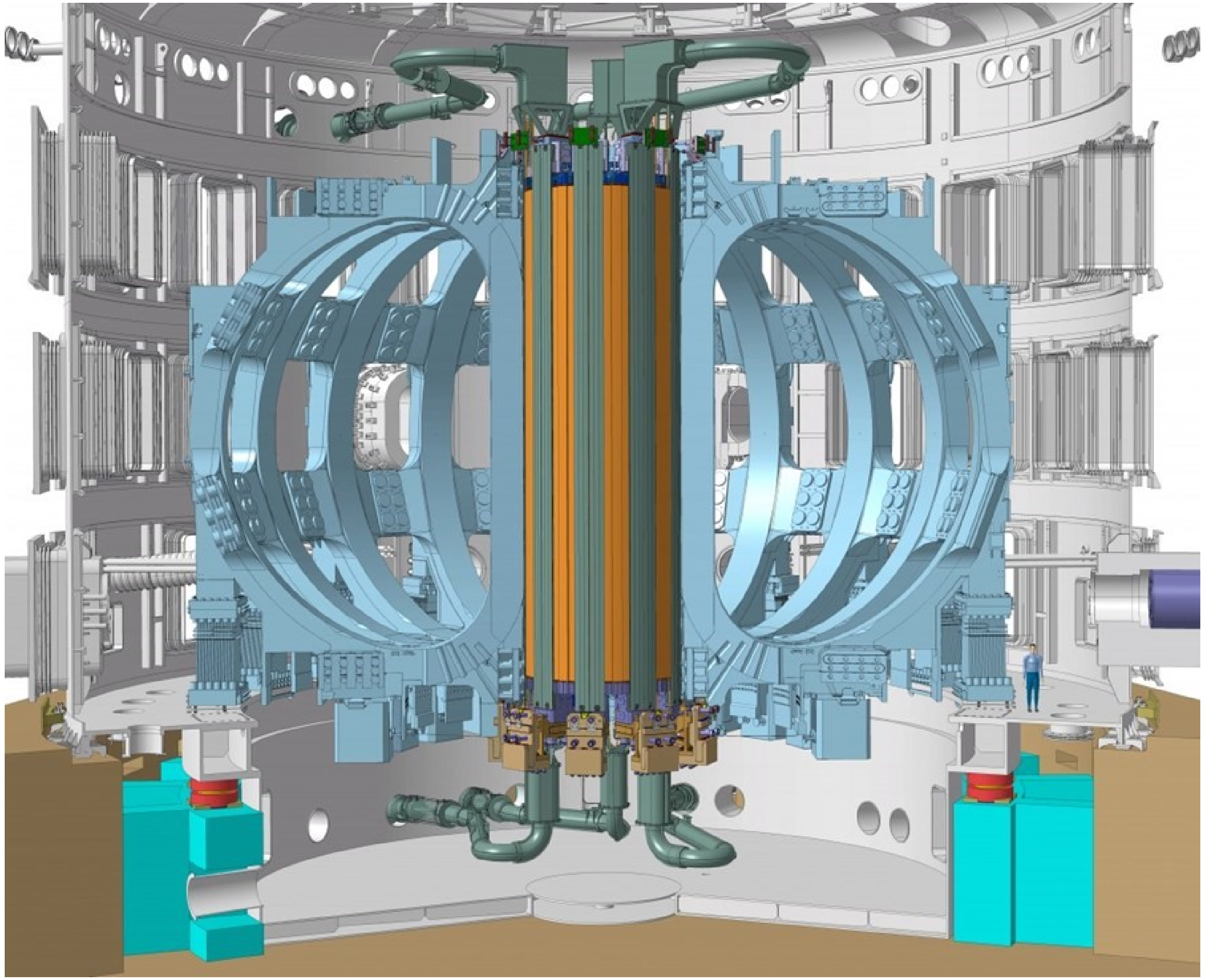


A GIANT
23 000 T

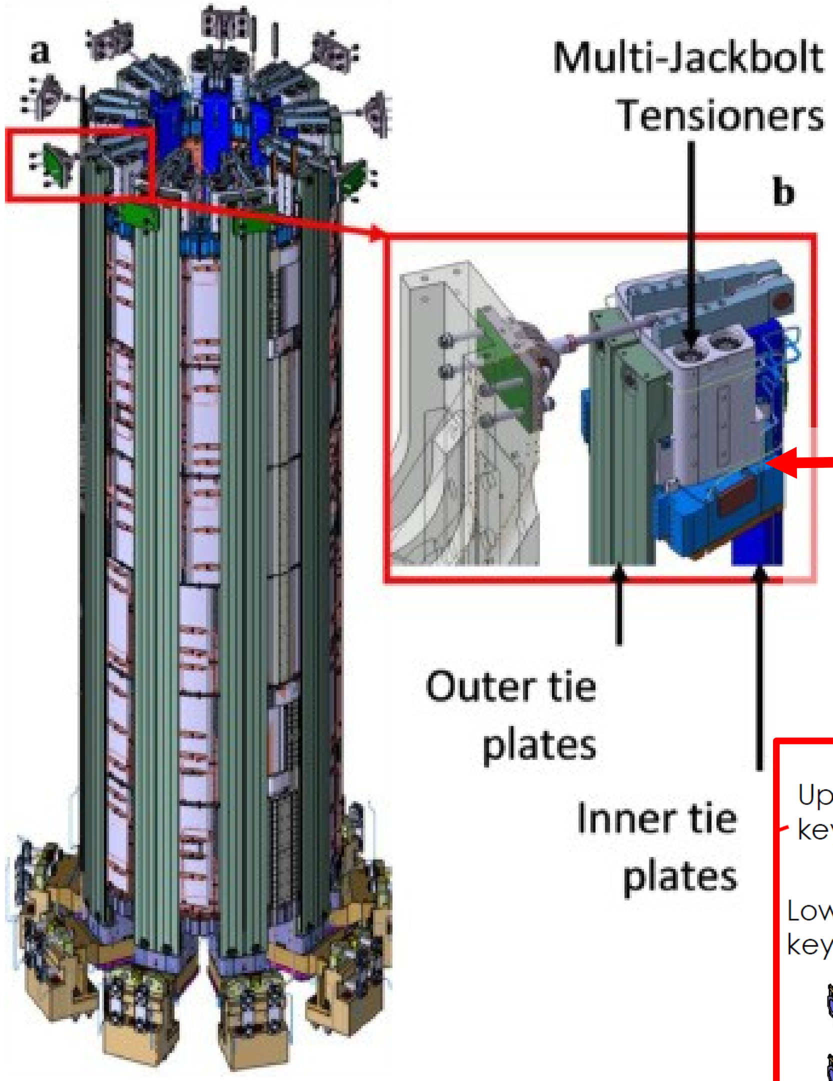
10X the core
of the sun
150 milllions ° C
plasma temperature

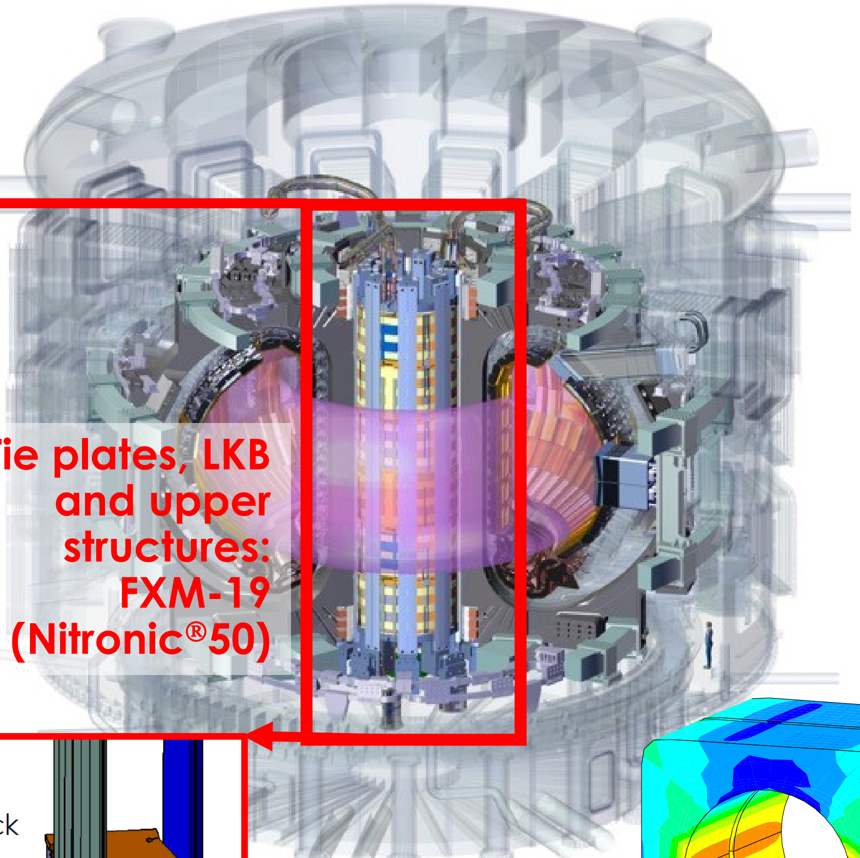
FUSION ENERGY
500 MW
Output power

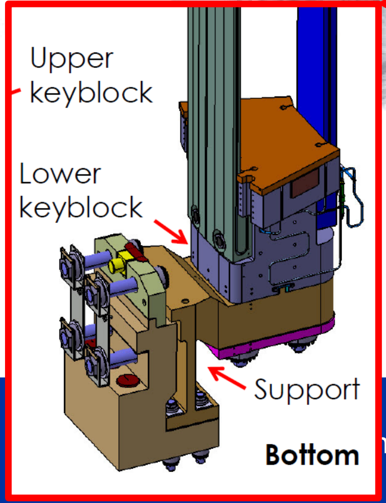
ITER Magnet system



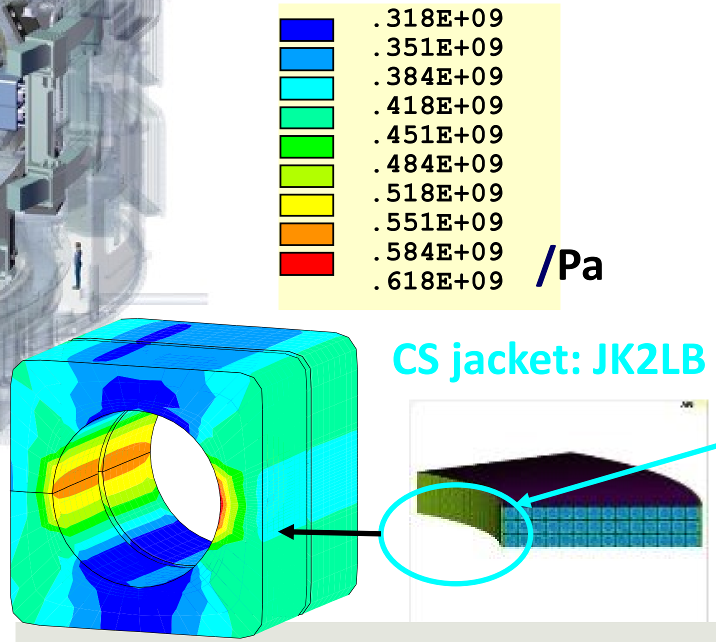
ITER Magnet system; the Central Solenoid and its precompression structure

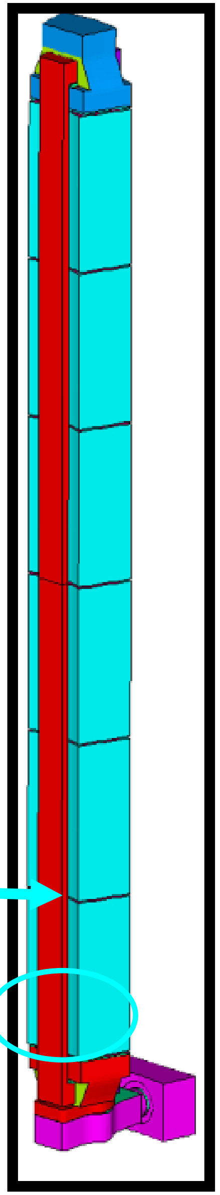






Central Solenoid Assembly Tooling Design Description, courtesy of S. Litherland, USIPO ↓



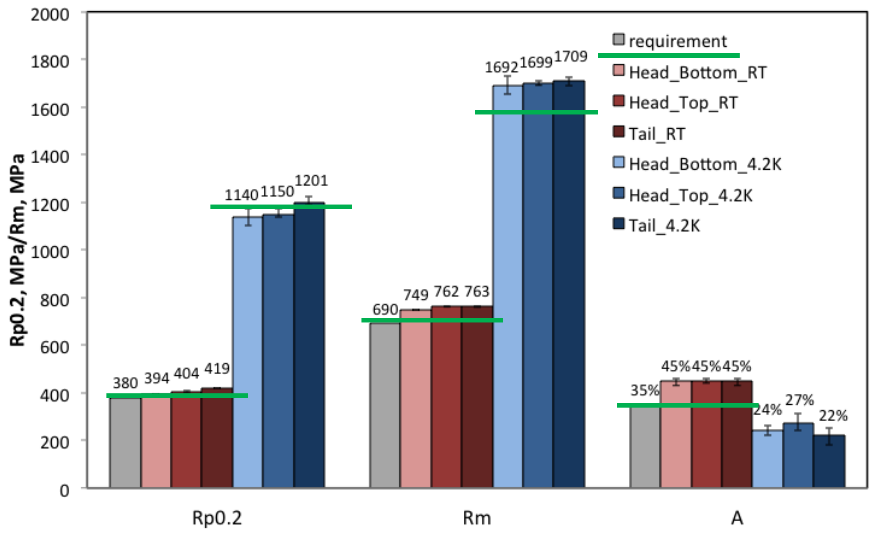


S. Sgobba et al., Metallurgical assessment of large size tensioning components for the precompression structure of the ITER central solenoid, Fusion Engineering and Design, Vol. 170, 2021, https://doi.org/10.1016/j.fusengdes.2021.112543

Material selection, ITER and fusion beyond ITER

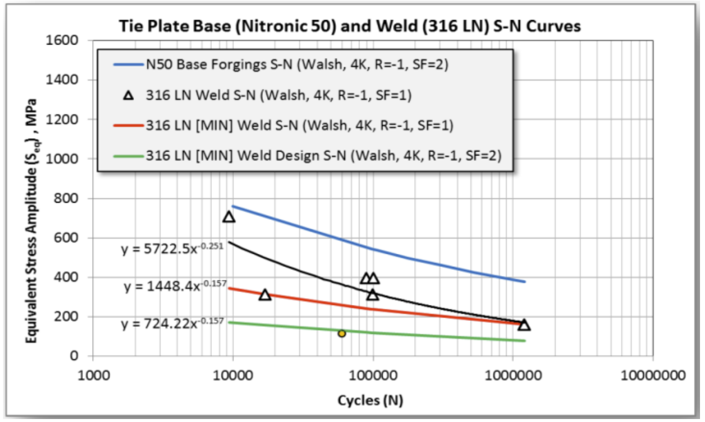
Mechanical properties

FXM-19



S.A.E. Langeslag et al., "Extensive Characterisation of Advanced Manufacturing Solutions for the ITER Central Solenoid Pre-compression System," Fusion Eng. Des. (2015), https://doi.org/10.1016/j.fusengdes.2015.06.007

	Specimen location	Specimen orientation	K_{IC} [MPa√m]	J_{IC} [N/mm]
Single piece forged	Head (top)	LT	170	130
	Head (bottom)	LT	190	161
		LS	197	172
	Tail (slab)	LT	188	157
		LT	226	239
Welded solution	weld	weld direction	112	62

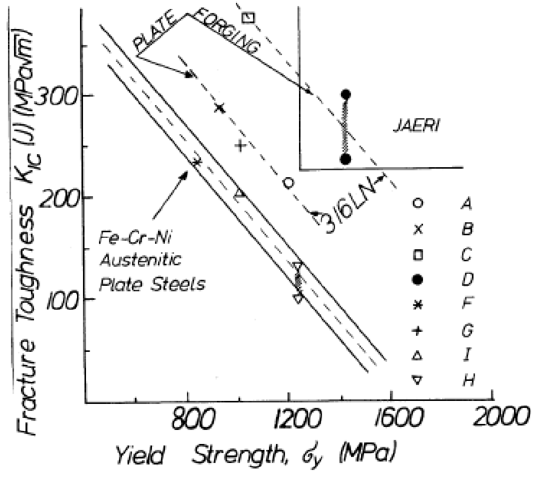


Courtesy of R.P. Walsh, NHMFL

Requirements at 4 K:

- Very high strength and toughness: Rp_{02} > 1200 MPa; Rm > 1600 MPa; K_{IC} > 130 MPa √m
- Fatigue resistance at cryogenic temperature
- Larger thermal contraction than central solenoid jackets (JK2LB) for an effective pre-compression at 4 K

FXM-19 (Nitronic®50) fulfils but with little margin the requirements for tensile properties and fracture toughness at RT and 4.2 K.

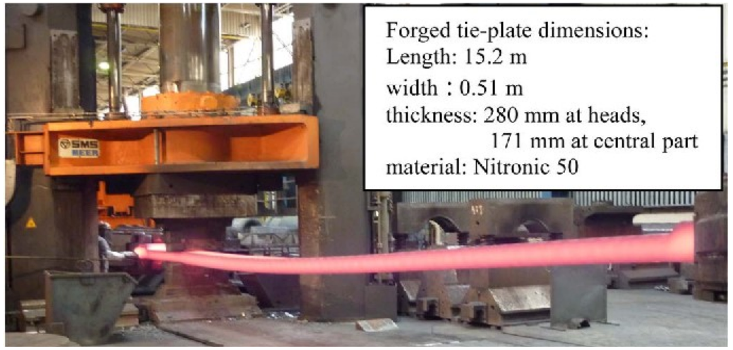


4 K toughness-strength relation of nitrogen strengthened stainless steels.

A. Nyilas, P. Komarek - Cryogenic Tensile and Fracture Properties of Structural Materials for Superconducting Magnets in Fusion Technology (1989)

Material selection, ITER and fusion beyond ITER

- Very large multidirectionally forged components

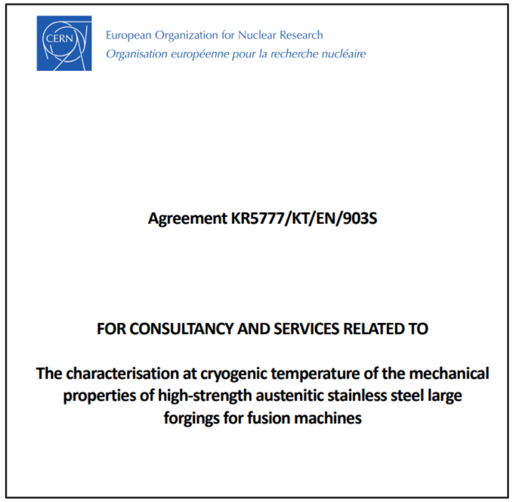


Tie plate single piece forging. Courtesy of Rolf Kind GmbH

Lower Support brackets: from material specification to 100% volumetric inspection. Courtesy of Monchieri

Lower key block in test assembly. Courtesy of US ITER and Petersen Inc.

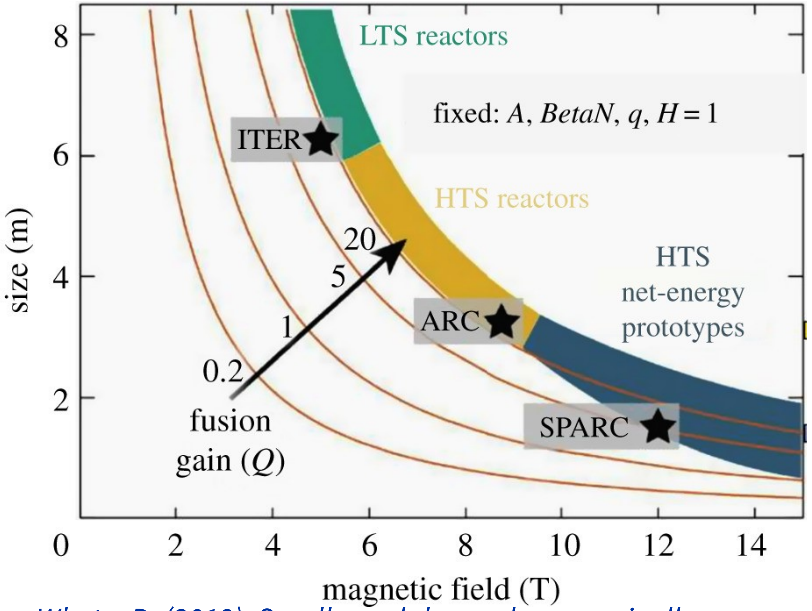
Fusion beyond ITER: the SPARC project


European Organization for Nuclear Research
Organisation européenne pour la recherche nucléaire

Agreement KR5777/KT/EN/903S

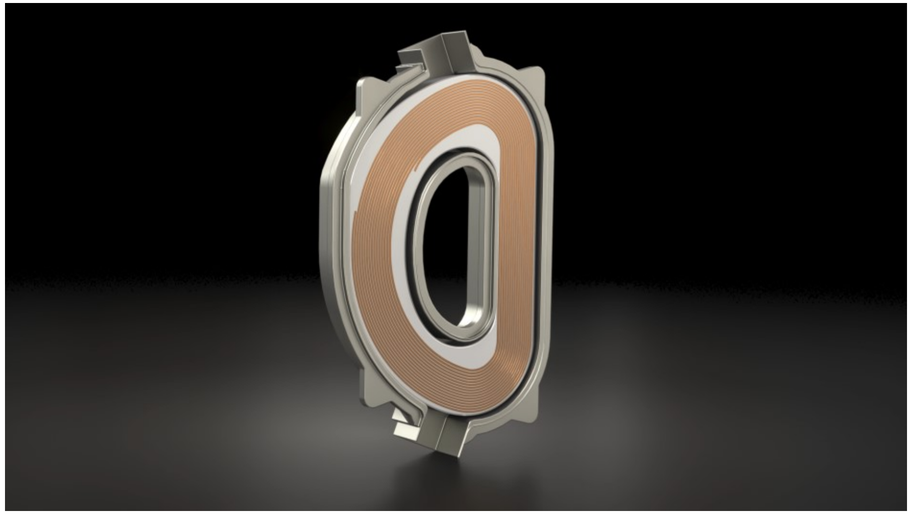
FOR CONSULTANCY AND SERVICES RELATED TO

The characterisation at cryogenic temperature of the mechanical properties of high-strength austenitic stainless steel large forgings for fusion machines



Whyte, D. (2019). Small, modular and economically attractive fusion enabled by high temperature superconductors. Philosophical Transactions of the Royal Society A, 377(2141), 20180354.

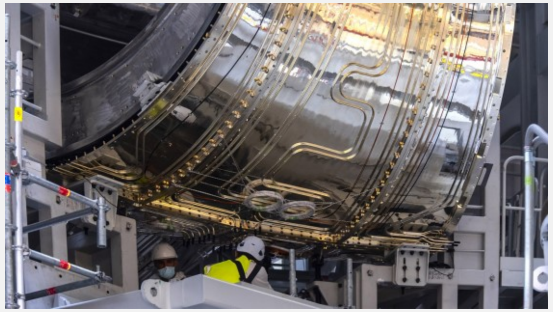
- **SPARC is a project of Commonwealth Fusion Systems (CFS), spin – off of MIT.**
- **Compact reactor (40 times smaller than ITER in volume).**
- **Relies on high temperature superconducting (HTS) magnets**



Ongoing cooperation contract with R. Kind, a company that specialises in the production of large size forgings in high-alloy stainless steels to precisely meet customer needs and find the optimal process for each piece. They are in charge of forging the casings of the toroidal magnets of SPARC. EN/MME-MM is providing expertise in cryogenic mechanical testing and material characterization.

Failure analysis and remediation: the ITER Vacuum Vessel Thermal Shields

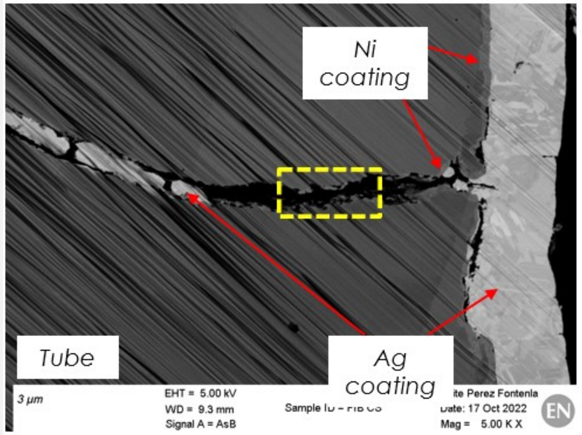
Leaks were found in an uninstalled TS, and **corrosion spots** have been observed in a large quantity both in installed and uninstalled TS cooling pipes' welds. These components are **very large panels** (2000 m^2), cooled down by ~**23 km of piping**, **stitch welded** for an improved thermal efficiency and **silver coated** to reduce their emissivity.

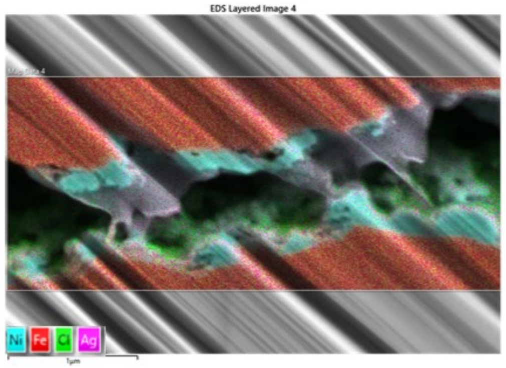


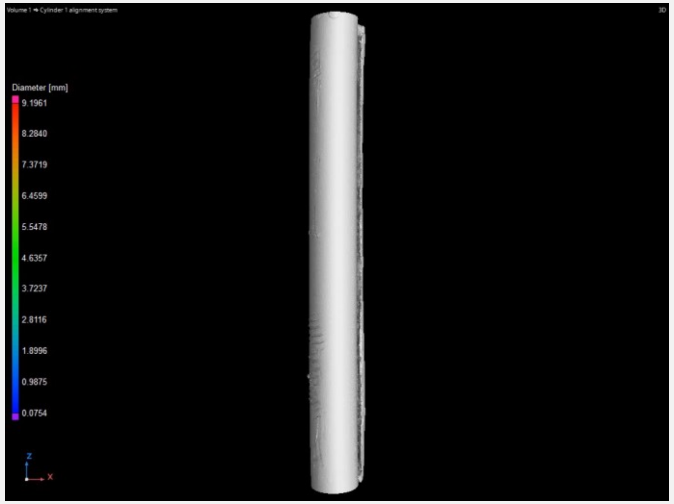
⥣ Silver coated thermal shields, with the stitch – welded cooling pipes

Investigations were based on **high resolution CT** (~ 150 CT scans), **metallographies** at targeted positions, and **advanced SEM** (including FIB).

FIB cut showing cavities underneath the Ag coating (top). The detection of Cl inside them was possible thanks to the X-max Extreme Detector with an extreme light element sensitivity ⇒

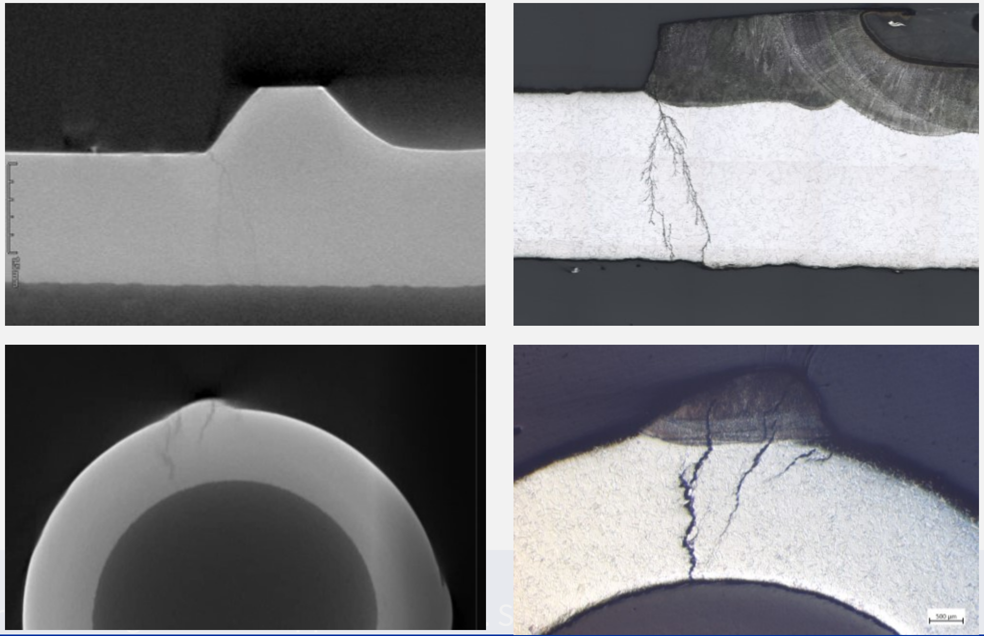






⥣ Computed tomography was used for the first time to show the 3D trajectory of the leak provoked by the SCC.

CT scans (left) and metallographic preparations (right) of the same positions showing through thickness cracks by SCC⇒



failures

S. Sgobba et al., *Analysis of the leakage events of the ITER actively cooled magnet system thermal shields pipes*, https://doi.org/10.1109/TASC.2024.3362746

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NEWS & MEDIA

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LATEST ITER NEWSLINE

THERMAL SHIELD REPAIR | WHERE ARE WE AT?

Fitting the vacuum vessel sectors like a jacket, lining the inner wall of the cryostat, or covering the sides of vertical coil gravity

ITER NEWSLINE -

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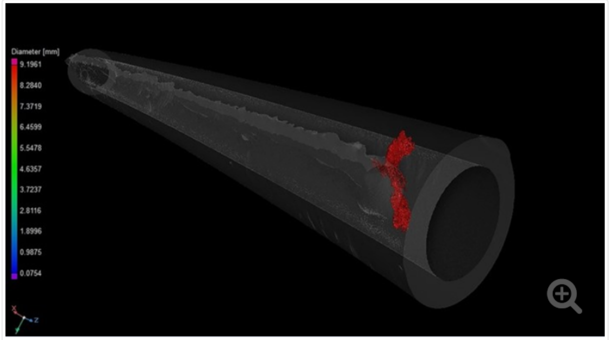
Vacuum technology

FINDING INFINITESIMAL NEEDLES IN A HAYSTACK

A cutting-edge X-ray process confirms microscopic cracking and keeps ITER on track to succeed.

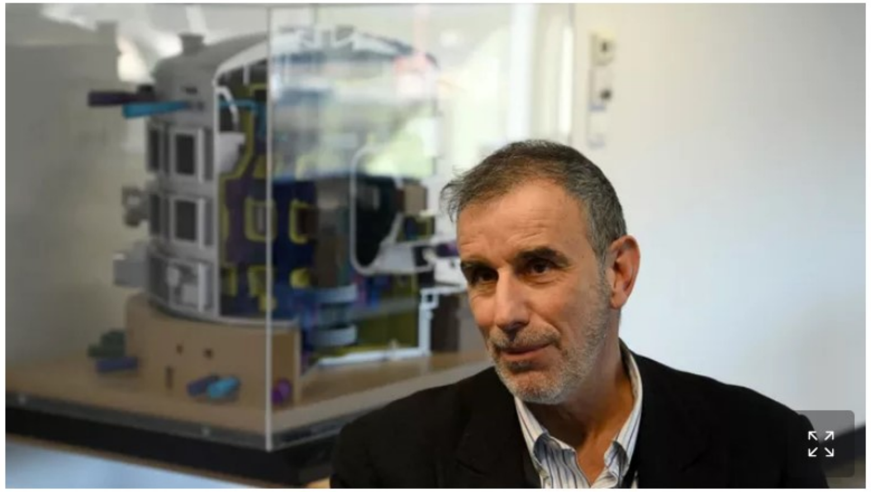


The location of a branched stress corrosion crack that crosses the entire cross section of a thermal shield cooling pipe is accurately identified by X-ray

Défauts et retards pour le projet international de fusion nucléaire Iter

Par Le Figaro avec AFP
Publié le 06/01/2023 à 17:50 , mis à jour le 06/01/2023 à 18:16

Écouter cet article 00:00/02:34



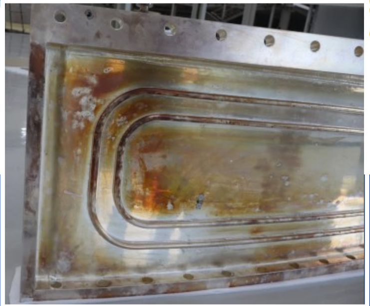
Pietro Barabaschi a été nommé à la tête du programme Iter en septembre 2022. *NICOLAS TUCAT / AFP*

Deuxième défaut relevé, des traces de corrosion sur les *«écrans thermiques»* qui doivent protéger de la très forte chaleur émise lors de la fusion. Ce qui pourrait aboutir à des fuites de l'hélium utilisé dans le circuit de refroidissement. Ces réparations vont retarder le projet. *«Ça n'est pas un processus qui prend des semaines, mais des mois, voire quelques années»*, a expliqué Pietro Barabaschi, qui doit élaborer d'ici la fin de l'année un nouveau calendrier des opérations.

Issue on Silver Coated Surface on VVTS#M OB Panel

NOH Chang Hyun & Mahipal Vasant mar...
Jan. 2024

VVTS-Qualification of remedial actions, Inox India, 28/02/24



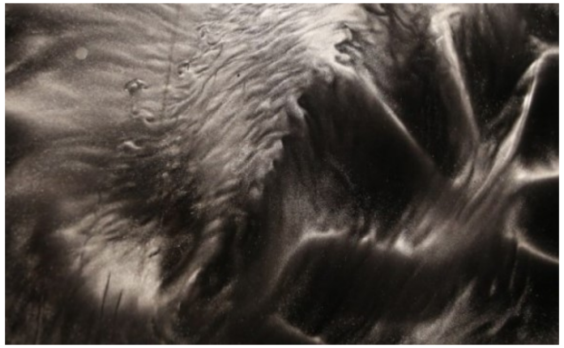
Conclusions

- Prevention of vacuum failures at CERN requires decades of anticipation
- Materials can seldom be “off-the shelf”
- Cost-driven solutions (1.4307 for the ITER VVTS; non ESR grades under the budget pressure of the LHC construction) are often not forgiving
- Future projects (SPARC) requiring high strength materials for cryogenic use at the limits of feasibility
- High strength grades may be not forgiving: irreproachable production and follow-up, starting from steelmaking, is paramount
- Stainless steels are not always stainless!
- All too often, “le mieux est l’ennemi du bien”: the best is the enemy of the good

CERN70
YEARS / ANS CERN

News

Yunchul Kim, paramagnetic particles, CERN Art and Science Summit, 30/01/24

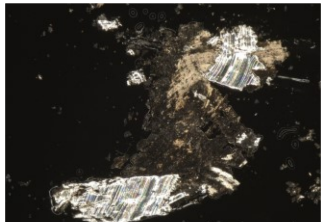


Yann Marussich, collide residency organised by Arts at CERN @ MME-MM on 21/09/24, ferrofluids

CERN at 70
Inspiring the Future

This year holds special significance for CERN as the Organization celebrates its 70th anniversary on 29 September 2024.

Johanna Bruckner, visualisation of CERN research@ MME-MM on 13/03/24



Joan Heemskerk, materiality of reading and writing @ MME-MM on 01/11/23