EPFL

FS 2023/24

MSE-422 – Advanced Metallurgy

Exercise 2 – Advanced steels

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Heat treatment of steels

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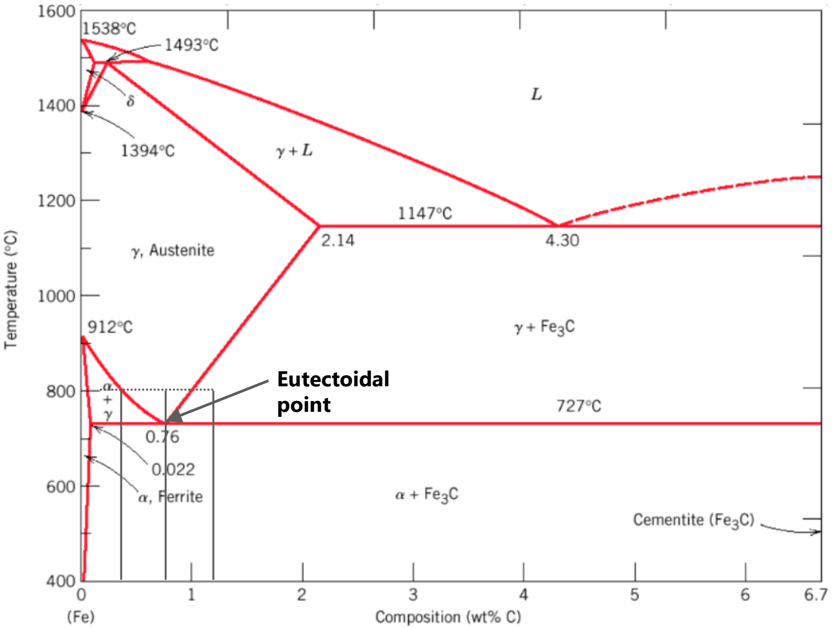
1. You have been recently hired by a renowned manufacturer of high-precision machine tools and you are responsible for the selection, heat treatment and characterization of the metallic materials used in the production.

a) You have to characterize three different carbon steels, containing 0.3 wt.% C, 0.76 wt.% C and 1.2 wt.% C, which are heat-treated at 800 °C. Are they hypo- or hypereutectoid according to the binary Fe-C phase diagram shown in Figure 1? What phases will result after quenching?

- **0.3 wt.% C: Hypoeutectoid**
- **0.76 wt.% C: Eutectoid**
- **1.2 wt.% C: Hypereutectoid**

- **0.3 wt.% C: Ferrite + Martensite**
- **0.76 wt.% C: Martensite**
- **1.2 wt.% C: Cementite + Martensite**



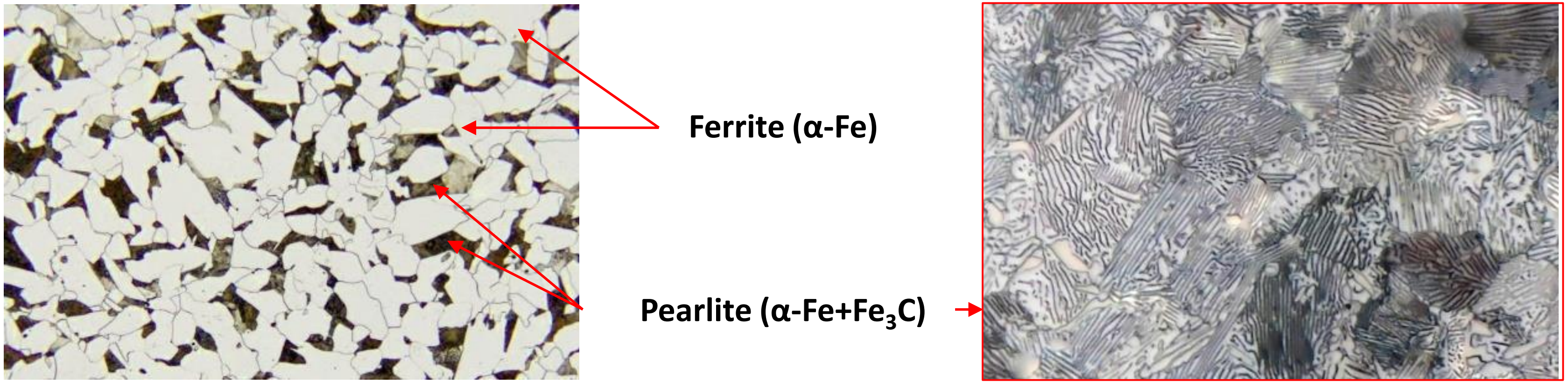
Heat treatment of steels

b) A colleague has prepared metallographic cross-sections of two other carbon steels and has taken some images with the optical microscope (Figure 2, b,c). However, she does not remember the **carbon content**. Can you help her (Tip 1: Ferrite contains 0.02 wt.% C, perlite contains 0.8 wt.% C. Tip 2: Use the area fractions…)? Label the phases.

- **Sample 1:** 90% Ferrite, 10% Pearlite
- 0.9 · 0.02 wt.% C + 0.1 · 0.8 wt.% C = 0.098 wt.% C

Sample 2: Fully eutectoid structure

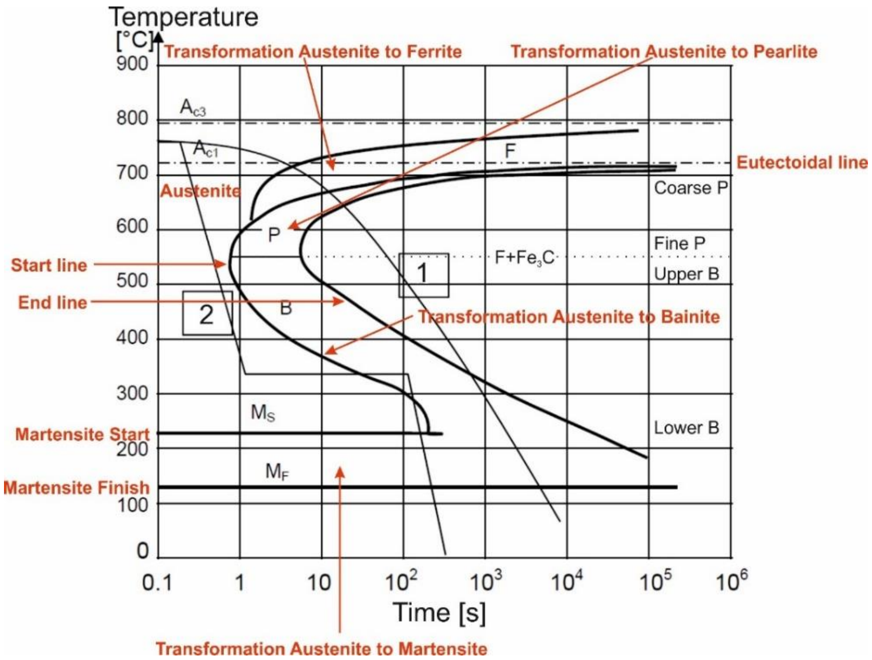
0.8 wt.% C



Heat treatment of steels

Your next job is to decide for a heat treatment cycle for a batch of forged shafts (l = 250 mm, ⌀ 35 mm) made from a low alloyed 30NiCrMo12-6 steel. You know that you have to austenize it first, followed by quenching in oil and subsequent tempering. Your colleague gives you the time-temperature-transformation (TTT) diagram for the steel, shown in Figure 3.

c) Explain the meaning of all lines and fields in the TTT diagram.



Heat treatment of steels

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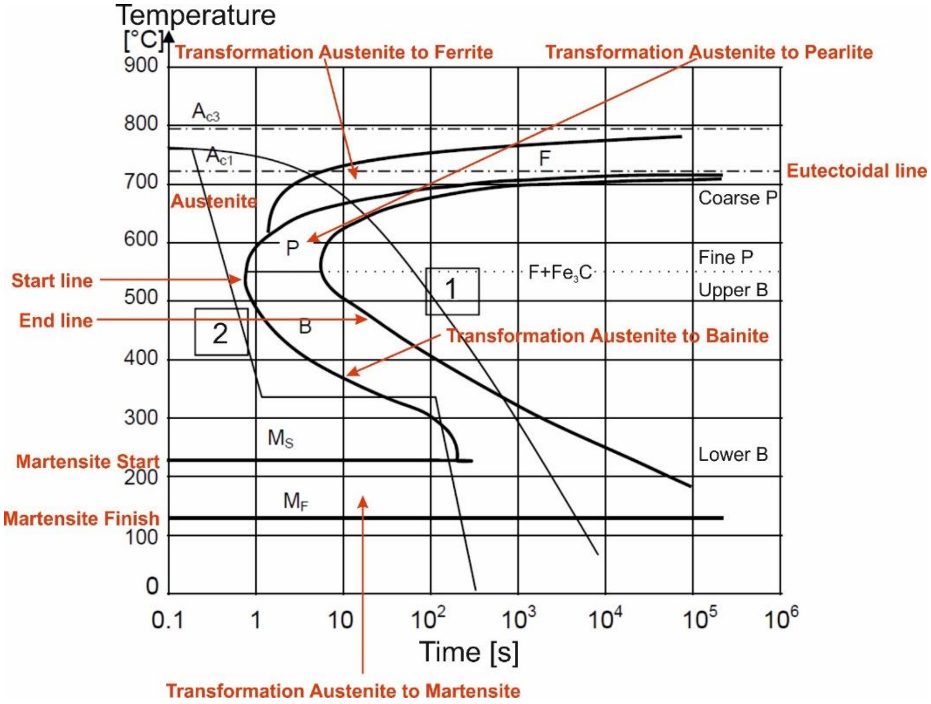
d) How can you tell the steel is hypo- or hypereutectoid from only the TTT diagram? Can you give the composition (30NiCrMo12-6)?

- There exists a ferritic region between Ac_1 and Ac_3
  → **Hypoeutectoid, < 0.8 wt.% C**

- **Composition of 30NiCrMo12-6:**
  0.3 wt.% C, 3.0 wt.% Ni, 1.5wt.% Cr, small undefined amount of Mo



For low alloy steels

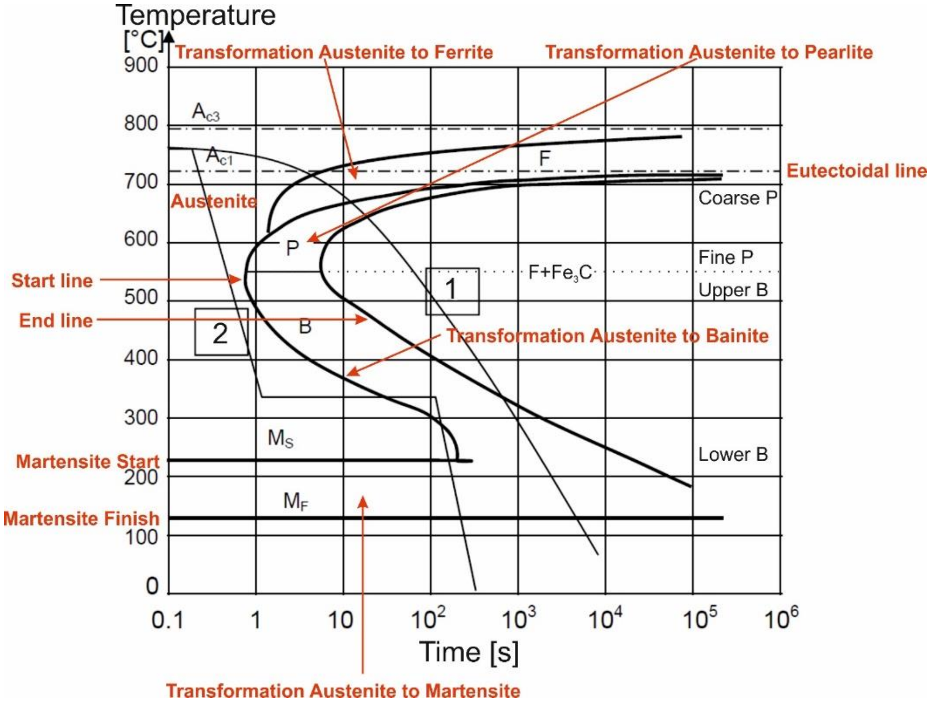
Amount of alloying elements
in wt.%/4 for Cr, Si, Mn, Ni, W
in wt.%/10 for Al, Mo, Nb, V, Ta, Ti, Zr, Cu
in wt.%/100 for P, S, N, C, Ce
in wt.%/1000 for B

Heat treatment of steels

e) From Figure 3, can we determine what phases form for the processing routes labeled 1 and 2? If so, try to estimate the final phase composition.

- **1:** No conclusion possible. TTT works only with isothermal transformations!

- **2:** At 330 °C, ~50% bainite forms out of austenite. By subsequent quenching, residual austenite transforms to martensite.



Heat treatment of steels

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f) For your steel, you know that an additional tempering step follows immediately on the previously conducted hardening. Would you rather choose a temperature of 200 °C or of 550 °C? Explain your choice. What is the effect of Mo and Cr with regard to an increase in strength at this temperature? Why temperatures around 300 °C and 500 °C typically have to be avoided when tempering steels?

1. **Step:** Hardening, homogenizing the microstructure (>Ac3, ≈830-860 °C)

2. **Step:** Quenching (oil, water, polymers, salt solutions)

3. **Step:** Tempering (underneath Ac3, **540-680 °C** → Tempered martensite/bainite + **Secondary (Mo,Cr)-Carbides precipitates** (enhance strength, reduce brittleness)

   (*Reject 200 °C, only martensite is tempered – no secondary carbides/nitrides/borides, hard but brittle)*
   Moreover, Cr and Mo retain the martensite by inhibiting the recovery and recrystallization of deformed grains

Heat treatment of steels

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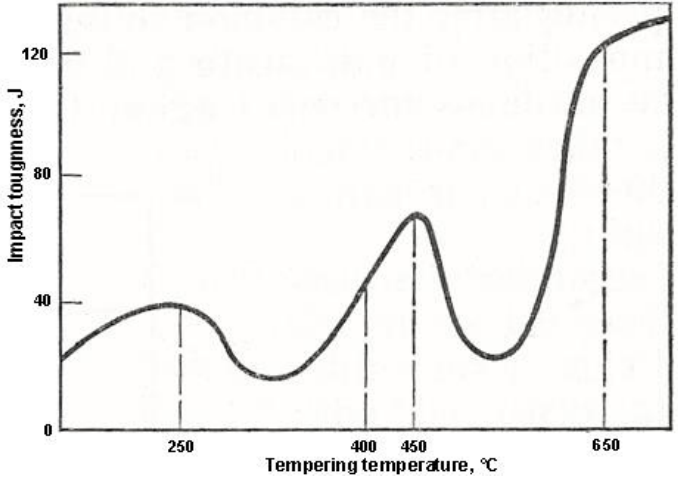

- **300 °C: "Blue brittleness":**
  C and N precipitate along GBs of α-Fe, low toughness and reduced formability

- **500 °C: Tempering embrittlement:**
  C precipitates along GBs of γ-Fe, (see above)

Stainless steels

2. Your job is to decide for a steel for the door of an industrial heat treatment furnace, used for ceramic dishes production, which is stable for temperatures up to 1100 °C. As the customer is happy to have an expert with him, he moreover asks you what would be your choice for the production of his premium kitchen knife selection.

a) Select one material from Table 1 for the door and one for the knives. Explain your choice based on the chemical composition and the requirement specifications.

Alloying concept	C	Cr	Mo	Ni	N	Cu	Others
	0.02	25.0	4.0	7.0	0.1-0.2	0.5-1.0	W: 0.5-1.0
	0.06	17.0	2.0	12.0	0.0-0.1	-	Ti/Nb: 5/10 x C
	0.46	13.0	-	-	-	-	Si, Mn: 0.0-1.0
	0.02	12.0	-	-	-	-	Ti: 0.3
	0.12	16.0	-	36.0	0.0-0.1	-	Si, Al: 0.5-2.0

Stainless steels

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②	**0.46**	**13.0**	**-**	**-**	**-**	**-**	**Si, Mn: 0.0-1.0**
	0.02	12.0	-	-	-	-	Ti: 0.3
①	**0.12**	**16.0**	**-**	**36.0**	**0.0-0.1**	**-**	**Si, Al: 0.5-2.0**

①

Door: High temperature resistance, resistance against oxidation

- **Ni** → Austenitic, fcc-lattice, high packing density leads to low atom mobility, resistance to creep, (noteworthy: minimum CTE at 36 wt.% Ni)
- **Cr** → Oxidation-resistant, stable Cr_2O_3 layer >12 wt.% Cr, stable Cr-carbides/-nitrides

②

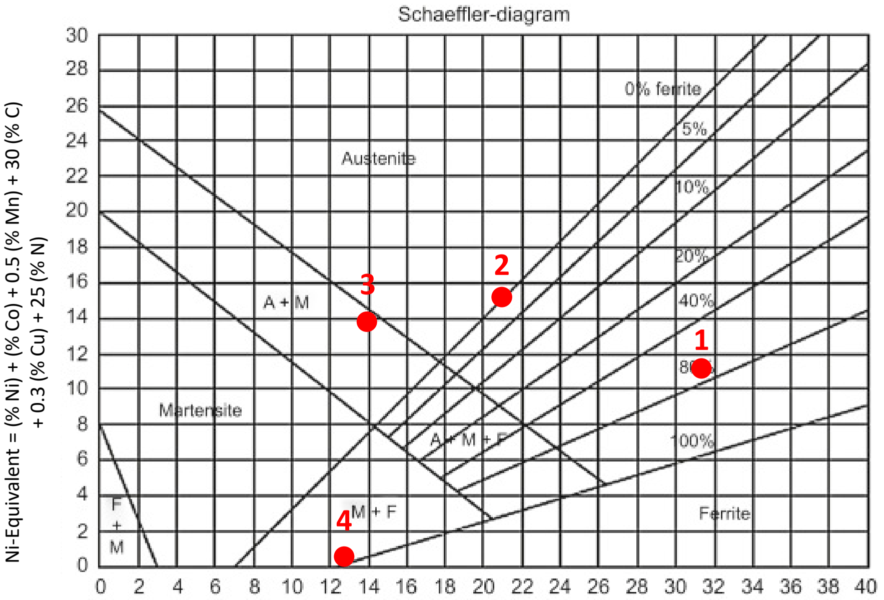
Knife: High hardness and wear resistance, resistance against oxidation, easy to grind

- **C** → Good hardness (martensitic structure + hardenability during HT)
- **Cr** → Oxidation-resistant
- **(Si, Mn**→ High affinity to oxygen, deoxidizer during steel production)

Stainless steels

b) As you know, stainless steels are grouped according to their microstructure/alloying concept. Note the corresponding alloying concept from the materials in Table 1 for each composition (tip: The Schaeffler diagram below might give you hints). Moreover, give the name according to the nomenclature.

Name/Alloying concept	C	Cr	Mo	Ni	N	Cu	Others
X2CrNiMoCuWN25-7-4 (Duplex)	0.02	25.0	4.0	7.0	0.1-0.2	0.5-1.0	W: 0.5-1.0
X6CrNiMoTi17-12-2 (Austenitic)	0.06	17.0	2.0	12.0	0.0-0.1	-	Ti/Nb: 5/10 x C
X46Cr13 (Martensitic hardenable)	0.46	13.0	-	-	-	-	Si, Mn: 0.0-1.0
X2CrTi12 (Ferritic)	0.02	12.0	-	-	-	-	Ti: 0.3
X12NiCrSi36-16 (Austenitic)	0.12	16.0	-	36.0	0.0-0.1	-	Si, Al: 0.5-2.0



Cr-Equivalent = (% Cr) + 2 (% Si) + 1.5 (% Mo) + 5 (% V) + 5.5 (% Al) + 1.75 (% Nb)+ 1.5 (% Ti) + 0.75 (%W)

Stress Corrosion Cracking of Steels (SCC)

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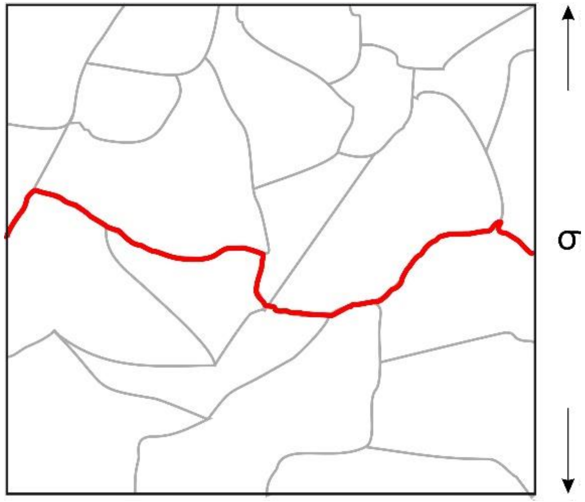
3. A customer uses tungsten inert gas (TIG) welded tubes made of the well-known AISI 304 (X5CrNi18-10) to transport gas in a petrochemical plant. After 5 years of service, he notices cracks near the welds. The tubes, however, were not exposed to all the conditions required for stress corrosion cracking (SCC).

a) What role does carbon play in the formation of corrosion in connection with the welding process mentioned?

- C forms carbides on the grain boundaries due to its affinity to Cr (e.g. $Cr_{23}C_6$, Cr_7C_3), reducing the Cr content in the vicinity of GBs to values <12 wt.%, thus making them susceptible to corrosive attacks.

Stress Corrosion Cracking of Steels (SCC)

b) Figure 5 shows the grain microstructure of the steel AISI 304. The sample is exposed to tensile stresses. Indicate where you would expect a crack path based on the information given above. Name the cracking mechanism.



(Other paths are possible, as long as short and perpendicular to stress direction)
Intergranular cracking/Intergranular fracture/Intergranular embrittlement.

Stress Corrosion Cracking of Steels (SCC)

c) If AISI 304L was used instead, an alloy having almost the same chemical composition as AISI 304, no cracks would occur. What elements are added to AISI 304L to prevent this type of cracking (give one example) and why?

E.g. Ti, Nb, as they have a higher affinity to C than Cr.

Stress Corrosion Cracking of Steels (SCC)

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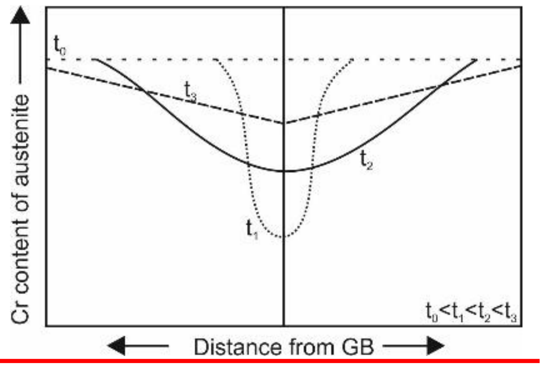
d) In some cases, local trans-crystalline stress corrosion cracking can also appear in so-called stain-less steels. Can you explain what happens during such SCC? In addition to the added elements mentioned in c), are there other approaches to achieve higher resistance to SCC?

1. **Corrosive environment:** Chlorides (e.g. in swimming pools), Sulfides (e.g. chemical industry), demineralized water at higher temperatures (e.g. boilers, due to depletion of protective oxide layer)
2. **Material:** Inclusions, precipitates, grain boundaries, Cr depleted regions, high dislocation density
3. **Tensile stresses:** Residual (e.g. from welding) or external loads, dislocation movement to localized regions on the surface break the passive Cr_2O_3 layer

How to avoid stress corrosion cracking?

- Addition of ≈ 2 wt.% Mo
- Addition of ≈ 40 wt.% Ni ($$)
- Heat treatment: Solution annealing or Stress Relief Annealing
- Introduce compressive stress by shot-peening

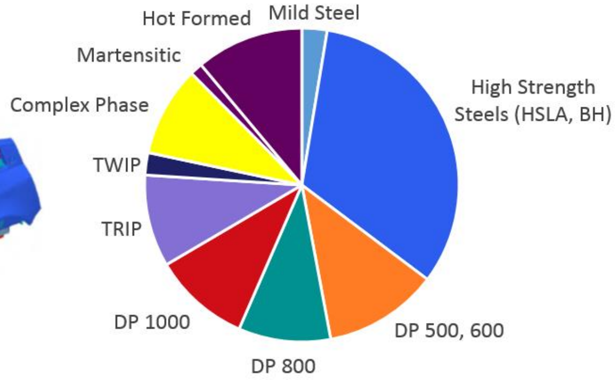


V. Kain, 5 - Stress corrosion cracking (SCC) in stainless steels, in: V.S. Raja, T. Shoji (Eds.), Stress Corrosion Cracking, Woodhead Publishing, 2011: pp. 199–244.

Advanced High Strength Steels (AHSS)

4. As a materials engineer in automotive engineering, you know that in recent years, multiphase AHSS have been increasingly used for structural parts in car body engineering. Based on their development time, a differentiation is made between 1st, 2nd and 3rd generation AHSS.

AHSS steels of the first generation are, for example, DP, PM, CP and TRIP steels. The latter contain up to 4 wt.% Mn. The special properties of such TRIP steels are based on the presence of about 10-25 vol.% retained austenite.



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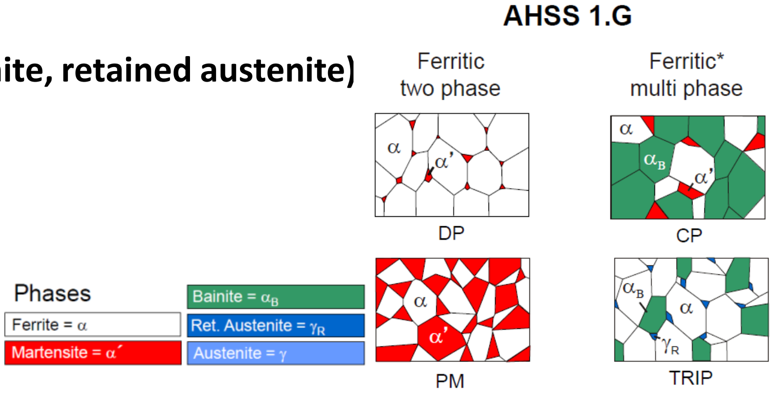
Advanced High Strength Steels (AHSS)

a) Name three advantages that TRIP steels offer over other 1st generation AHSS in terms of formability and crash safety in car bodies. What is the reason for their high strength? What phases are present?

1. **Distinct work hardening during forming due to TRIP effect**
2. **Reduction of sheet thickness and associated weight reduction**
3. **Martensitic transformation during crash reduces residual kinetic energy of impact**

- **Reason for high strength:** Martensitic transformation during deformation induces distinct work hardening.

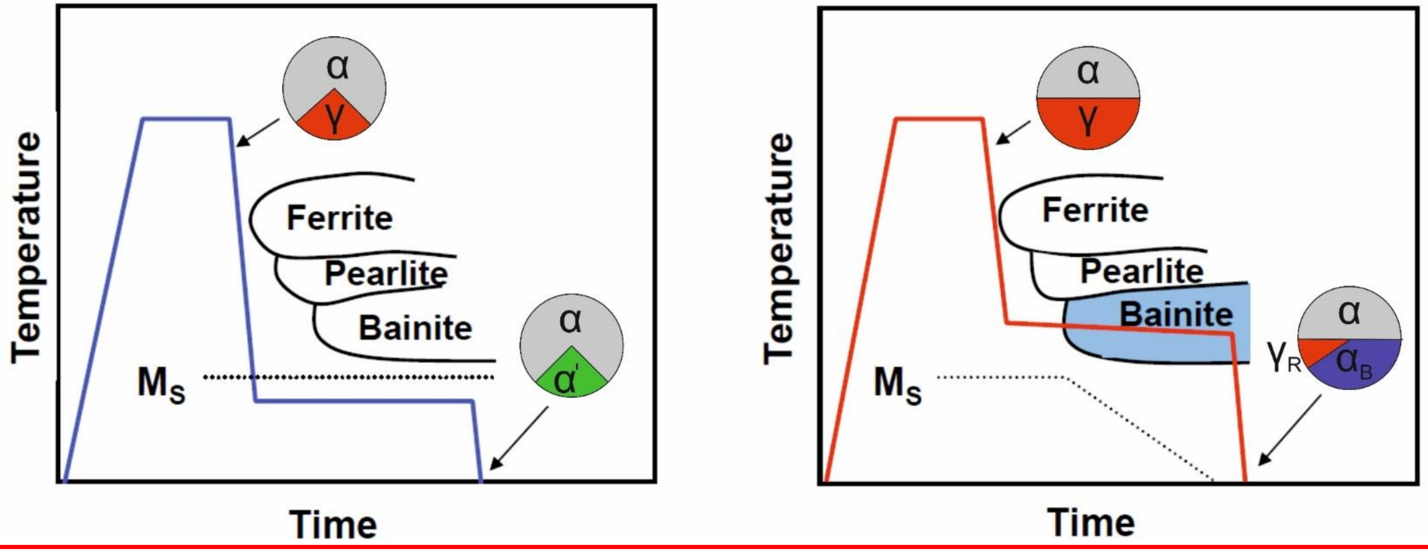
- **Ferritic multiphase matrix (Ferrite, bainite, retained austenite)**



Advanced High Strength Steels (AHSS)

b) Figure 6 shows processing routes for the production of two different AHSS grades of the first generation, which have almost the same chemical composition, but different phases after heat treatment. Match the steel grades and indicate the predominant phases in the pie chart.

Dual phase steel (DP) **TRIP steel**



Advanced High Strength Steels (AHSS)

c) A cold-rolled side impact bar made from TRIP steel, containing 0.2 wt.% C, is intercritically annealed in the two-phase region between Ac1 and Ac3. After quenching from the austenitic region, which should typically lead to a martensitic microstructure, retained austenite is found in the TRIP steel. Explain how it is possible for there to be any retained austenite at all in a steel with 0.2 mass % carbon. What is meant by "partitioning" in this context? Is this retained austenite thermodynamically stable? Is this retained austenite mechanically stable?

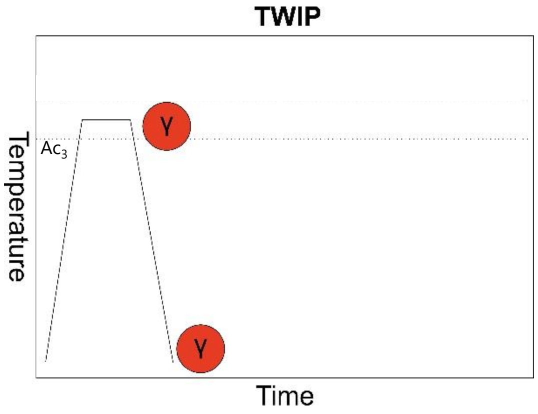
C-Partitioning → Adjustment of C equilibrium between α and γ phase by C redistribution during intercritical annealing → Carbon-enriched austenite can be retained

Retained austenite is thermodynamically stable, but mechanically instable

- Si prevents carbide formation → C within bainite cannot form carbides → Diffuses into austenite
- C content of austenite rises and, concurrently, M_s sinks
- When M_s reaches room temperature, the austenite remains completely as retained austenite after cooling

Advanced High Strength Steels (AHSS)

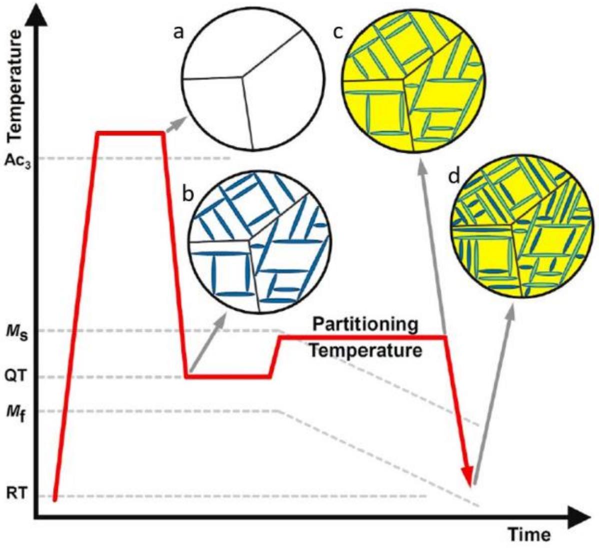
d) Based on your background knowledge, you suggest it would be better to use a second generation (e.g. TWIP steels) instead of the first generation AHSS from c). Draw a schematic diagram of the heat treatment for TWIP steels in the graphic below and name the phase fractions present after each heat treatment step in a pie chart (in a similar way as Figure 6). What are the advantages of TWIP steels in terms of formability, crash safety, and durability compared to TRIP steels?



- **Formability:** High strain, at the same time high work hardening effect
- **Crash Safety:** After deep-drawing, high amounts of γ-Fe left for TWIP/TRIP effect
- **Durability:** High fatigue resistance due to high work hardening

Supplement

Figure below demonstrates the heat treatment of a type of 3rd generation AHSS. In general, it has a composition of 1-1.5 wt.% Mn, 0.1-0.3 wt.% C, and 0-1.5 wt.% Al. (Exam 2023-2024)



a) Name the steel grade.
What are the phases shown in the microstructures of a-d in Figure 4?
Explain briefly what happened during the "partitioning" process.
Why did the temperatures Ms and Mf decrease during partitioning? (3P)

Quenched and Partitioned (Q&P) steels
a: austenite;
b: austenite and martensite
c: carbon-enriched austenite and carbon-decreased martensite
d: carbon-enriched austenite, carbon-decreased martensite, and martensite
During partitioning, carbon transports from martensite to austenite.
M_s and M_f decrease because the increase of carbon content in austenite can stabilize the austenite phase and lower the martensite formation temperature.

Supplement: Quenched and Partitioned (Q&P) steels

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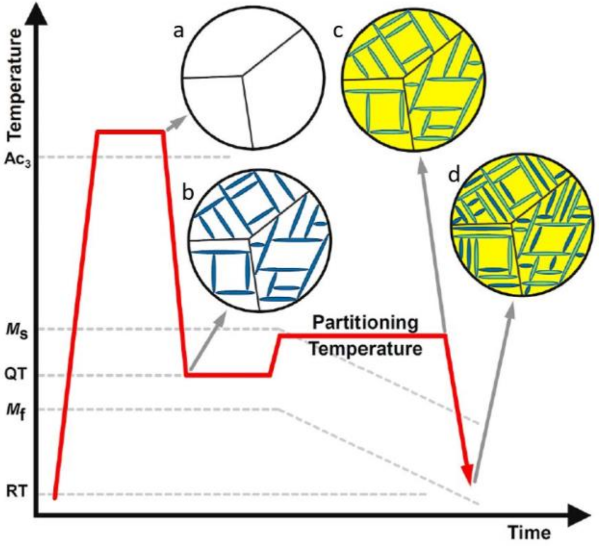

b) The volume fraction of the retained austenite at stage "d" in the above Q&P steel was observed to first increase and then decrease with increasing quench temperature (QT in Figure 4). Provide a possible explanation for this phenomenon. (2P)

When the QT starts to increase from Mf, the volume fraction of untransformed austen-ite increases. However, if the QT is too high, the partitioning process cannot effective-ly pump carbon into the austenite phase because of its high volume fraction, resulting in low stability of the untransformed austenite (the decreases in Ms and Mf are less pronounced). Therefore, more austenite transforms to martensite during the final quenching, i.e., less retained austenite.