INVESTIGATION OF MICROPOROSITY IN SINGLE-CRYSTAL NICKEL-BASE SUPERALLOYS BY DIFFERENT EXPERIMENTAL TECHNIQUES

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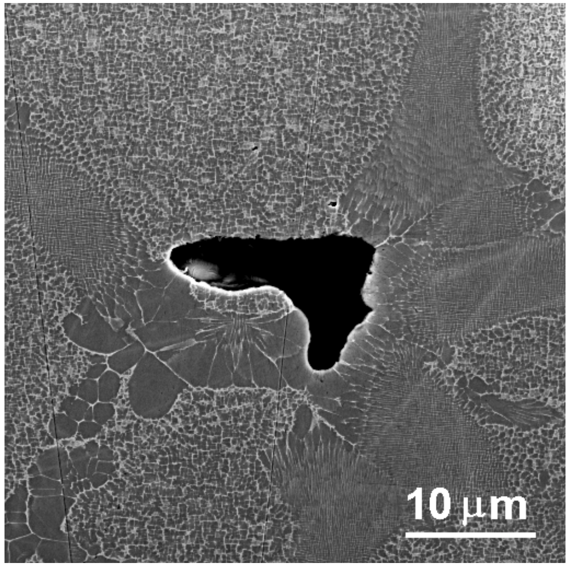
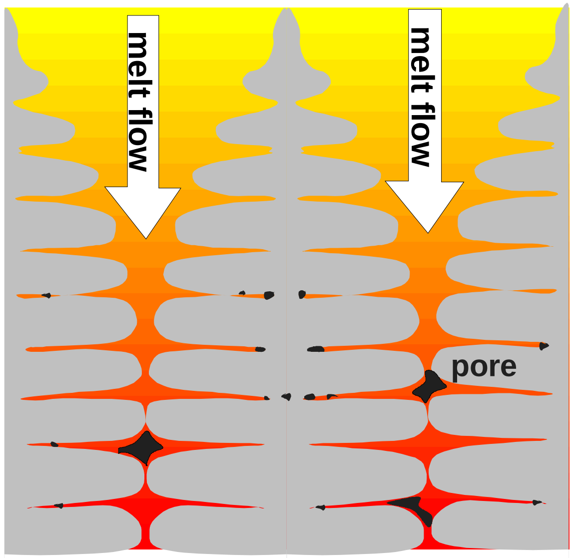
Porosity in single crystal superalloys can be classified into 3 different kinds of pores:

- **Solidification: S-pores**
- **Homogenisation: H-pores**
- **Creep deformation: D-pores**

Solidification → S-pores



CMSX-10, as cast



Pore formation mechanism

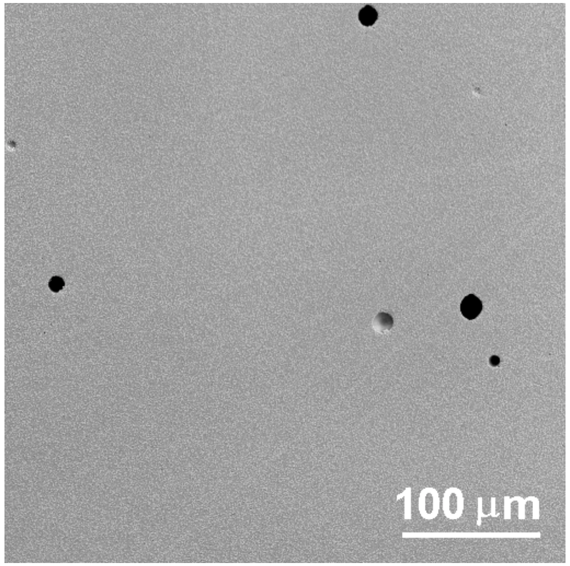
Dendritic growth

↓

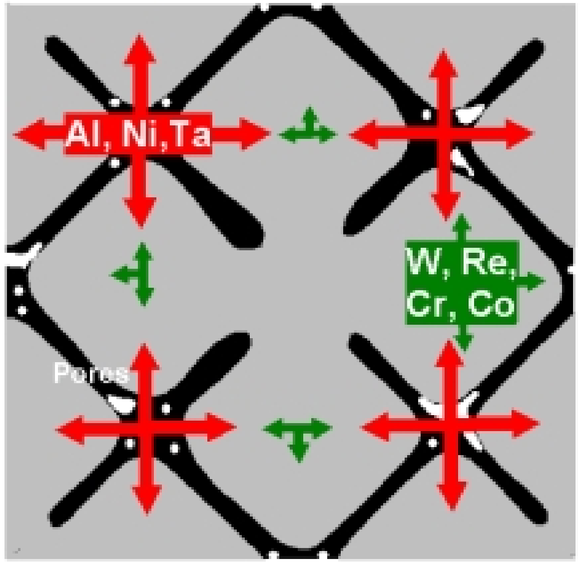
Shrinkage pores

in interdendritic regions

Homogenisation → H-pores



CMSX-10, heat treated (HT)



Diffusion fluxes during HT

Dendritic segregation

↓

Homogenisation

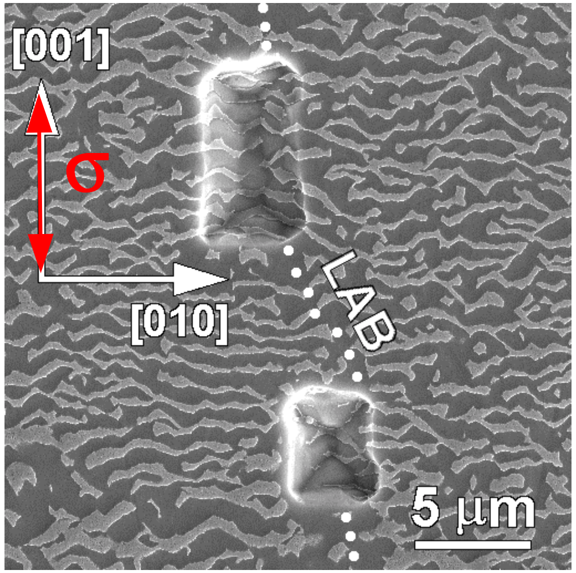
Different diffusion rates

↓

H-pore formation by

Kirkendall effect

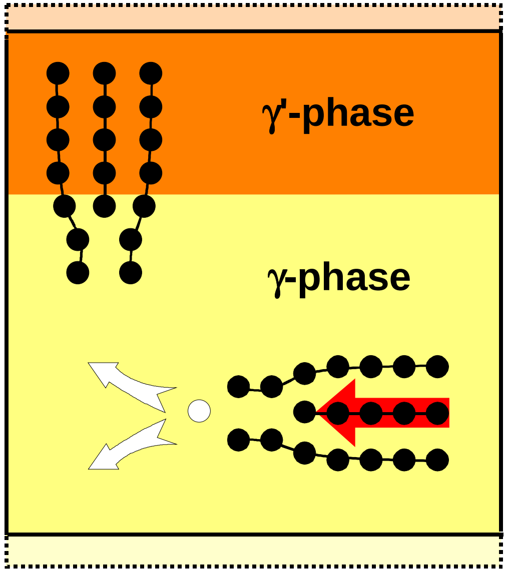
Creep deformation → D-pores



CMSX-10, 120 MPa, 654 h

Facetted pores at a

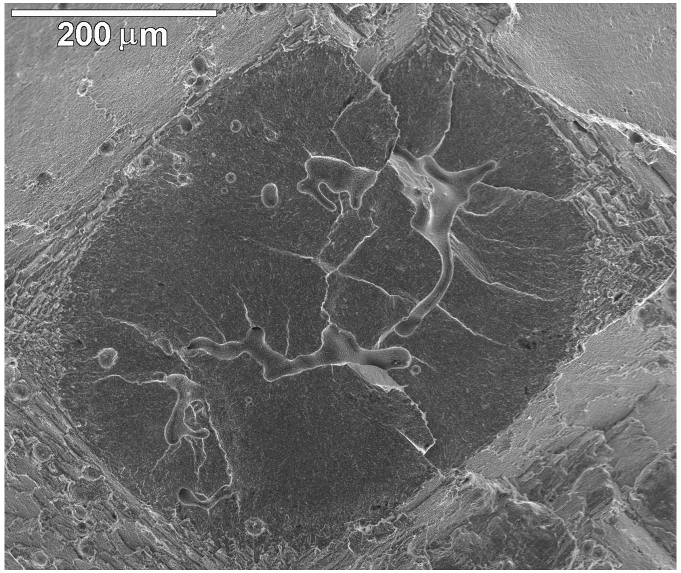
low angle boundary



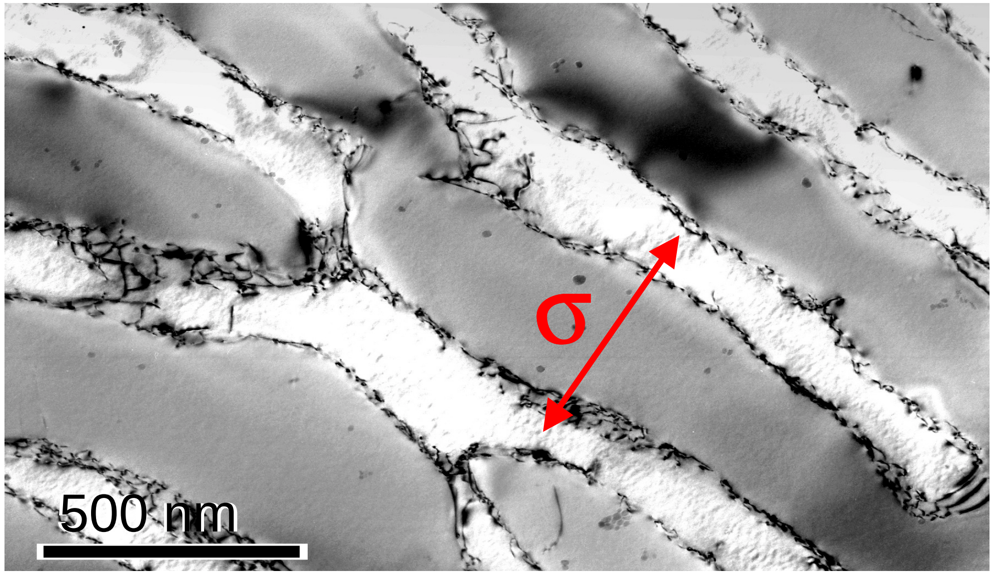
Mechanism of D-pore formation

HT-creep
↓
Formation of γ'-rafts
↓
Dislocations climb along the γ/γ'-interface
↓
Vacancy generation
↓
Growth of D-pores with crystallographic shape

Why is pore characterization important ?



Crack surface with C-pore
CMSX-4, LCF, 700 °C, $\Delta\varepsilon$ = 1.2%, R=-1



Dislocations in rafted γ/γ'-structure
SRR99, 980°C, 200 MPa, 115 h

- **S and H pores are important for the mechanical properties. → Fatigue behaviour.**
- **D pores are generated during creep. → Deformation mechanism.**

Why is pore characterization difficult ?

- **H and D pores are small (about 10 μm). → Difficult preparation.**

- **Small volume fraction of pores (0.1%→ 0.5%). → High accuracy. (Better than 0.01 absolute %)**

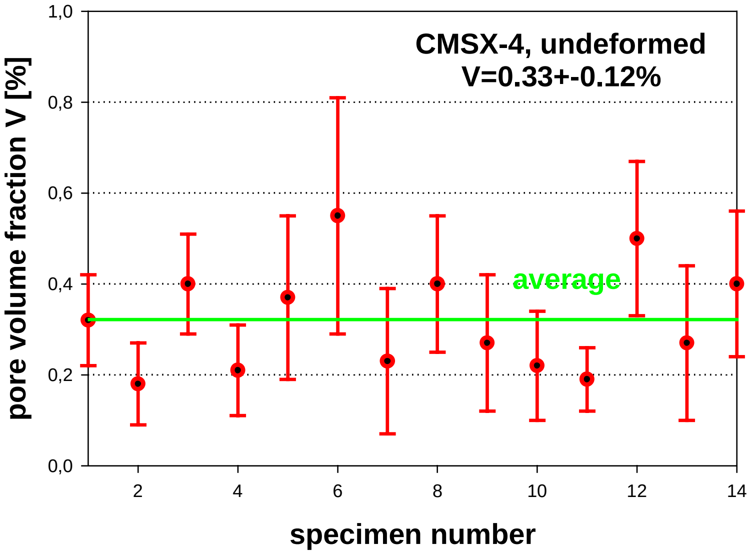
- **Pores are distributed inhomogeneously. → 3D position characterization.**

- **D pores are not spherical. → 3D shape characterization.**

Quantitative metallography

- •Light microscopy
- •CMSX-4, undeformed
- •14 specimens from one mold
- •28 micrographs from each specimen (25,5 mm^2 per specimen)

LM, binary image



Result: Inhomogeneous pore distribution has strong effect on the results.

Density measurements

When porosity increases density decreases: $\frac{\Delta V}{V} = -\frac{\Delta \rho}{\rho}$

Methods used:

Gas pycnometery: Helium, 134 MPa, about 10 g superalloy material

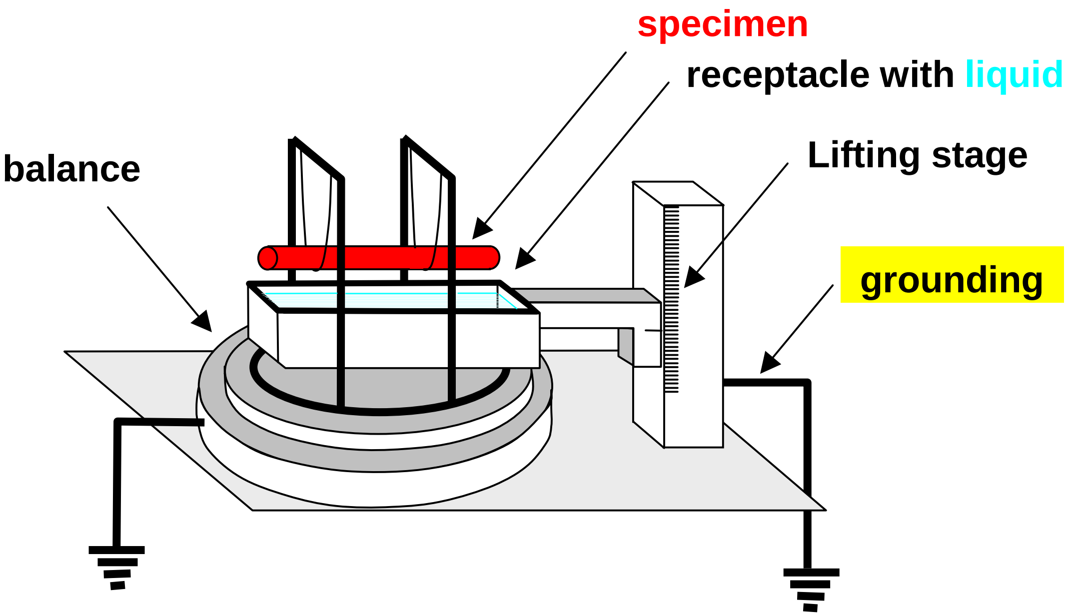
Archimedes method: Water or Dodecan, about 200 g superalloy material,

Specimen highly polished: $\Delta h = \pm 2.5\,\mu m$

Accuracy of balance: $\Delta m = \pm 0.1 mg$

Stability of temperature: $\Delta T = \pm 1°C$

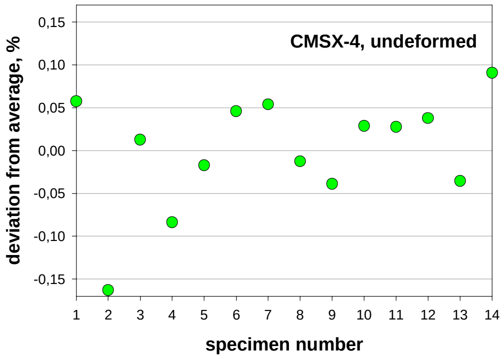
Archimedes method



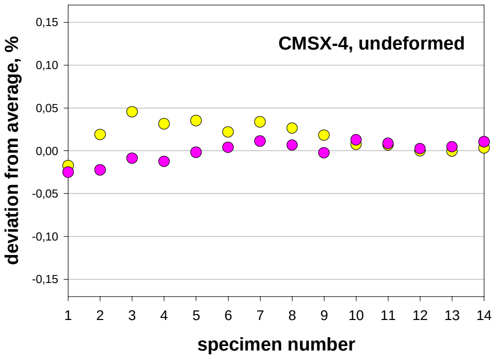
To attain high accuracy it is very important to:

- **control immersion depth of specimen in liquid.**
- **avoid electrostatic forces between liquid and specimen.**

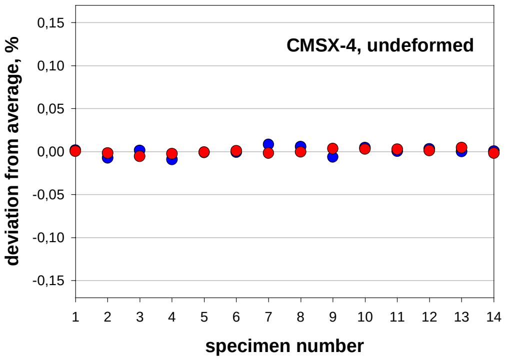
Comparison of methods for density measurement



Gas pycnometer



Archimedes meth. with dodecan



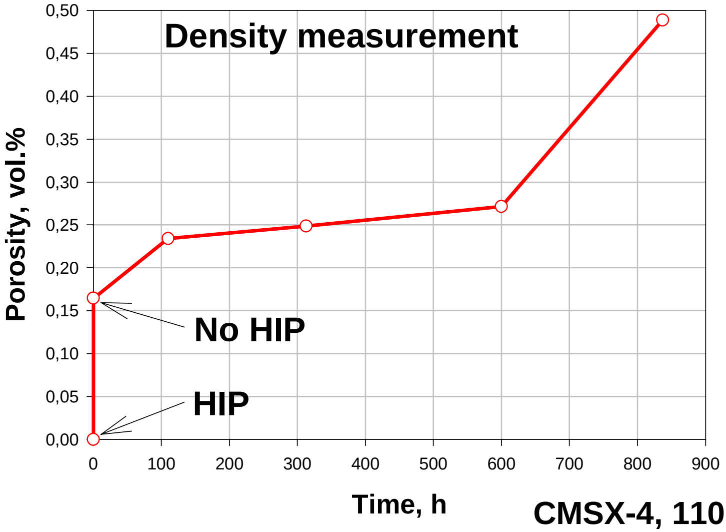
Archimedes meth. with water

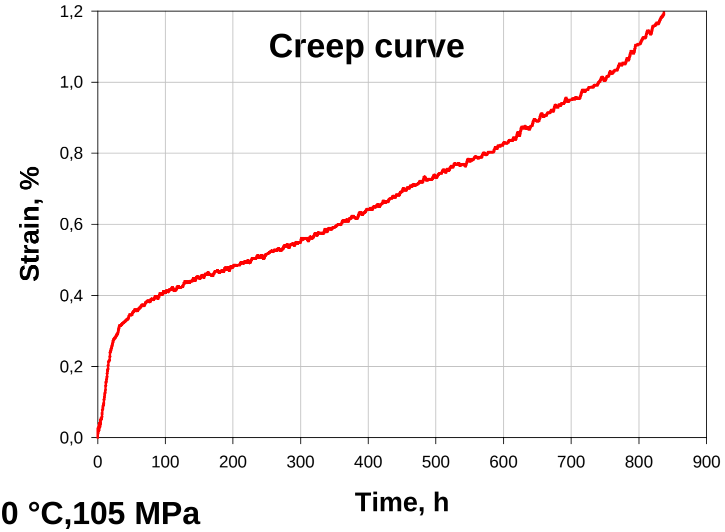
The highest accuracy is attained with

the Archimedes method using water:

Standard deviation of the average density: $3.6 \cdot 10^{-5}$%

Effect of HIP-ing and creep on porosity





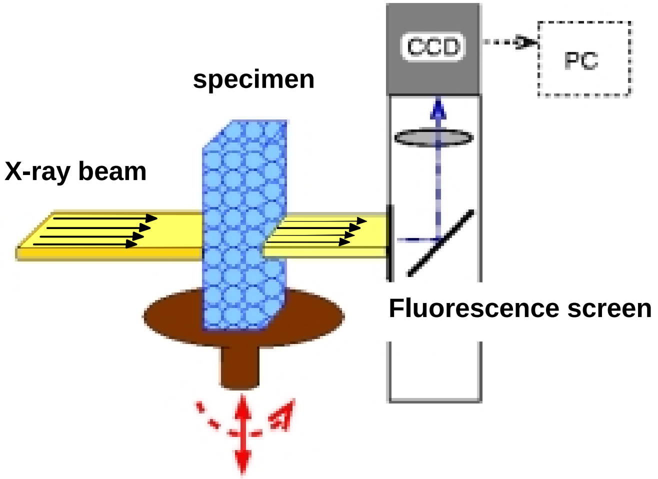
CMSX-4, 1100 °C,105 MPa

- •HIP significantly reduces the porosity of CMSX-4.

  (After HIP no more pores detectable by LM.)

- •The increase of porosity at high temperatures and low stresses follows the creep curve.

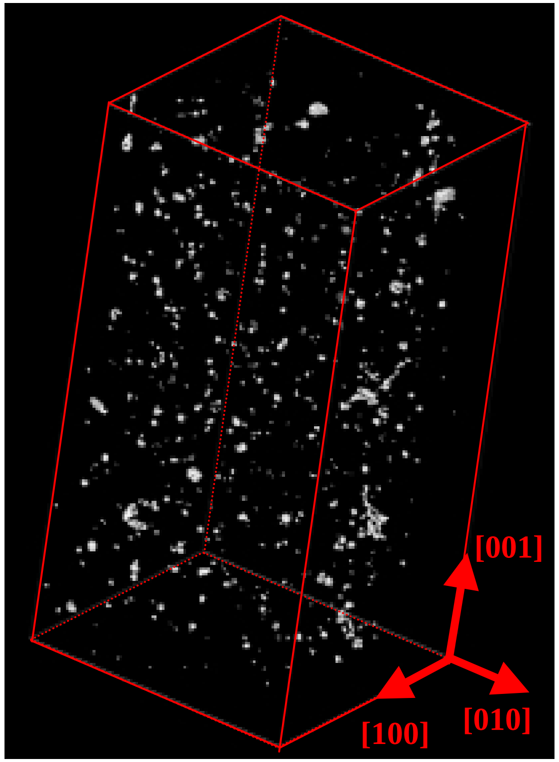
Tomography of Superalloys



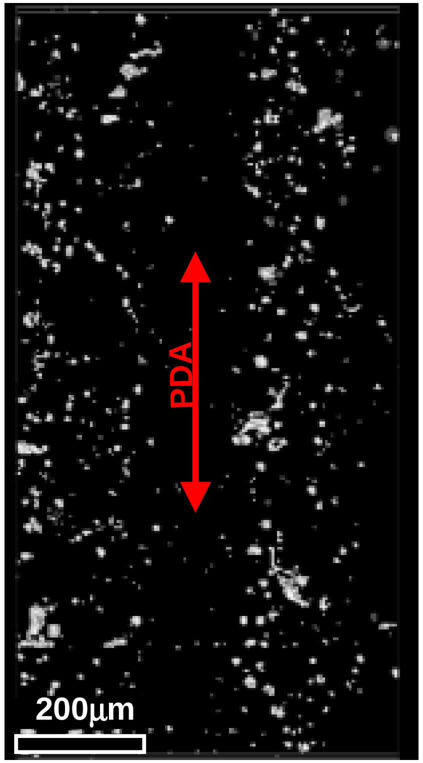
Measurements performed at the European **S**ynchrotron **R**adiadion **F**acility**-Grenoble**

- **Beam line ID 19**
- **X-ray energy 50 kV**
- **Voxel size 0.7µm**
- **Scanned specimen volume ≈ 0.5 x 0.5 x 1.0 mm^3**

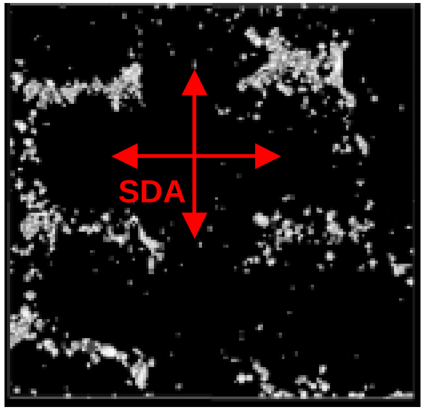
Distribution of pores



3D view



Projection in (100) plane



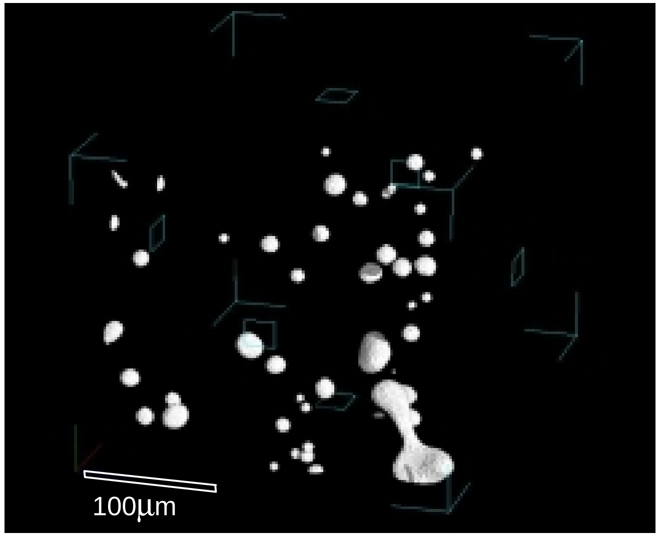
Projection in (001) plane

CMSX-4,

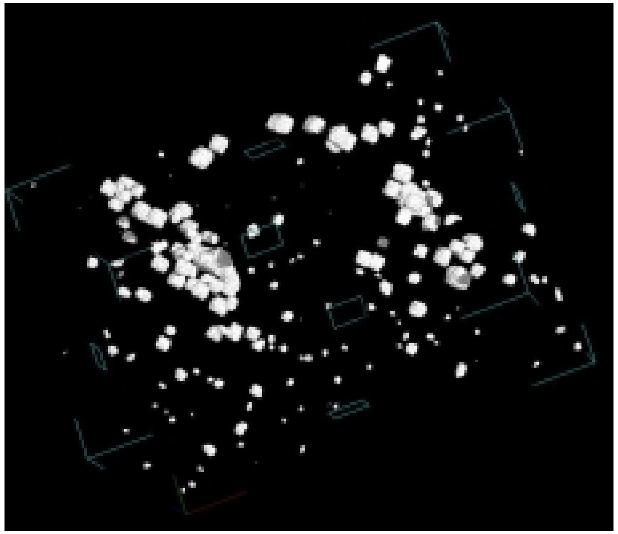
1100 °C, 117 MPa,
392 h

Pores concentrated in the interdendritic area

Shape of pores



CMSX-4, undeformed

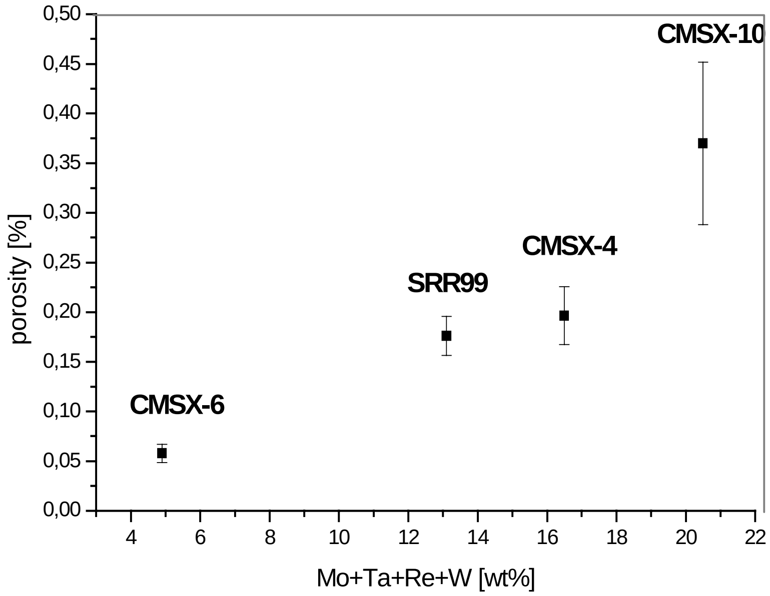


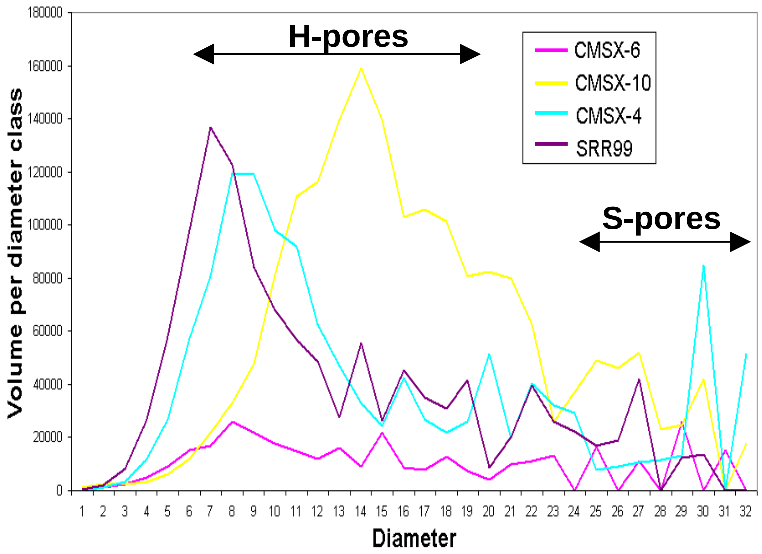
CMSX-4, crept at 1100° C, 117 MPa, 392 h

Pores are round after heat treatment

and facetted after creep deformation

Alloy composition → Porosity





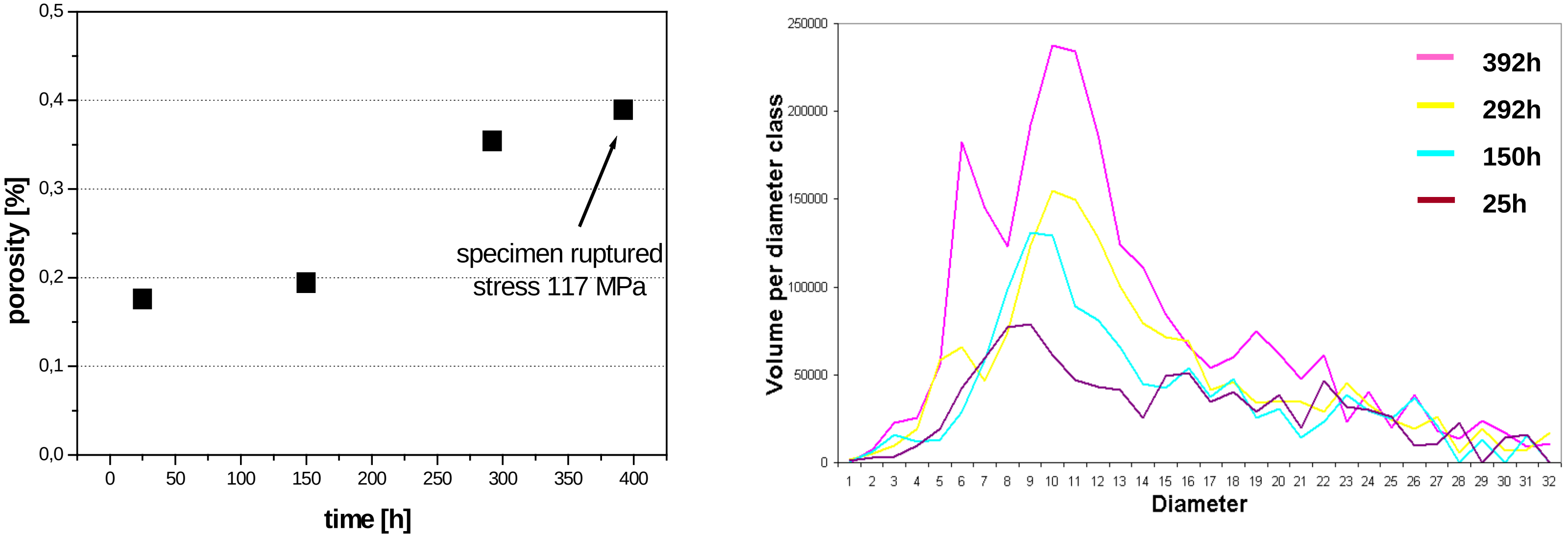
With increasing concentration of the refractory elements Mo, Ta, Re, W porosity and size of pores increase.

Reason for porosity increase

Superalloy	Mo+Ta+W+Re [wt%]	Heat treatment		Porosity [%]
		Max. Temp [ºC]	Total time [h]	
CMSX-6	4,9	1280	10	0.058
SRR99	13,1	1303	5	0.18
CMSX-4	16,5	1303	9	0.20
CMSX-10	20,5	1366	20	0.37

Higher content of refractory element needs a heat treatment with higher temperatures and longer times.

Creep → Porosity

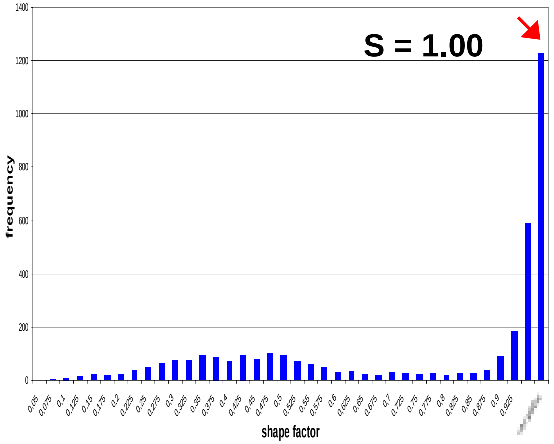


CMSX-4, 1100 °C, 120 MPa

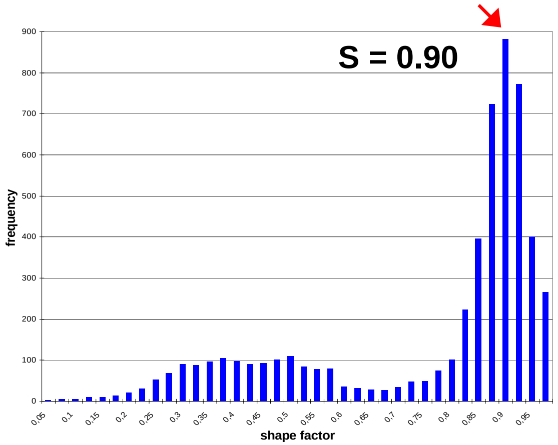
Porosity increases during creep.

In the size distribution a second peak develops at small pore sizes.

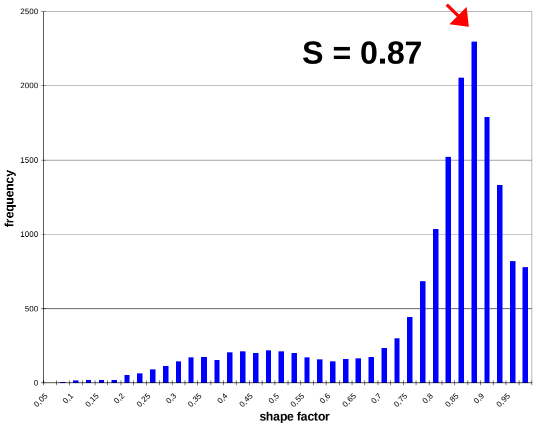
Creep → Pore shape



CMSX-4, undeformed



CMSX-4, 1100°C, 120 MPa, 150 h



CMSX-4, 1100°C, 120 MPa, 392 h

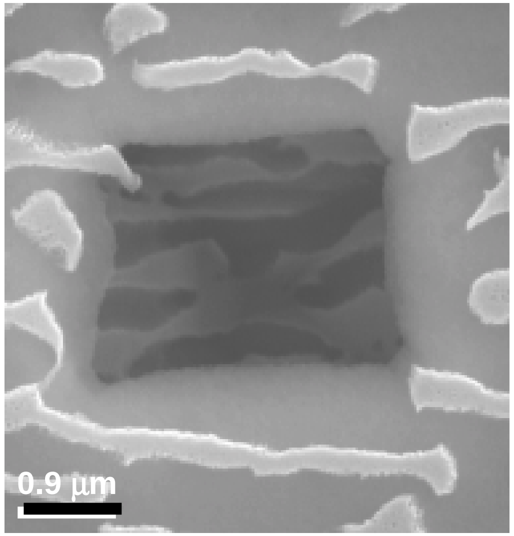
Shape factor: $$S = 6 \cdot \sqrt{\pi} \cdot \left(\text{volume} / \sqrt{(\text{surface area})^3}\right)$$

S = 1.00 : sphere

S = 0.72 : cube

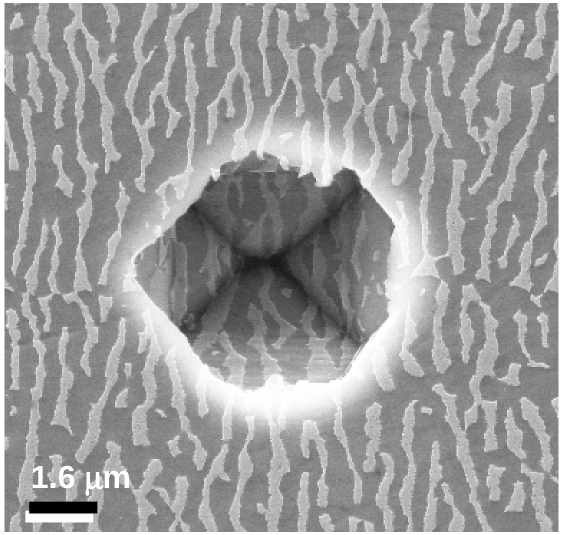
Shape factor decreases during creep

Shape of creep pores



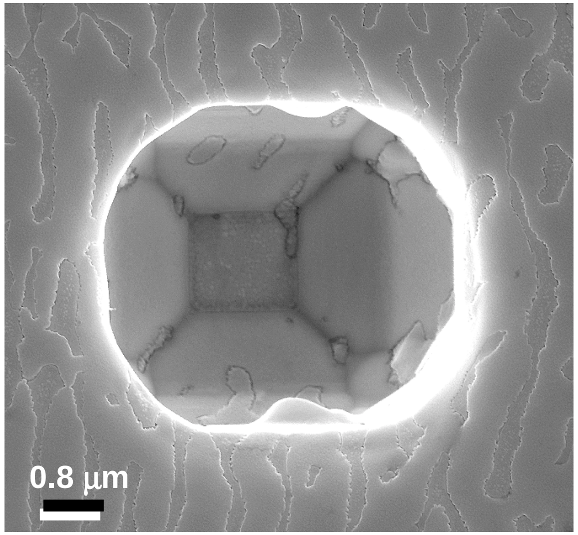
CMSX-4, 1100°C, 120 MPa, 292 h

{100} facets



CMSX-4, 1100°C, 120 MPa, 150 h

{110} facets



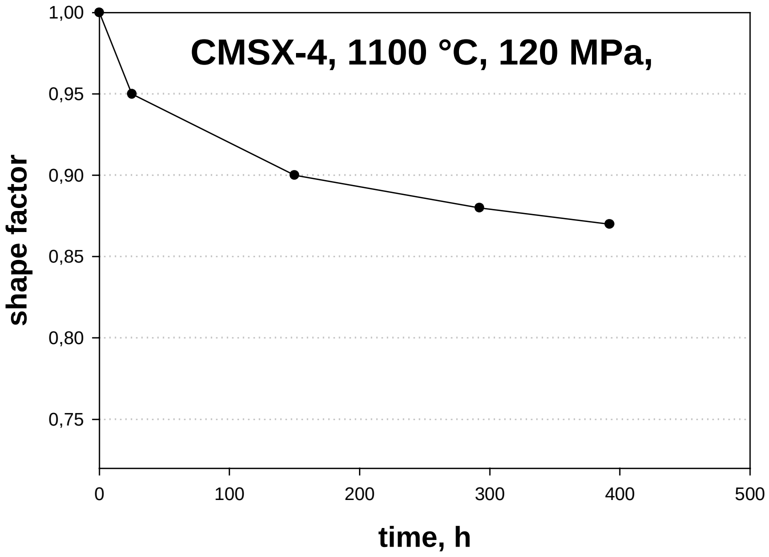
CMSX-10, 1100°C, 120 MPa, 200 h

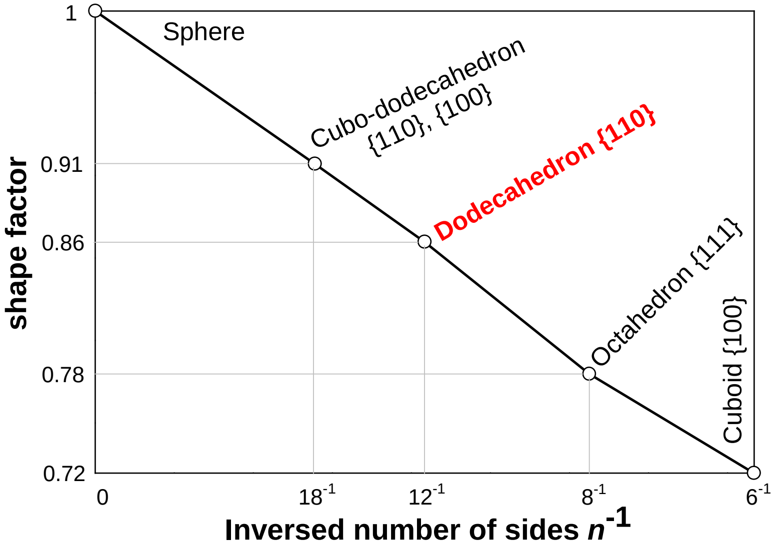
{110} and {100} facets

Creep temperature (1100 °C) is low compared to the homogenisation temperatures (about 1300°C).

⇒ Surface energy at creep temperature is anisotropic.

Shape factor⇔ Pore shape





The changing shape factor indicates a transformation from spherical shape to facetted shape with {110} and {100} facets.

Summary

- In single crystal superalloys there are 3 classes of pores: Solidification pores, homogenisation pores and creep pores.
- Quantification of porosity by classical metallografy is difficult, because of small pore size, low pore volume fraction and inhomogeneous distribution.
- Differences in porosity can by measured very exactly by the Archimedes method.
- High resolution tomography allows to quantify porosity and to characterize three-dimensionally position and shape of each single pore.
- Pores are concentrated in the interdendritic region.
- Porosity increases with the concentration of refractory elements Mo, Ta, W, Re.
- Porosity increases with creep strain.
- Pore shape transforms during creep from spheres into polyhedrons.