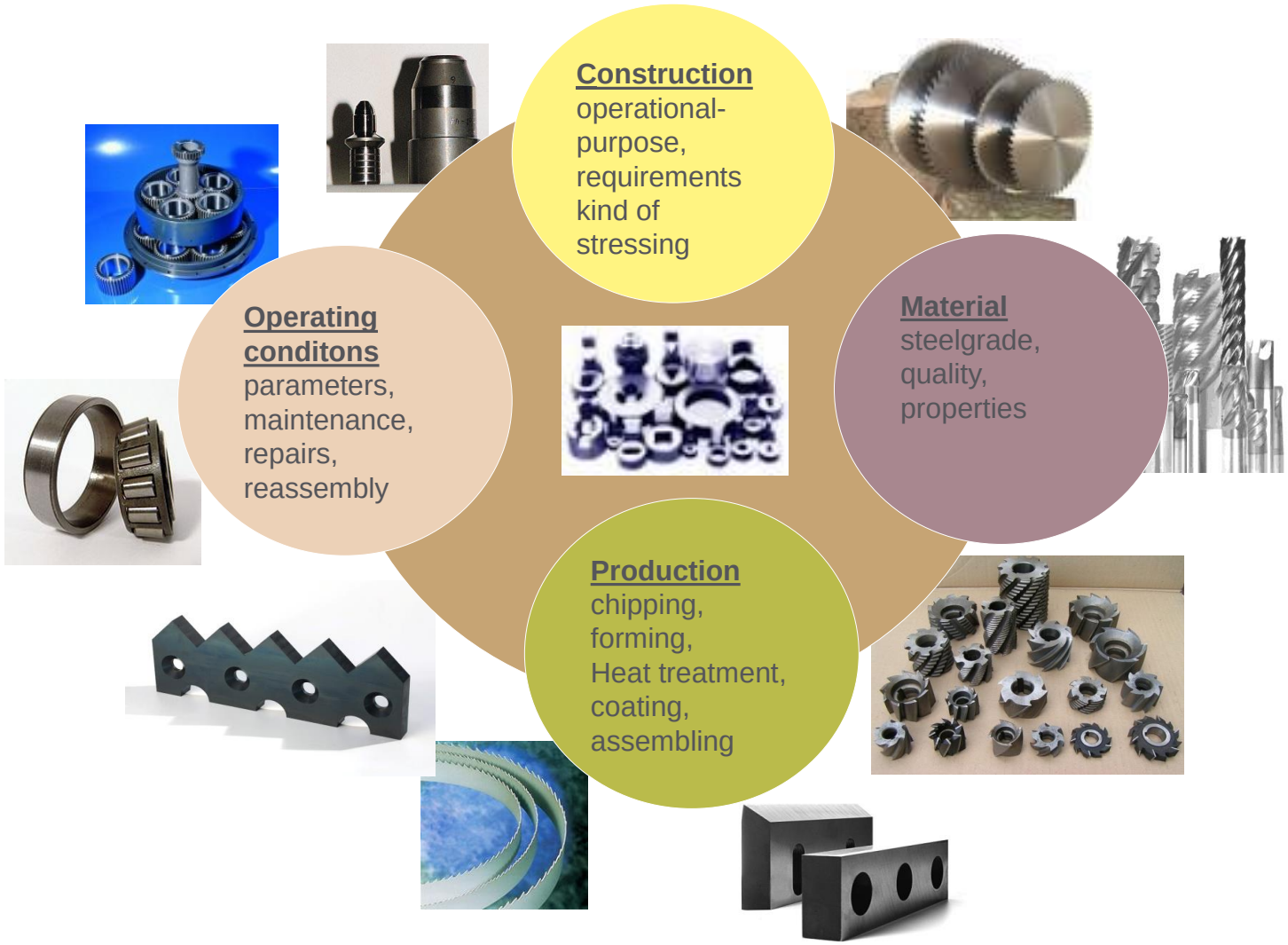
Cryogenic Treatment of Metals

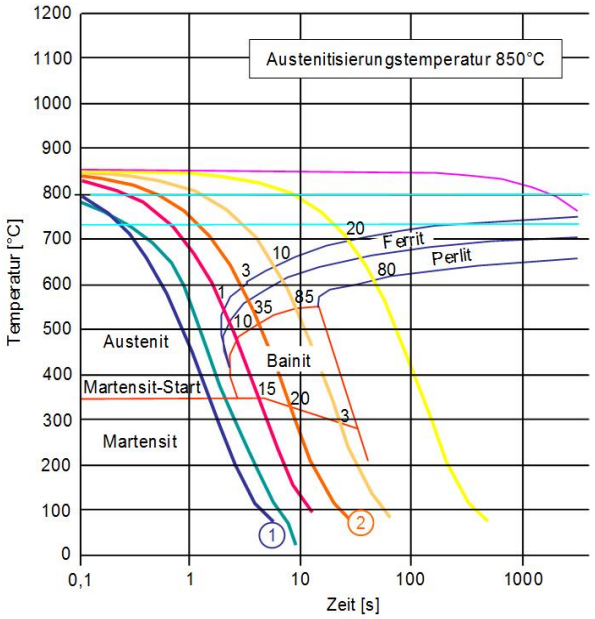
Recent scientific investigations and implementation in industrial heat treatment processes

Västerås, 20/09/2017 Georg Lehmkuhl Air Liquide CWE, Krefeld

Impact on Lifetime



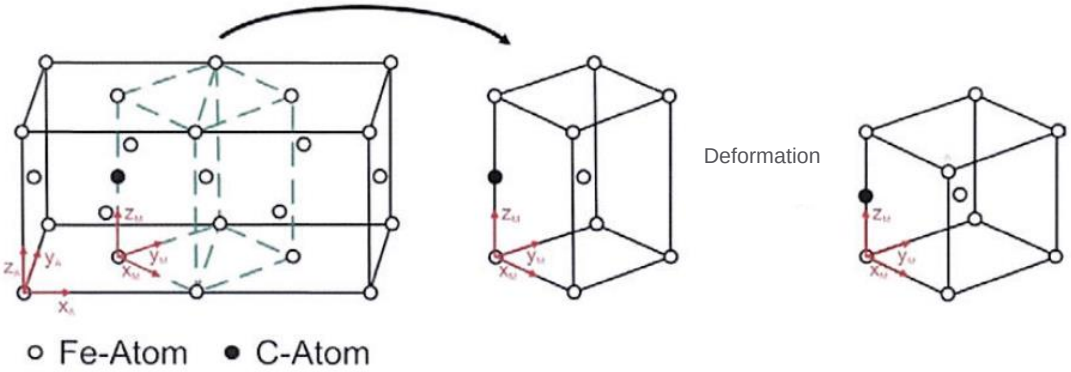
Martensitic transformation



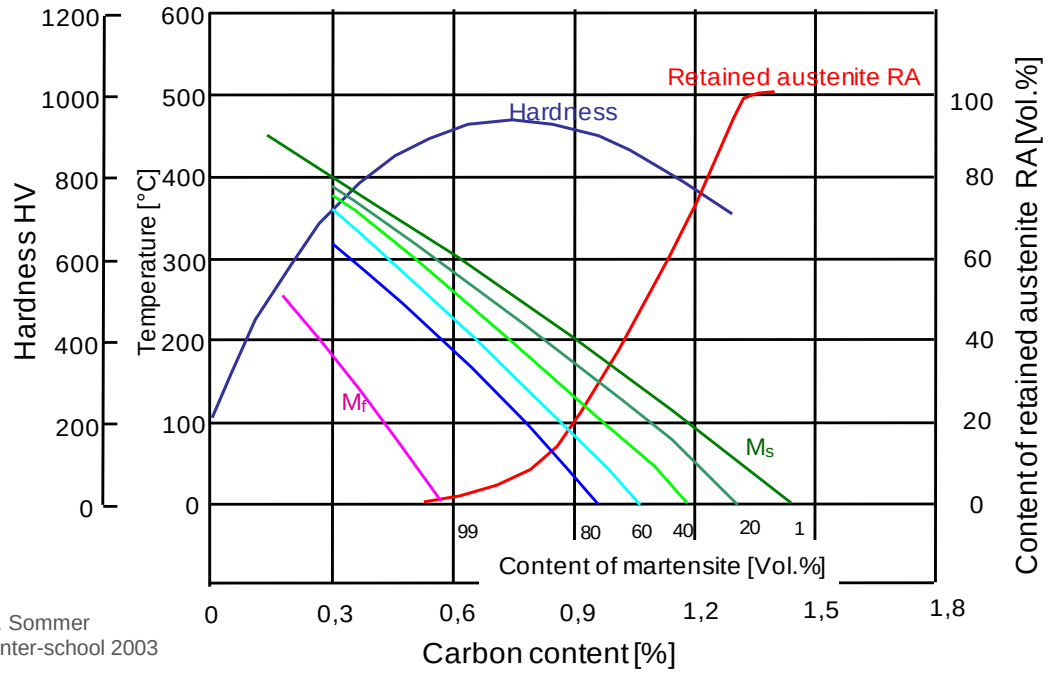
Hardening means to quench the steel very quickly after austenitising and transform the austenit into a martensitic structure.

Hardness of martensite is mainly driven by the enforced embedded carbon atoms and residual stresses.

The critical quenching rate must be achieved.



The formation of retained austenite RA



Dr. Sommer
Winter-school 2003

Martensite start **Ms** and martensite finish **Mf** temperatures are mainly affected by the carbon content and the alloying elements of the steel.

If **Mf** boundary is not reached during quenching, retained austenite RA will remain.

Hardness ⇓

In many cases, the structure of hard martensite and soft retained austenite affects lifetime of steel

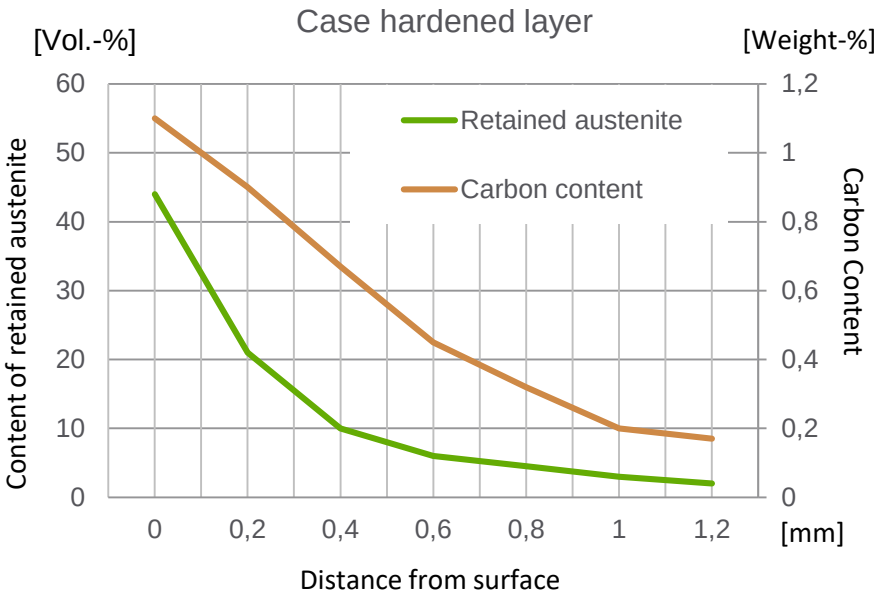
Wearing quality ⇓

During operating time, heating up and cooling down cycles can cause uncontrolled transformation of retained austenite to martensite

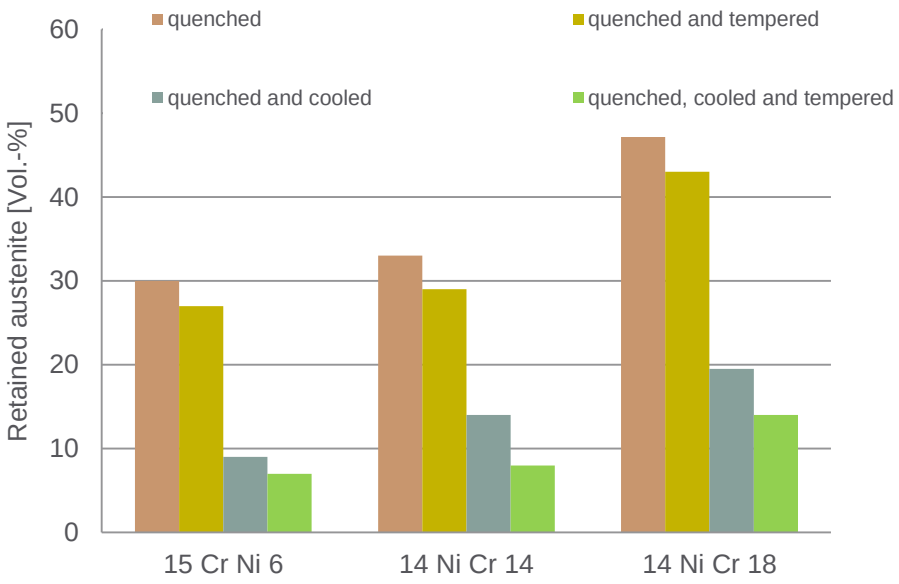
⇒ Volume expansion

Dimension stability ⇓

Case hardening combined with cryogenic treatment



Nach O. Schwarz u.a.



During carburising the surface is enriched with RA

Cryogenic treatment reduces RA significantly

Benefits from cryogenic treatment directly after quenching

- Quality improvement:
  - Significant reduction of RA content
  - Homogenisation of RA content
  - Prevention of RA stabilisation
  - Increase of hardness
  - Dimension stability
- Costs savings:
  - Substitution of 1or 2 following tempering cycles

Aircraft

Precision Gears
15 NiCr 13

Engineering

Precision bolts +
bushings 16MnCr5

Bearing

Bearings
100Cr6

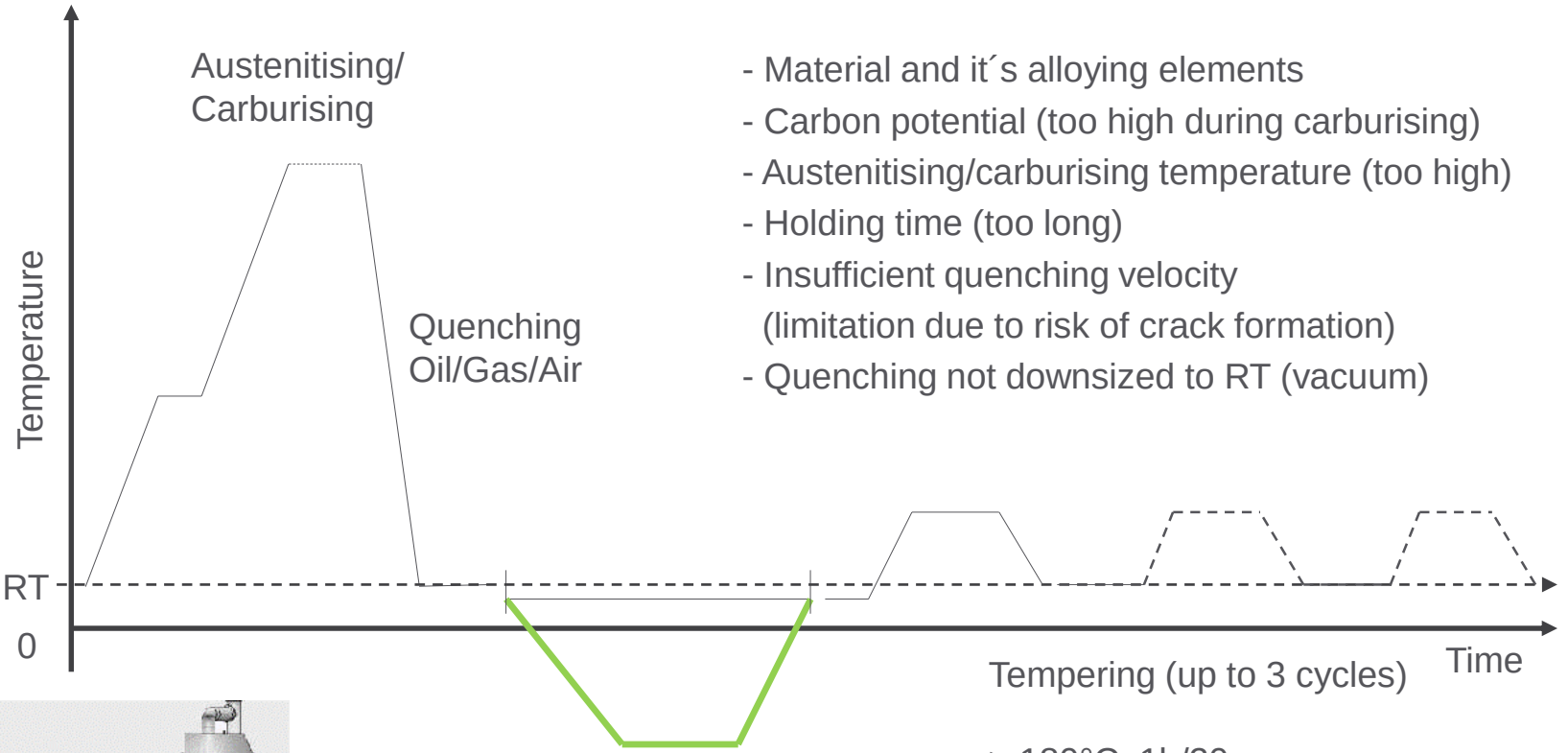
Automotive

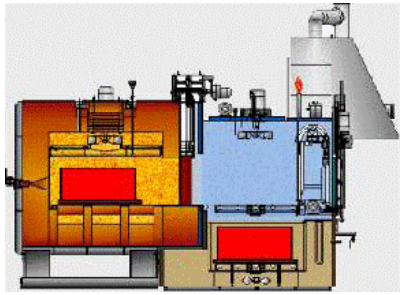
Diesel injection
nozzles18CrNi 8

Implementation of Cryogenic Treatment CT

Effects on content of retained austenite RA:

- Material and it´s alloying elements
- Carbon potential (too high during carburising)
- Austenitising/carburising temperature (too high)
- Holding time (too long)
- Insufficient quenching velocity
  (limitation due to risk of crack formation)
- Quenching not downsized to RT (vacuum)



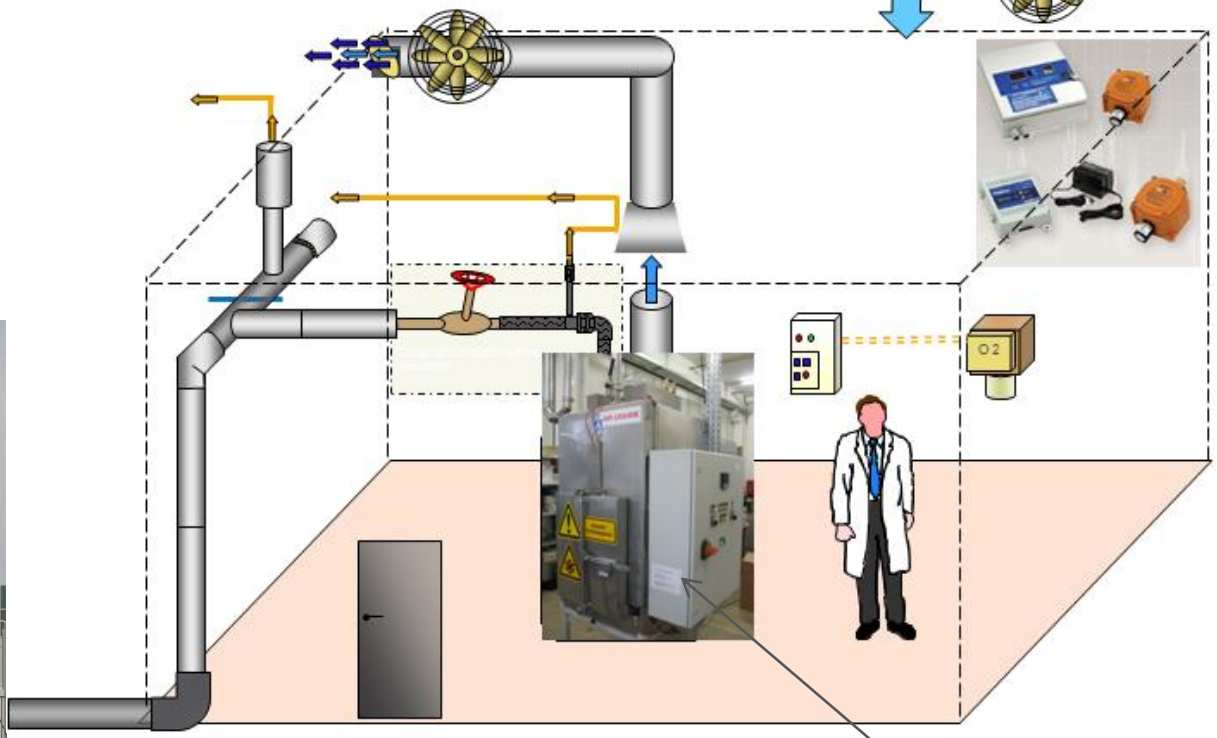


Overview installation for cryogenic treatment

Liquid nitrogen
(LIN) supply 1-3 bar

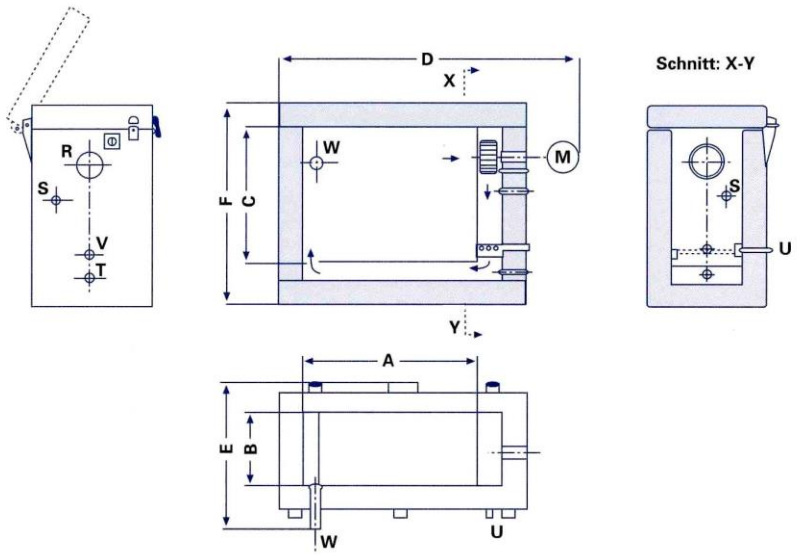
or

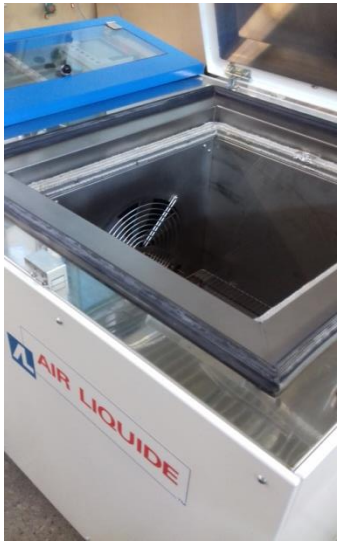
Air ventilation



Cryogenic chamber

Standard cryogenic equipment for cooling





ALNAT Cryo Top-Lid Chamber ACKT 1900 S

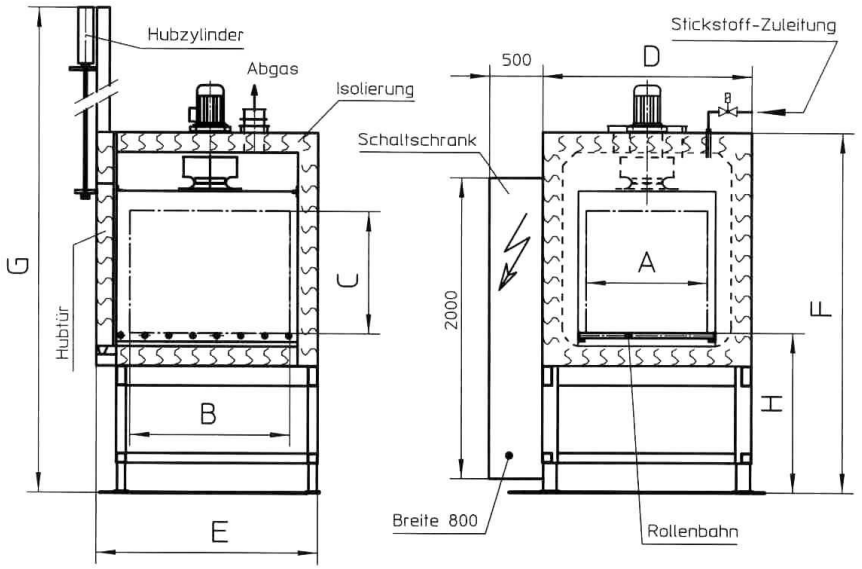
- Direct injection of liquide nitrogen (LIN)
- Integrated ventilation sytem
- Operating temperatures from -180 to +100°C
- Uniform temperature distribution (±5°C)
- Program controller for cooling/heating cycles
- Recording function for documentation

User: hardening shops, assembly by shrinkage automotive, aircraft-, bearing-, tooling-industries etc.

Specific cryogenic equipment for cooling and tempering

Hot/Cold working chamber ACKW 420

Continuous working freezer ACCF 475



- Individual design
- Batch size is adapted to furnace dimensions
- Integration in automatic driven furnace lines (PLC controlled)
- Operating temperature from -140°C up to +550°C
- Tempering under nitrogen atmosphere
- Powerful ventilation turbine(s)
- Uniform temperature distribution (<±5°C)

Aerospace Material Specification: AMS 2750E compliance

- **AMS 2750 E "Pyrometry" specification:**
  - Defines pyrometric requirements for thermal processing equipment used for heat treatment
  - Covers temperature sensors, instrumentation, system accuracy tests and temperature uniformity survey

Classe de four	Homogénéité dans la répartition des température	
	°C	°F
1	+/- 3	+/- 5
2	+/- 6	+/- 10
3	+/- 8	+/- 15
4	+/- 10	+/- 20
5	+/- 14	+/- 25
6	+/- 24	+/- 50

Classe de four	Difference maximum SAT	
	° C	° F
1	+/- 1,1	+/- 2
2	+/- 1,7	+/- 3
3	+/- 2,2	+/- 4
4	+/- 2,2	+/- 4
5	+/- 2,8	+/- 5
6	+/- 5,6	+/- 10

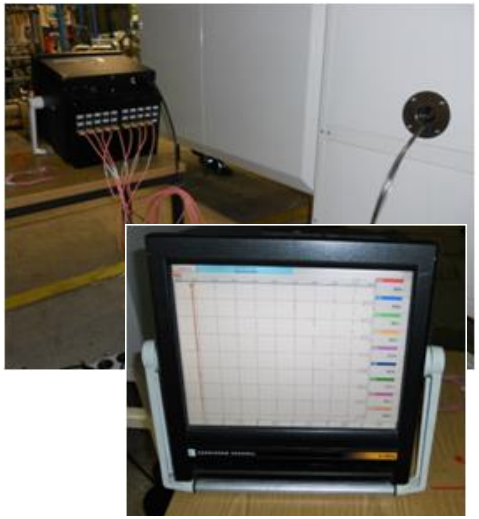
- **Cryogenic chambers compliance with AMS 2750E**
  - Definition of MIN/MAX temperature range
  - Definition of required tolerance (equipment class)
  - Different thermocouples sensors required for:
    - Control, Policeman, Min, Max, Load
  - Calibration for thermocouples (ISO/IEC17025)
  - UKAS calibration of instruments
  - SAT: System Accuracy Test
  - TUS: Temperature Uniformity Survey
  - Documentation/certificates

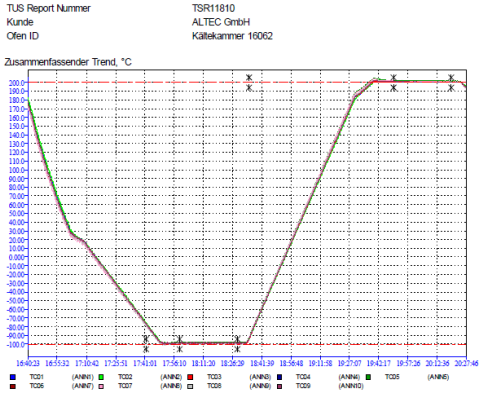
UKAS CALIBRATION

Cryogenic chambers: AMS 2750 compliance

SAT: **S**ystem **A**ccuray **T**est
TUS: **T**emperature **U**niformity **S**urvey

High temperature uniformity:
- Class 2 in standard
- Class 1 possible with custom optimisation



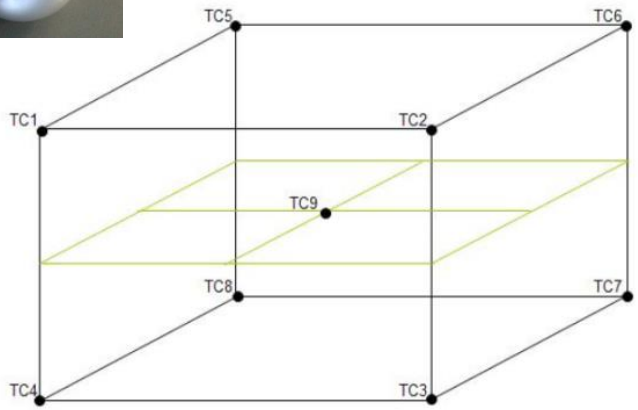


UKAS calibration

Continous Quality Improvement: CQI-9 compliance

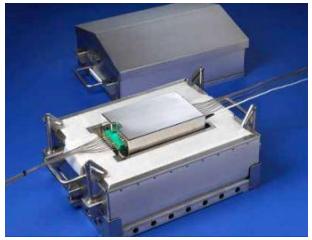
- **Self assessment** to create a global quality standard in Automotive Industry

Number of TUS elements depends on the volume of working space



Specific design of cryogenic chambers

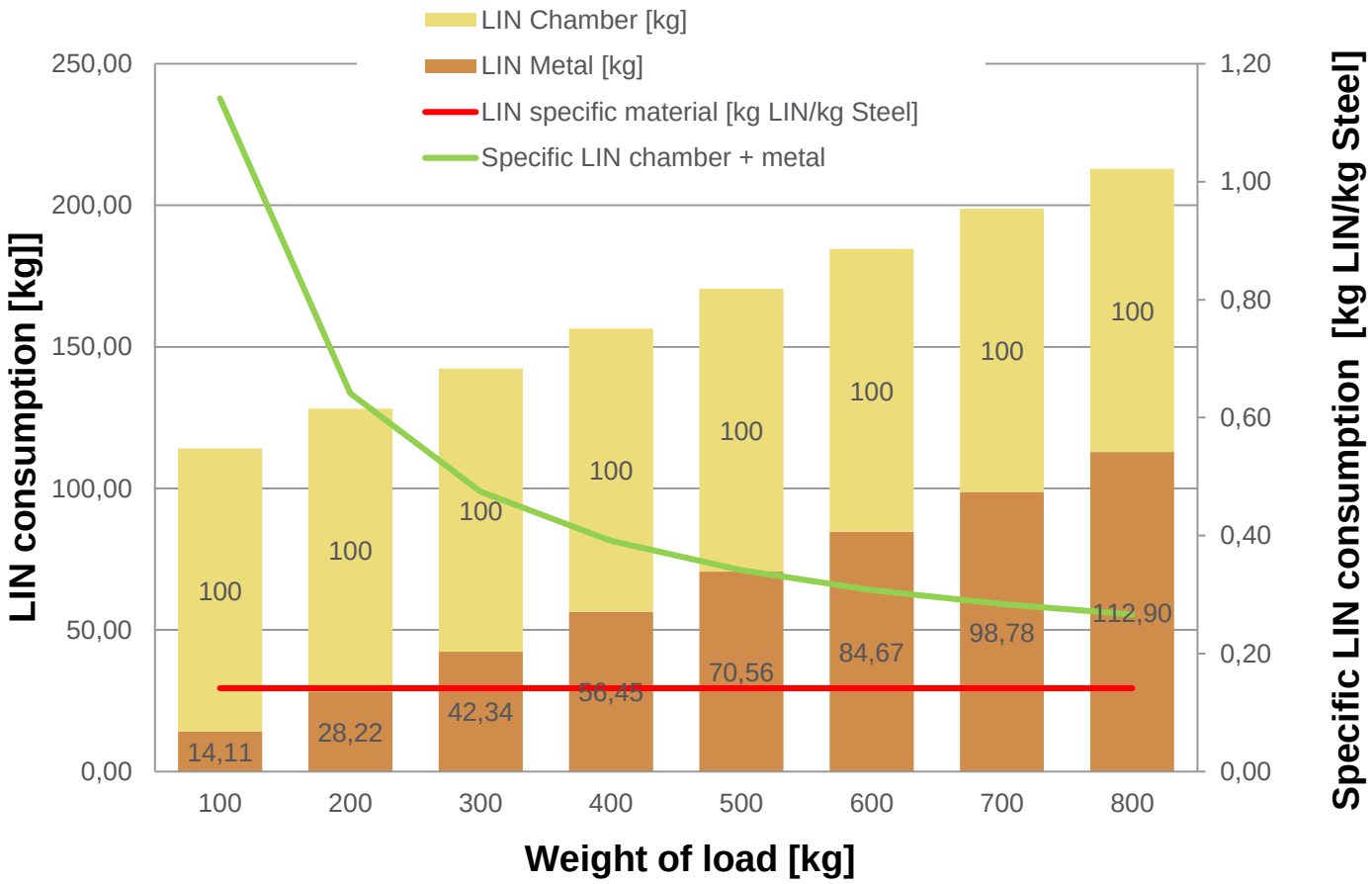
- Precise temperature control for cooling and heating
- Integrated recirculation fans or turbines
- Use of calibrated thermocouples
- Use of calibrated instruments
- Use of tamper resistant recorders/documentation
- Safety and Reliability



Operating efficiency (exemplary)

LIN = f(material+chamber+operating conditions)

Processing: 1.00 h cooling from +20°C to -80°C
1.00 h holding at - 80°C

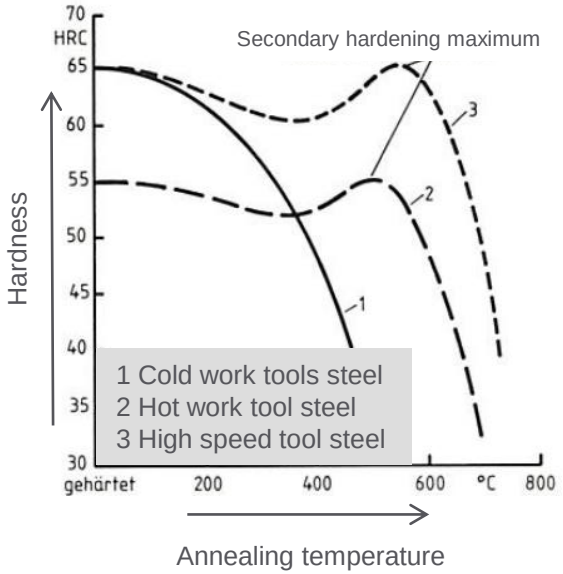


New development: Deep Cryogenic Treatment (DCT)

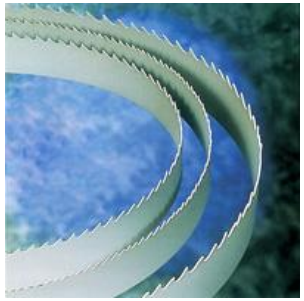
DCT is preferabely applied to improve **wear resistance and lifetime** of tools, considering the enhancement of mechanical properties like hardness, toughness and fatigue resistance.

Theories:

- Nearly complete transformation of retained austenite to martensite
- Optimal conditioning of martensite and **precipitation of fine carbides**
- Reduction of residual stresses



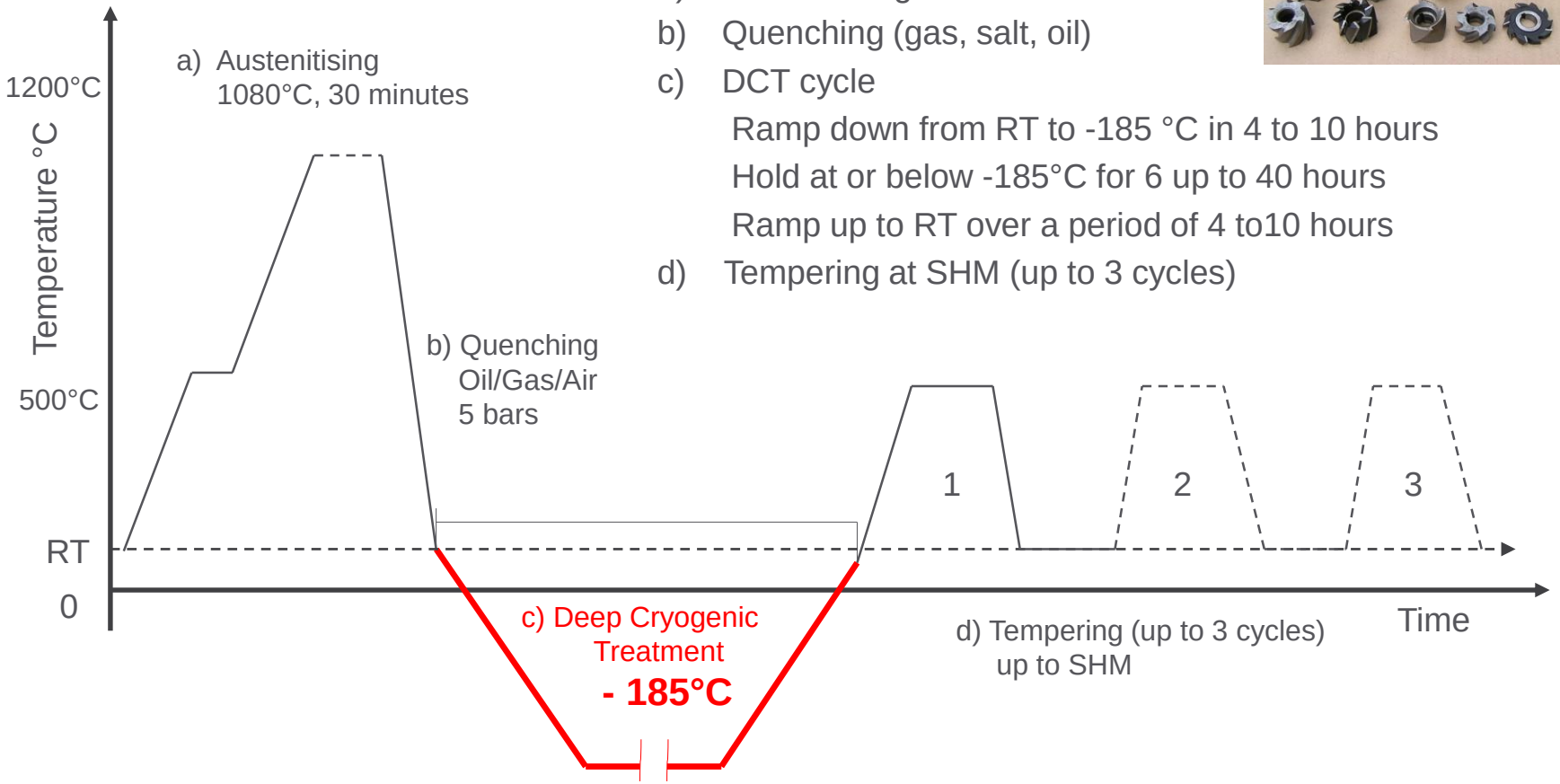
Steel	Material-No.	Application
Cold work tool Steel	1.2379	Machine Cutting tools, stamps, pressing tools, chisel, cutter block, etc.
High Speed Steel	1.3202	Drilling bits, reamer, chisel, milling tools, saw blades etc.
Hot work tool Steel	1.2343	Die shapes, extrusion die



How to apply Deep Cryogenic Treatment ?

DCT is used within conventional heat treatment processes

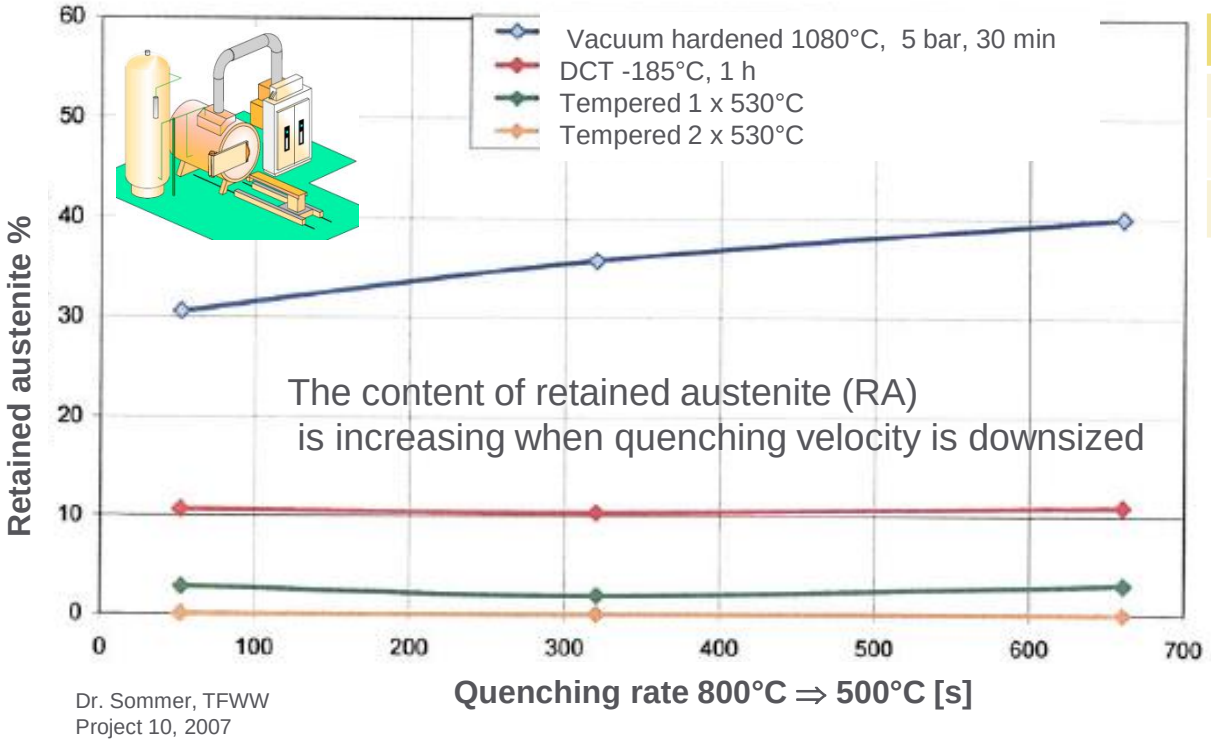
a) Austenitising
b) Quenching (gas, salt, oil)
c) DCT cycle
   Ramp down from RT to -185 °C in 4 to 10 hours
   Hold at or below -185°C for 6 up to 40 hours
   Ramp up to RT over a period of 4 to10 hours
d) Tempering at SHM (up to 3 cycles)



Vacuum isolated vessel for Deep Cryogenic Treatment

- Minimal losses of nitrogen at low temperatures and long holding times
- Processcontroller for regulation and documentation
- Parts are exposed above liquid nitrogen below -185 °C
  (no contact with LIN)

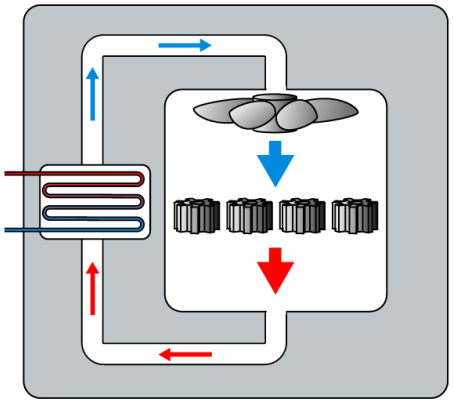
Benefits from Deep Cryogenic Treatment



Dr. Sommer, TFWW
Project 10, 2007

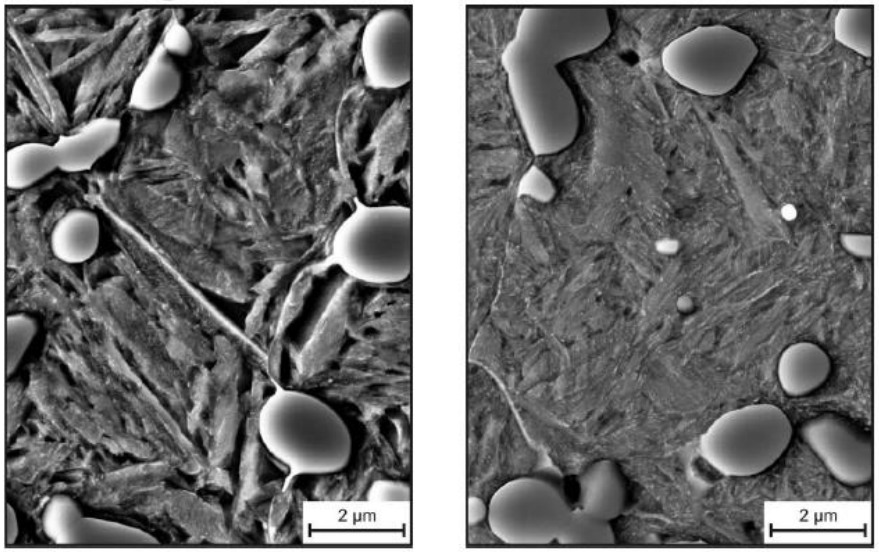
1.2379		X153CrVMo12			
C	Cr	V	Mo	Si	Mn
1,53	12,00	0,95	0,80	0,30	0,40

Axles of different diameters Ø 150 -350 mm



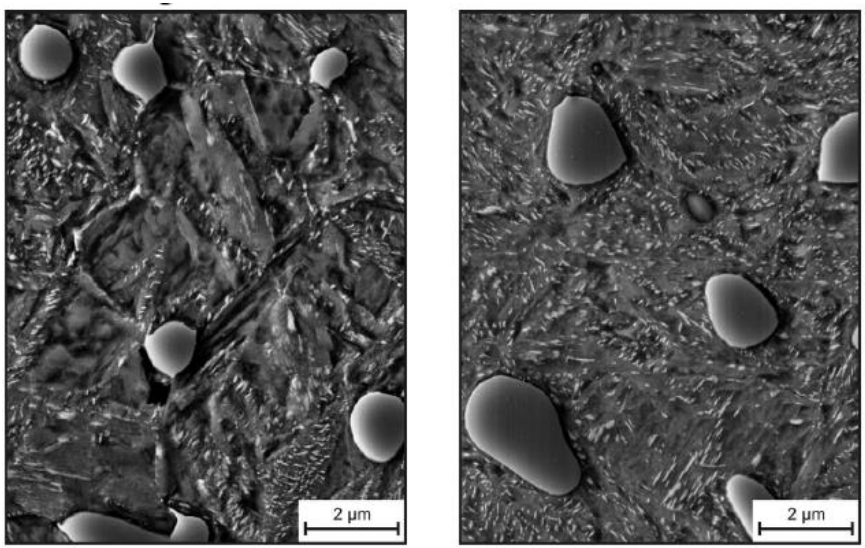
DCT:
- Reduces the content of RA significantly (under quantification limit)
- Homogenises the content of RA caused by downsized quenching velocities
- Replaces 1 or 2 tempering cycles

DCT - Precipitation of secondary carbides (REM)



Konventionell T_A = 1080°C $T_{anl.}$ = 180°C Cryo

1.2379



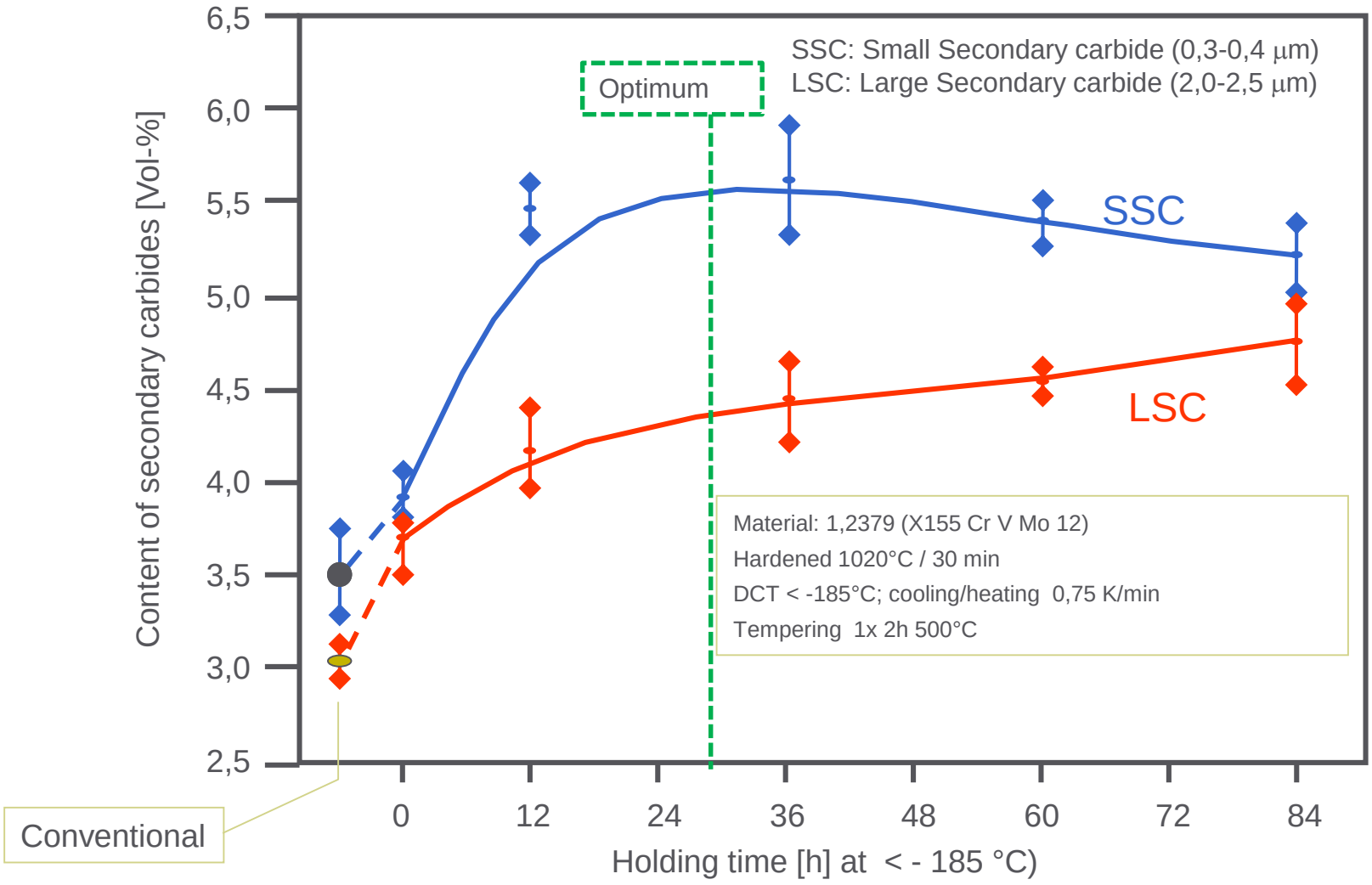
Konventionell T_A = 1080°C $T_{anl.}$ = SHM Cryo

DCT with high tempering at SHM:

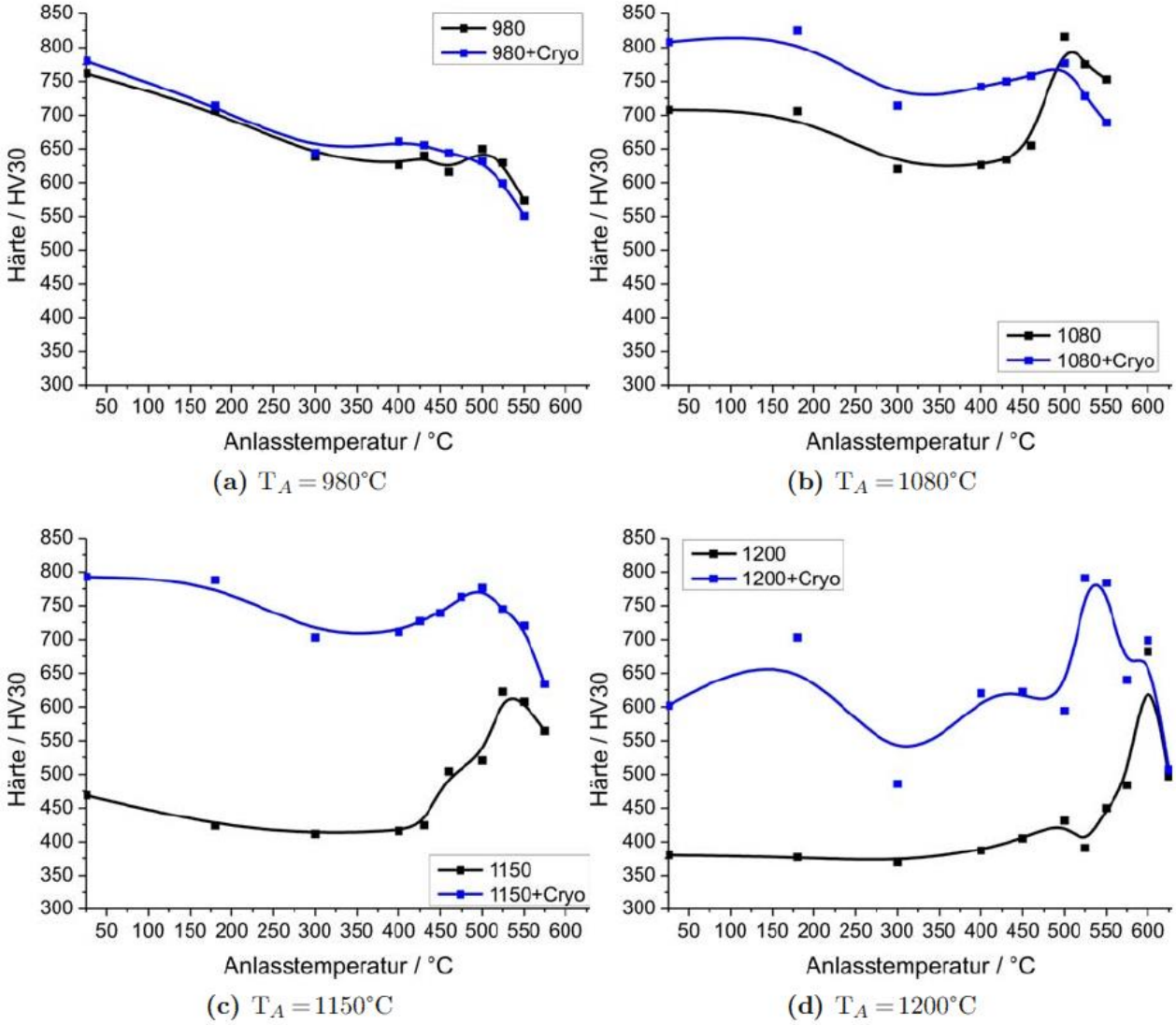
- Tough tempered martensite
- Fine distribution and high amount of secondary carbides
- Martensite laths of DCT samples are even thinner

Ruhr-Universität Bochum, RUB,
REM study CryoDuran

DCT: Formation of carbides



Deep Cryogenic Treatment: Tempering diagram

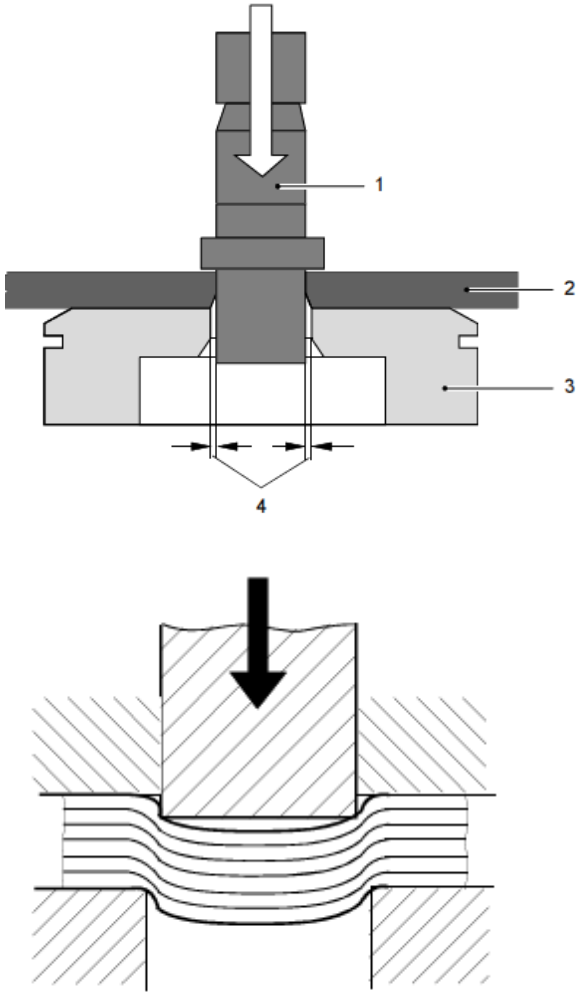


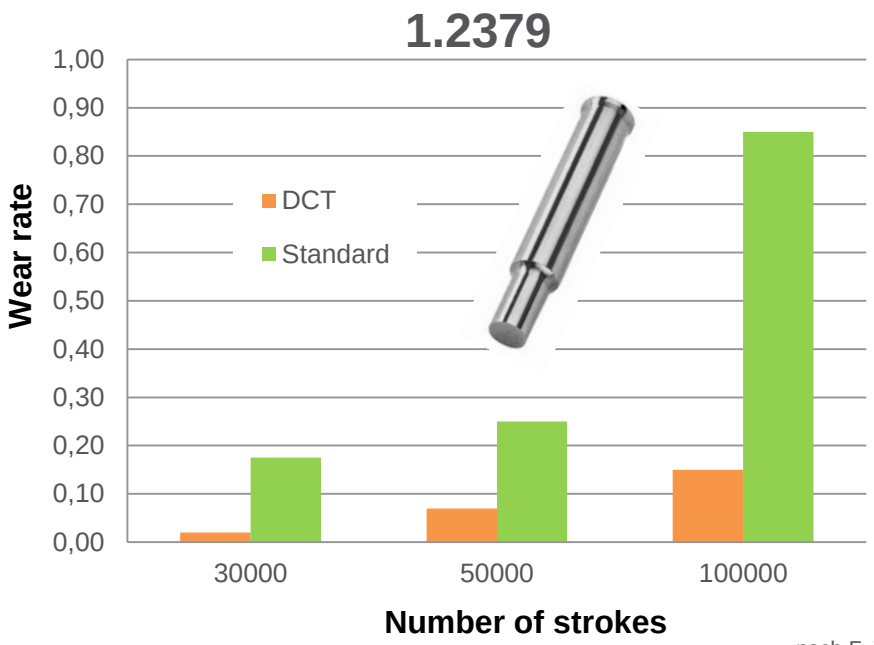
Material: 1.2379

- Influence of austenitising temperature TA
- Tempering behavior is modified (Displacement of SHM)
- Hardness is increasing by DCT
- DCT is recommended directly after quenching (Stabilisation of RA at RT)

Ruhr-Universität Bochum (RUB), Abschlussbericht CryoDuran)

Influence of DCT on wear resistance of stamps (test)



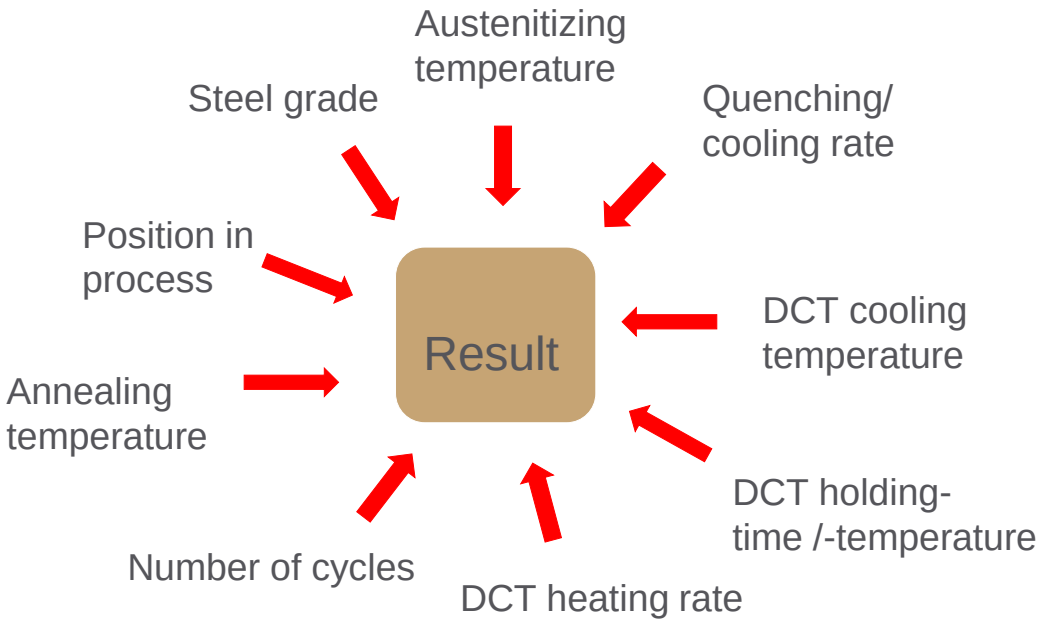


nach F. Wendl et AL
HTM Mat 66

- Angle curve improves with number of strokes
- Cryogenic treated stamps show lower abrasion and disruption
- Significantly enhancement of tool life

Improvement of wear resistance up to 30%

Cryogenic treatment: New possibilities in heat treatment



- Tribological properties of metals will be effected substantially when applying (deep) cryogenic treatment.
- Controlled martensitic formation and the precipitation of well distributed carbides is the key to form a homogenous microstructure, to improve hardness and wear resistance.
- Further investigations have to be done to determine the best processing parameters for each material in combination with heat treatment.

Thank you for your attention

Georg Lehmkuhl
Air Liquide CWE