



An Application of Powder Metallurgy for the LHC

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TS-MME-MM

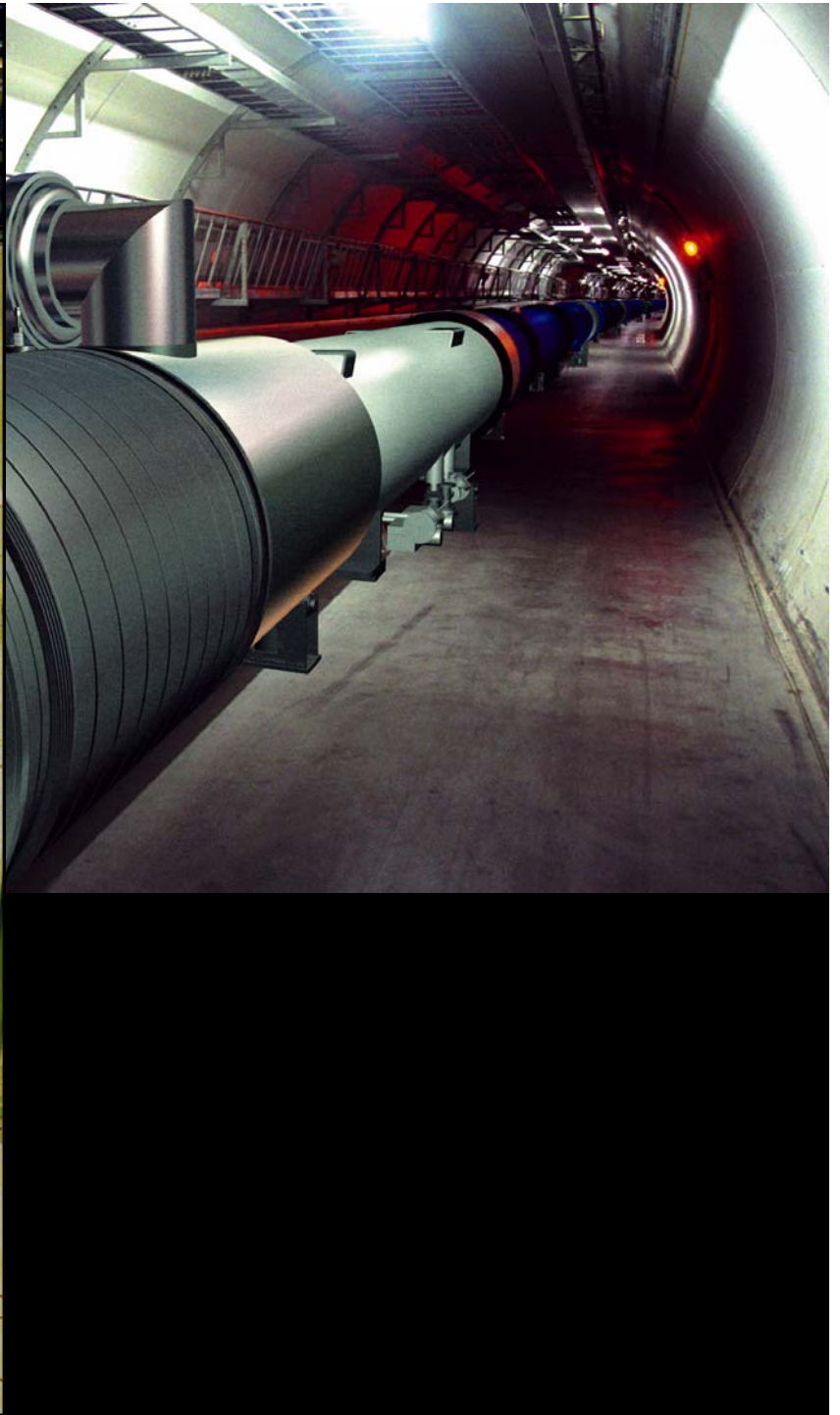
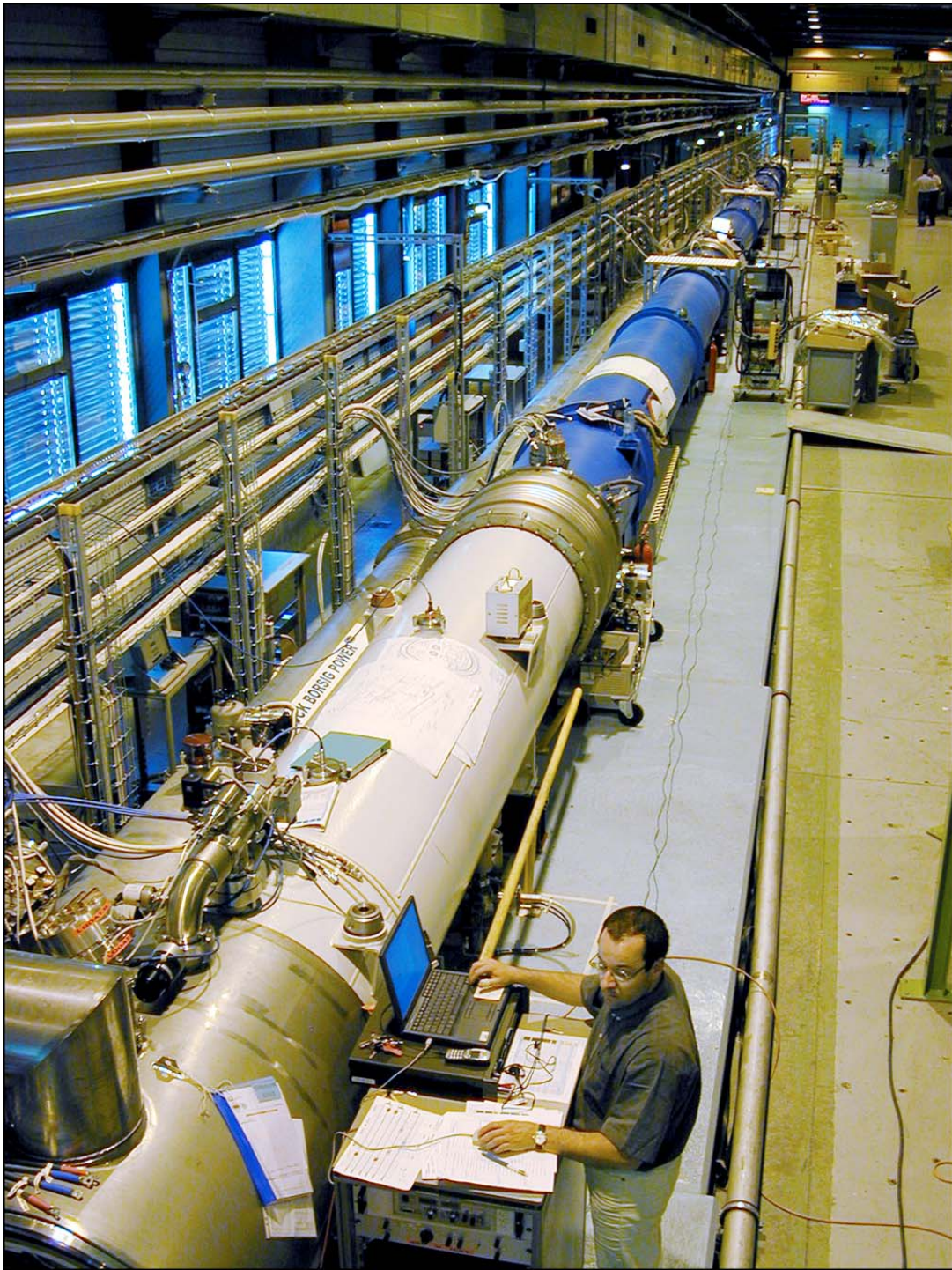
TS Workshop
4-6 May 2004
Archamps, FR



Plan

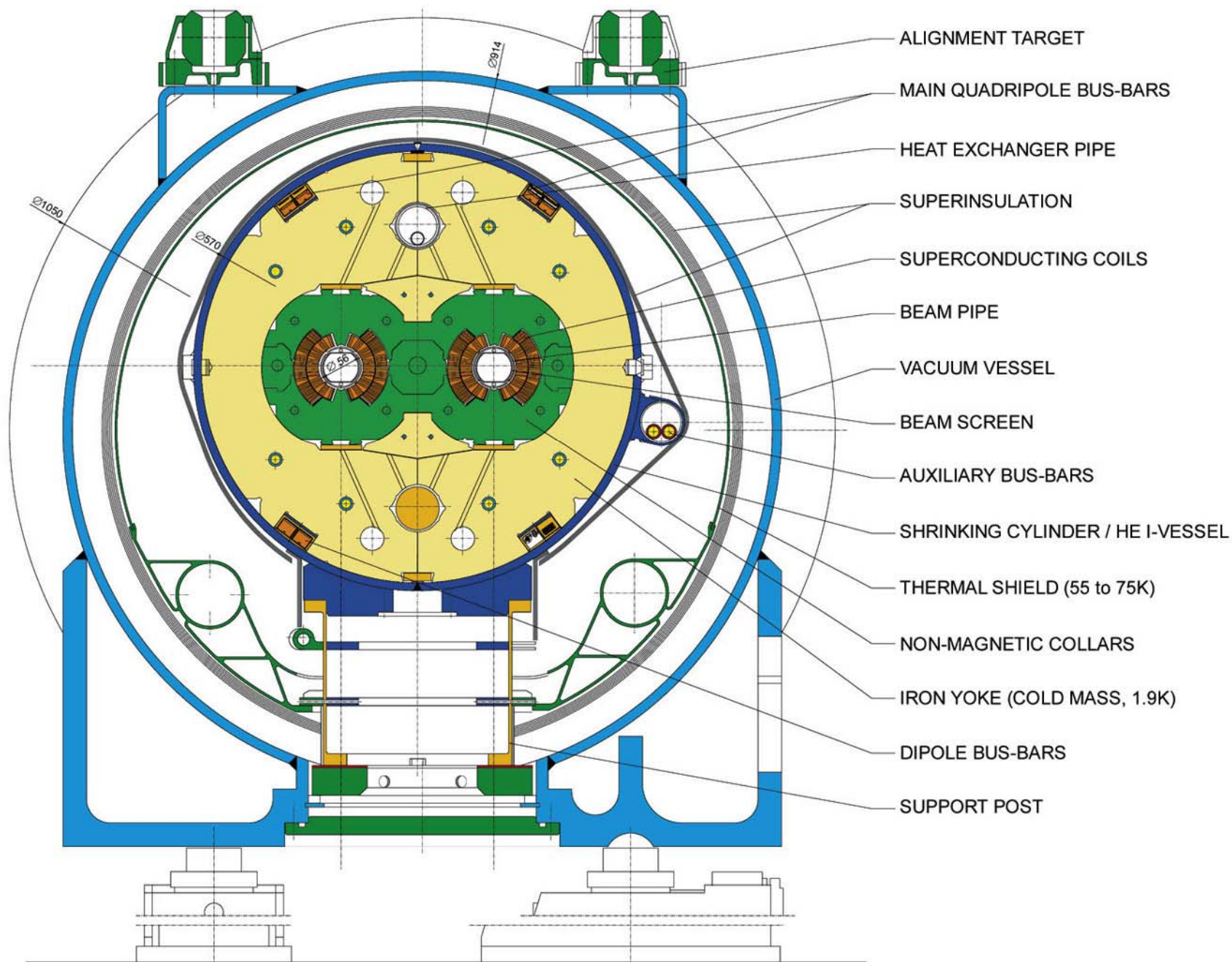


- The end covers
- Requirements
- Welded, cast, forged, PM solutions
- The Metso 316LN PM covers
 - Microstructure*
 - Properties at 4.2 K*
 - Weldability*
- Image analysis as a quality control tool
- Discussion and conclusion



LHC DIPOLE : STANDARD CROSS-SECTION

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





What is a magnet cover ?



o an austenitic stainless steel dome equipped with protruding nozzles

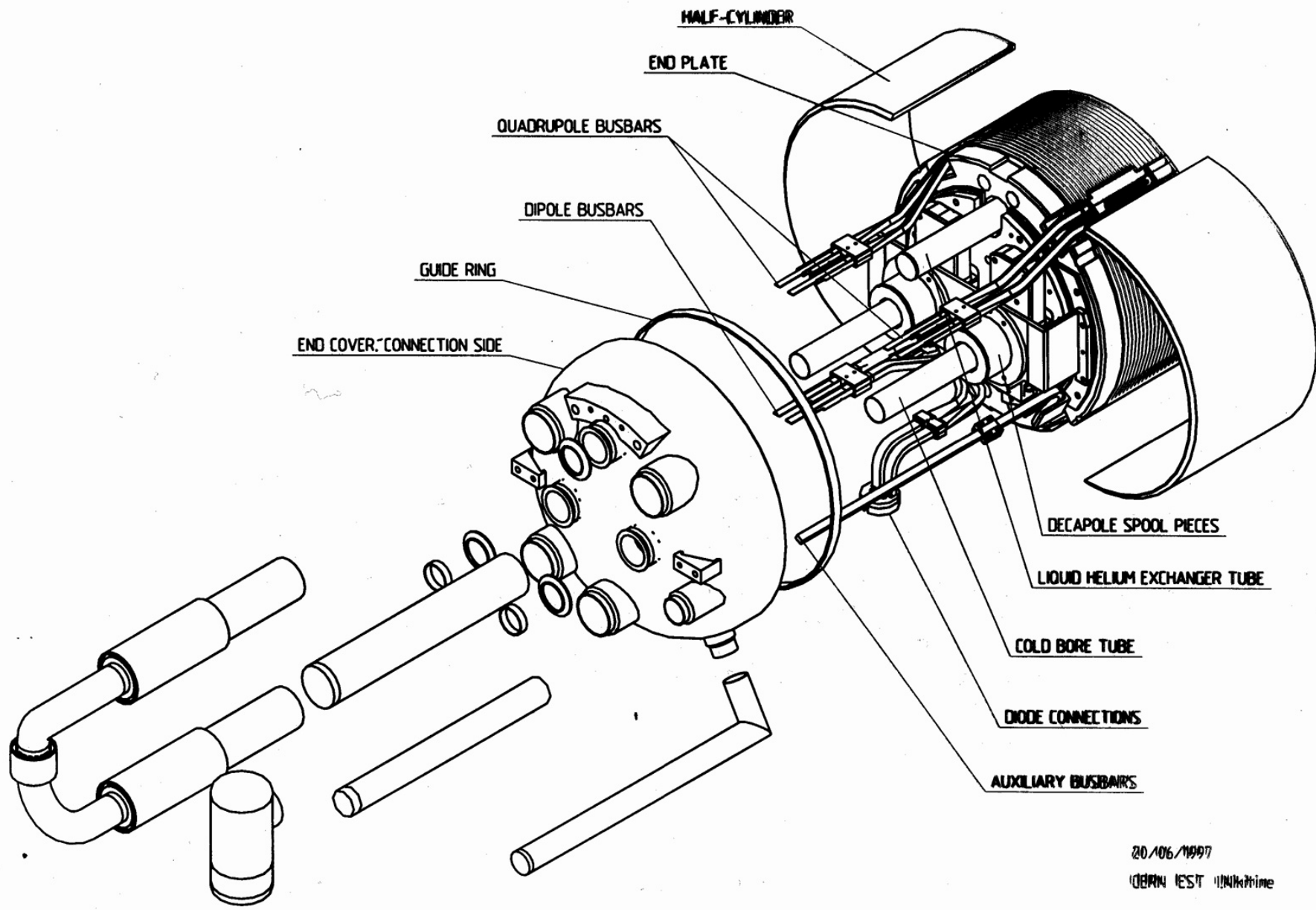
o the nozzles are foreseen for the passage of the cold bore and the different cryogenic lines

o it allows to close the cold mass of the cryodipoles

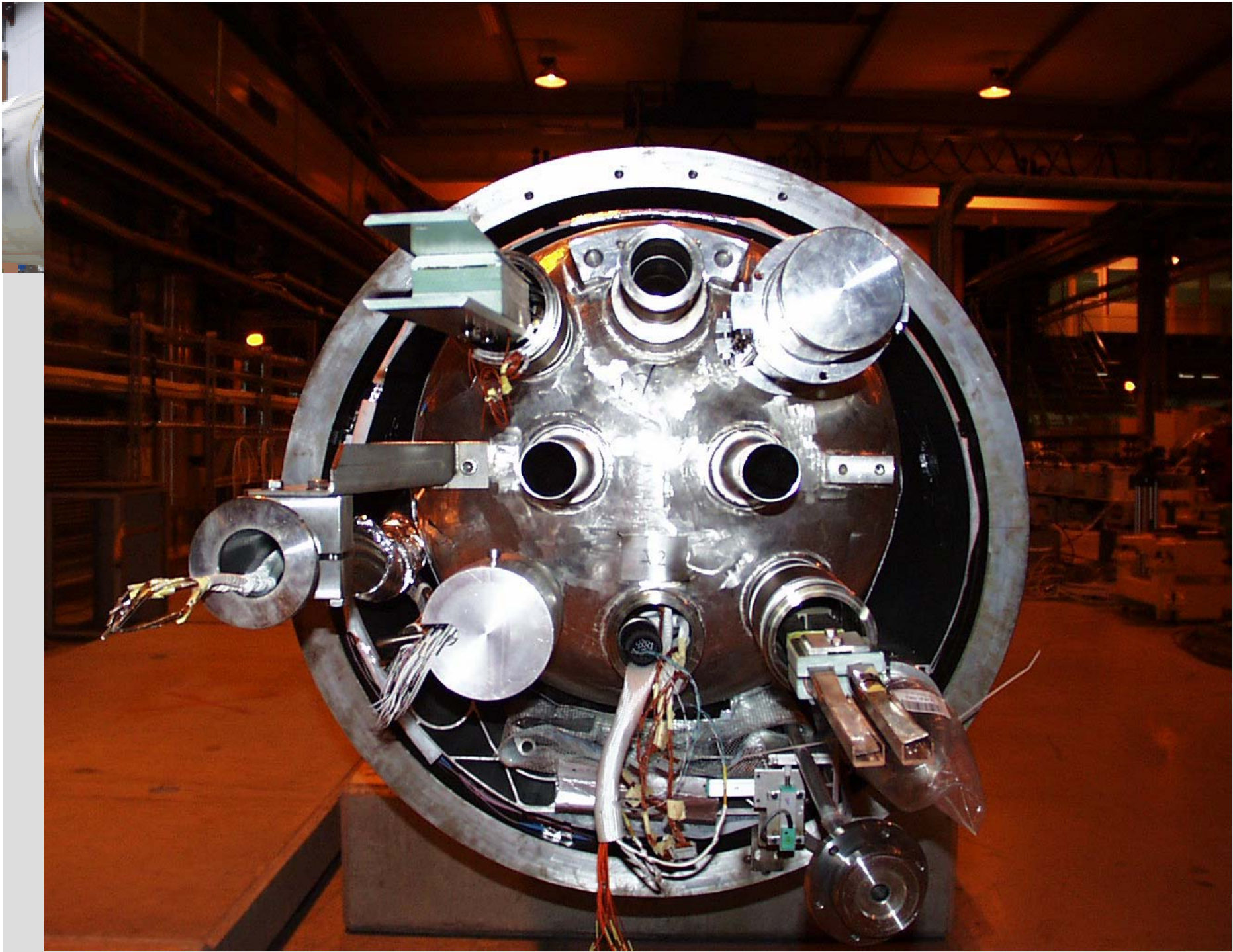
o is welded onto the shrinking cylinder (MIG, circular)

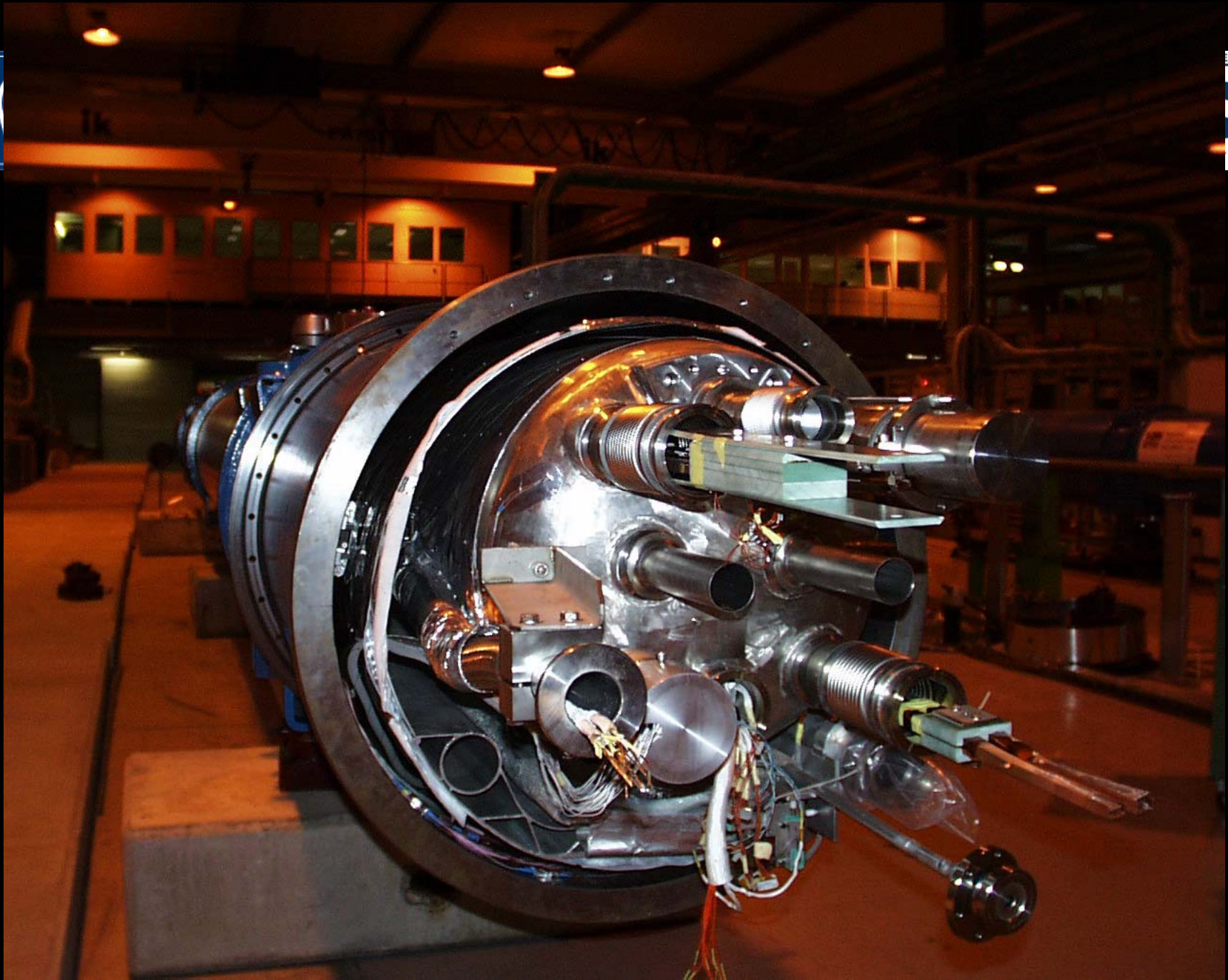
o the nozzles are welded onto the interconnections pipes (TIG, orbital)

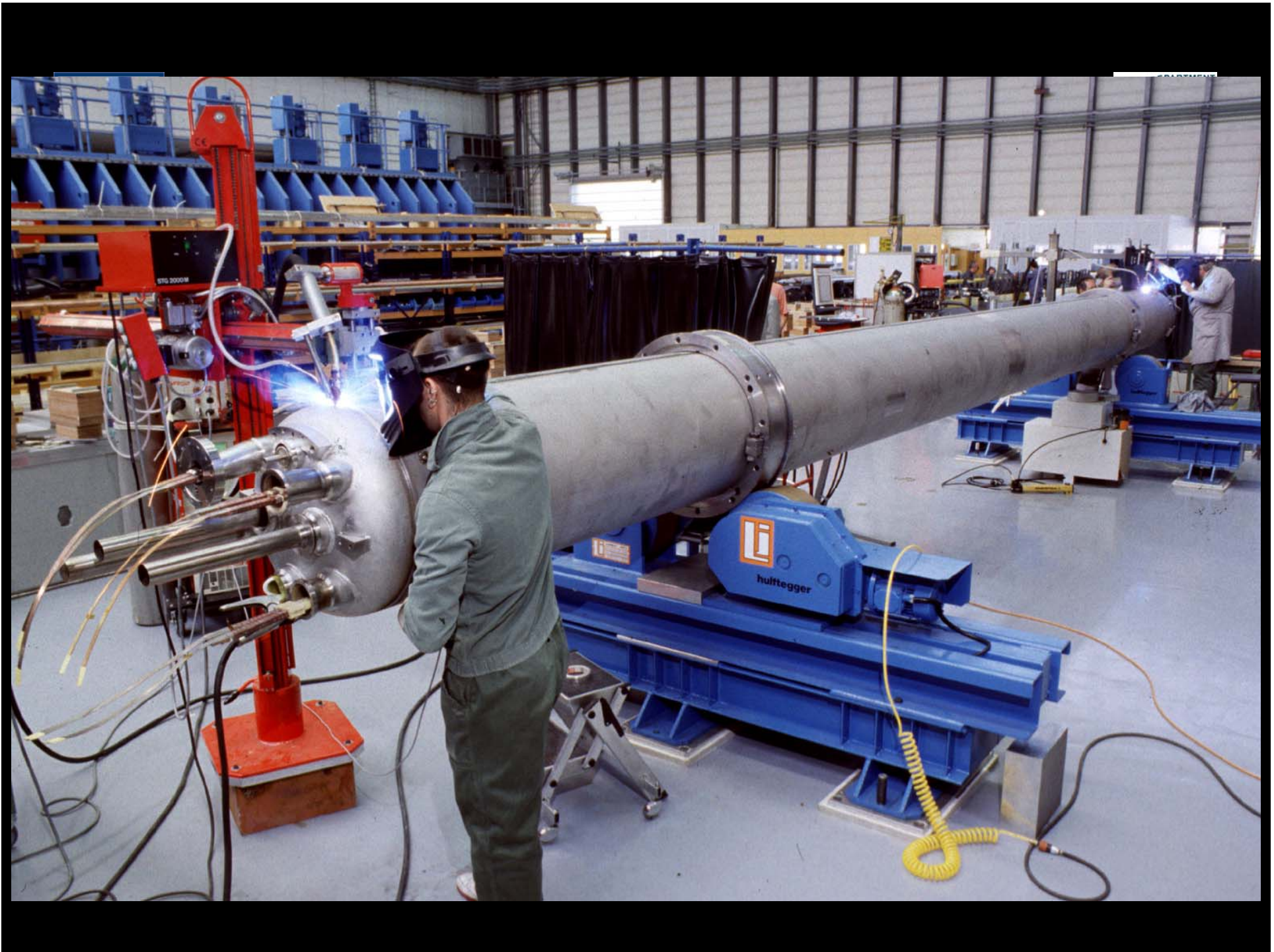
END COVER AND DIPOLE. CONNECTION SIDE



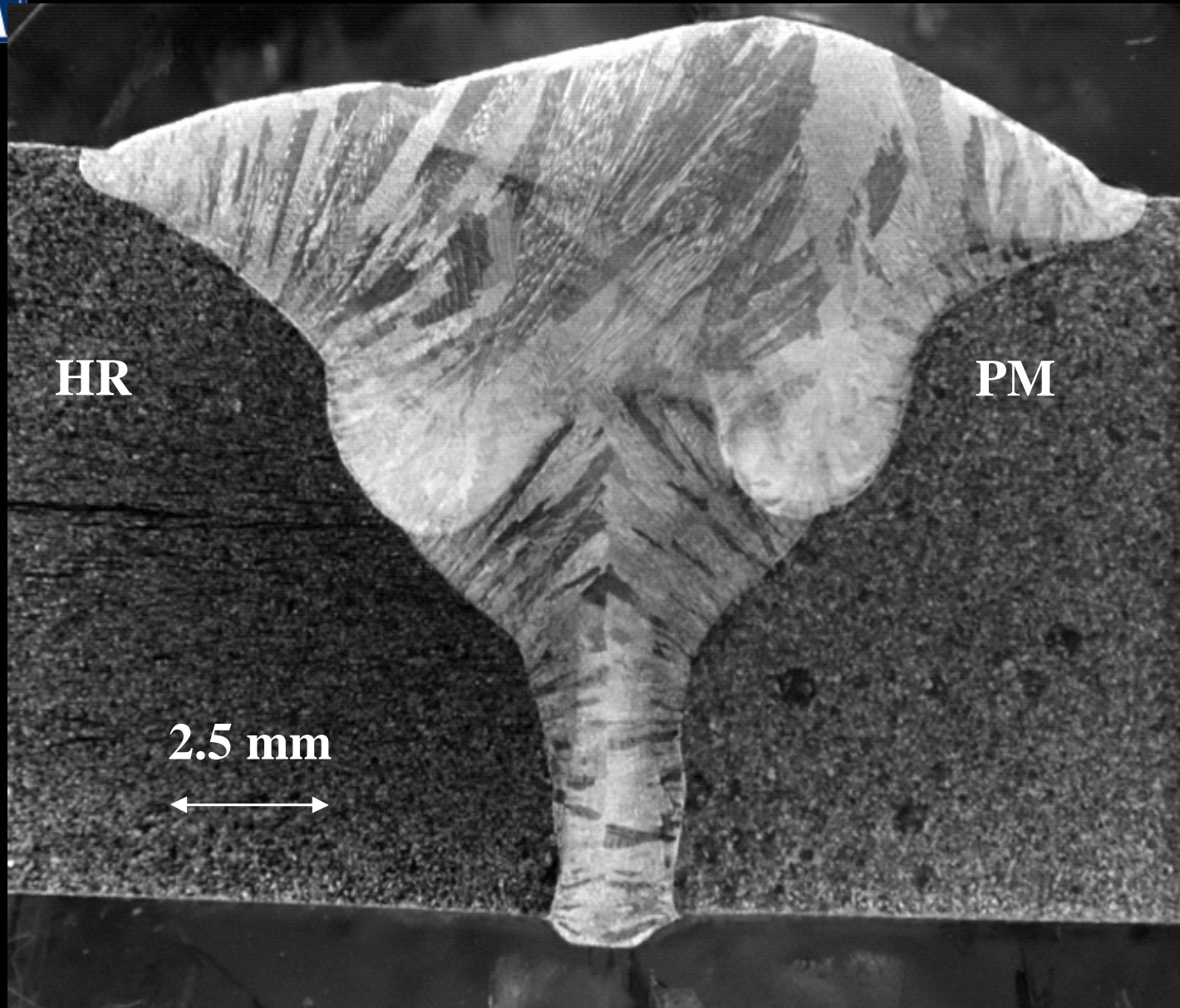
20/06/1997
GERM TEST 11/1/97







PM 316LN, circular MIG weld



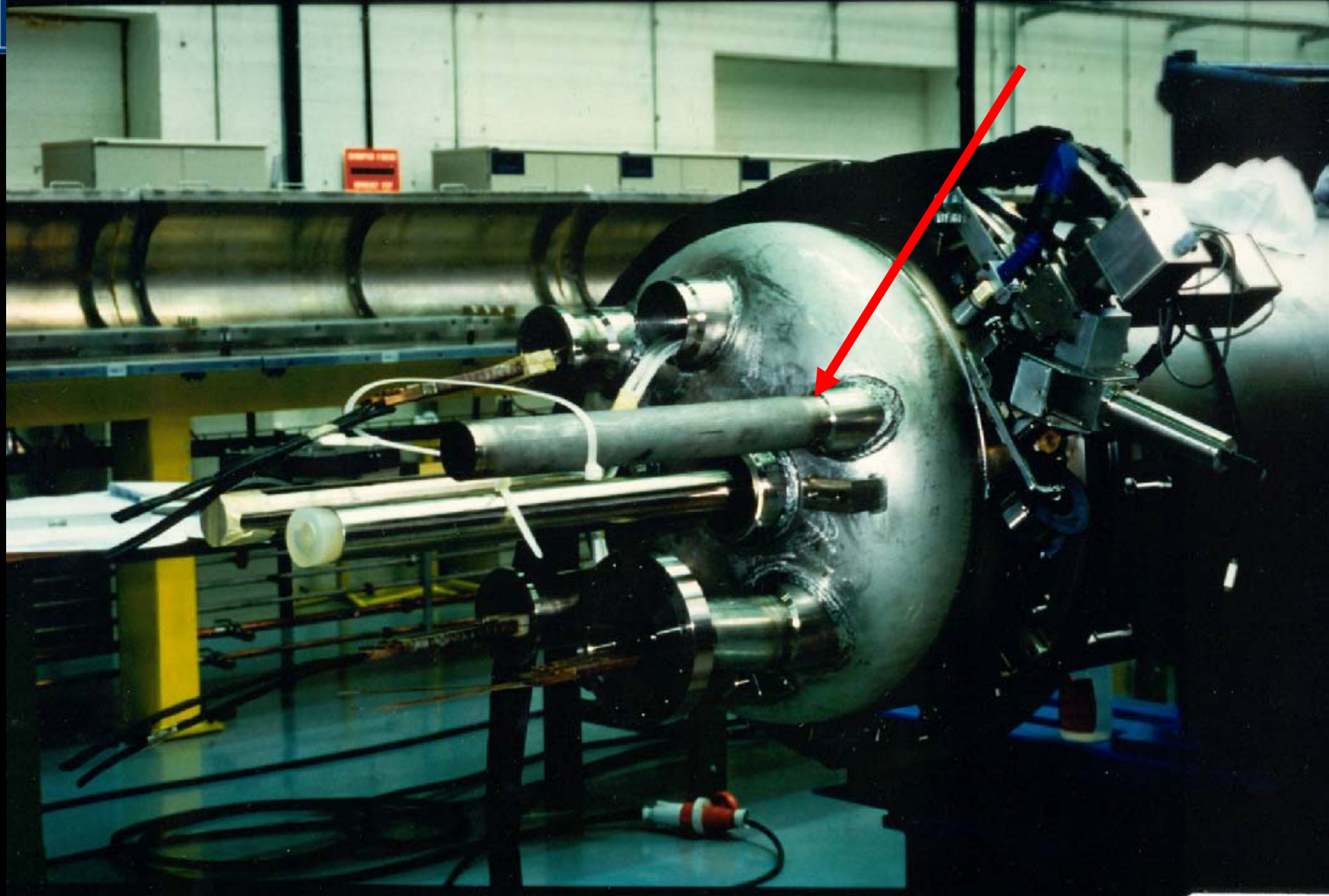
HR

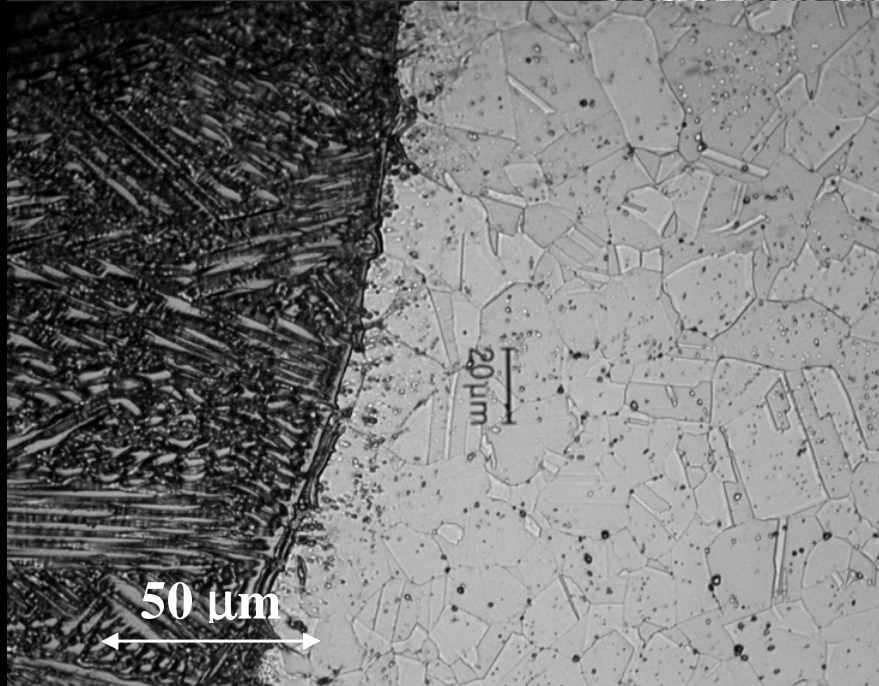
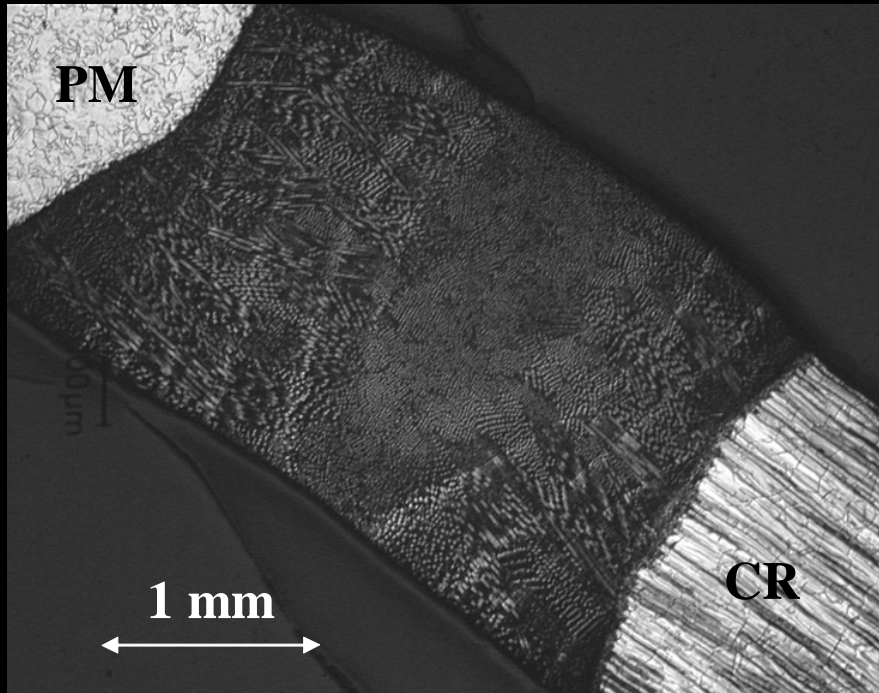
PM

2.5 mm



316LN, orbital TIG weld





PM 316LN, orbital TIG weld

Welding details :

Travel speed:	1.5E-02m/s	Arc length:	1.5E-03m
Peak current:	68 A	Tip angle	45°
Base current:	32 A	Gas flux and flow rate:	
Peak time:	0.2 s	Shielding:	Ar, 1/6 l/s
Base time:	0.2 s	Backing:	Ar, 1/12 l/s



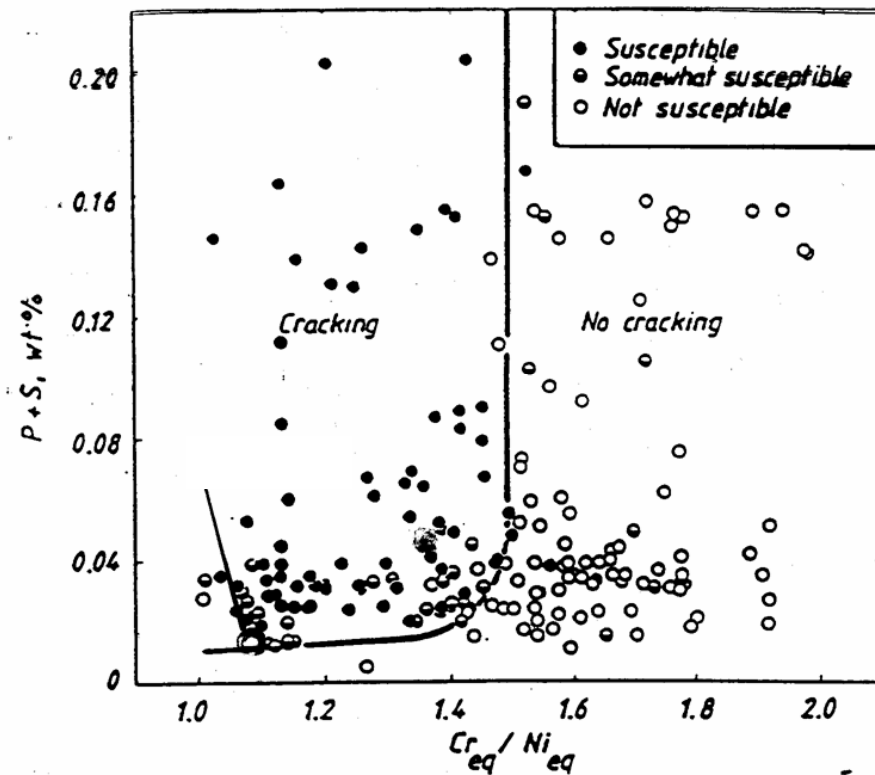
Dimensions and working conditions



- **depth : 194 mm**
- **diameter : 580 mm**
- **wall thickness : 12 mm**
- **weight : approx. 50 kg**

- **leak tight to gaseous He at 300 K under 2.6 MPa (*test pressure*)**
- **leak tight to superfluid He at 1.9 K under 0.13 MPa (*operating pressure*)**
- **25 thermal cycles 1.9 K \Rightarrow 300 K \Rightarrow 1.9 K (*over 20 years*)**

DIAGRAMME DE SUUTALA (Établi pour le procédé TIG)



$$\frac{Cr_{eq.}}{Ni_{eq.}} = \frac{Cr + Mo + 1,5Si + 0,5Nb}{Ni + 30C + 0,5Mn}$$

- 1



ontaneous

McHenry H.I., The Properties of Austenitic Steels at Cryogenic Temperature

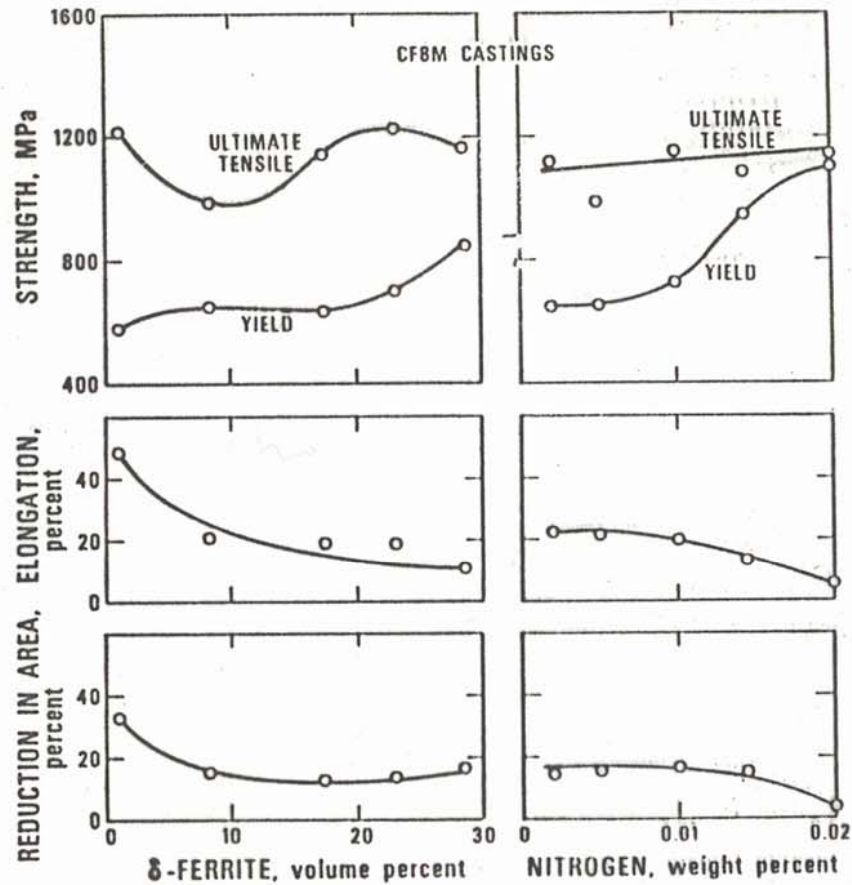


Fig. 18. The effect of δ -ferrite and nitrogen content on the tensile properties at 4 K of CF8M austenitic stainless steel castings. The castings in the δ -ferrite study contained 0.05% N and the castings in the nitrogen study contained $9 \pm 1\%$ δ -ferrite.³¹

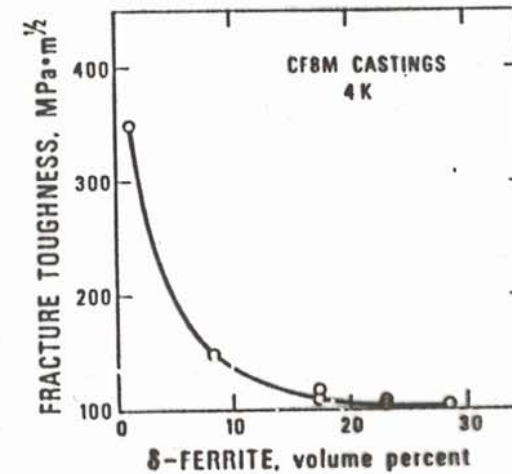


Fig. 19. The effect of δ -ferrite on the 4-K fracture toughness of CF8M austenitic stainless steel castings containing 0.05% N.³¹



Requirements - 2



- **Low temperature ductility**

⇒ impact toughness 120 J/cm² at 4.2 K

- **Leak-tightness**

- **Homogeneity of properties within the thickness**

- **Compatible with its environment**

⇒ coefficient of thermal contraction \approx wrought 316LN



HIPed AISI 316LN end covers for CERN LHC project (courtesy of Metso)



Main manufacturing stages

- **inert gas atomization of AISI 316LN powder**
- **capsule manufacture**
- **powder filling, evacuation and sealing**
- **hot isostatic pressing (1180 °C, 100 MPa, 4 h)**
- **can removal by pickling**
- **heat treatment (1060 °C, 2 h, water quenching)**
- **machining**
- **NDT by 100 % ultrasonic and liquid penetrant inspection**





HIPed AISI 316LN end covers for CERN LHC project (courtesy of Metso)



**After capsule
removal by
pickling and heat
treatment, before
machining**





HIPed AISI 316LN end covers for CERN LHC project (courtesy of Metso)



Machined

COMPONENTA

Componenta Nisamo Oy: 3D measurements, from AT-MAS/FB 7153 visit report (19/08/03)





HIPed AISI 316LN end covers for CERN LHC project (courtesy of Metso)



**Liquid penetrant
inspection**





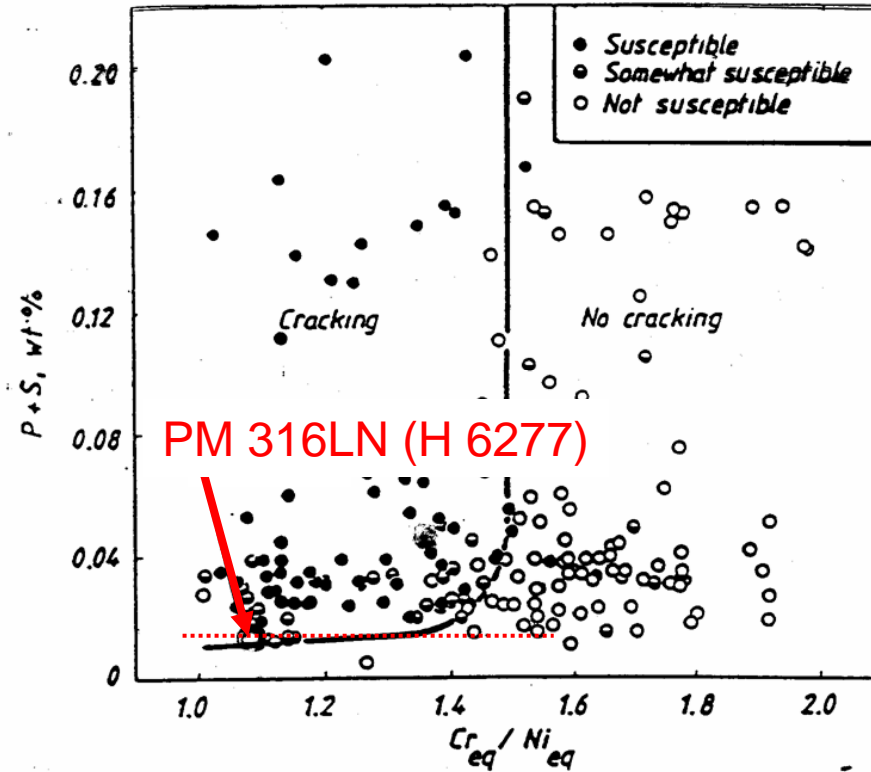
HIPed PM 316LN		Metso	CERN Specification	
		<i>H 6277</i>	<i>Min.</i>	<i>Max.</i>
<i>Composition (w%)</i>	C	0.017		0.030
	Si	0.59		1.00
	Mn	0.71		2.00
	S	0.005		0.015
	P	0.012		0.040
	Ni	13.07	12.00	14.00
	Cr	16.98	16.00	18.00
	Mo	2.53	2.00	3.00
	O	0.011		
	N	0.185	0.15	0.20

Typical Oxygen levels

<i>in 316LN:</i>	Couturier et al. (1998)	200 ppm
	Dellis et al. (1996)	195 ppm
<i>in 304L:</i>	Appa Rao and Kumar. (1997)	400 ppm
<i>in aust. SS</i>	Zou and Grinder (1982)	300 to 4500 ppm
<i>in 304L</i>	Dunkley (1981)	1200 to 7800 ppm



DIAGRAMME DE SUUTALA (Établi pour le procédé TIG)



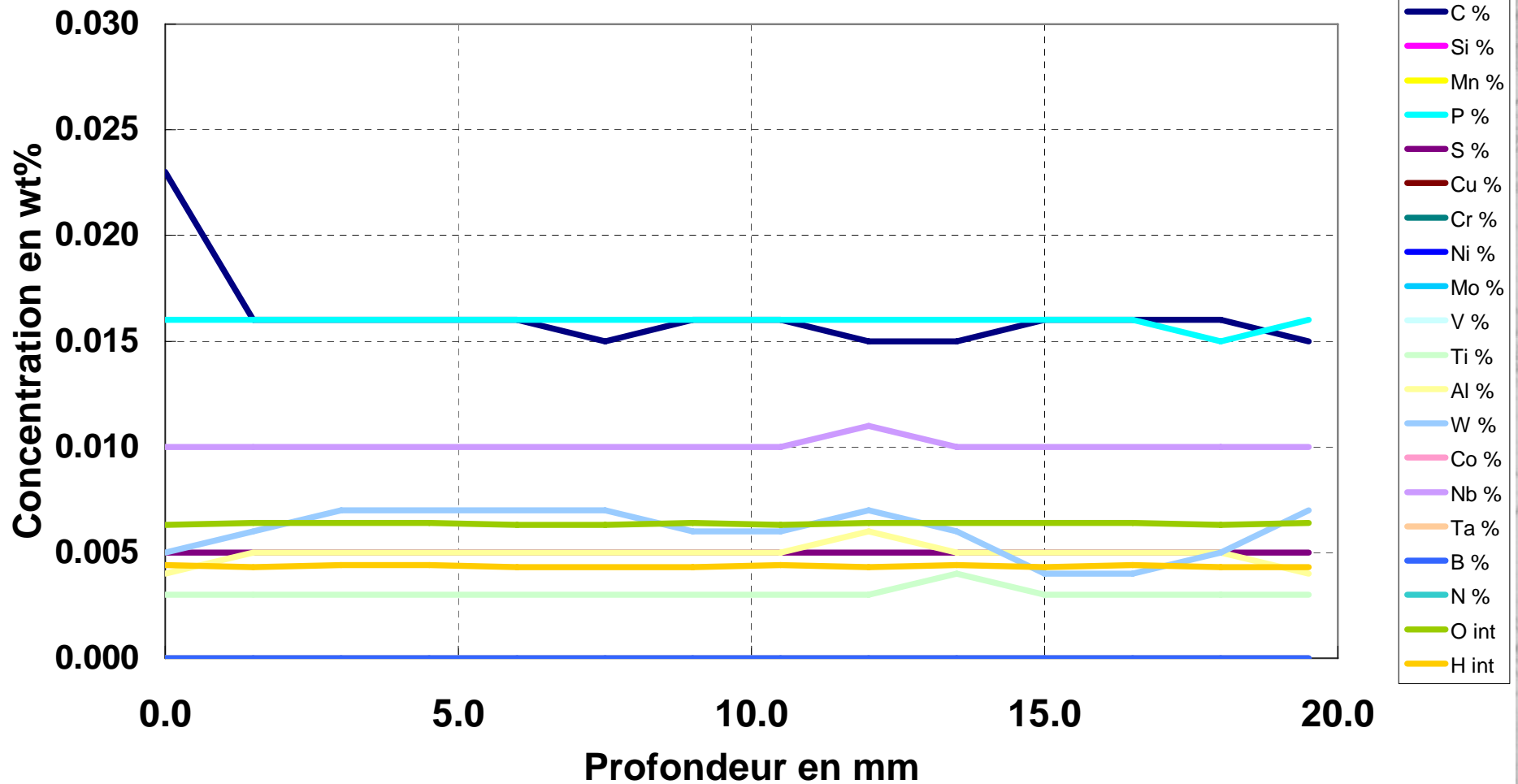
$$\frac{Cr_{eq.}}{Ni_{eq.}} = \frac{Cr + Mo + 1,5Si + 0,5Nb}{Ni + 30C + 0,5Mn}$$



GDOES, composition profile

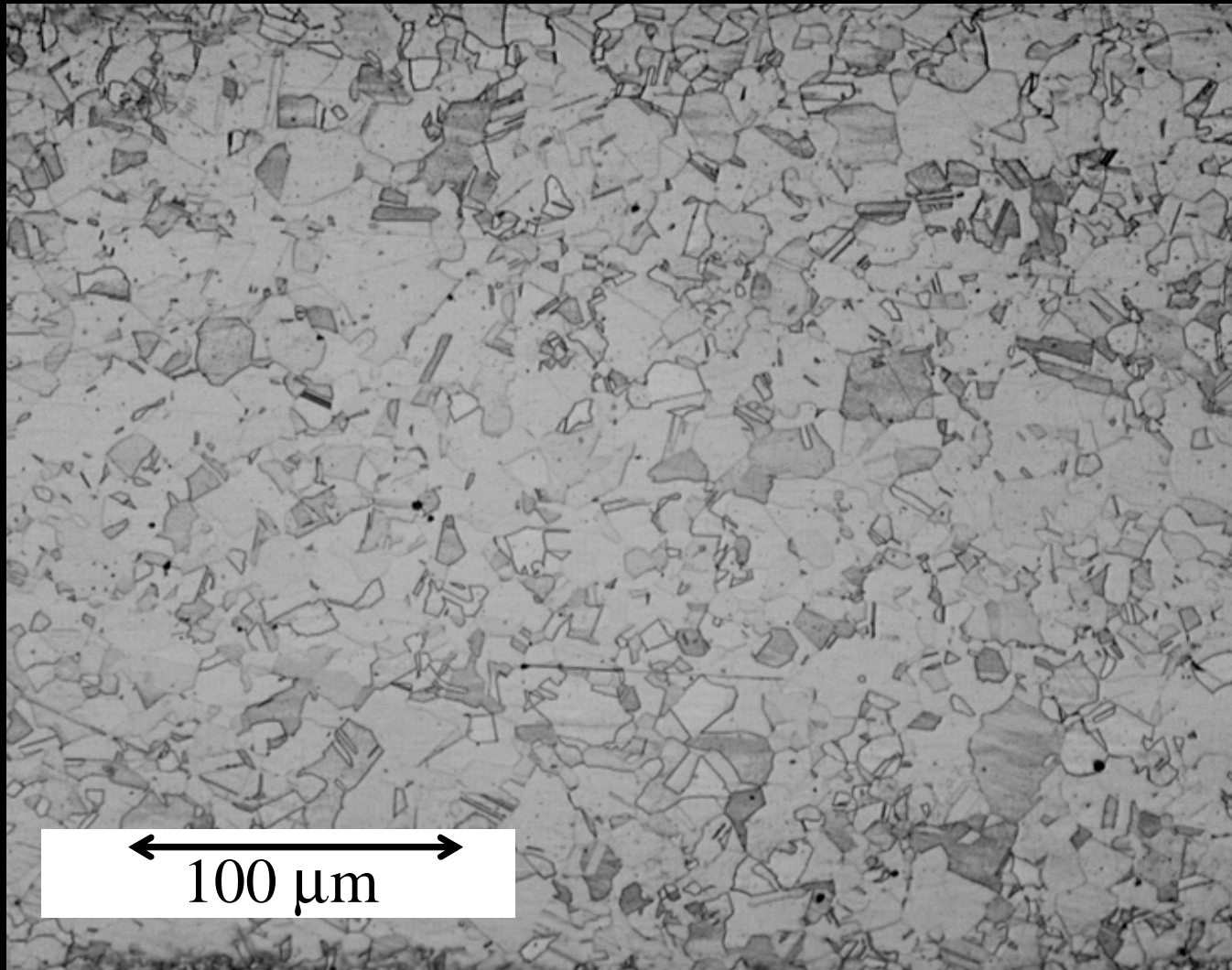


Echantillon GRAC





Microstructure

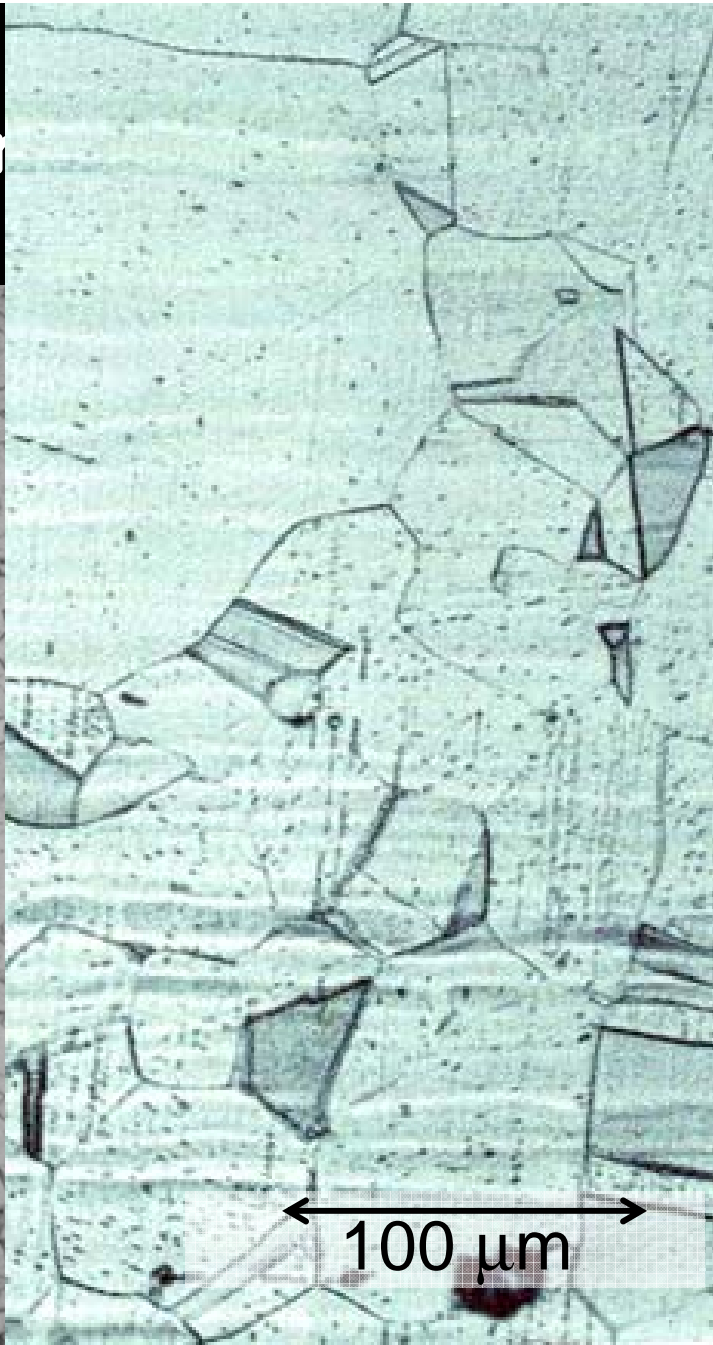
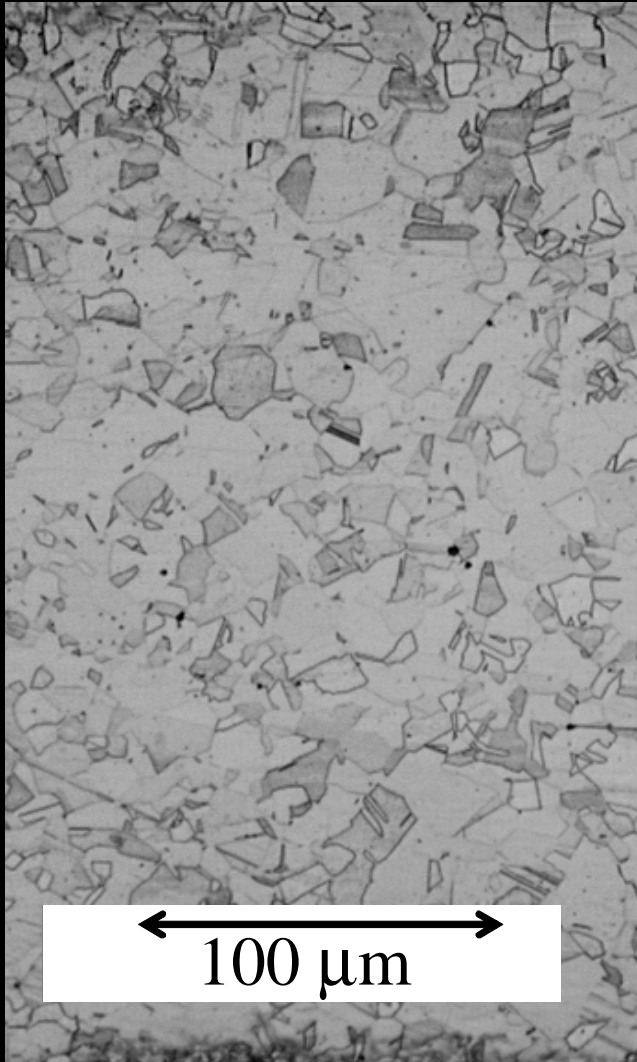


PM 316 LN – Metso, Grain size according to ASTM E112: N° 6 to 7



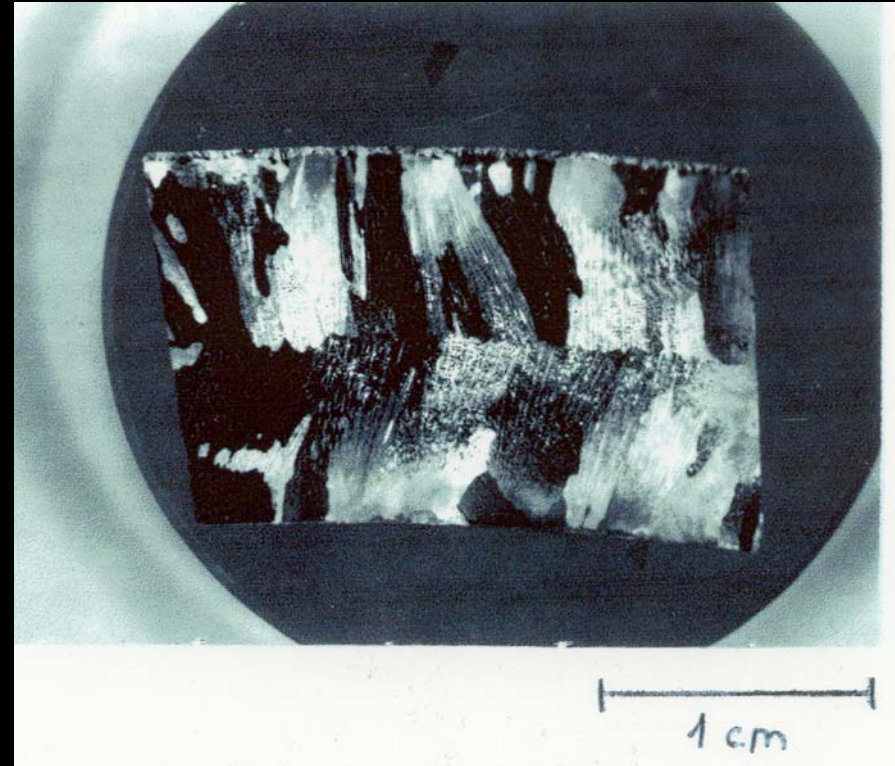
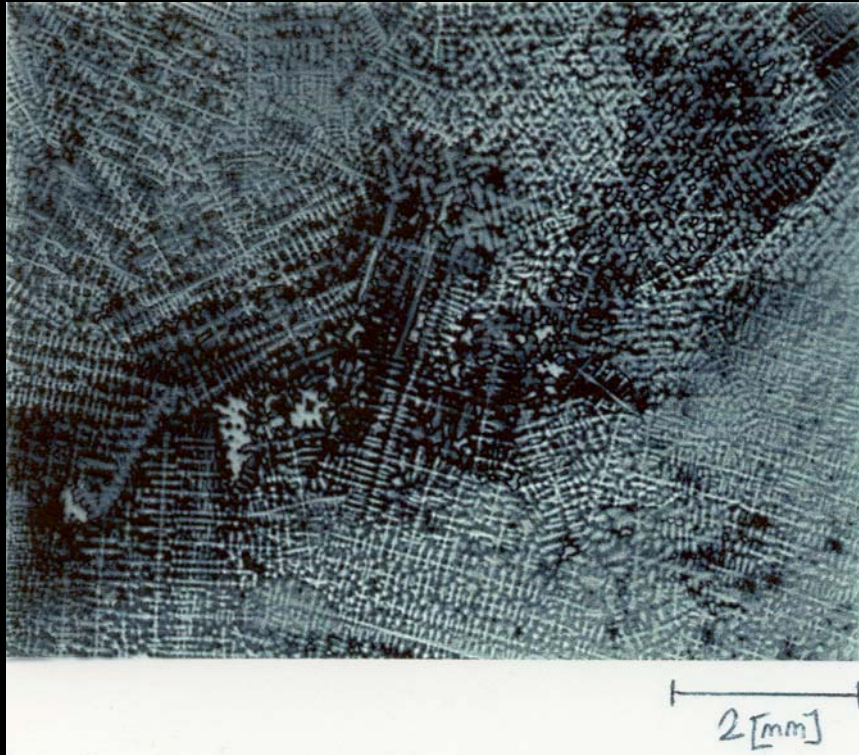
Microstr

1:





Microstructure, comparison 2: cast

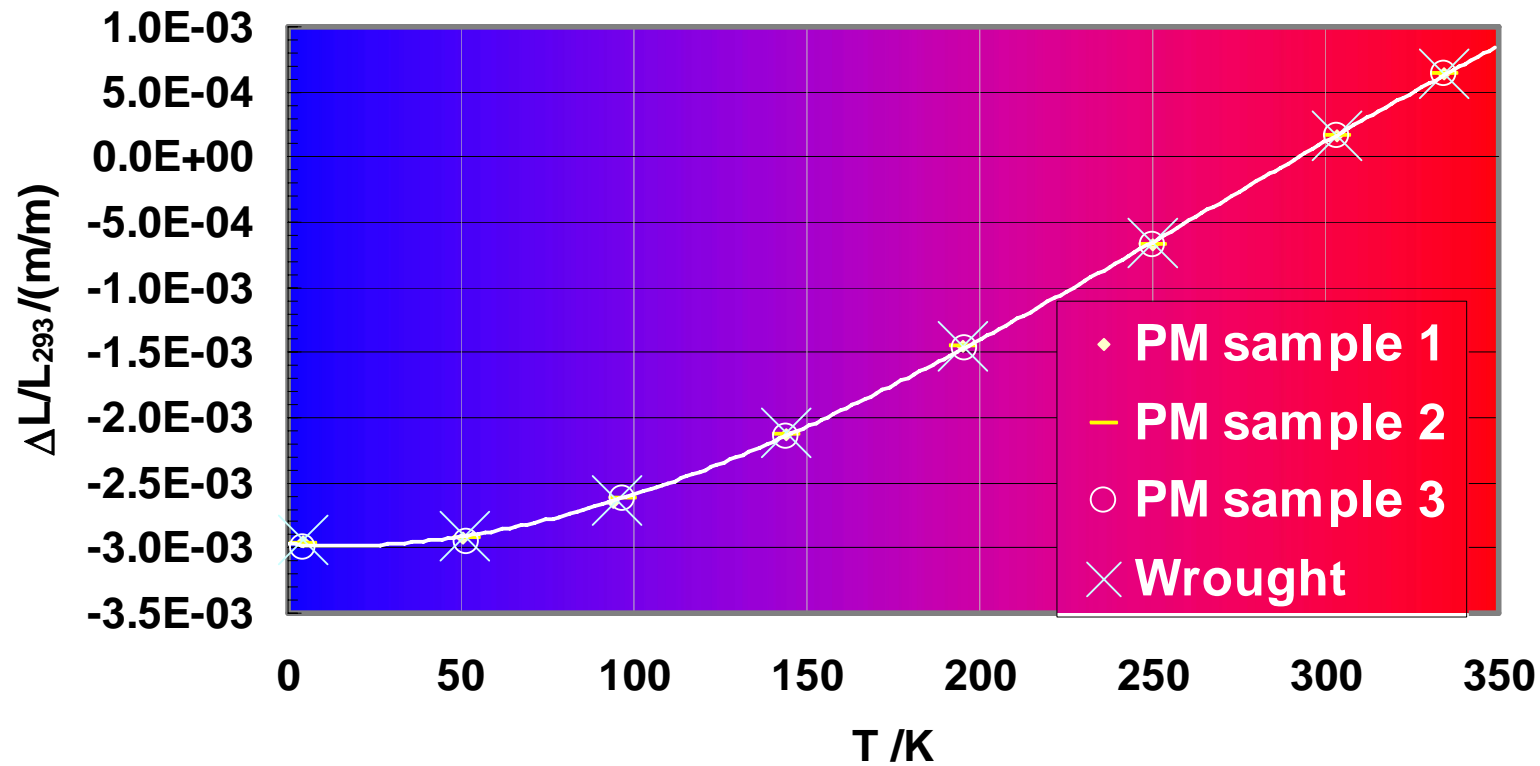




Thermal contraction

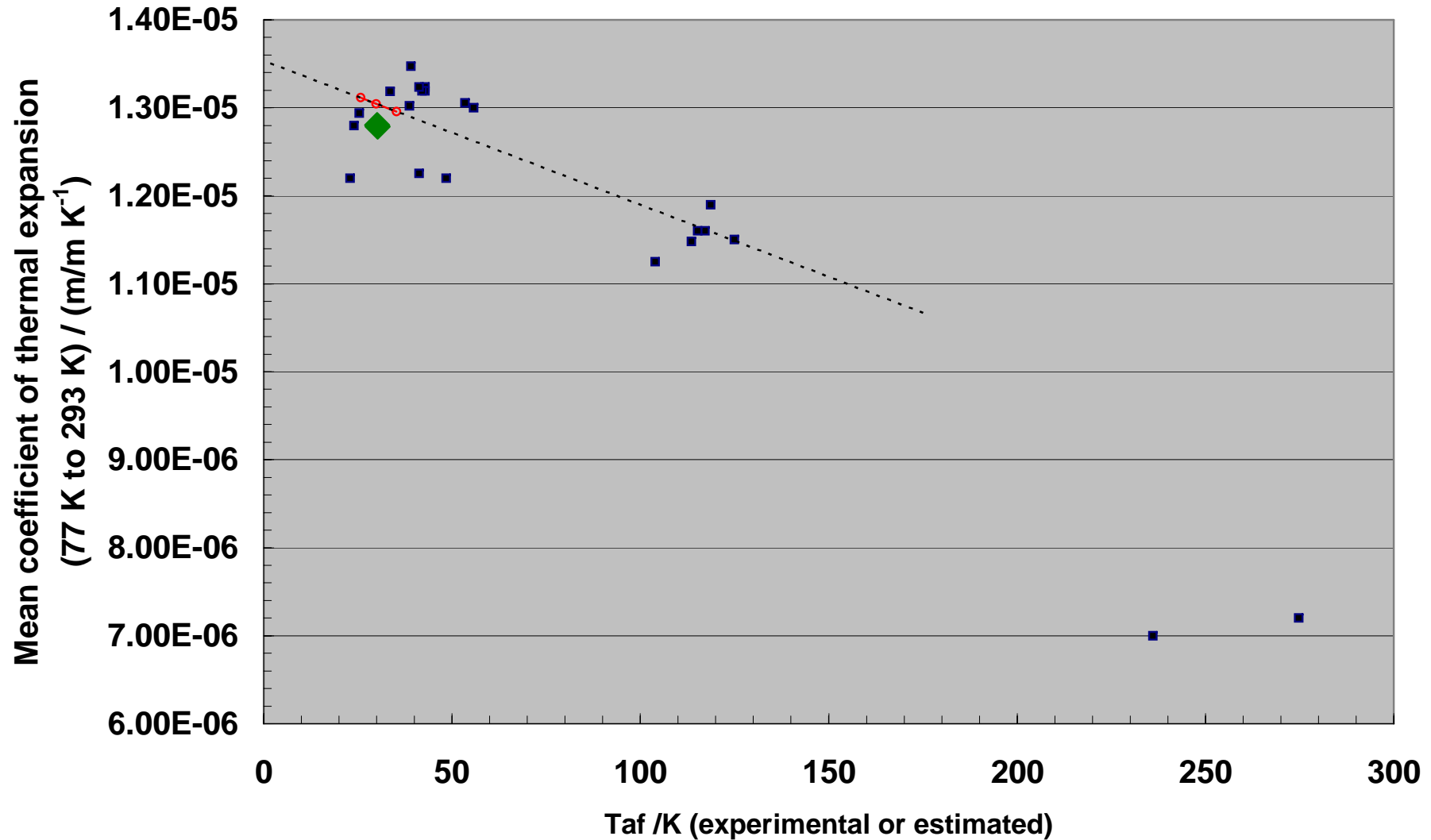


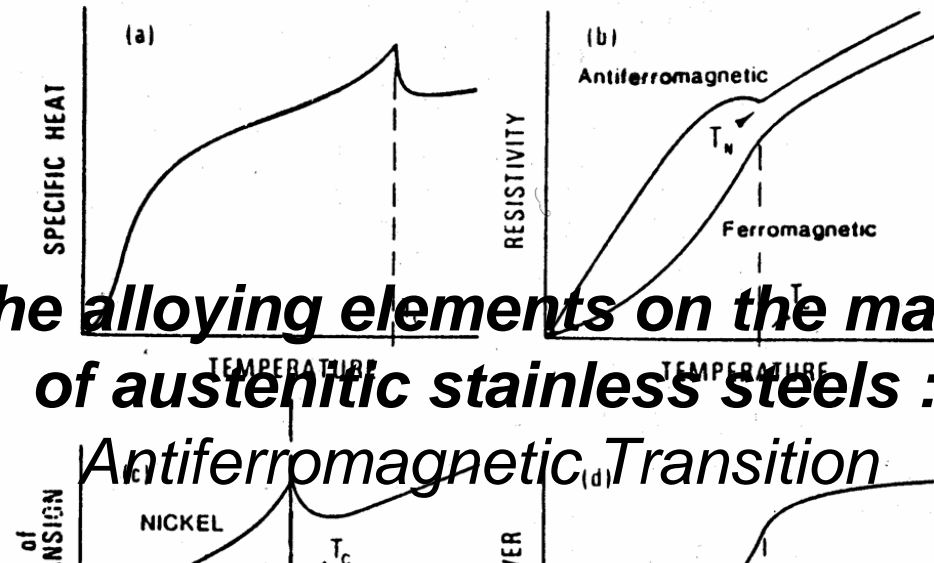
Compared thermal contraction
of wrought and PM 316 LN stainless steel





Thermal contraction

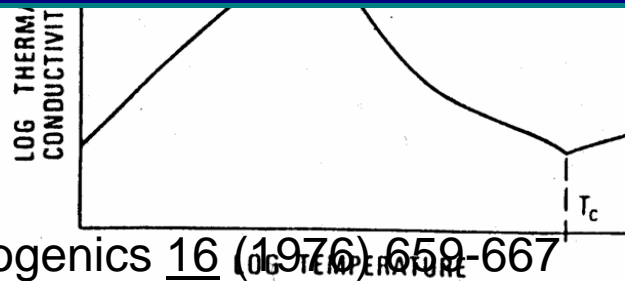




Influence of the alloying elements on the magnetic stability of austenitic stainless steels : Antiferromagnetic Transition

“Warnes equation”⁽¹⁾

$$T_{af} / K = 90 - 1.25 Cr - 2.75 Ni - 5.5 Mo - 14 Si + 7.75 Mn$$



(1) : Warnes et al., Cryogenics 16 (1976) 659-667

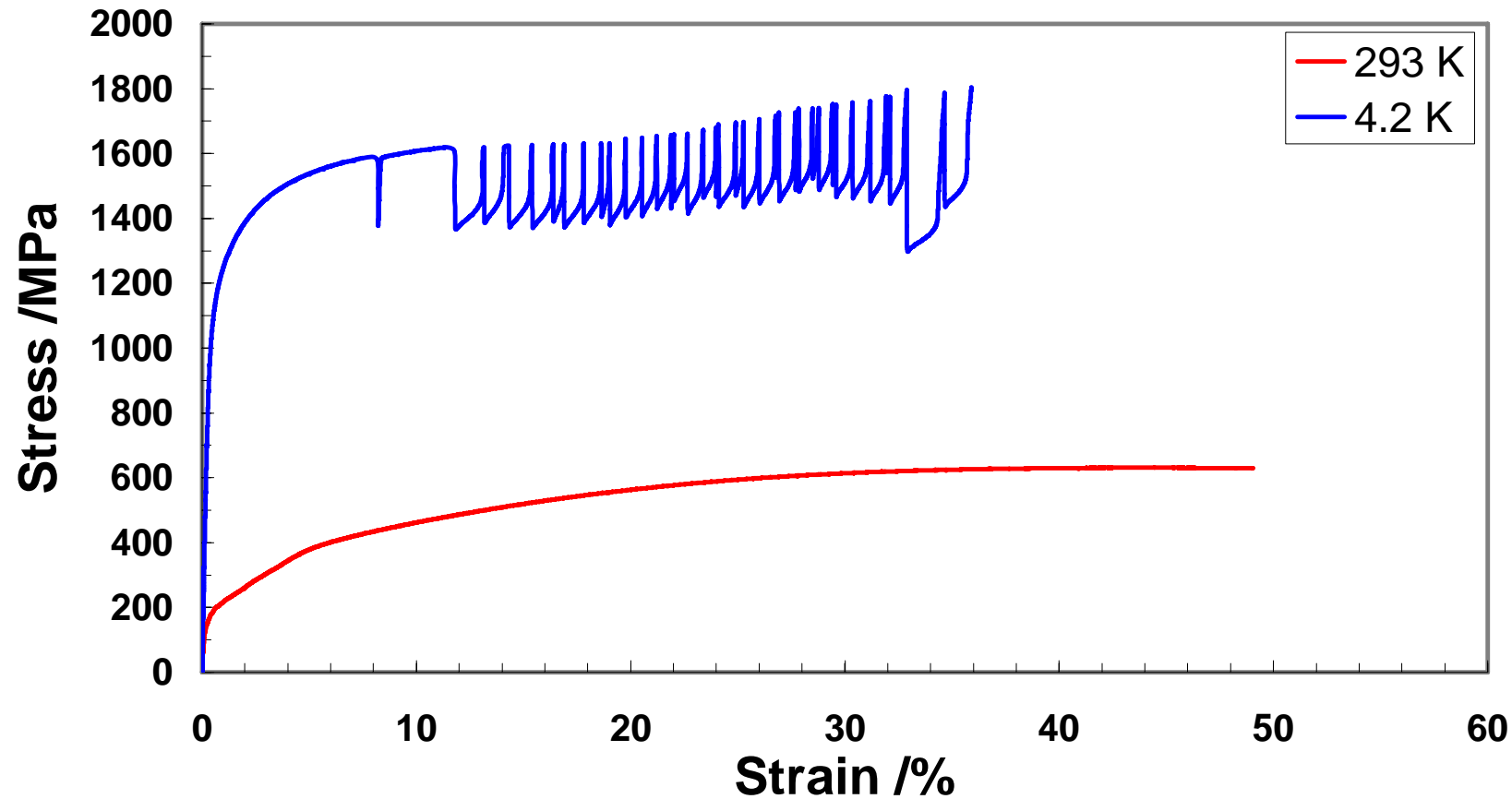
Figure 6.16 The effect of ferro- and antiferromagnetic transitions on a number of material properties.



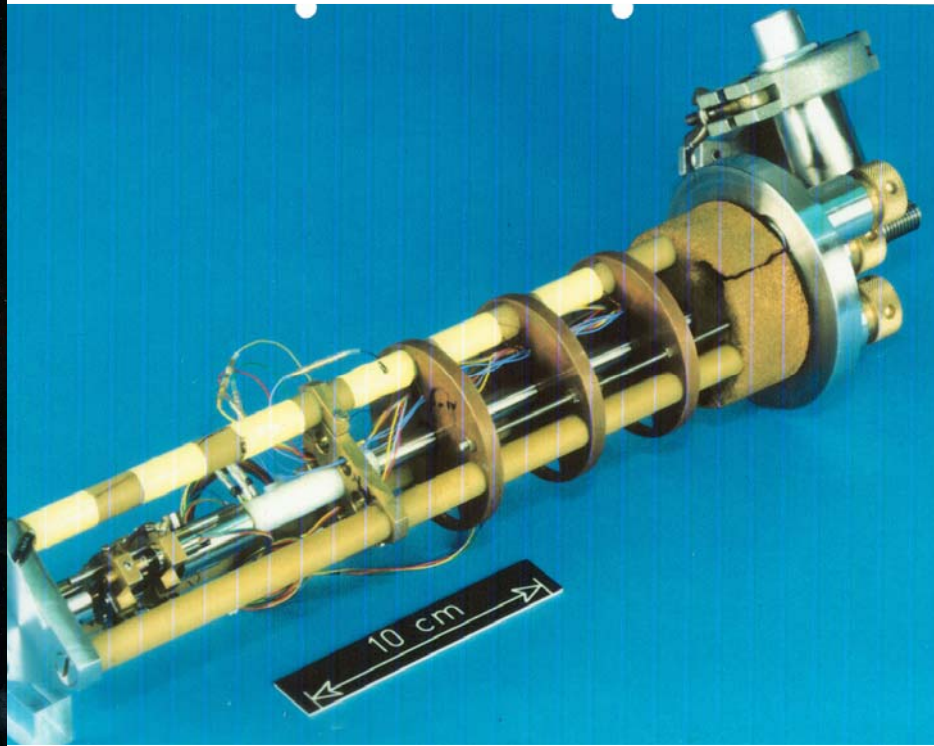
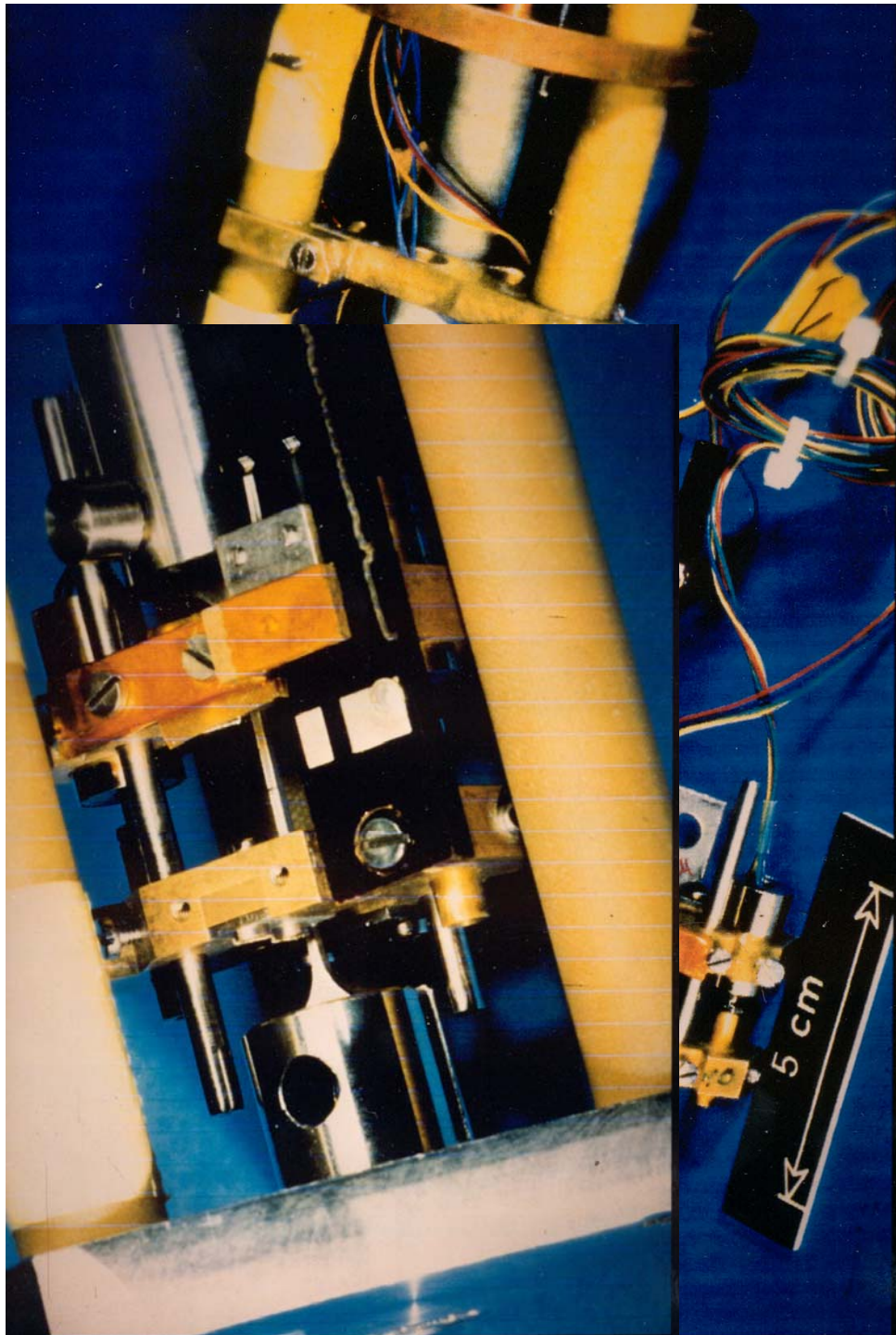
Mechanical properties: tensile



PM 316 LN
Tensile curves measured
at 293K and 4.2K

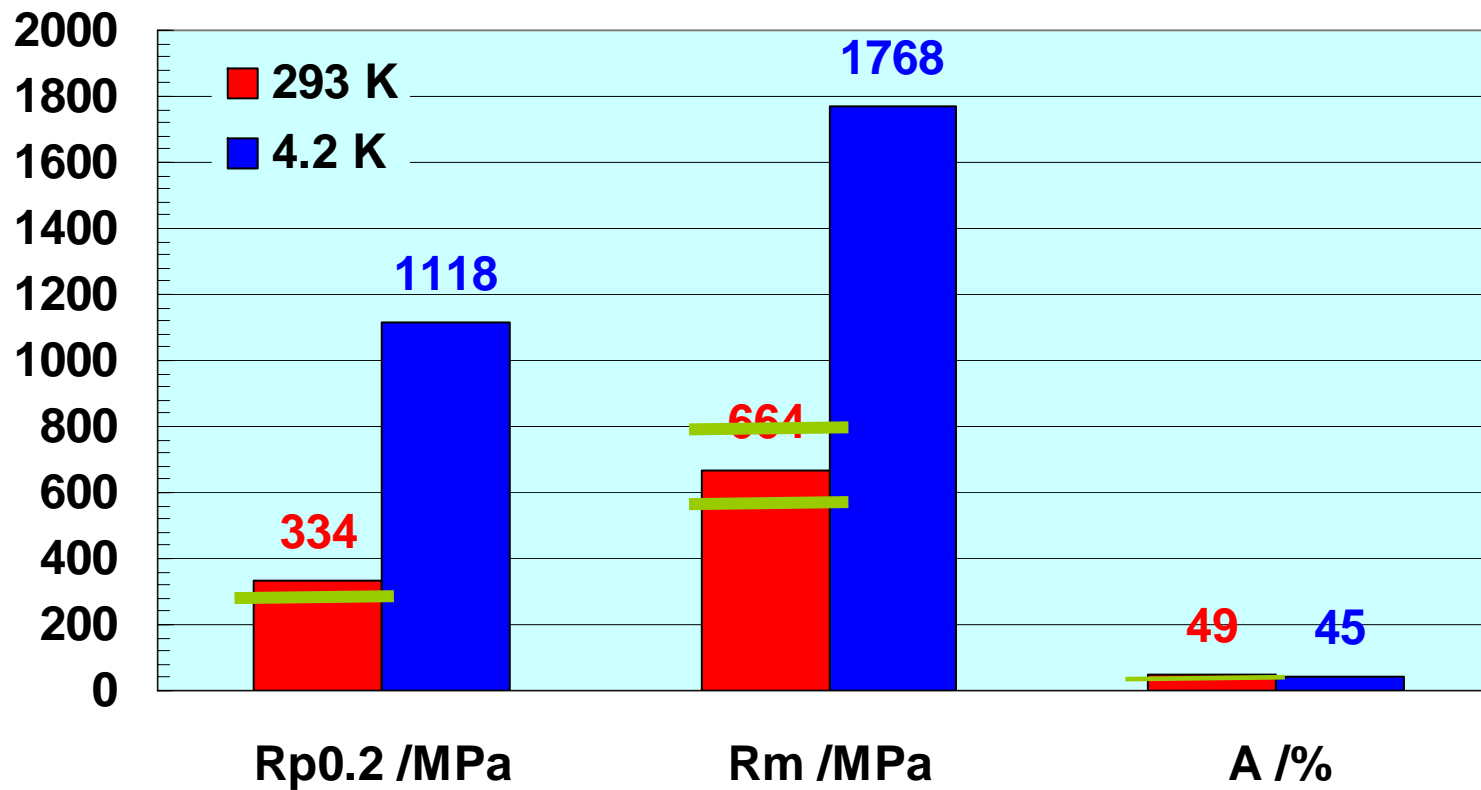


g at 4.2 K



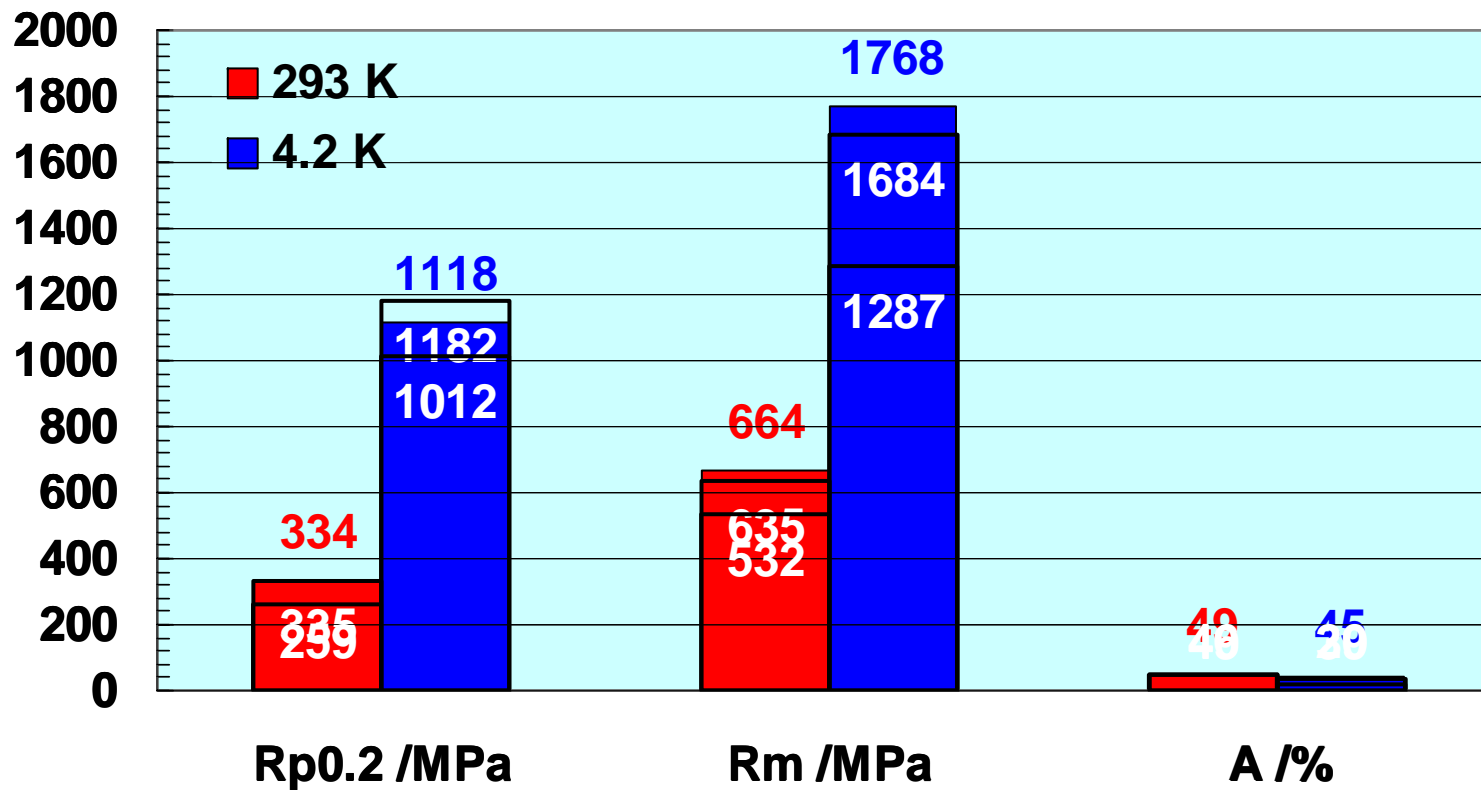


Compared tensile properties of PM 316 LN at 293 K and 4.2 K



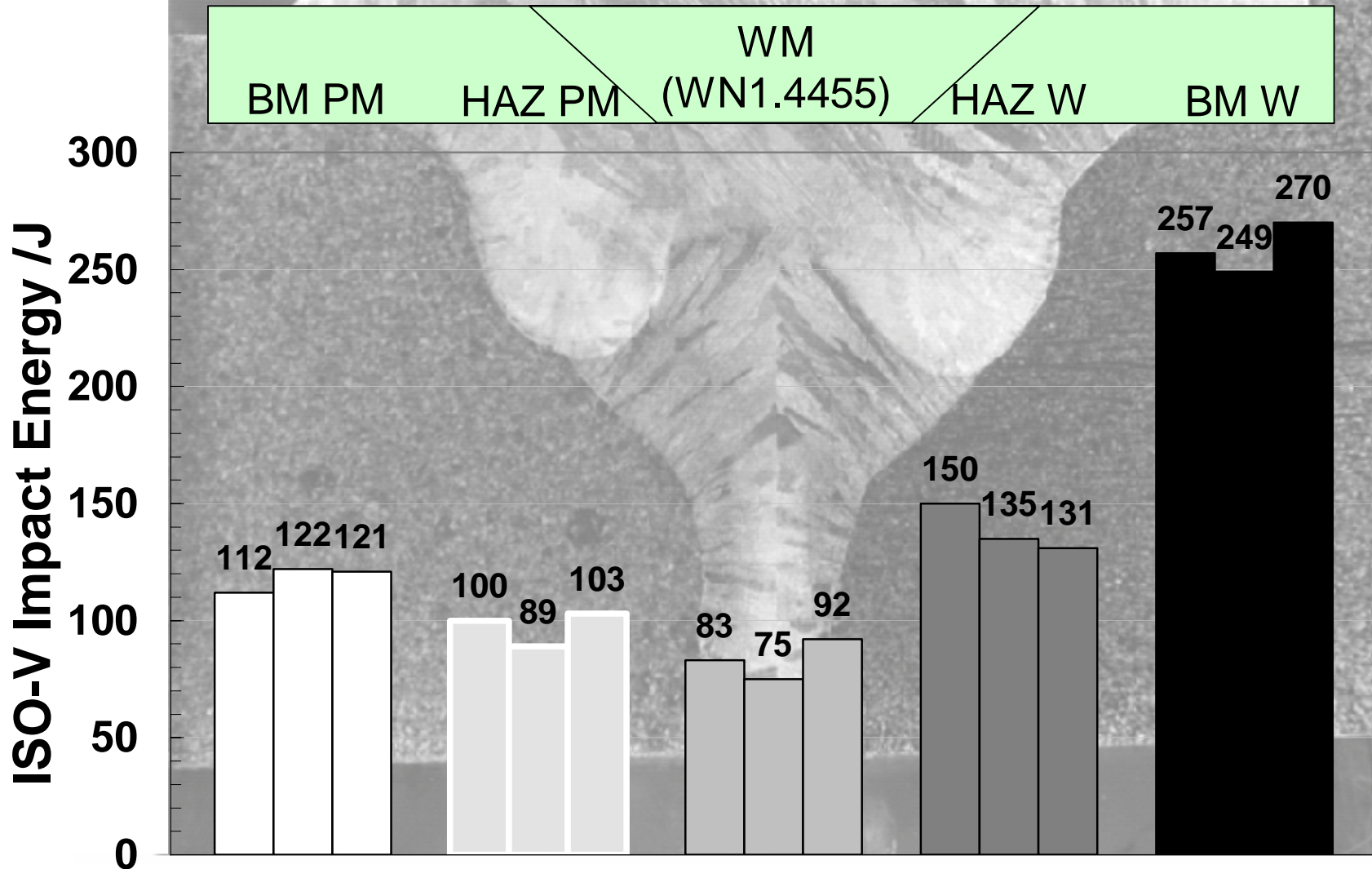


Compared tensile properties of EN 1616 NL at 293 K and 4.2 K





ISO-V impact energy at 4.2 K

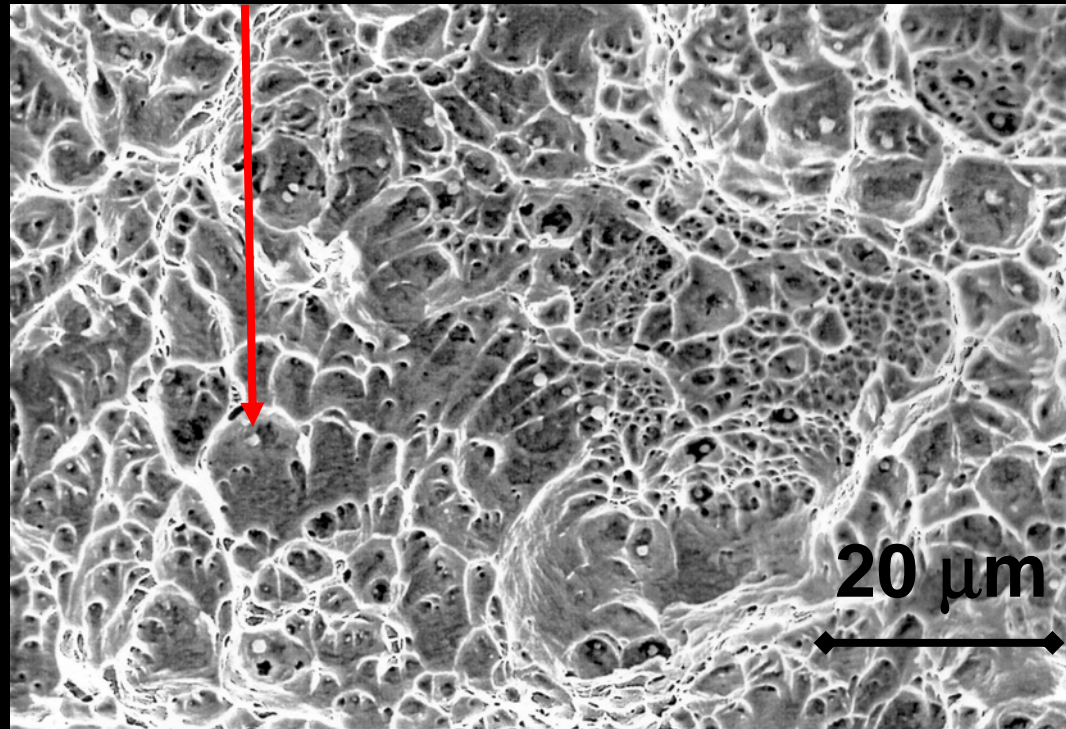




Fractographic analysis

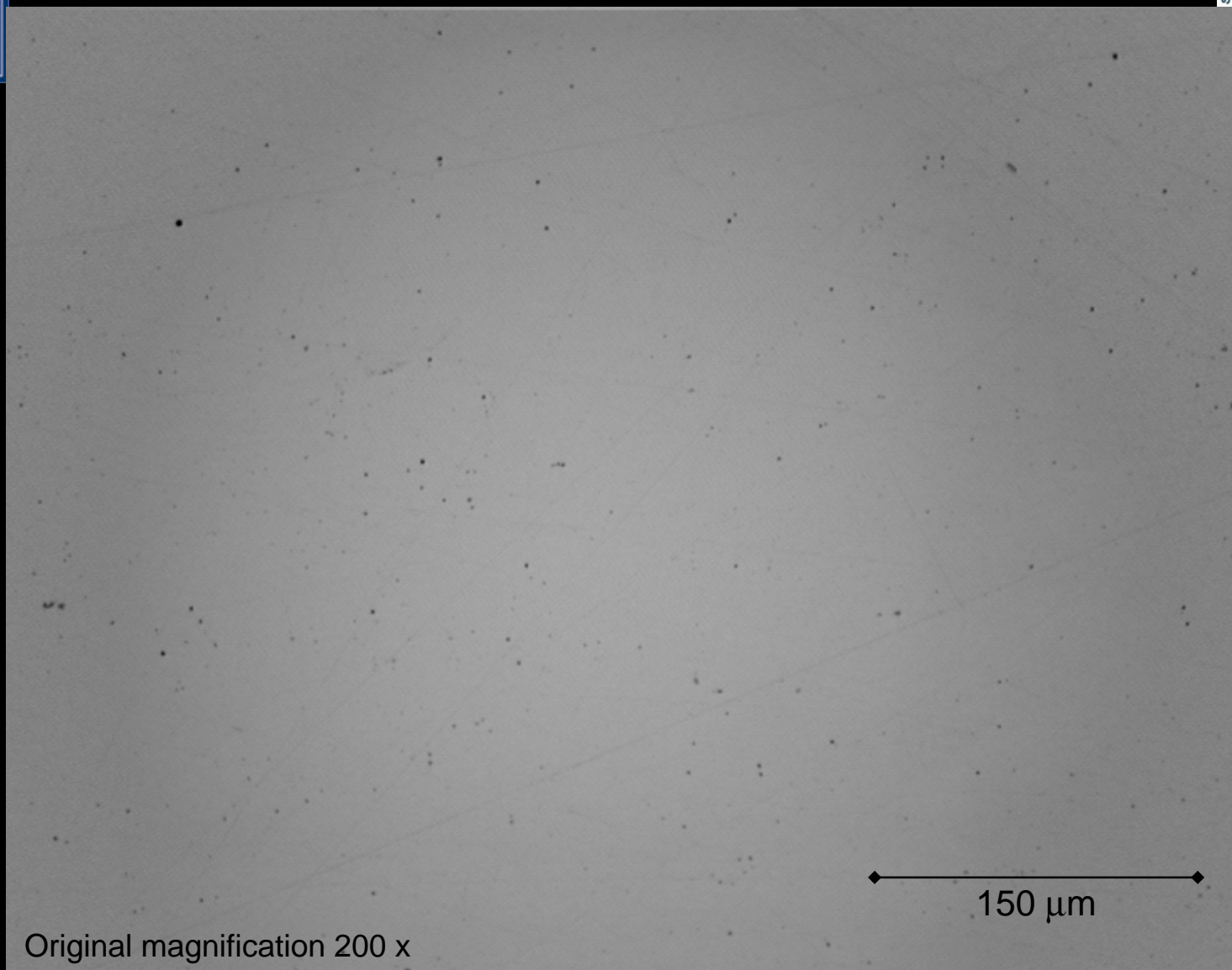


Oxides within dimples



Localized ductility (Couturier 99)

Inclusions



Original magnification 200 x

150 μm

Mainly globular-type



Standard Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis¹

This standard is issued under the fixed designation E 1245; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.



INTRODUCTION

This practice may be used to produce stereological measurements that describe the amount, number, size, and spacing of the indigenous inclusions (sulfides and oxides) in steels. The method may also be applied to assess inclusions in other metals or to assess any discrete second-phase constituent in any material.

1. Scope

1.1 This practice describes a procedure for obtaining stereological measurements that describe basic characteristics of the morphology of indigenous inclusions in steels and other metals using automatic image analysis. The practice can be applied to provide such data for any discrete second phase.

NOTE 1—Stereological measurement methods are used in this practice to assess the average characteristics of inclusions or other second-phase particles on a longitudinal plane-of-polish. This information, by itself, does not produce a three-dimensional description of these constituents in space as deformation processes cause rotation and alignment of these constituents in a preferred manner. Development of such information requires measurements on three orthogonal planes and is beyond the scope of this practice.

1.2 This practice specifically addresses the problem of producing stereological data when the features of the constituents to be measured make attainment of statistically reliable data difficult.

1.3 This practice deals only with the recommended test methods and nothing in it should be construed as defining or establishing limits of acceptability.

1.4 The measured values are stated in SI units, which are to be regarded as standard. Equivalent inch-pound values are in parentheses and may be approximate.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- E 3 Methods of Preparation of Metallographic Specimens²
- E 7 Terminology Relating to Metallography²

¹ This practice is under the jurisdiction of ASTM Committee E-4 on Metallography and is the direct responsibility of Subcommittee E04.14 on Quantitative Metallography.

Current edition approved Jan. 15, 1995. Published March 1995. Originally published as E 1245 – 88. Last previous edition E 1245 – 89.

² Annual Book of ASTM Standards, Vol 03.01.

E 45 Test Methods for Determining the Inclusion Content of Steel²

E 768 Practice for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel²

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this practice, see Terminology E 7.

3.2 Descriptions of Terms:

3.2.1 *detected feature*—the oxide, sulfide, or other second-phase constituent of interest that is isolated for measurement by adjustment of the threshold setting to its particular range of gray level.

3.2.2 *exogenous inclusions*—those inclusions that arise from entrapment of foreign matter within the ingot and are distributed in a nonuniform, unpredictable manner.

3.2.3 *feature-specific measurements*—individual measurement of each detected feature in the field of view.

3.2.4 *field measurements*—simultaneous measurement of all detected features in the field of view.

3.2.5 *licker method*—the procedure of alternating between the live video image and the detected image while altering the gray-level threshold range to establish the optimum discrimination and detection of the inclusions.

3.2.6 *gray level*—the range of neutral colors between white and black on the monitor screen that corresponds to the feature to be detected.

3.2.7 *indigenous inclusions*—those inclusions that arise from the natural precipitation of insoluble nonmetallic phases during or after solidification (sulfides) or from combination with the residual oxygen content before or during solidification (oxides) and are distributed throughout the ingot in a relatively predictable manner.

3.2.8 *lot*—a unit of material processed at one time and subjected to similar processing variables.

3.2.9 *morphology*—the shape and size of a microstructural phase or constituent.

3.2.10 *stereological methods*—the procedures used to characterize three-dimensional microstructural features based on measurements made on two-dimensional sectioning planes.



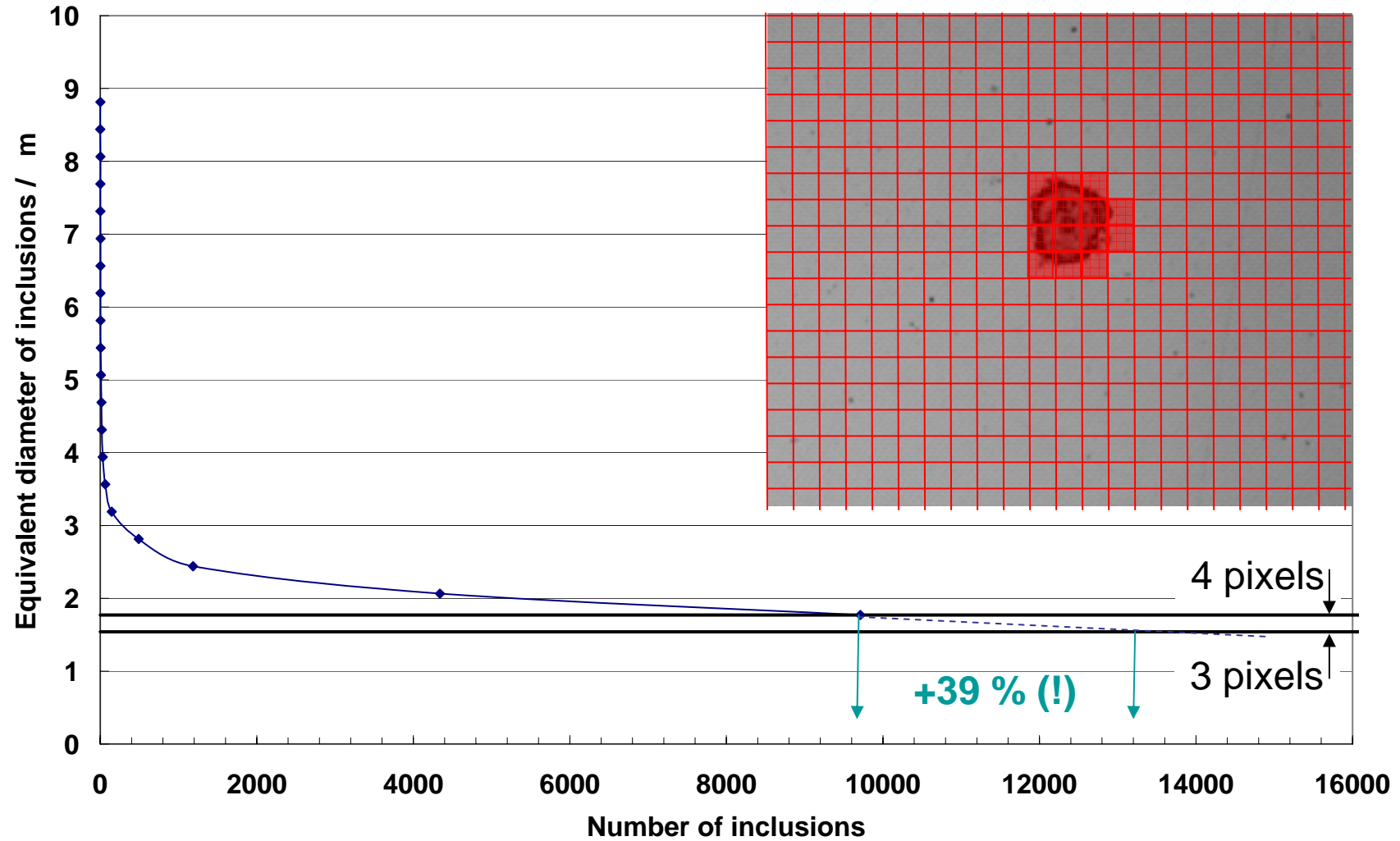
CERN specification (acc. to ASTM E1245-95)

	Symbol	Units	Max. allowed mean value	Max. allowed value in a single field
Area fraction of inclusions (mean value, value per field)	\bar{A}_A, A_A	%	0.20	7
Number of inclusions per unit area (idem)	\bar{N}_A, N_A	1/mm ²	230	1200
Average area of the inclusions (idem)	\bar{A}, \bar{A}	μm ²	20	2000
Mean free path of the inclusions (idem)	$\bar{\lambda}, \lambda$	μm	Min. 20	min. 8
Number of interceptions of inclusions per unit length of scan line (idem)	\bar{N}_L, N_L	1/mm	70	120
Diameter of the inclusions (idem) ¹⁾	none	μm	5	150 ²⁾

- 1) Additional required parameter not mentioned in ASTM E1245, defined as the inclusion equivalent diameter
- 2) Maximum allowed equivalent diameter of a single inclusion

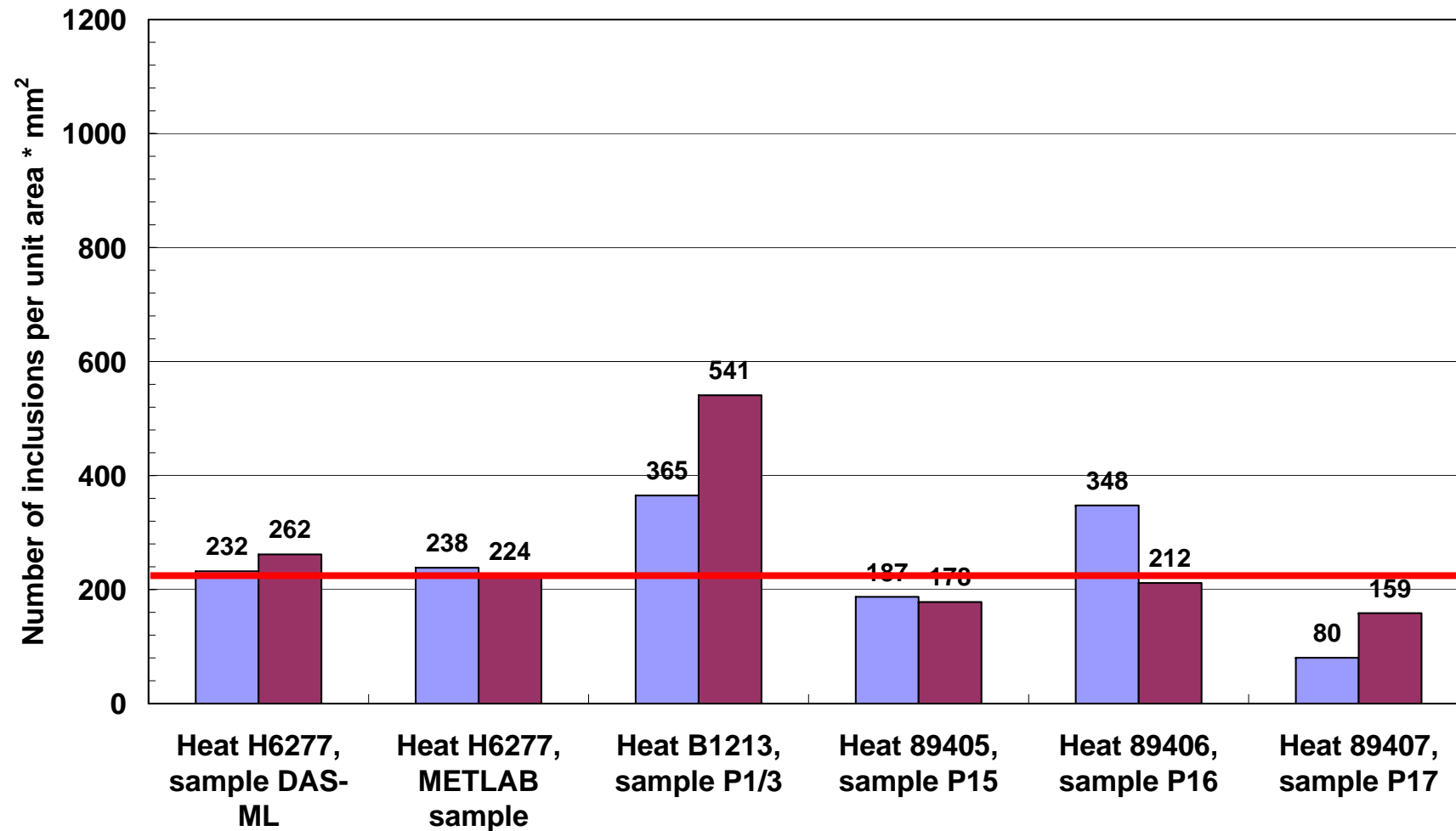


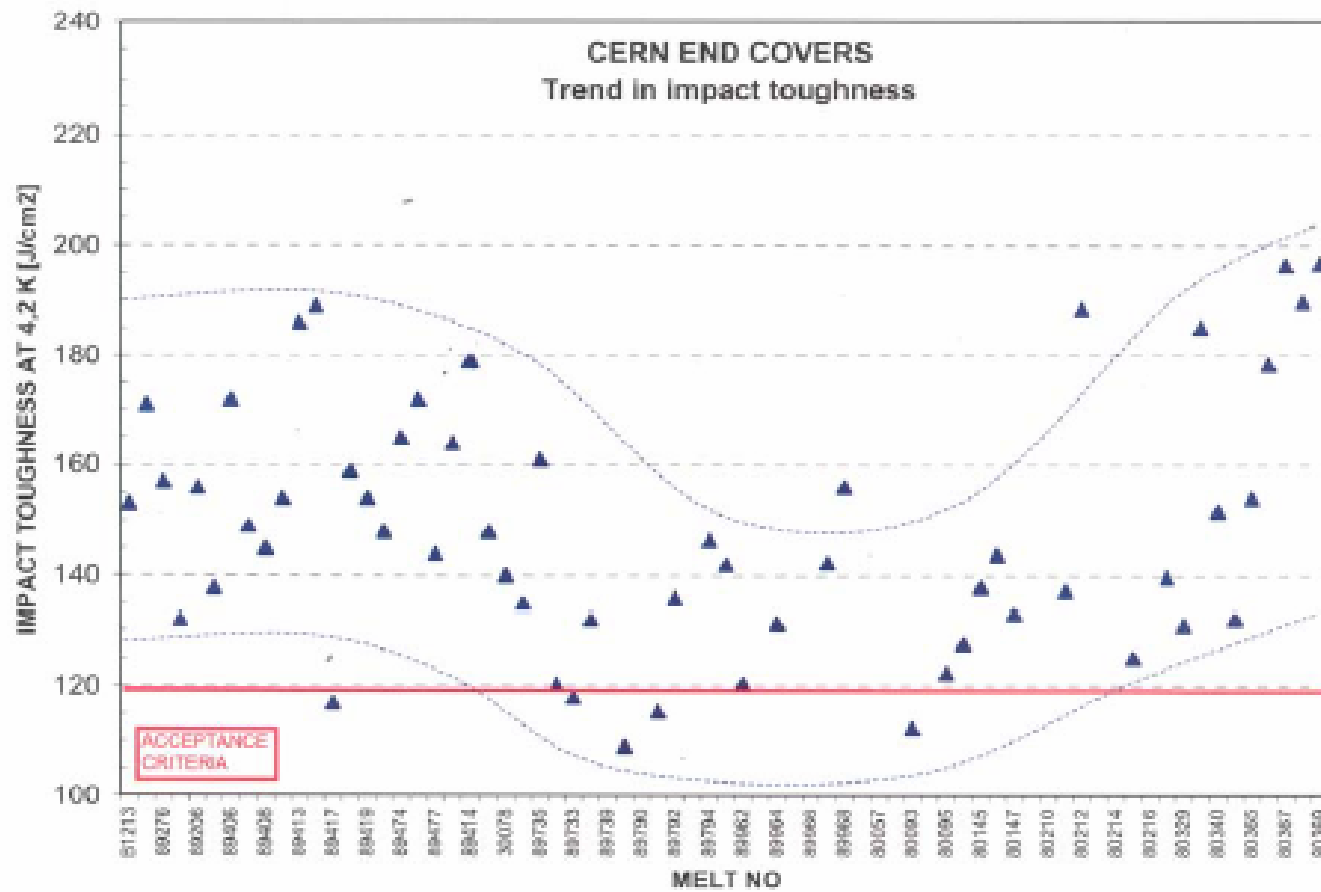
Distribution of the diameter of inclusions
(H6277, METLAB Sample, CERN measurement, threshold: 205)





Effect of threshold, CERN measurements.
Threshold 123 (205); Anal. On (Off); Min. accepted 4 pix.



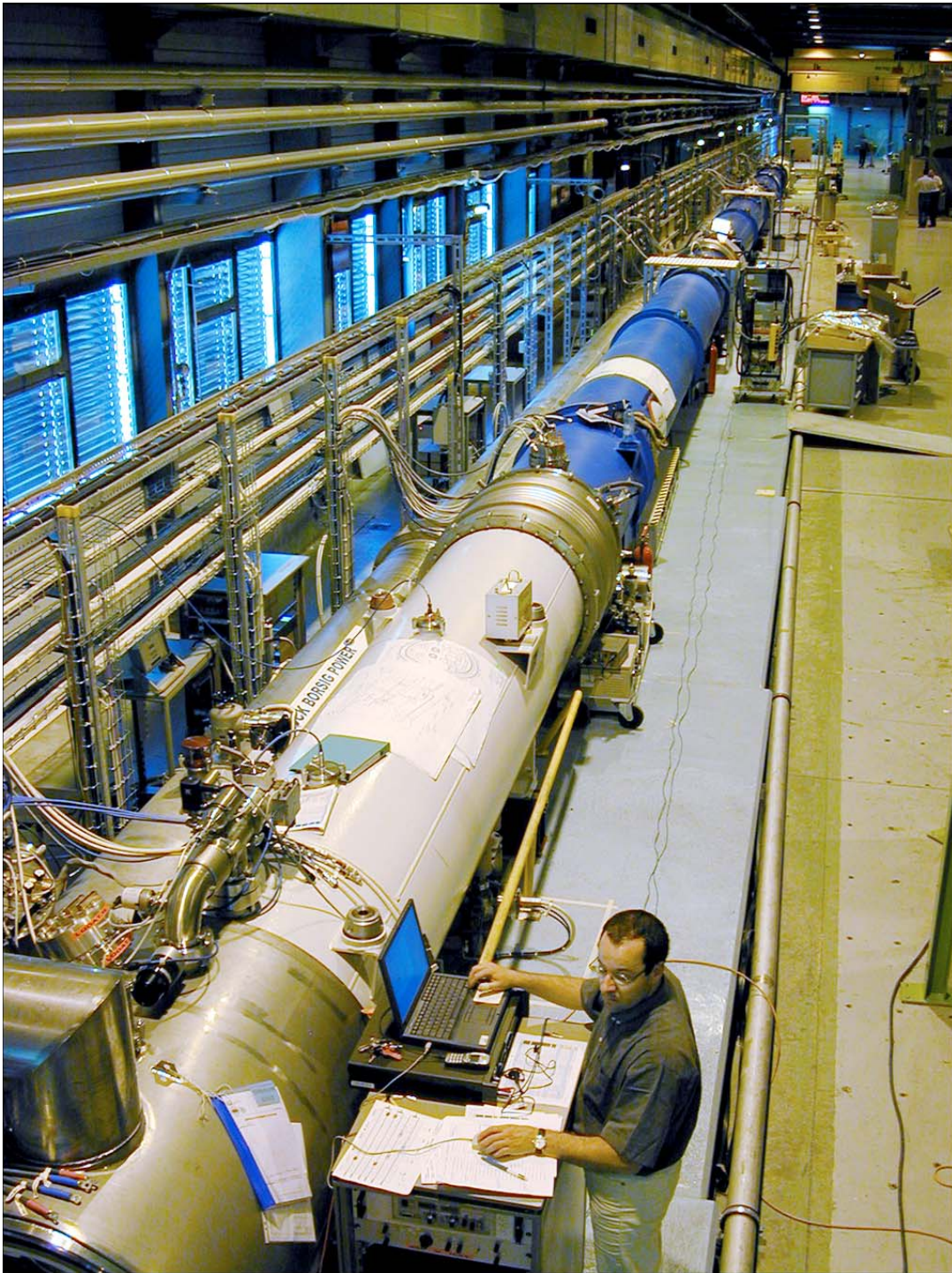




Magnet end covers

Compared advantages of possible fabrication techniques

	<i>welded</i>	<i>closed die forged</i>	<i>cast</i>	<i>PM</i>
<i>Microstructure</i>	-	++	-	++
<i>Tensile properties</i>	+	+	-	++
<i>Impact toughness at 4.2 K</i>	+	++	+	+
<i>Near net shaping</i>	++	--	+	++
<i>Reliability, NDT</i>	--	++	--	++
<i>Competitiveness, small series (tools)</i>	+	--	+	+
<i>Competitiveness, large series (tools)</i>	+	+	+	+



ons



ve technique

plex shape components
on, readily weldable

with PM covers
in the “string”







TRAIN DE LAMINEURS DANS UN LAMINOIR DE MONTIGNY-SUR-SAMBRE.

Dessin de Constantin Menner.

Concutiat motus, quis fulgura ducat hiatus,
 Unde torquent nubes, quo lumine floreat arcus;
 Hæc mihi quærenti, si quid deprendere veri
 Mens valet, expediat. Lapis est cognomine Magnes,
 Decolor, obscurus, vilis : non ille repexam
 Cæsariem regum, nec candida virginis ornat
 Colla, nec insigni splendet per cingula morsu :
 Sed nova si nigri videas miracula saxi,
 Tum pulchros superat cultus, et quidquid Eois
 Indus litoribus rubra scrutatur in alga.

C. L. F. PANCKOUCKE.

Exegi monumentum ære perennius.
(Hor., Od. lib. III, ode 30.)



demens de la terre, et, déchirant la nue, en fait jaillir
 l'éclair, et fait gronder la foudre; quelle lumière colore
 l'arc d'Iris. moi aussi, je cherche à résoudre ces grands
 problèmes; et, si votre esprit peut entrevoir la vérité,
 éclairez mes doutes. Il est une pierre noire, sombre et
 dédaignée: l'Aimant est son nom. Elle ne pare point la che-
 velure des Césars, ni le cou éblouissant de la jeune vierge;
 elle ne rattache pas d'une agrafe brillante la ceinture du
 guerrier; mais, si vous considérez les prodiges inconnus
 de cette pierre grisâtre, elle aura plus de prix à vos yeux
 que ces belles parures, que les richesses recueillies par
 l'Indien sur les grèves rougeâtres de l'océan Oriental.

TOME SECOND

PARIS

C. L. F. PANCKOUCKE, ÉDITEUR

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