RUHR-UNIVERSITÄT BOCHUM





Fundamental Aspects of Materials Science and Engineering (FAMSE)

Exercise 2

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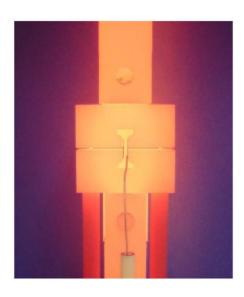
Outline

- (1) Creep Fundamentals
- (2) Ni-base Superalloy Single Crystals
- (3) Martensitic Transformation & Shape Memory Alloys (SMA)
- (4) SMA Fracture Mechanics & Basics of Structural Fatigue



Part I

Creep Fundamentals



1.1 What is creep?

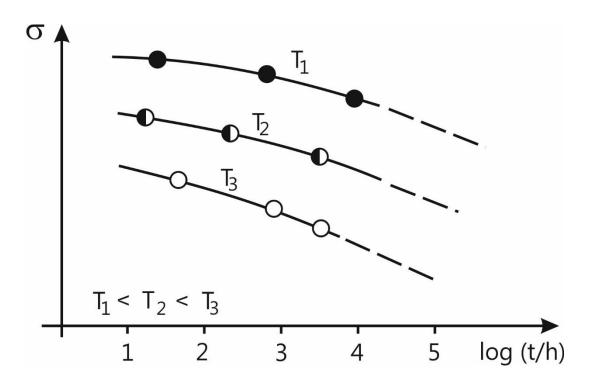


Creep is the time dependent plastic deformation under high temperature and load.

Low temperature deformation	High temperature deformation
Edge dislocations cannot climb	Edge dislocations can climb
Minimal stress needed for a plastic deformation (yield strength)	Plastic deformation at any stress
Increase in stress necessary for additional pl. deformation	Constant stress → creep deformation
Governed by dislocation motion	Dislocation motion Diffusion Sliding of grain boundaries
Not time dependent	Time dependent deformation

1.2 What is a stress rupture plot (text and drawing)?





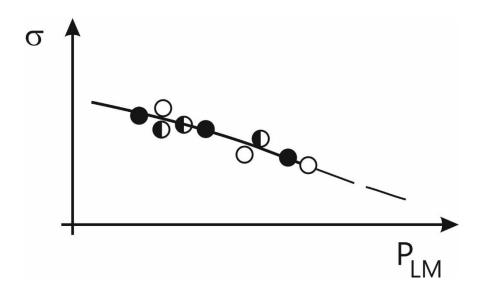
- Important for lifetime estimation (critical stress for required life time)
- Engineers need this for temperature and stress designs
- Extrapolation for very long life time

1.3 What is the Larson Miller Parameter?



- Phenomenological parameter, which allows to plot experimental results from different temperatures onto one master curve.
- P_{LM} is a temperature compensated time

Larson Miller Plot



$$P_{LM} = T(C + log t)$$

LARSON MILLER PARAMETER:

T = temperature

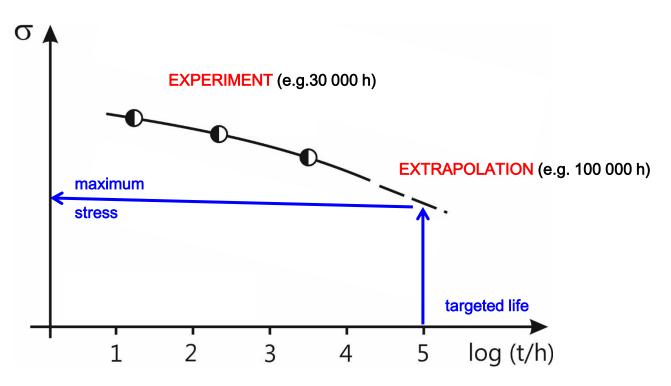
C = constant (often: 20)

t = time

1.4 Why is there a need to extrapolate from short term creep data into the long term regime (text and drawing)?



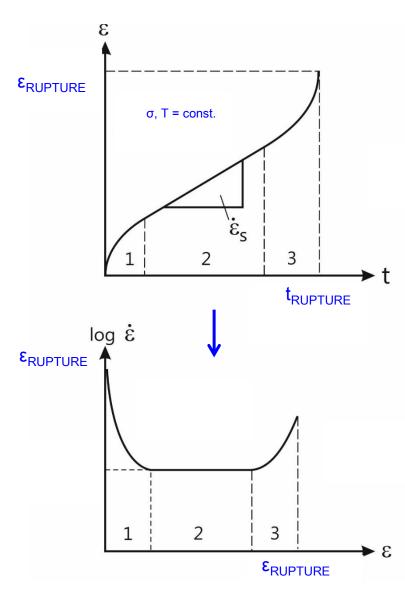
T = const.



- estimate critical stress for a required target life
- extrapolation for very long rupture times, which would exceed experimental testings

1.5 How does a generic creep curve look like (plots: strain vs. time, log strain rate vs. strain)?





3 stages of creep must be marked!

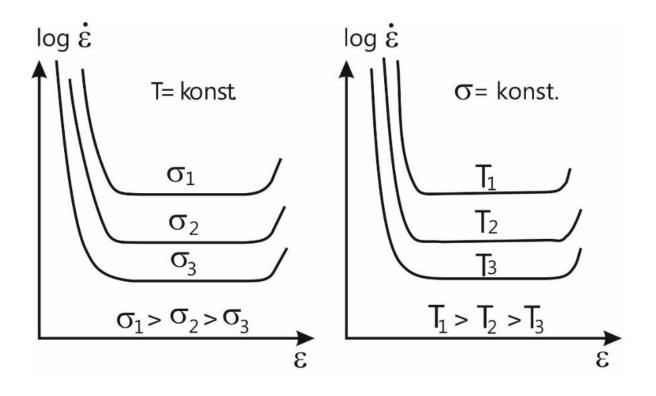
- Primary: increasing dislocation density
- <u>Secondary:</u> dynamic equilibrium between dislocation formation and annihilation

$$d \rho_{total} = 0 \longrightarrow r \cdot dt + h \cdot d \varepsilon = 0$$

<u>Tertiary:</u> pore formation, Ostwald ripening, ...

1.6 Explain the dependence of creep-curves on stress/temperature changes (text and drawing)?





- An increase in stress leads to an increased creep-rate
- An increase in temperature leads to an increased creep-rate

1.7 How does the secondary creep rate depend on stress and temperature (equation)?



Sherby-Dorn equation

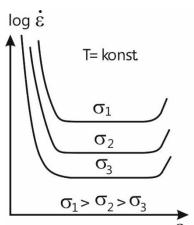
$$\dot{\varepsilon}_s = A' \cdot \sigma^n \cdot \exp\left(-\frac{Q_{\rm app}}{RT}\right)$$

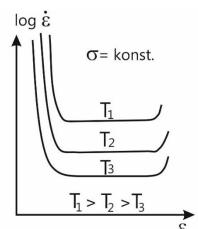
Symbols:

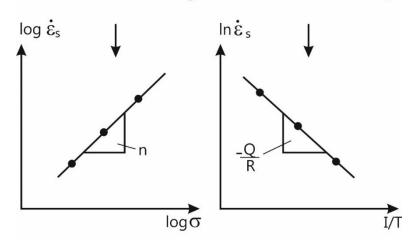
- ε'_s secondary creep rate
- A' constant
- σ stress
- n stress exponent (e.g. 5-10)
- Q_{app} apparent activation energy (e.g. 500 kJ/mol)
- R,T gas constant and temperature

1.8 How can the stress exponent n and activation energy Q_{app} be determined?









$$\dot{\varepsilon}_{\min} = A' \cdot \sigma^n \cdot \exp\left(-\frac{Q_{\text{app}}}{RT}\right)$$

Calculation rules for logarithm Equation of a line with slope n

$$\underbrace{\log(\dot{\varepsilon}_{min})}_{y} = \underbrace{n}_{m} \cdot \underbrace{\log(\sigma)}_{x} + \underbrace{\log\left[A' \cdot \exp\left(-\frac{Q_{\mathrm{app}}}{RT}\right)\right]}_{b}$$

$$\dot{\varepsilon}_{\min} = A' \cdot \sigma^n \cdot \exp\left(-\frac{Q_{\text{app}}}{RT}\right)$$

Calculation rules for logarithm

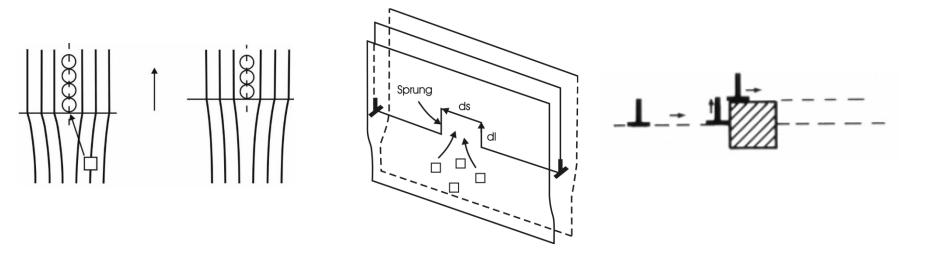
Equation of a line with slope -Q/R

$$\underbrace{\ln(\dot{\varepsilon}_{min})}_{y} = \underbrace{-\frac{Q_{app}}{R}}_{m} \cdot \underbrace{\frac{1}{T}}_{x} + \underbrace{\ln(A' \cdot \sigma^{n})}_{b}$$

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1.9 What role plays dislocation climb in creep?

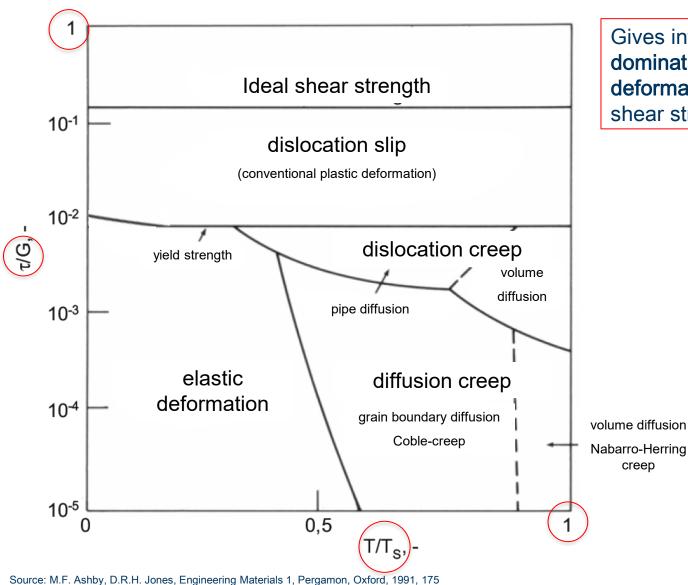




- Creep mechanism involving dislocation climb (vacancy diffusion)
- Often creep rate controlling mechanism
- When glide is blocked, climb enables dislocations to bypass these obstacles by moving out of their slip plane into a adjacent plane.
- Climb becomes faster at higher temperatures as diffusion rates increase

1.10 Schematically draw an Ashby map and explain the principle behind it.



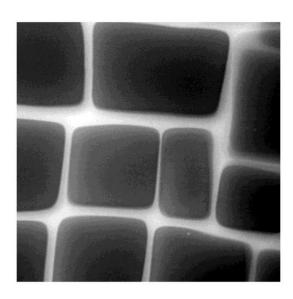


Gives information about the dominating mechanism of deformation in dependence of shear stress and temperature.



Part II

Ni-base Superalloy Single Crystals



2.1 Name five alloy elements which are typically contained in superalloy single crystals except for Ni.



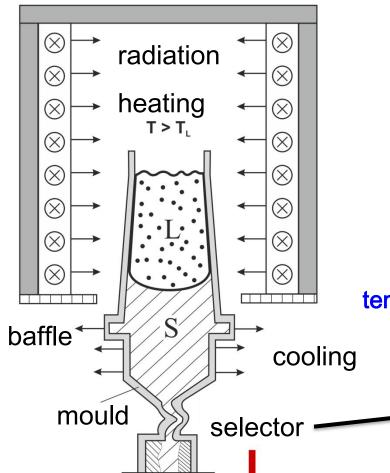
- **Ti**: forming γ'
- AI: forming γ'
- Re: Significantly enhances high-temperature creep strength
- Co: Improves hot corrosion resistance and stabilizes the microstructure
- W: solid solution strengthening, improves creep resistance
- **Ta**: Strengthens the γ' phase and improves oxidation resistance

Fe: substitute for Ni → cheaper

2.2 How does the Bridgman process work (drawing)?

pull out rate





water cooling

Solidification in <001>

$$\dot{T} = \frac{dT}{dt} = \frac{dT}{dz} \cdot \frac{dz}{dt} = G \cdot v$$

G = temperature gradient (K/mm)

v = Solidification speed (mm/min)

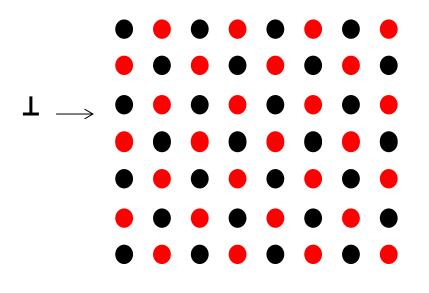
temperature gradient

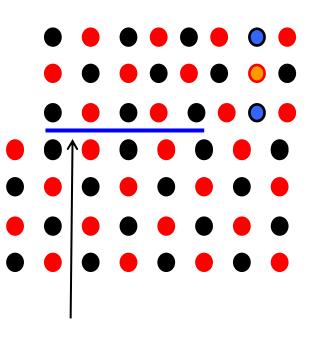
Seeding is also possible!

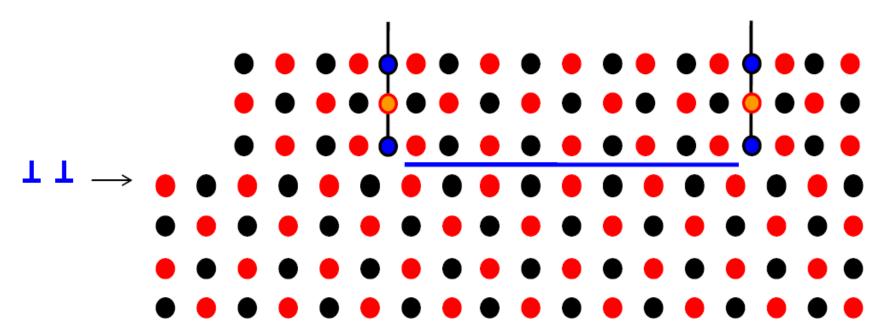
2.3 What is the problem with a dislocation entering an ordered crystalline phase?



The <u>order</u> is affected/destroyed. An <u>antiphase boundary</u> is created, which is connected with an <u>increase in energy</u>. Hence, it is easier for a "superdislocation" to glide through the particle by pairwise cutting. A superdislocation exists of <u>two dislocations</u> of <u>same sign</u>.





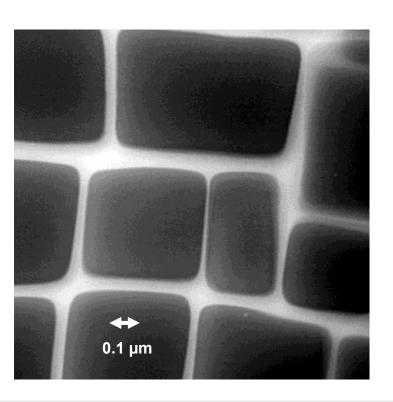


2.4 Why do γ'-precipitations form cubes?

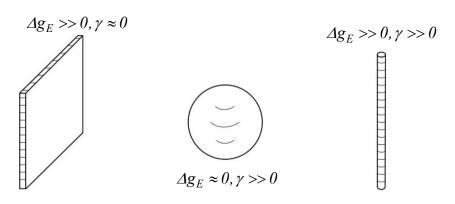
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The precipitate morphology is a compromise between **surface energy** and the energy stored by the **elastic deformation** of the surrounding matrix material in order to minimize the total stored energy of the system.

In Ni-base super alloys the γ/γ phases are coherent, the atoms of the two phases occupy one common lattice. The **interfacial energy minimizes in [100]** direction.

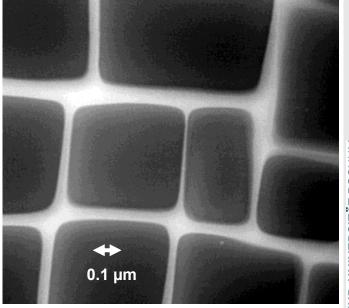


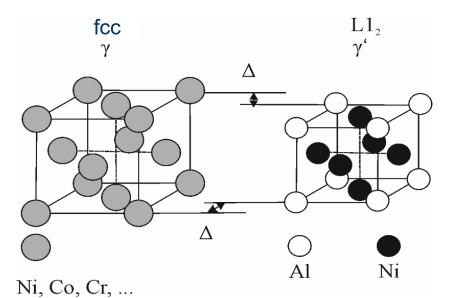
$$\Delta G(r) = \frac{4}{3}\pi r^3 \Delta G_v + 4\pi r^2 \gamma_{ph} + \frac{4}{3}\pi r^3 \Delta G_m$$
 volume surface mechanical



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2.5 What are the γ- and γ'-phases in Ni-based superalloys? Draw the unit cells and explain the two basic differences.





γ

Non-ordered solid Solution

Matrix phase

Misfit

Ni, Co, Cr, Mo, W, etc.

Y

Ordered phase

Coherent precipitation

Misfit

Al/Ti & Ni

 Ni_3X

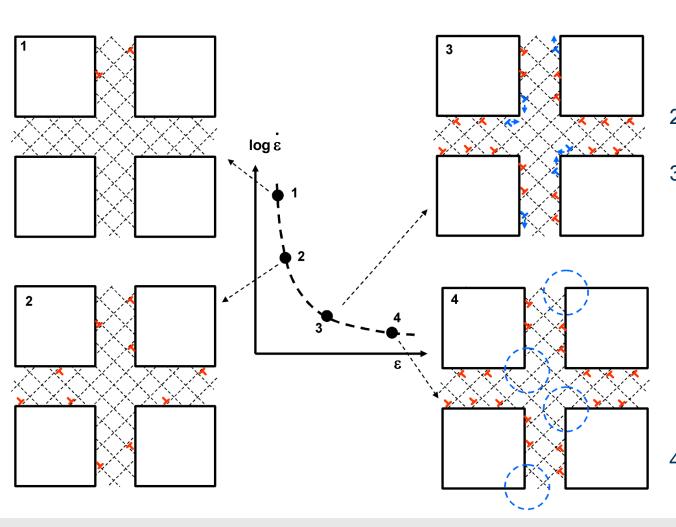
 $\delta = \frac{2 \cdot \left(a_0(\gamma') - a_0(\gamma)\right)}{a_0(\gamma') + a_0(\gamma)}$

 Δ = misfit

δ = misfit parameter

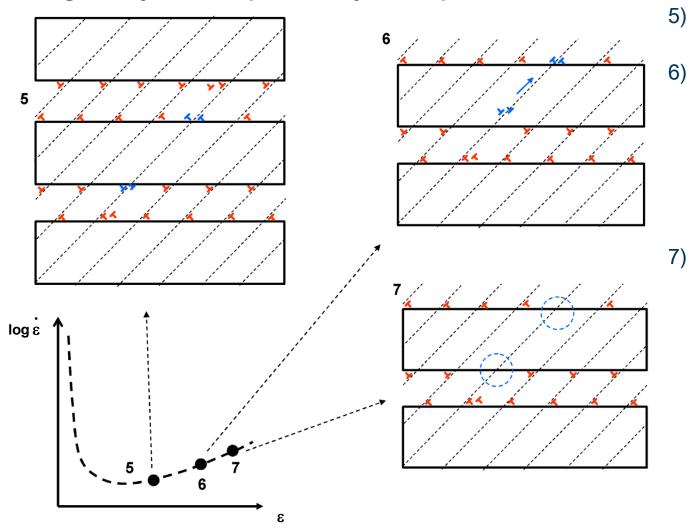
a₀=lattice parameter

2.6 Provide drawings which explain the early (decreasing creep rates) and later (from creep rate minimum) stages of single crystal super alloy creep.



- Decrease of creep rate, dislocations density p increases, dislocations are build up at interfaces
- 2) Hardening due to increasing ρ
 - High ρ -> high internal back stress, it becomes more and more difficult to force additional disloc. into the channel. recovery starts at the corners of γ particles: dislocation climb takes place
 - Annhilation of dislocations of opposite signs

2.6 Provide drawings which explain the early (decreasing creep rates) and later (from creep rate minimum) stages of single crystal super alloy creep.

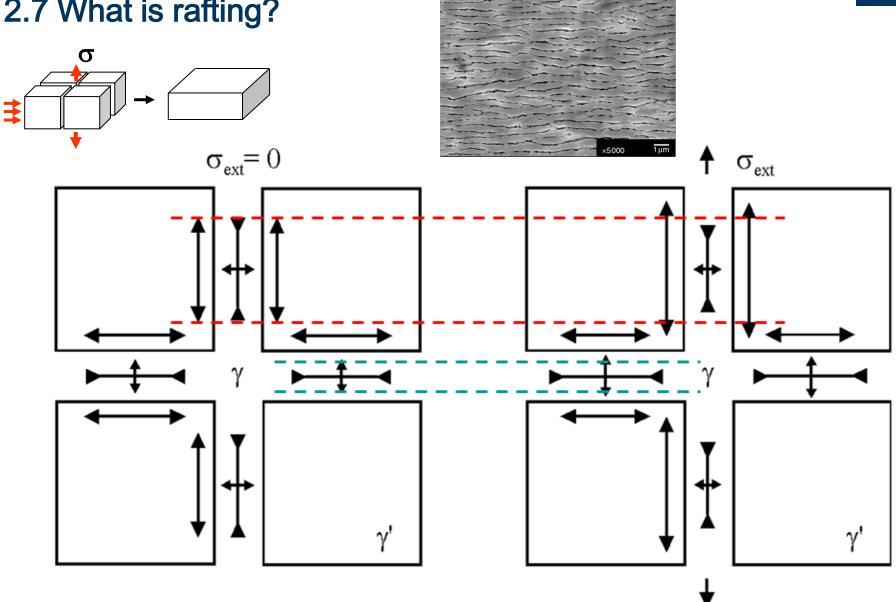


- Rafting of y´particles
 - Dislocations of same sign can glide pairwisely through the particle. Inbetween an antiphase boundary is build.
 - Annihilation of dislocations on the other side of particle of opposite sign

Sources: Wu 2016, Parsa 2015



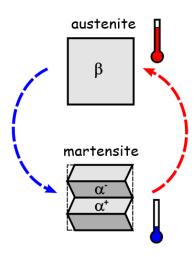
2.7 What is rafting?





Part III

Martensitic Transformation & Shape Memory Alloys (SMAs)





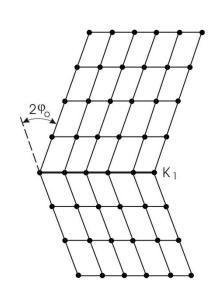
3.1 Why is martensite in steel hard while martensite in NiTi shape memory alloys is soft?

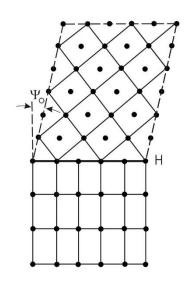
- Steel martensite is hard, twin boundaries cannot move
- SMA martensite is soft, twin boundaries are mobile

3.2 What does martensitic transformation and twinning have in common? What are the differences?

Crystal structure of **twinning**:

identical on both sides of plane K₁





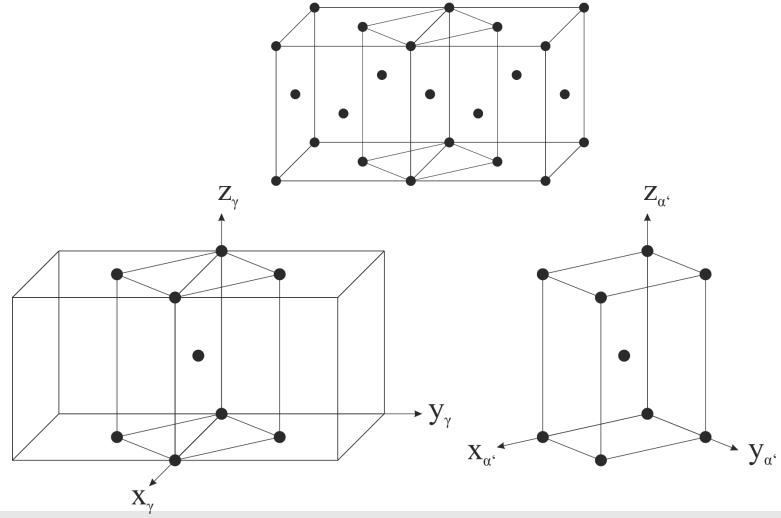
Crystal structure of martensitic transformation:

different on both sides of plane H

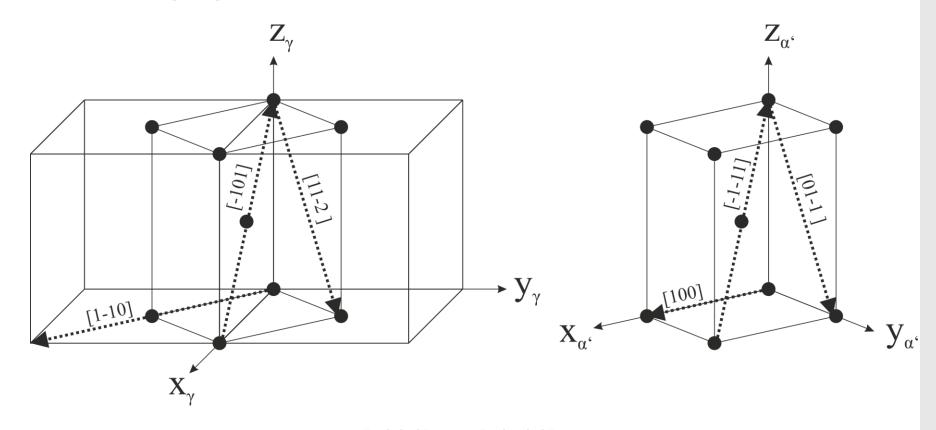
The formation of martensite is a special type of twinning, where the lattice changes.



3.3 Sketch the Bain orientation relationship for a diffusionless martensitic phase transformation from the austenite! Determine the crystallographic directions/planes in the body centered tetragonal unit cell, which are parallel to (111)γ, [-101]γ, [1-10]γ and [11-2]γ!



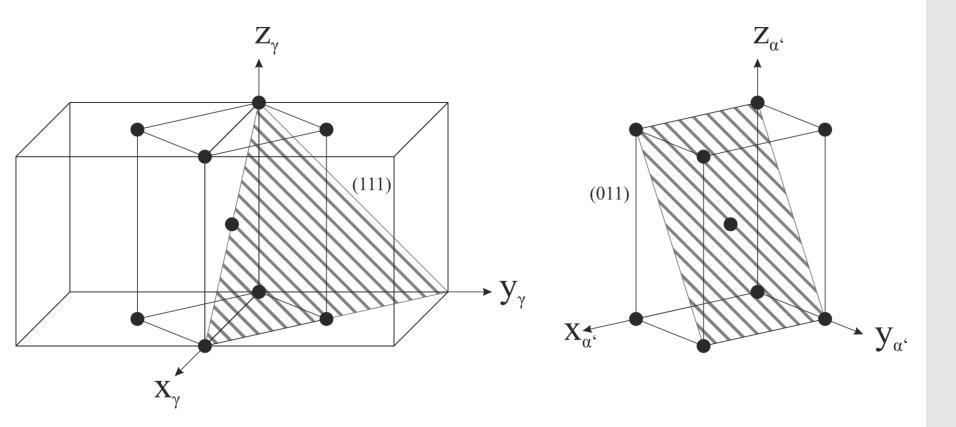
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$$[-101]_{\gamma} \rightarrow [-1-11]_{\alpha'}$$

 $[1-10]_{\gamma} \rightarrow [100]_{\alpha'}$
 $[11-2]_{\gamma} \rightarrow [01-1]_{\alpha'}$

3.3 Sketch the Bain orientation relationship for a diffusionless martensitic phase transformation from the austenite! Determine the crystallographic directions/planes in the body centered tetragonal unit cell, which are parallel to $(111)\gamma$, $[-101]\gamma$, $[1-10]\gamma$ and $[11-2]\gamma$!

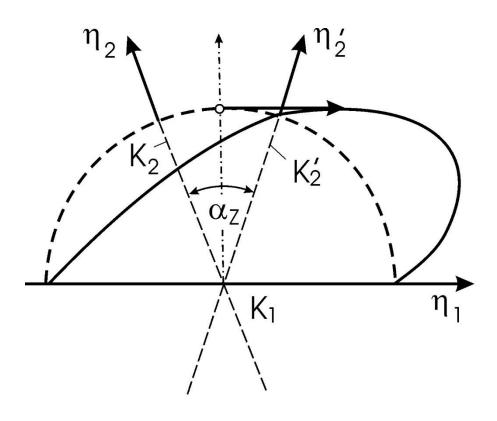


$$(111)_{\gamma} \rightarrow (011)_{\alpha'}$$

3.4 Why are accommodation processes required?

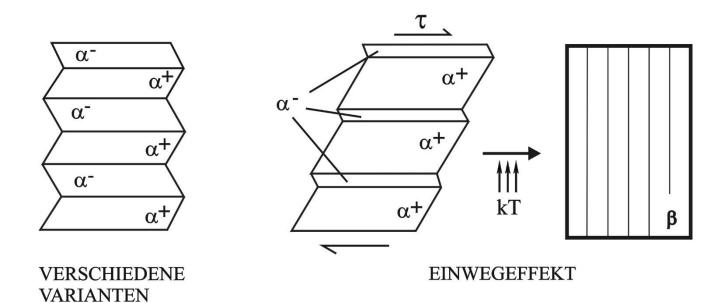
They help to keep the increase in elastic strain energy at a minimum.

3.5 What are the invariants of a martensitic transformation?



invariants during twinning: equatorial plane K_1 and shear direction η_1 . Plane K_2 remains on a circle! rotates by α_2 , but does not get distorted.

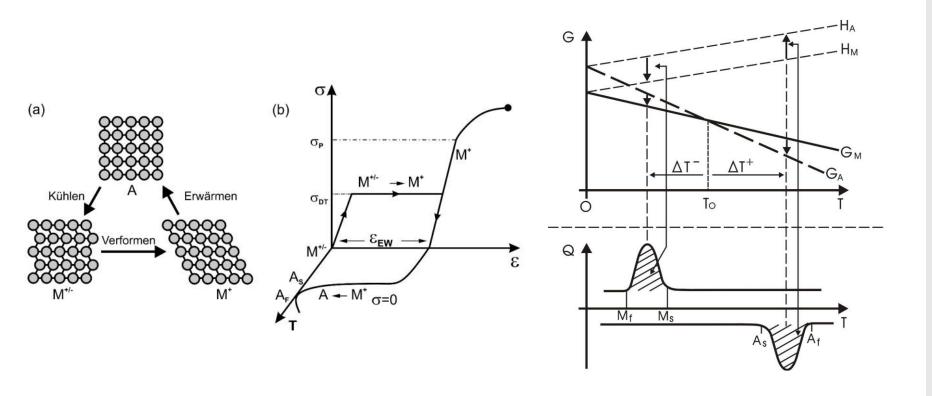
ONE WAY EFFECT:



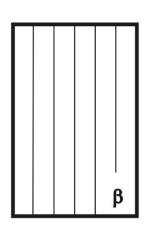
Deformation and growth of favorably oriented variants

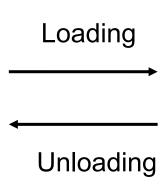
Martensite variants must return into one and only Austenite lattice

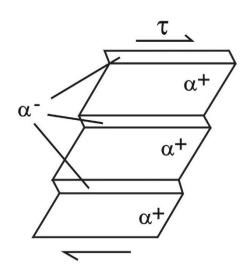
ONE WAY EFFECT:



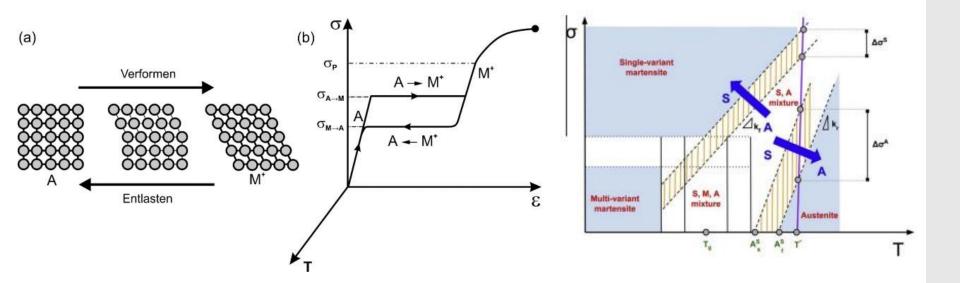
PSEUDOELASTICITY:



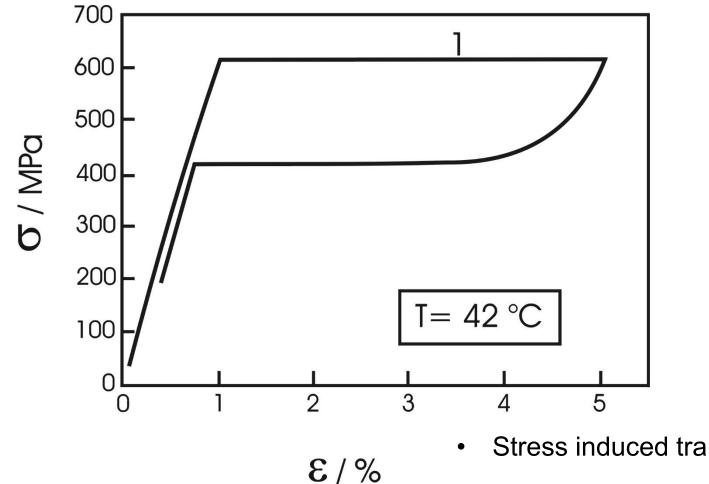




Stress induced martensitic transformation.



3.7 Provide the Clausius Clapeyron equation? How does it help to describe the strong temperature dependence of the pseudoelastic plateau stress in the loading part of a loading/unloading experiment?

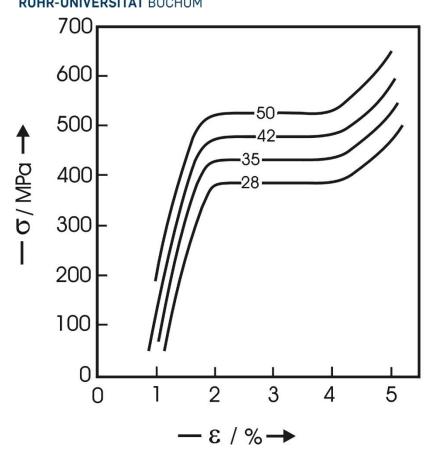


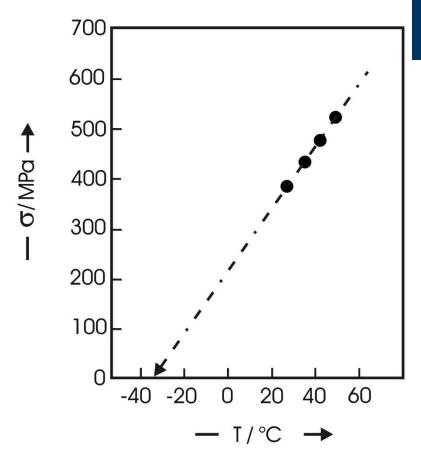
- Stress induced transformation
- High temperature dependence



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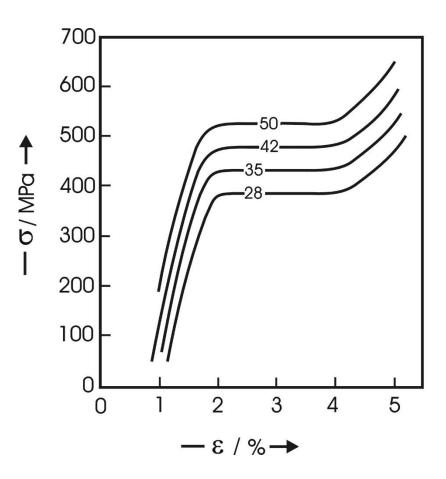


CC-equation for SMAs

$$(dp / dT)_{equilibrium} = \frac{\Delta H_m}{T_m \cdot \Delta V_m}$$

$$\left(d\sigma_{plateau} / dT\right) = \frac{\Delta S}{\varepsilon} = \frac{\Delta H}{\varepsilon \cdot T}$$

3.7 Provide the Clausius Clapeyron equation? How does it help to describe the strong temperature dependence of the pseudoelastic plateau stress in the loading part of a loading/unloading experiment?



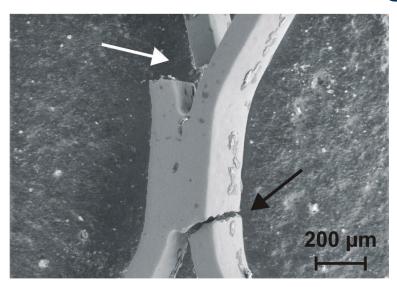
Why do we have higher stress plateaus with increasing temperature?

- stress plateaus: stress induced martensite (pseudoelasticity)
- The higher the temperature the more stable the austenite (we are far away from M_s, see question 6)
- Hence, in order to induce martensite we have to apply higher stresses
- If T is too high, we exceed the yield strength (plastic deformation) and pseudoelasticity can not be ontained.



Part IV

SMA Fracture Mechanics & Basics of Structural Fatigue



4.1 What is the crack extension force G, how does it relate to the stress intensitiy factor K?

Crack extension takes place under elastic energy release. The crack extension force G is defined as

$$G = -\frac{d(\Delta U_E)}{2 \cdot da},$$

where ΔU_E the elastic energy and da the length of the crack intension is. The G value for which crack extension in crack mode I takes place is G_{IC} . For plane stress we can derive

$$G = \frac{\pi \cdot a \cdot \sigma^2}{E},$$

with stress σ and Young's Modulus E. As engineers we often use the stress intensity factor K, which is

$$K_I = \sigma \cdot \sqrt{\pi \cdot a}$$
.

for crack mode I. Combining the crack extension force G with the stress intensity factor K yields

$$G_I = \frac{K_I^2}{E}.$$



4.2 What information on elementary processes in front of cracks in SMAs can one obtain using in-situ scanning electron microscopy (SEM), a thermocamera and synchrotron radiaton?

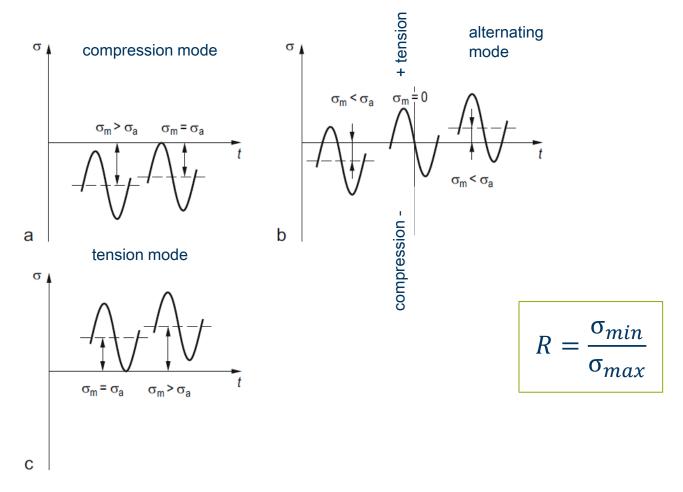
Thermocamera can detect local changes in temperature. This is an indicator for a phase transformation, but it could be something else. By synchrotron radiation we get crystallographic information, e.g. during the phase transformation diffraction peaks/rings of the new phase appear. This is the proof.

With in-situ SEM we can observe crack propagation and plastical deformation (slip bands etc.).

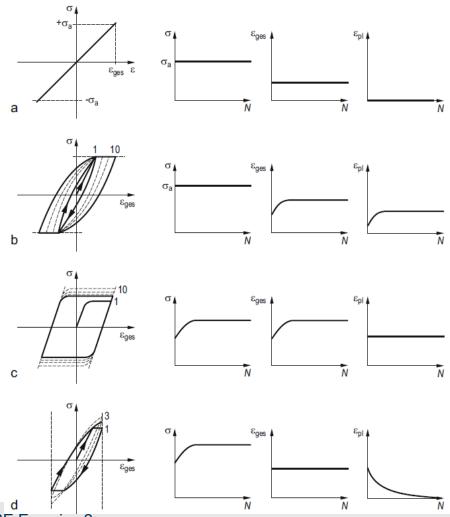
4.3 What is the difference between structural and functional fatigue?

- <u>Structural fatigue</u>: rupture, total loss,
- <u>Functional fatigue</u>: loss of certain properties, p.e. changes in shape, changes of transformation behavior

4.4 Draw σ(t) diagrams for the three possible cases of loading of a fatigue experiment. Highlight the following parameters: σmax, σmin, σa. What is the definition of R?



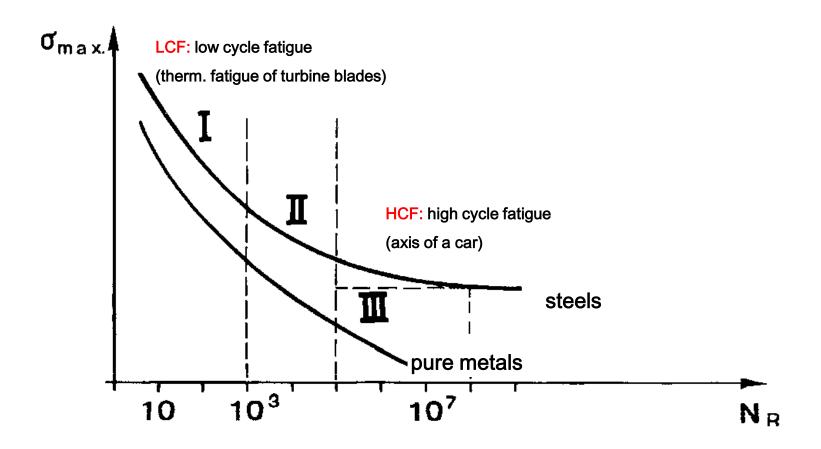
- 4.5 The performance of fatigue testing under tension and/or compression can be stress or strain controlled. Please draw under the consideration of a)-d) stress-strain diagrams, which describe the curve evolution from the beginning of the first changing in load until saturation, respectively. Additionally draw for each case three single diagrams for σ_a , $\varepsilon_{tot} = \varepsilon_{el} + \varepsilon_{pl}$ and ε_{pl} in dependence of the number of load cycles N. For this holds σ_m =0.
 - a) $\sigma_a = \text{const.}$, $\varepsilon_{pl} = 0$,
 - b) σ_a = const., material softens,
 - c) ϵ_{pl} = const., material hardens,
 - d) ε_{tot} = const., material hardens.



4.6 Why do we observe a stress strain hysteresis during fatigue testing?

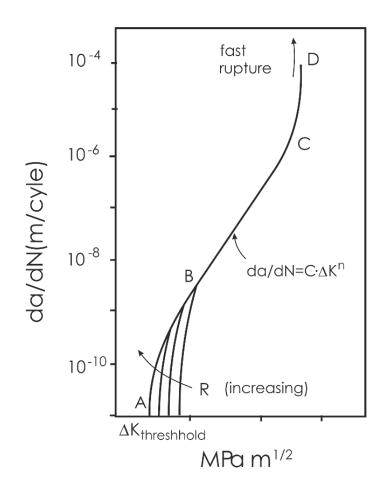
Because there is an energy loss during loading and unloading caused by local plastic deformation (dissipation). Hence, the material becomes warm.

4.7 What is a Wöhler curve?



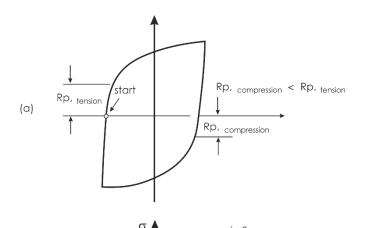
Describes the rupture in dependence of stress and cycle number.

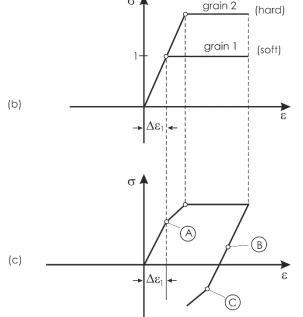
4.8 What is the Paris law?



- \circ describes ΔK controlled crack growth
- \circ $\frac{da}{dN}$ changes of crack length in dependence of N
- o constants: *C,n*
- $\Delta \sigma = \sigma_{max} \sigma_{min}$, external applied load
- o even if $\Delta \sigma$ = const., ΔK increases because *a* grows
- o if ΔK increases, $\frac{da}{dN}$ also increases

4.9 What is the Bauschinger effect?





(a) Bauschinger Effekt:

Yield stress in tension is not the same as yield stress in compression!

(b) and (c) Masing's explanation:

Soft grain yields earlier than hard grain (A).

Wenn hard grain starts yielding, soft grain has already plastically deformed.

On unloading we reach a point, where the soft grain is already fully unloaded, while the assembly is still in tension (B).

Soft grain gets into compression, when the assembly is still in tension..

This is why in compression, soft grain starts to yield earlier (**C**).

4.10 What is the microstructural origin of functional fatigue?

Thermo/mechanical cycling introduces lattice defects, which interact with the martensitic transformation. These defects can stabilized the stress induced martensite which is not being recovered. This yields functional fatigue.



Thanks for your attention!