

Control of heat treatment quality

Heat treatment project

CTU in Prague, FME
Materials Engineering

Thermal processing of metal alloys

Control changes of mechanical, physical, electrical and other properties – always connected with structural or substructural changes in treated material

Each heat treatment consist of three main periods:

- heating on at required temperature
- dwell at temperature
- cooling

Aim of heat treatment is to achieve more or less thermodynamically equilibrium state (stable or metastable)

Thermal processing of metal alloys

Heat treatment (temperature effect)

- annealing (to get more stable state)
reduce internal stress, reach softer and ductile structure, ...
- hardening (to get metastable state)
Increase of strength, hardness, wear resistance, ...

Thermomechanical treatment

(effect of temperature and deformation)

control of final structure and mechanical properties

Chemical heat treatment

(effect of temperature and changes of the chemical composition)

to get different properties of surface layer as in core of the piece
– higher hardness, better wear or corrosion resistance, ...

All steps of heat treatment must be provided at proper conditions!

Heating

- controlled by heat conductivity of material and heat transfer conditions
 - deformation, insufficient temperature in core

Dwell time

- controlled by required structural changes by diffusion.
homogeneous austenite, dissolving of minority phase (carbides)...
- prolonged time – negative effect - grain coarsening, oxidation, decarburization, ...

Cooling rate

- controlled by required microstructural changes (CCT, TTT diagrams), reducing internal stresses, ...
 - to slow - coarser and softer microstructure, tempering embrittlement, ...
 - to fast – high internal stresses, quench cracking , intensive deformation, ...

Requirements on pieces after heat treatment

Appearance - surface quality - oxidation, deformation

Geometry - dimensions, deformations

Properties - hardness testing
- microstructure verification

Homogeneity - presence of cracks (sound, impact check, NDT)

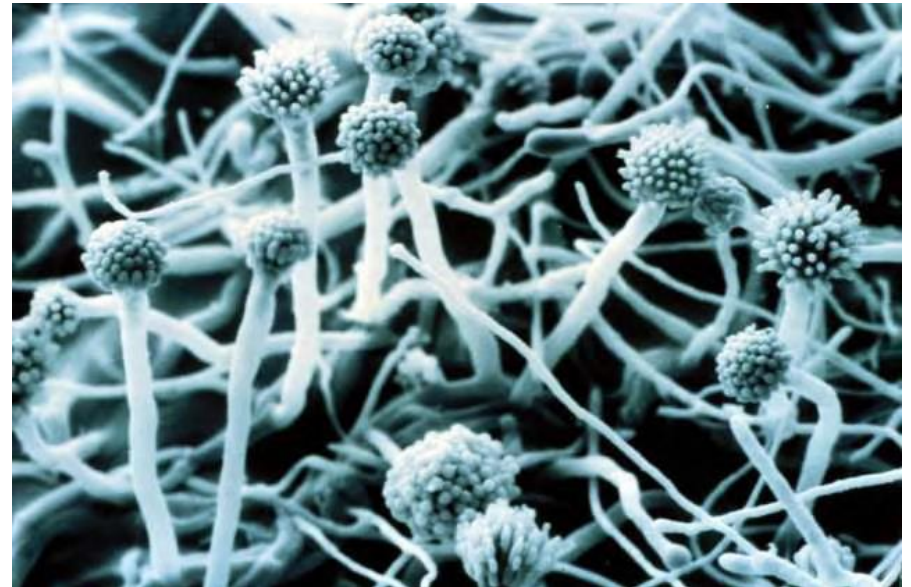
Hardness testing

- Hardness is determined in drawings or by other technical documentation.
- Hardness measurement is typical and the most used method for monitoring of heat treatment results and confirmation of customer requirements.
- Standard static indentation methods are used
 - **Brinnell** mild hard and soft materials, heterogeneous materials - surface
(Al alloys, Cu alloys, cast irons , annealed steels, ...)
 - **Rockwell** hard materials and mild hard materials - surface
quenched steel, surface quenched steel, carburized steel, ...
 - **Vickers** soft, mild soft, hard materials (including ceramics)
hardened layer depth measurement, thin sections, coatings, selective measurements on microstructure components, ...

Microscopic evaluation

- **Hardness measurement is significant but not conclusive result of the heat treatment quality.**
- **Microstructure analysis and documentation is necessary to declare, that the heat treatment does not negative influence on the properties – exclusion of undesirable defects in microstructure.**
- **Necessary method for measurements and evaluation of the depth of hardened layers or coatings.**

Macro - micro



Macrostructure

- 1) heterogeneity of chemical composition of alloys and mixtures at different cross sections**
- 2) macroscopic structural units formed during crystallization or solidification**
- 3) macroscopic structural units formed of metal forming, shaping of non-metals and materials for joining by welding, brazing or adhesive bonding or other processing technologies**

Macrostructure

- 4) the heat affected depth of the heat transfer or the surface layers of the mass transfer (diffusion)**
- 5) depth of surface damage due to corrosion or wear**
- 6) fractures produced in operation by external forces or environments**

Microstructure

- a) qualitative and quantitative phase composition, which is preferably determined by diffraction methods**

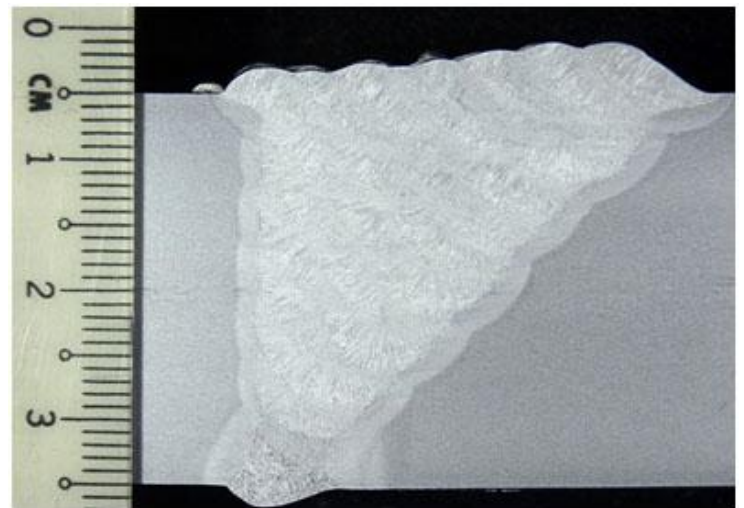
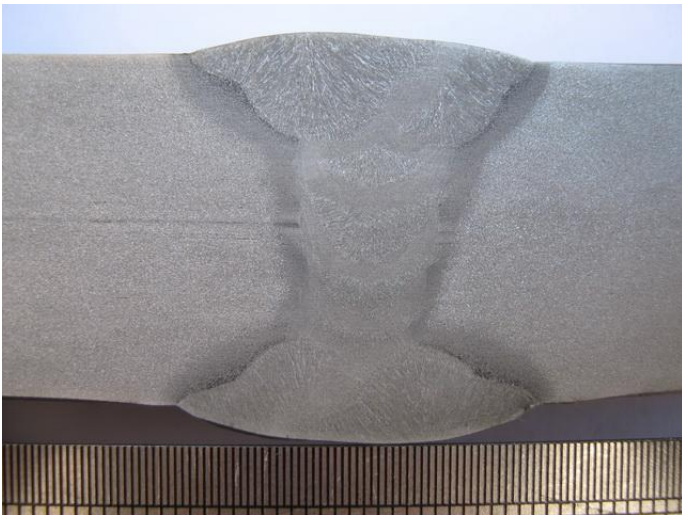
- b) types and proportional fractions of microstructural components and other microstructural features determined by imaging methods**

Microstructure

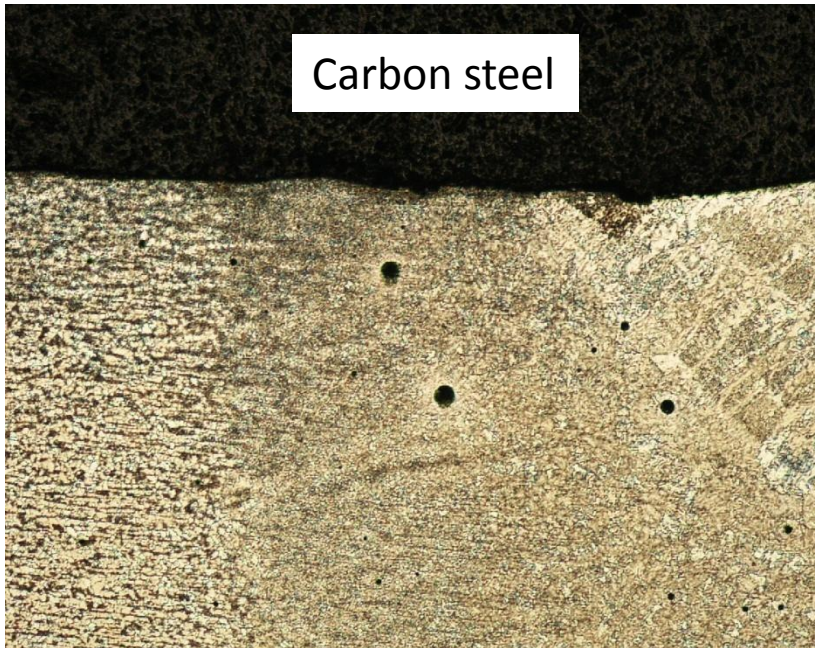
- c) morphology of microstructural components and units, ie. their shape, size, distribution and preferred orientation (texture)**

- d) qualitative and quantitative characteristics of lattice defects, macromolecules, or amorphous regions, which are also known as substructure.**

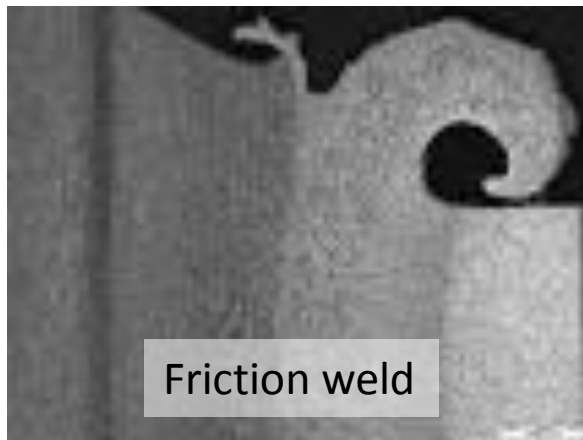
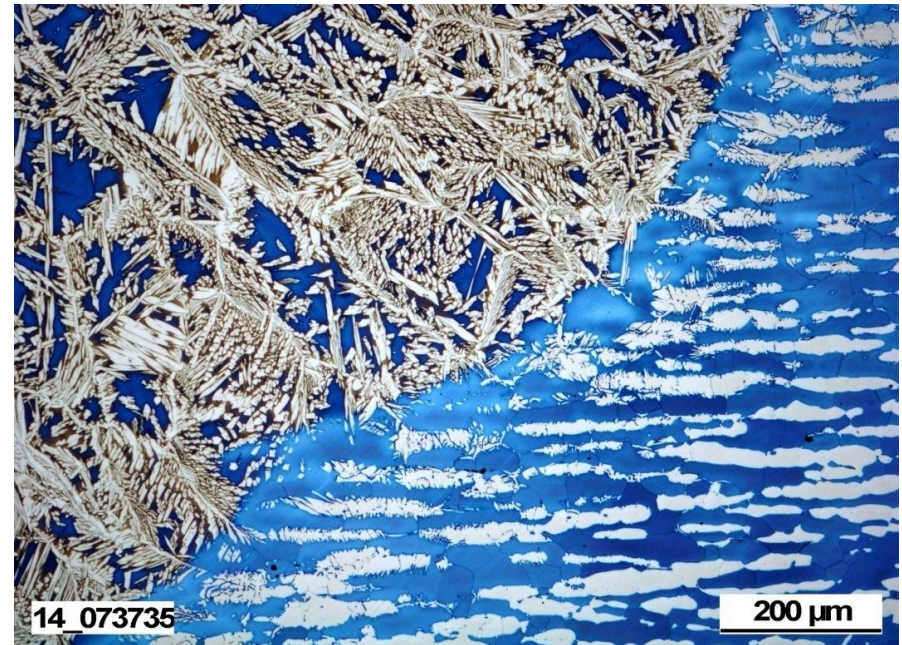
WELD



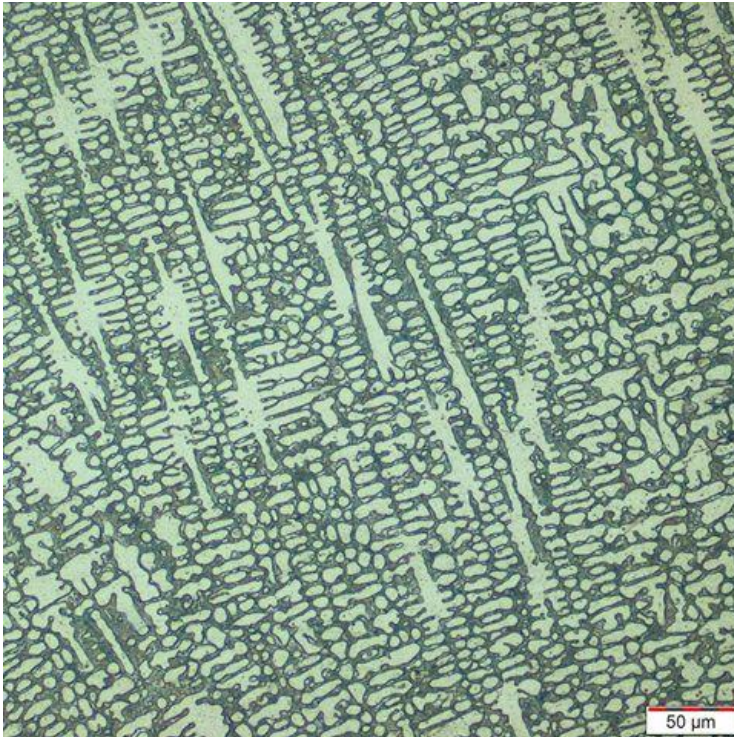
WELD



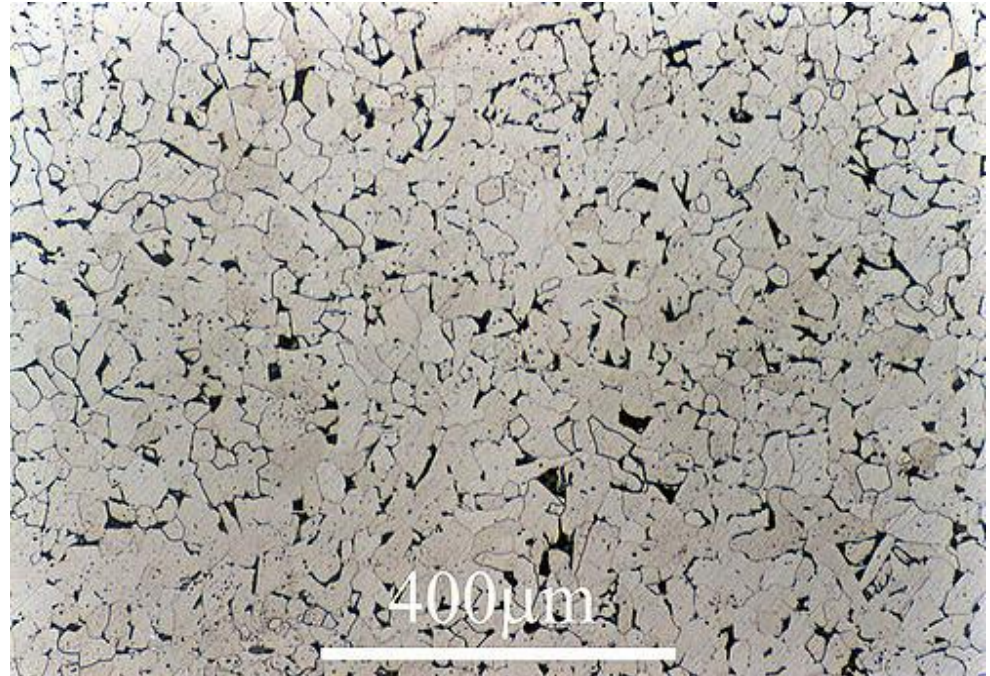
Duplex (austenitic-ferritic stainless steel)



WELD



Weld metal

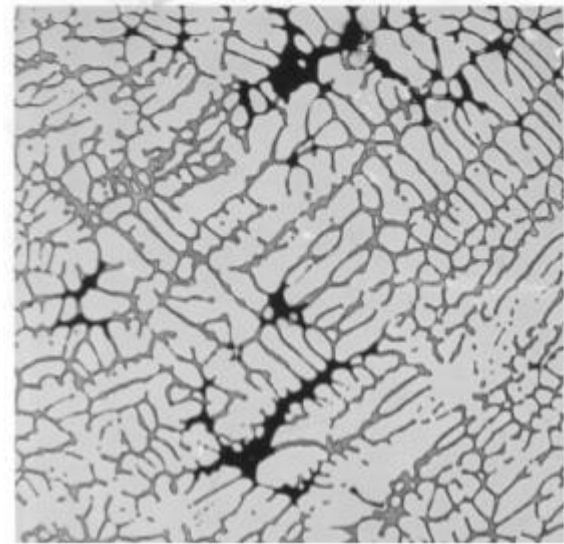


Parent (base) metal
Low carbon ferritic-pearlitic steel

Casting

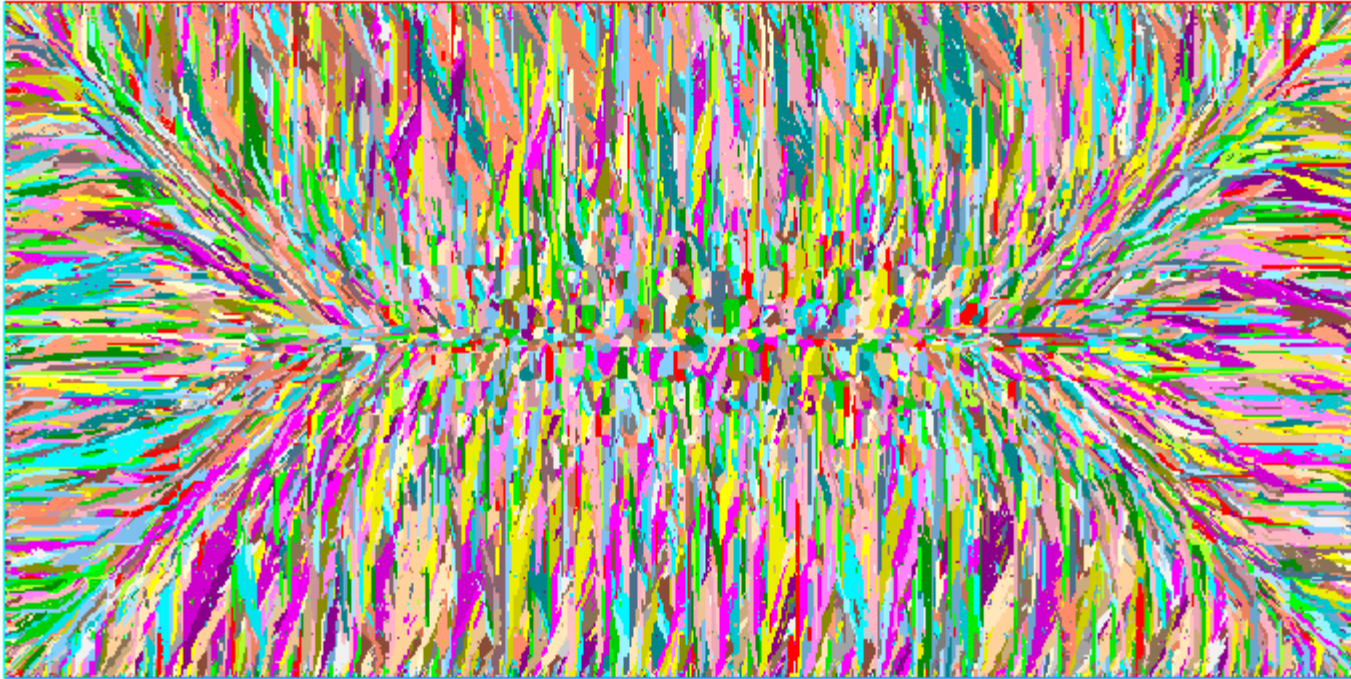


Shrinkage porosity

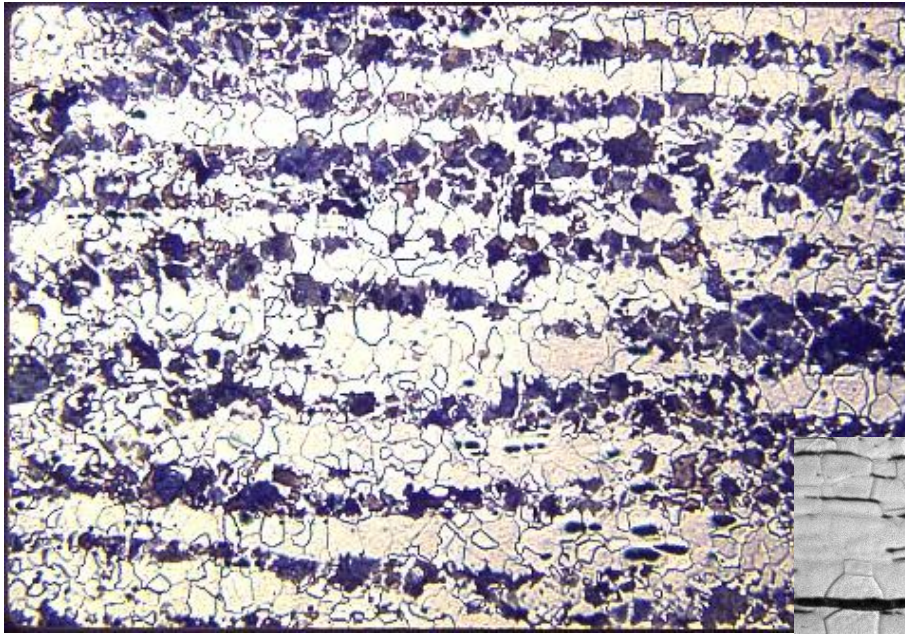


Casting

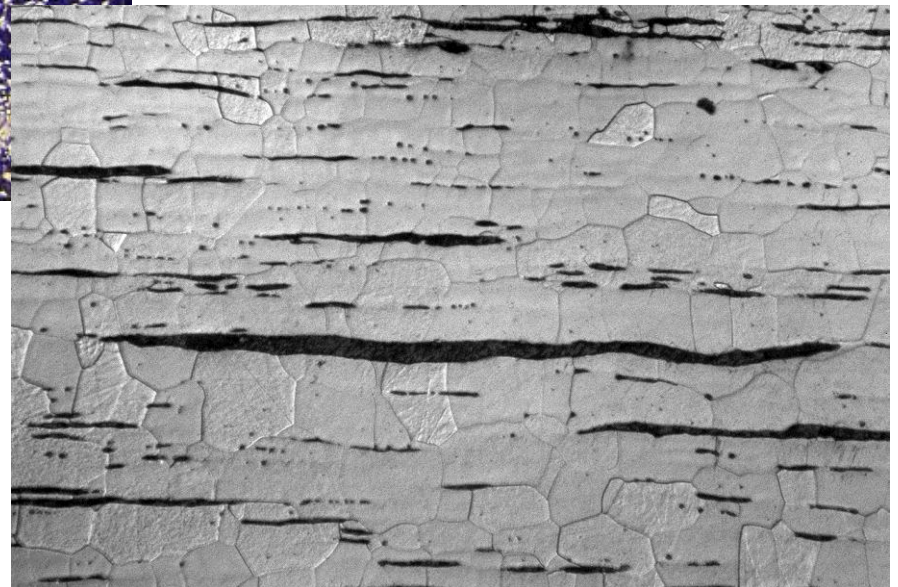
Grain growth in casting



Forming (rolling)

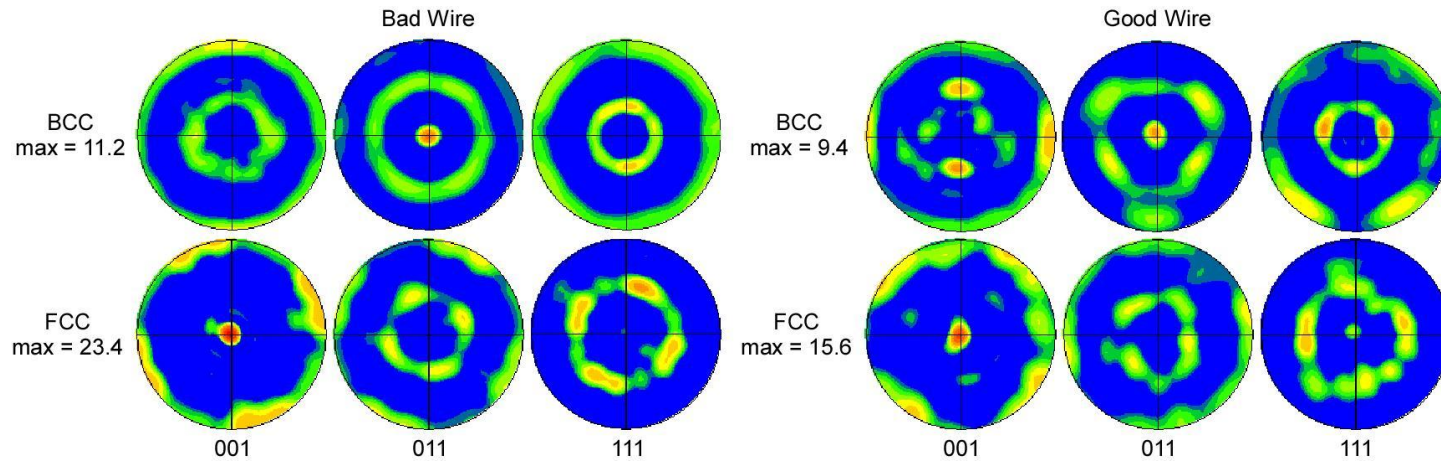
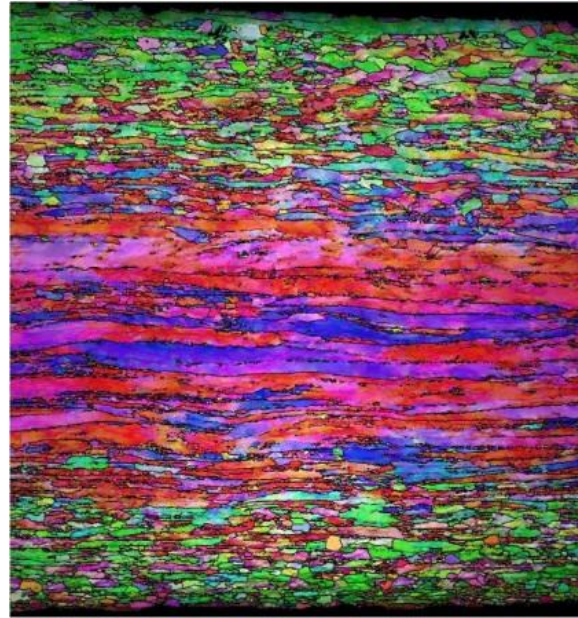


Bands of pearlite (dark grains)

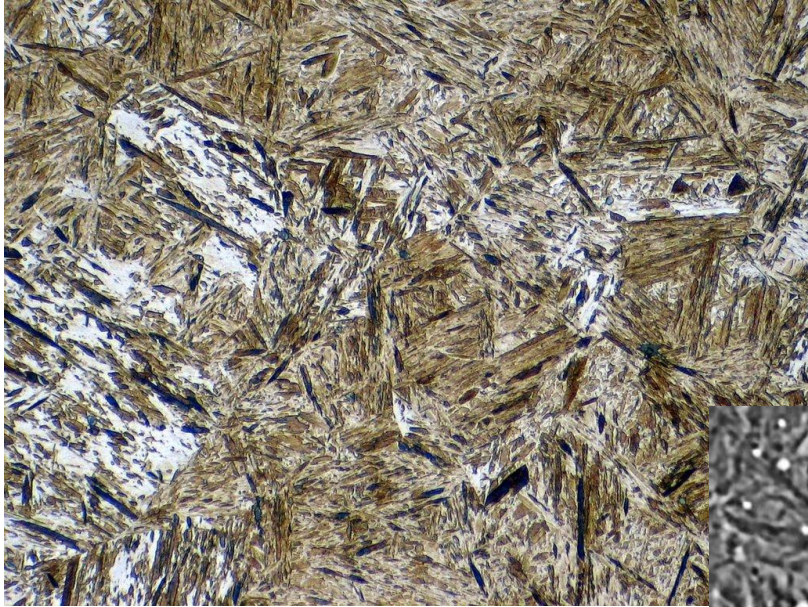


Linespacing of MnS inclusions

Texture - preferred orientation

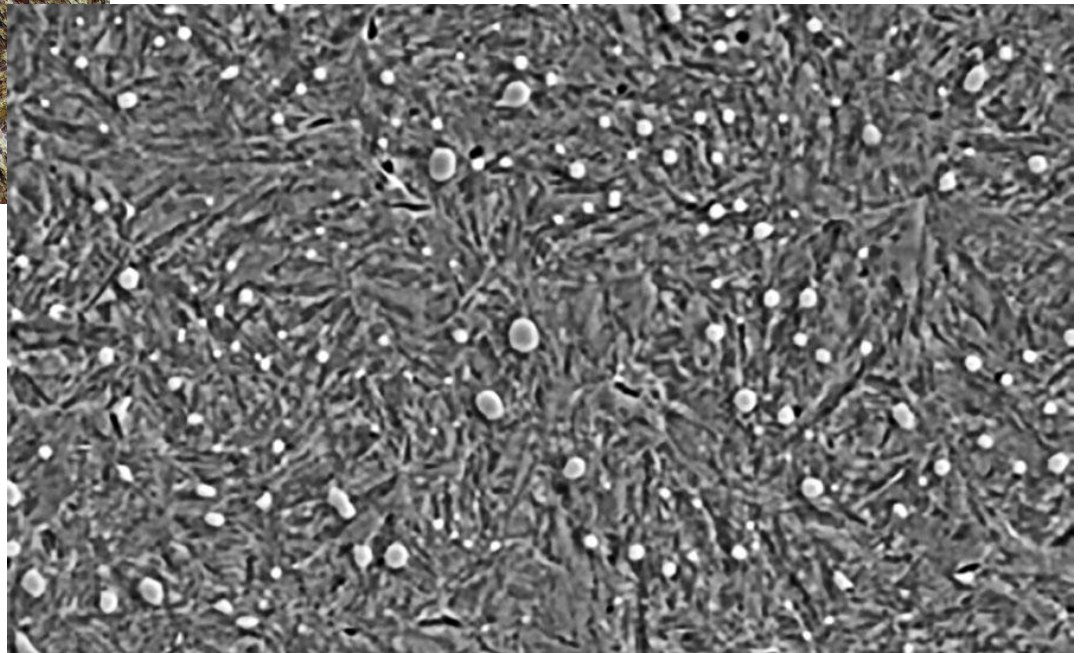


Martensite



Low alloyed steel

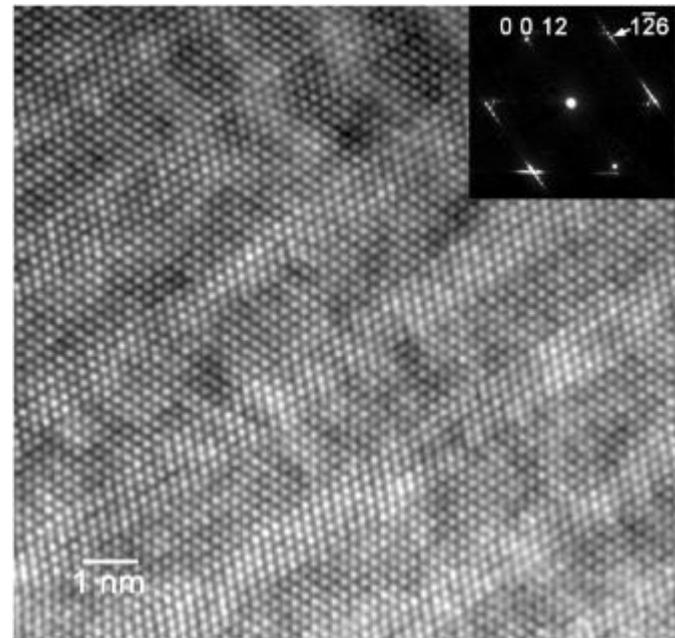
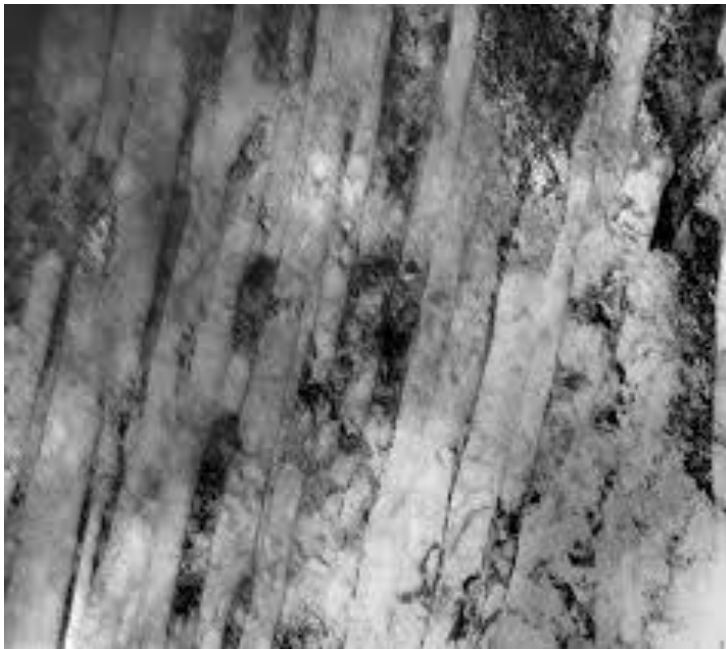
High speed steel
martensite and carbides



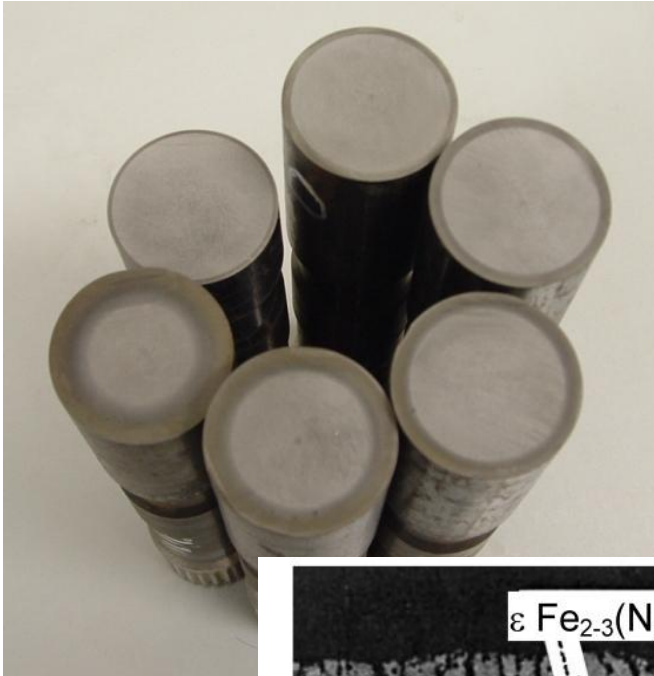
Martensite

substructure

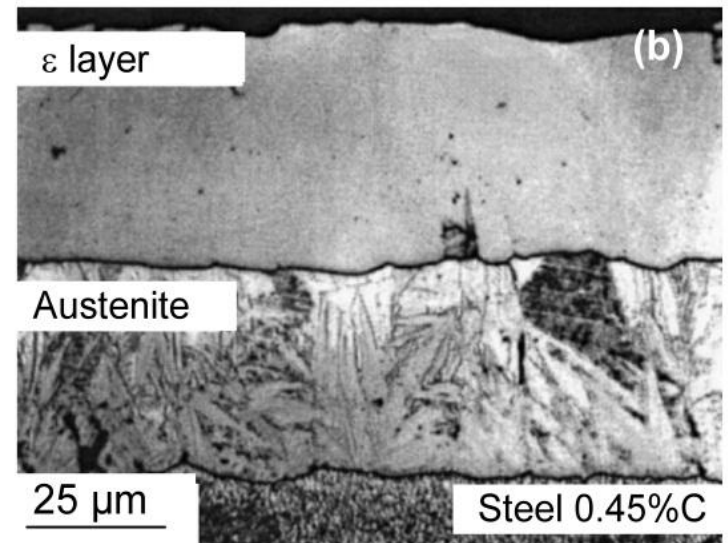
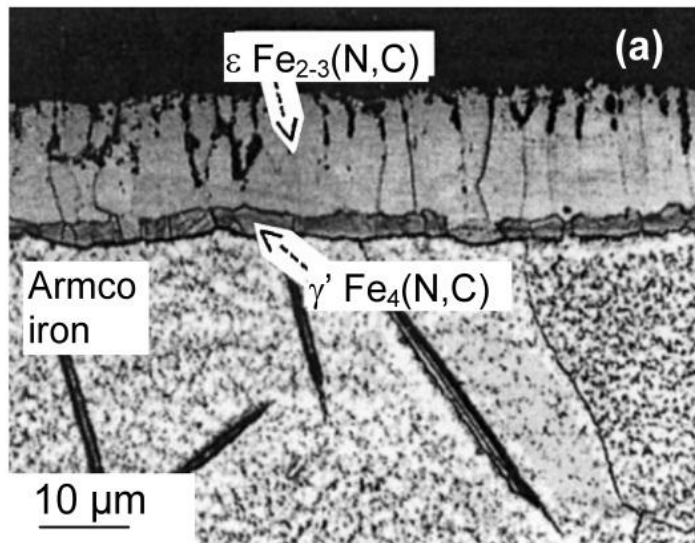
TEM and HRTEM microscopy



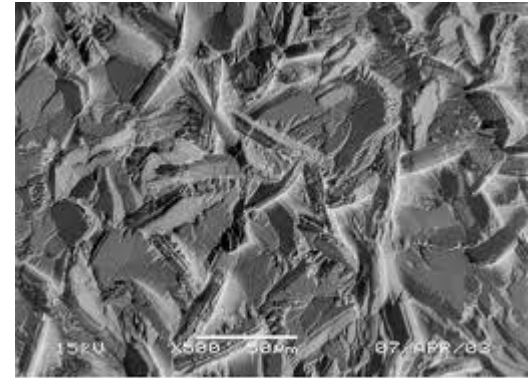
Layers - chemical heat treatment



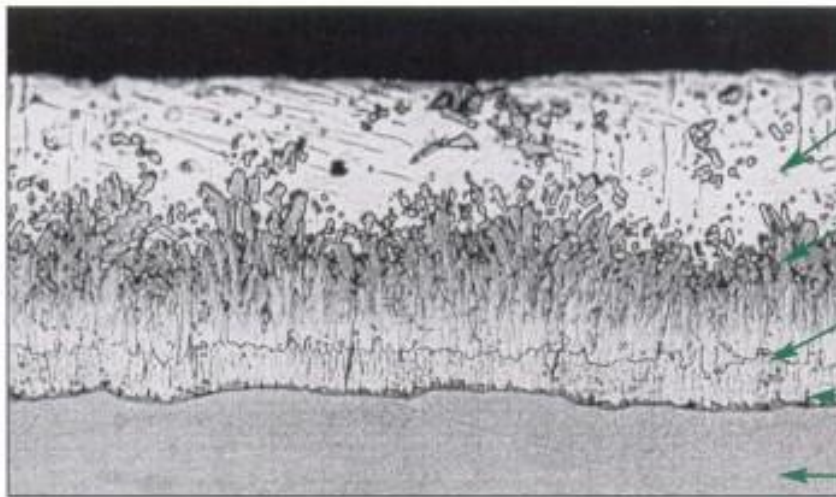
Nitrocarburised steel



Coatings

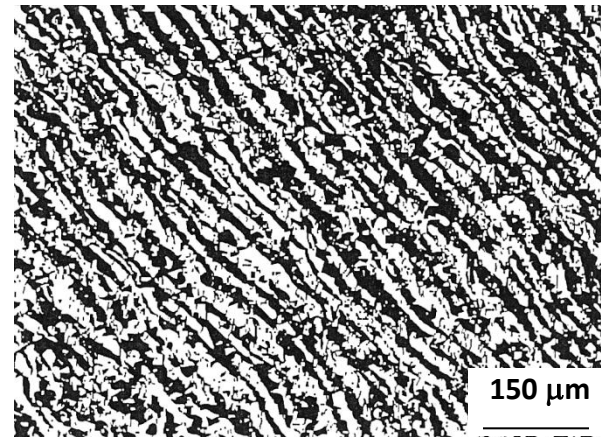
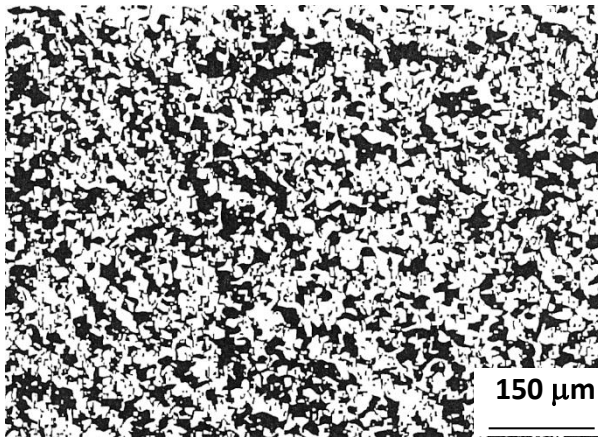
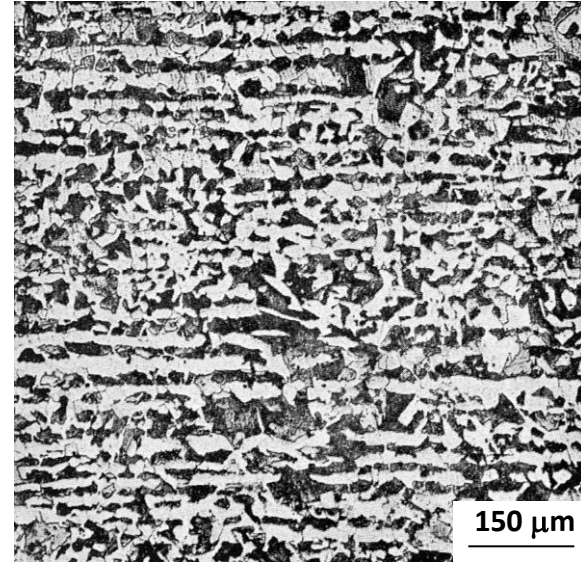
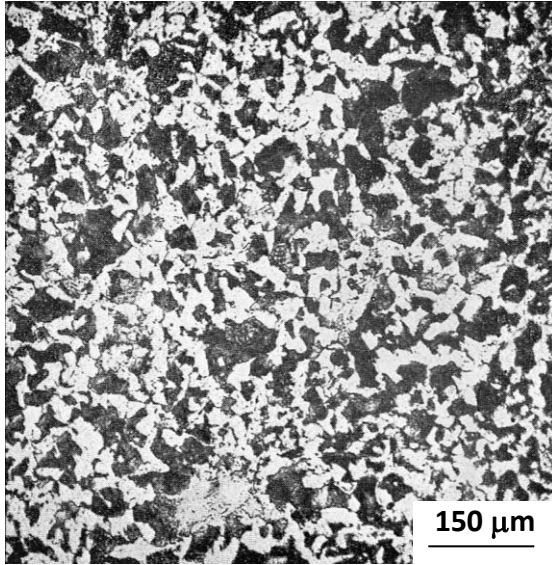


SEM of heavy zinc phosphate crystal (500X)



- Eta**
(100% Zn)
70 DPN Hardness
- Zeta**
(94% Zn 6% Fe)
179 DPN Hardness
- Delta**
(90% Zn 10% Fe)
244 DPN Hardness
- Gamma**
(75% Zn 25% Fe)
250 DPN Hardness
- Base Steel**
159 DPN Hardness

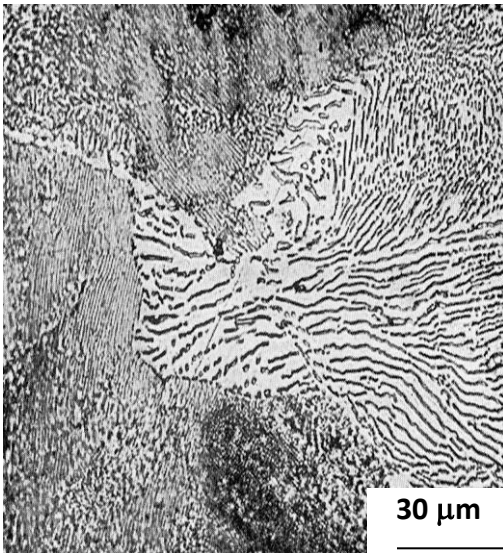
Microstructures after normalizing



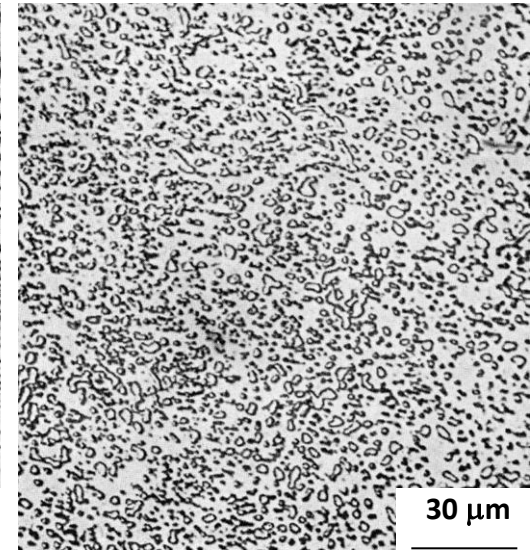
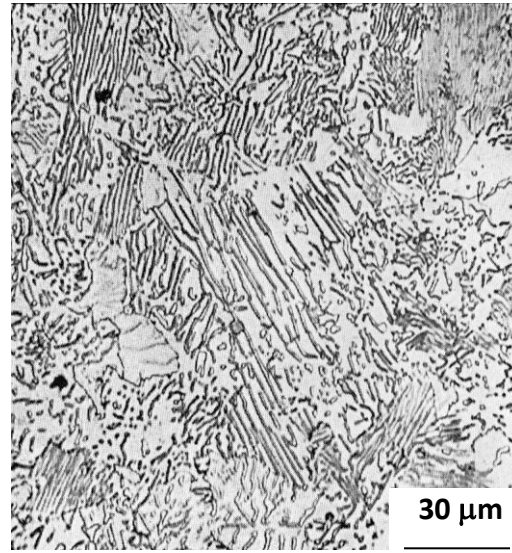
Pearlite spherodisation

influence of time at elevated temprature (700 °C)

SOFT ANNELING

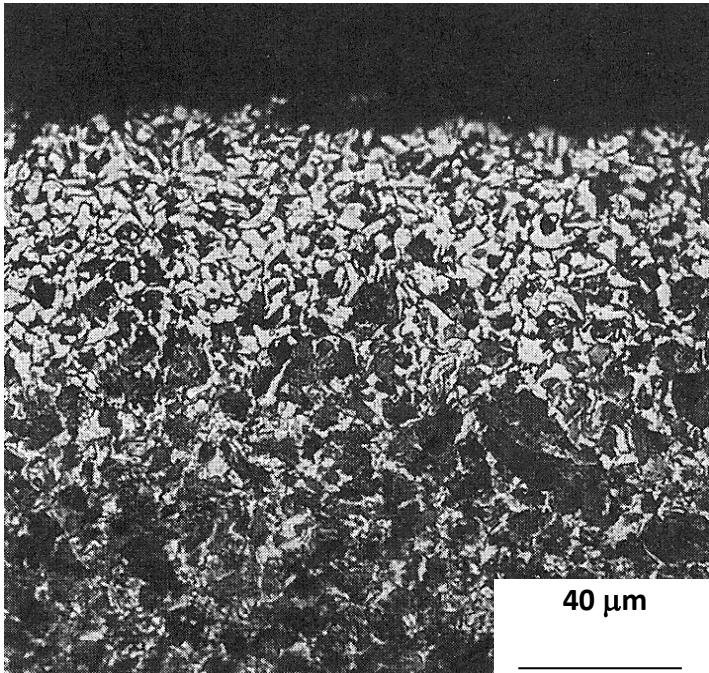


Lamellar pearlite

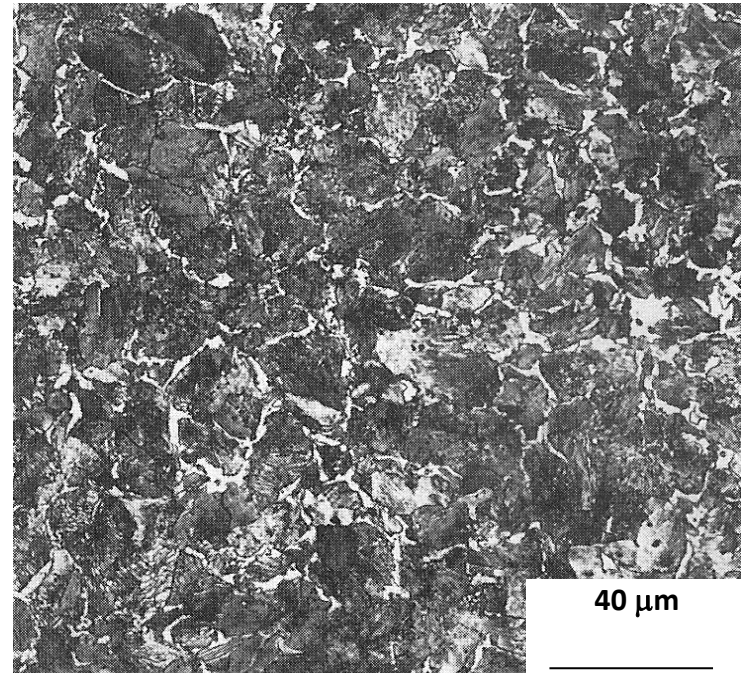


Globular pearlite

Decarburisation



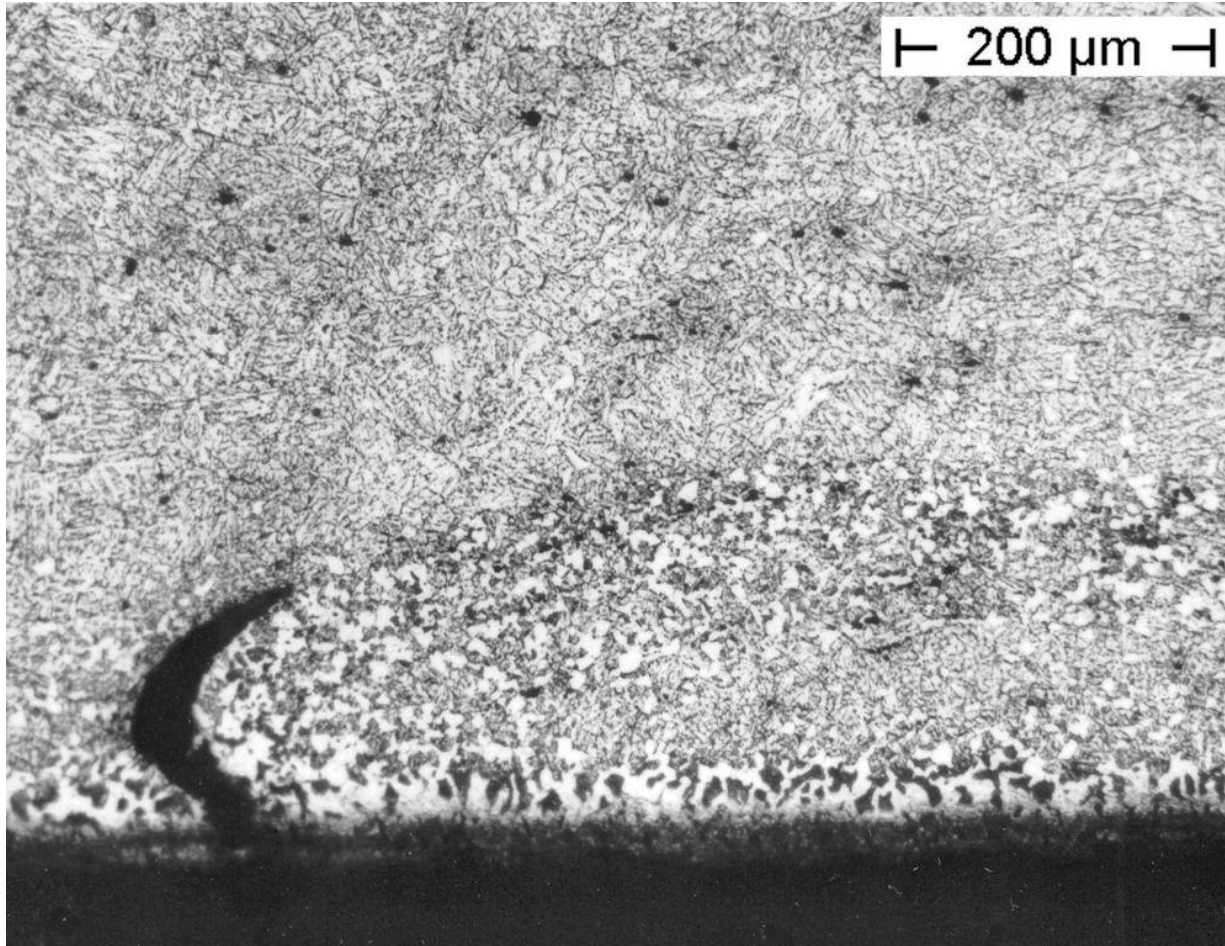
Surface decarburisation



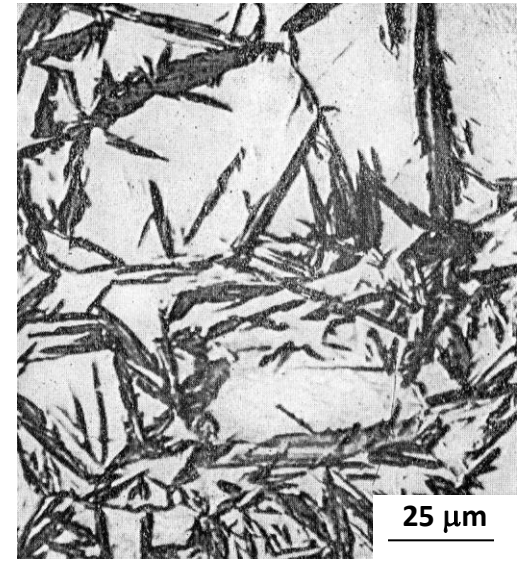
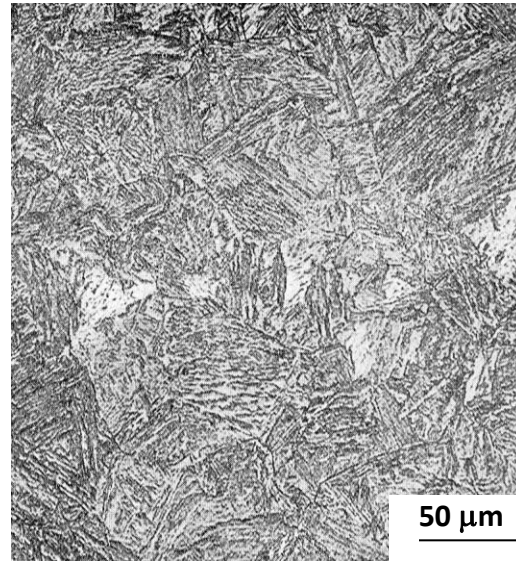
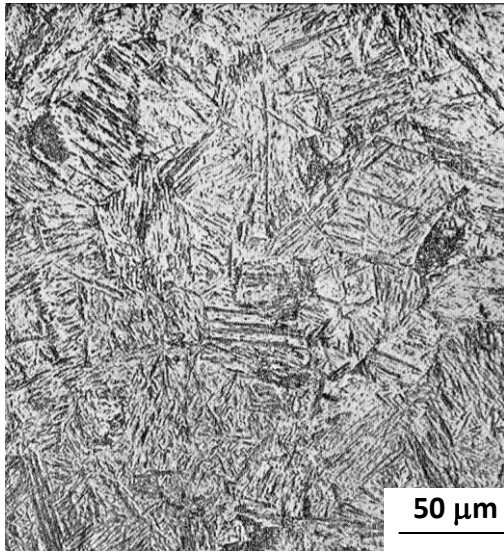
Core decarburisation

Decarburisation, overlap

forging, shot peening etc.

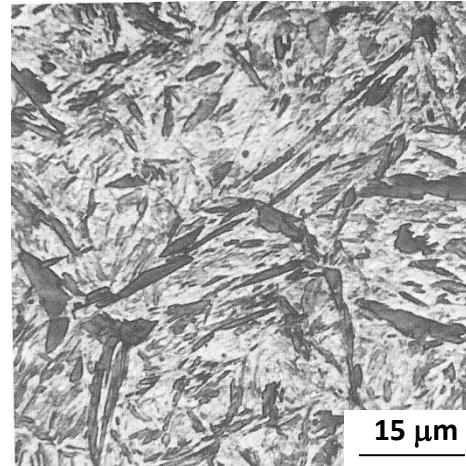
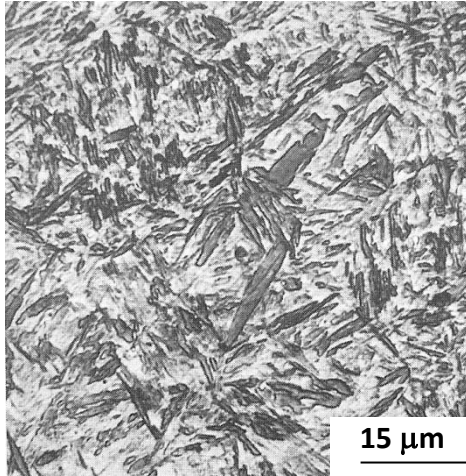


Martensite

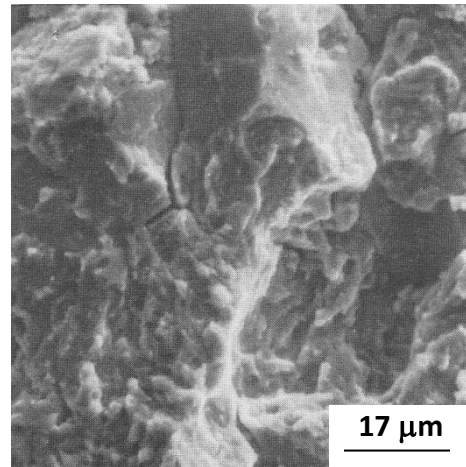
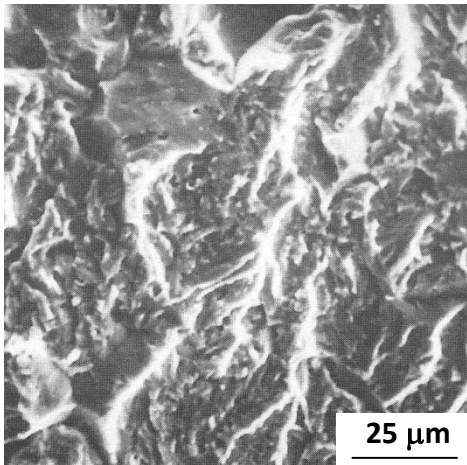


Martensite and retained austenite

Quenching - overheated

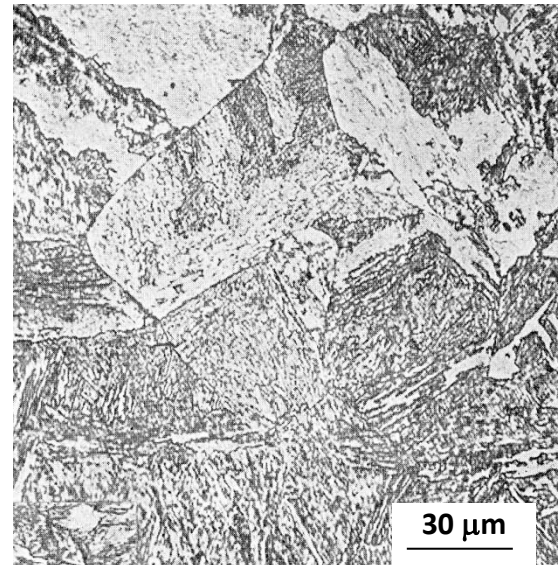
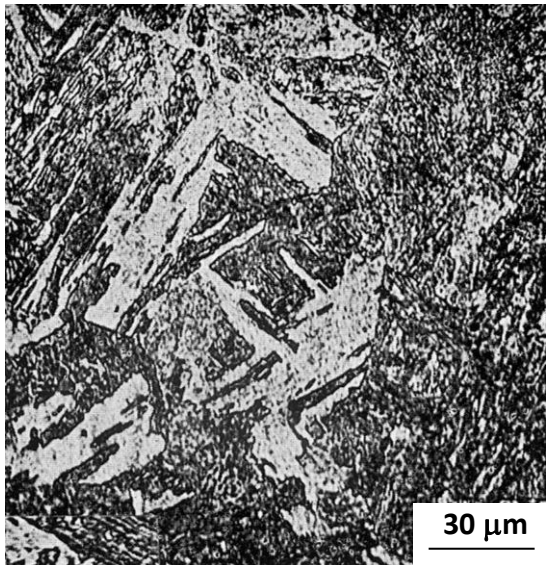
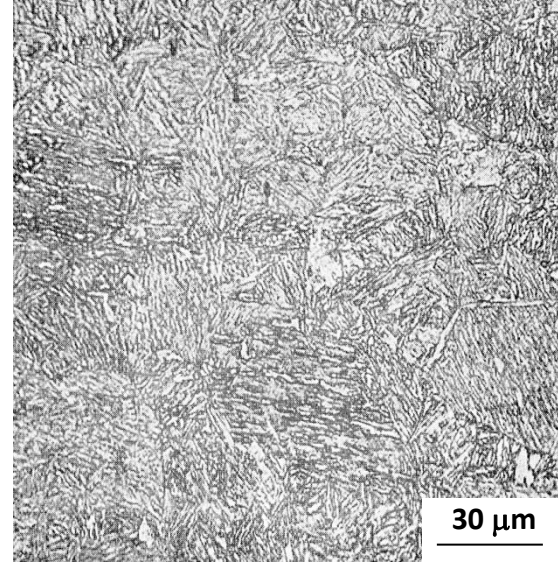
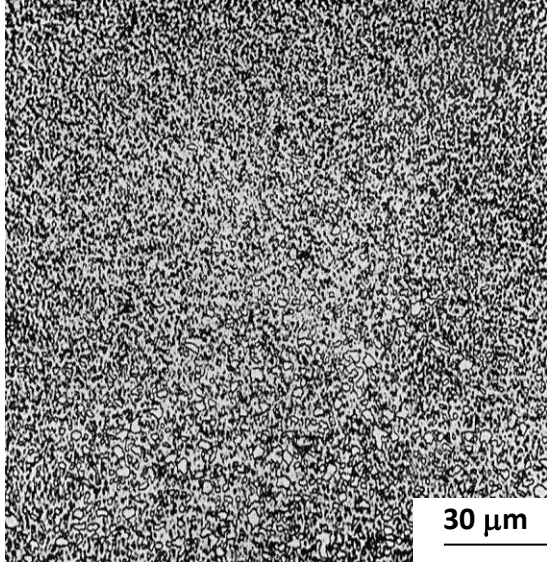


coarse martensite



intergranular fracture

Tempering - sorbite



Measurement of depth of hardened layers on metallographic sections

1. Carburised or carbonitrided parts (EN ISO 2639)

Hardness Limit = 550 HV

CHD (Eht) = Distance from surface to point where hardness is 550 HV

2. Induction or flame hardened parts (EN 10328, ISO 3754)

Hardness Limit = 80 % × (Minimum) surface hardness.

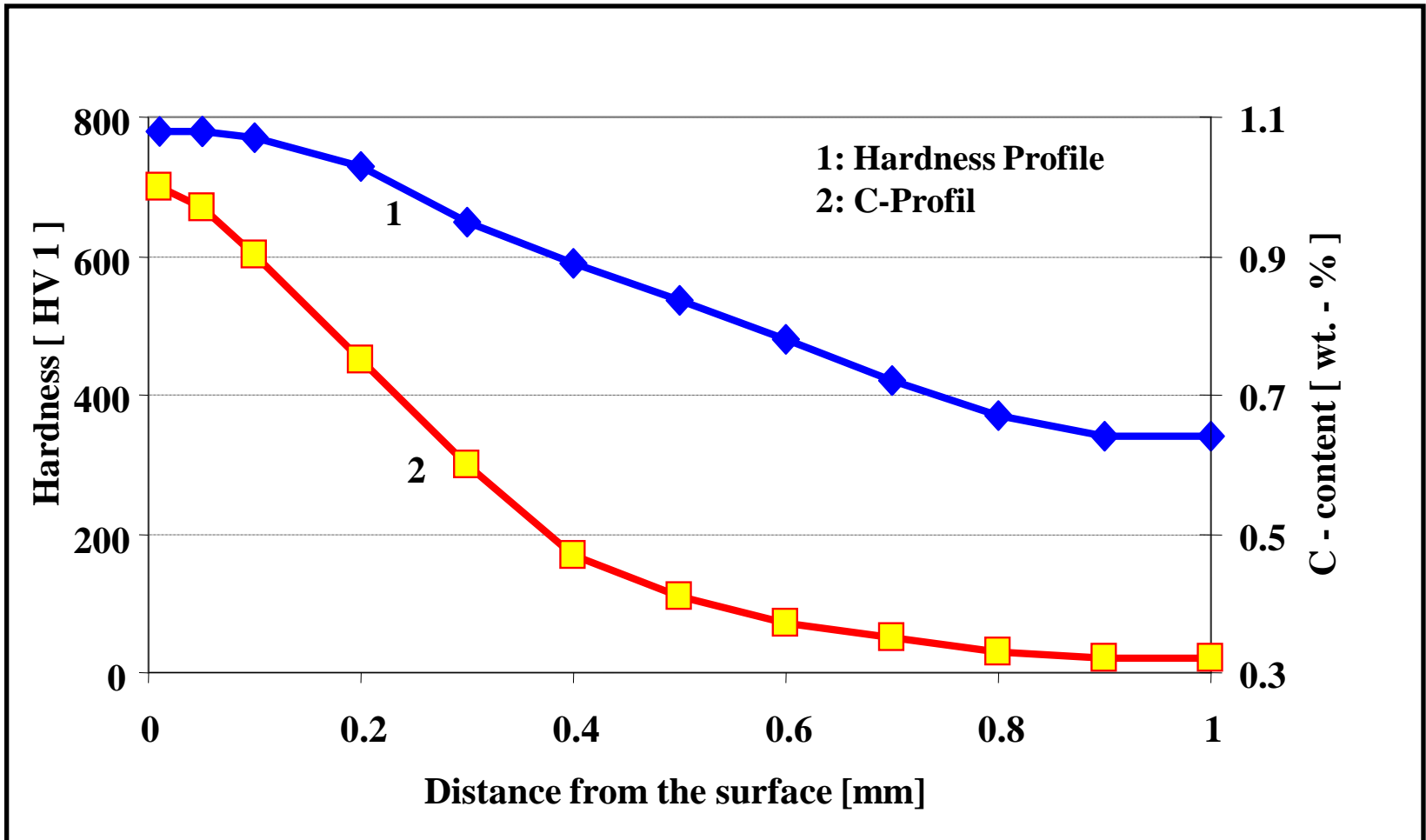
CHD (Rht) = Distance from surface to point where hardness is 80 % of (minimum) surface hardness.

3. Nitrided parts (DIN 50190-3)

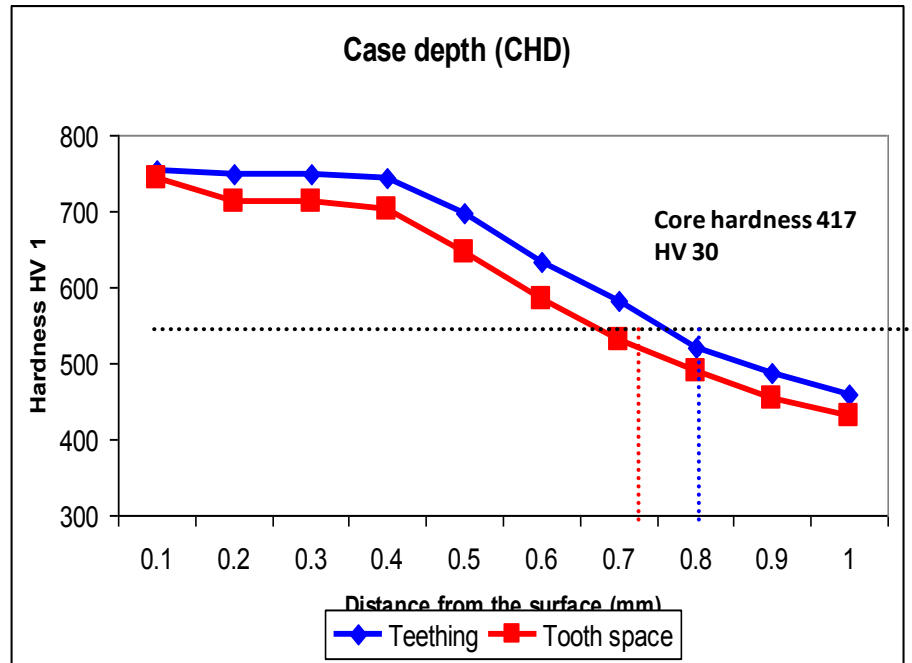
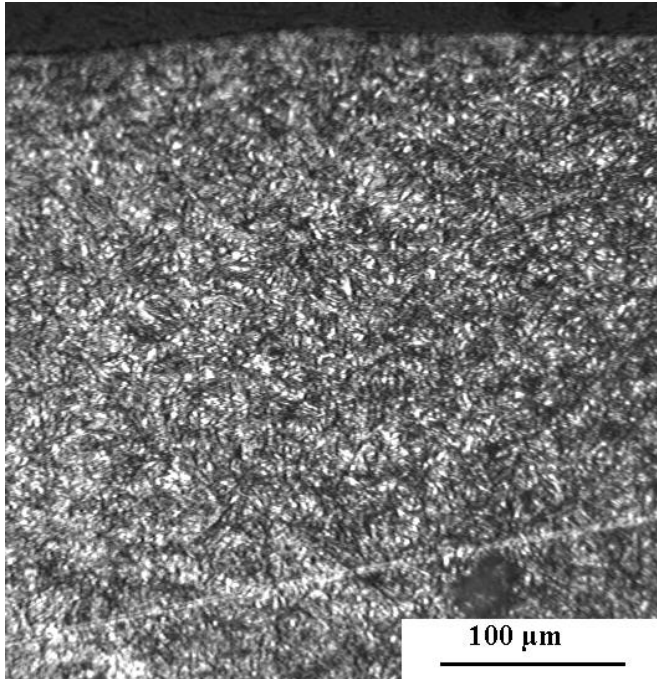
Hardness Limit = Core Hardness +50 HV.

CHD (Nht, NCD) = (Max.) Distance from the surface to the point where hardness is 50 HV₁ above core hardness

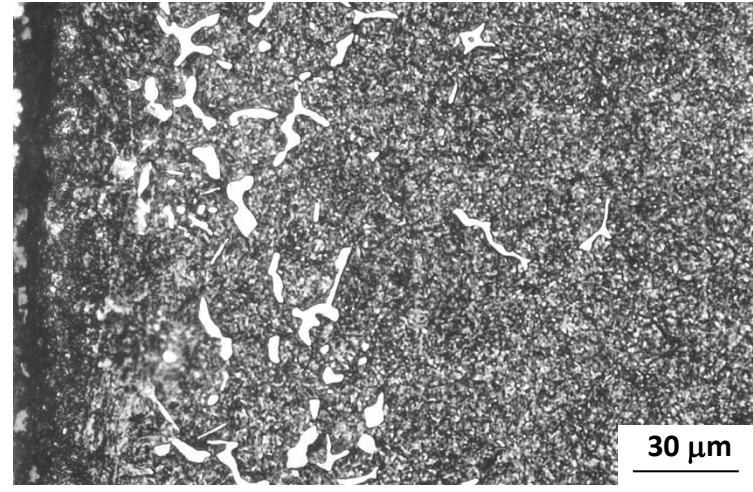
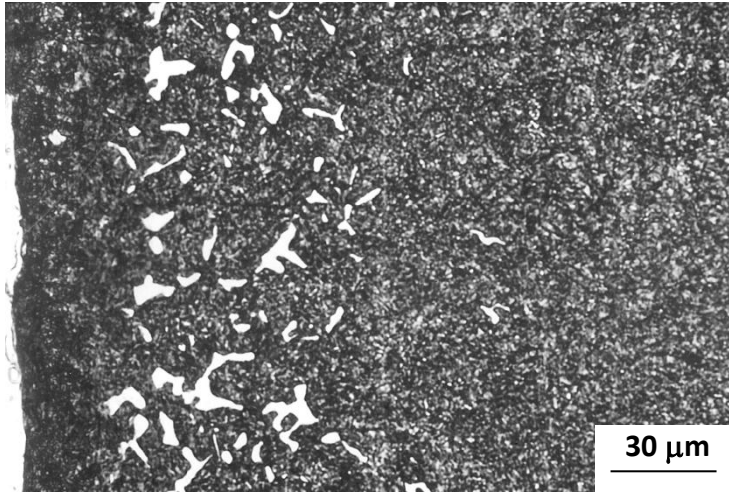
Case hardening



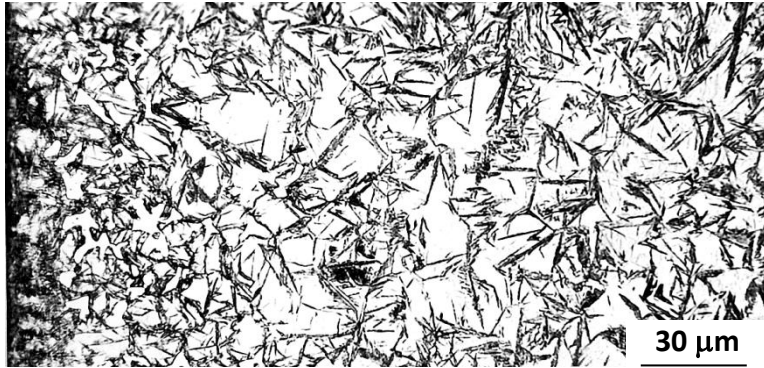
Case hardening



Case hardening - defect



Net of secondary cementite on grain boundaries of prior austenite



Retained austenite, net of secondary cementite

Typical nitrided layer

oxide layer, 1-2 μm :

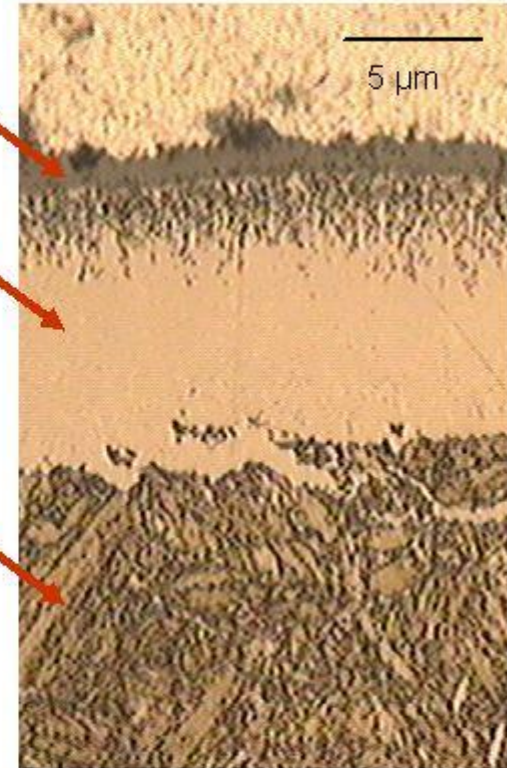
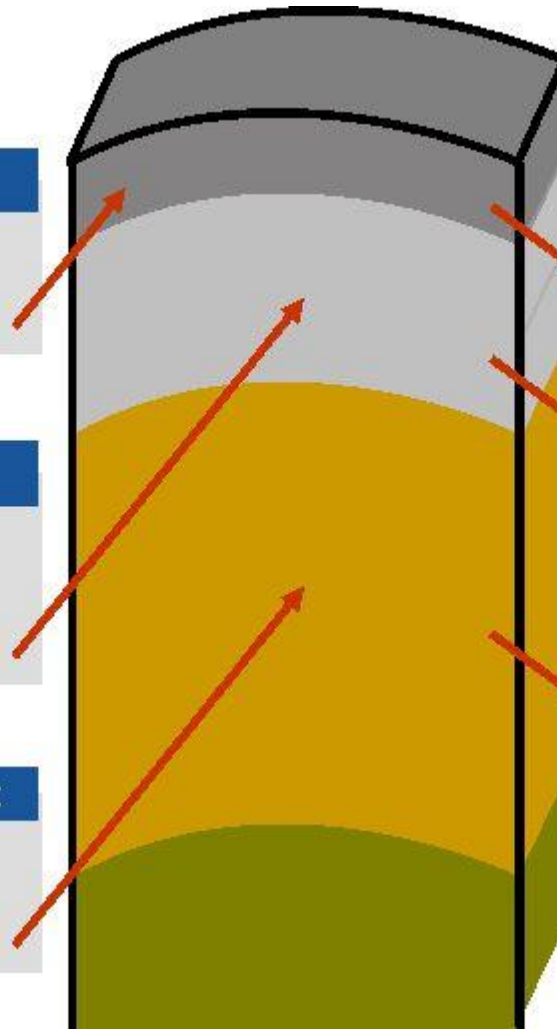
- running-in layer
- friction decreasing
- corrosion resistant

white layer, 5-20 μm :

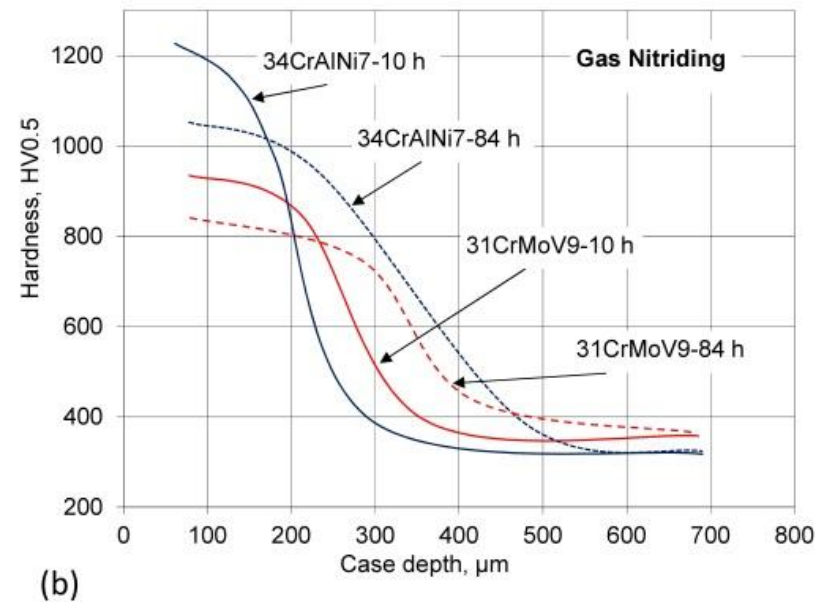
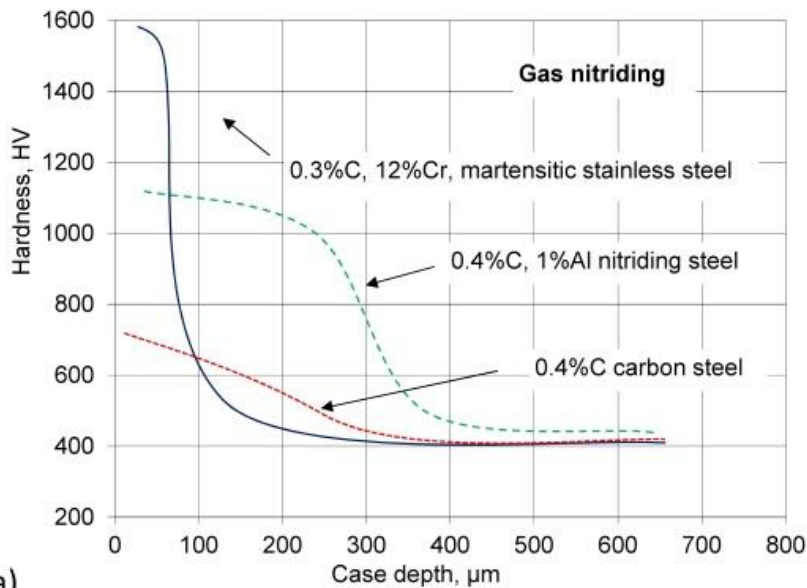
- protection against abrasive and adhesive wear
- low friction coefficient
- high hardness

diffusion zone, 10-1000 μm :

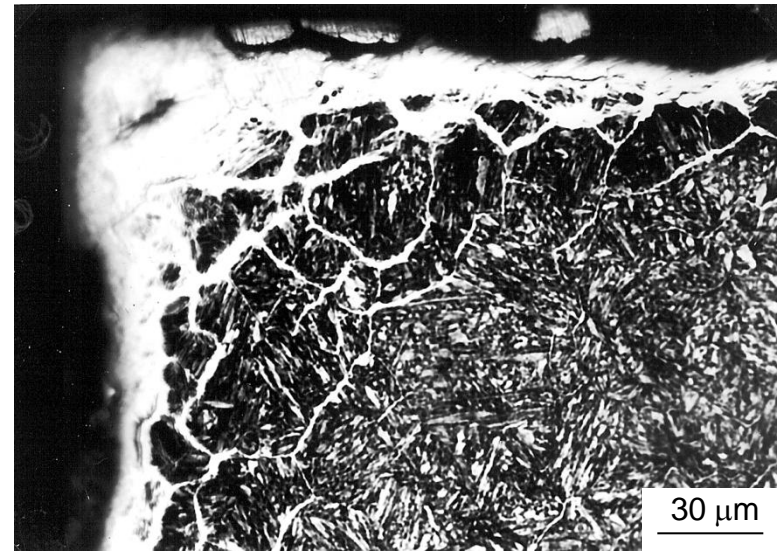
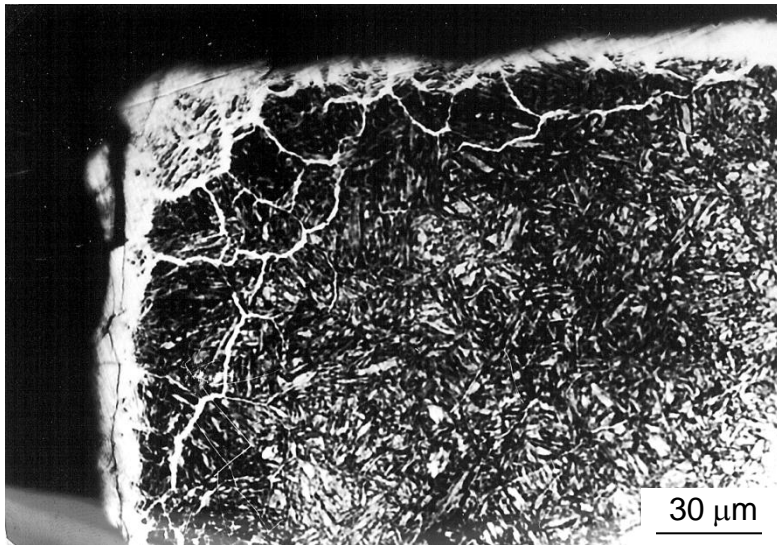
- high compressive stress
- high fatigue strength
- hardness higher than substr.



Hardness course of nitrided layers



Defect of nitriding



Net of nitrides on grain boundaries of prior austenite