Ref. Ares(2023)2333987 - 31/03/2023

ACHIEF Training material

DTNM/LV/2023/019

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The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N° 958374.

Artificial intelligence for HEA material selection

Tom Andersson

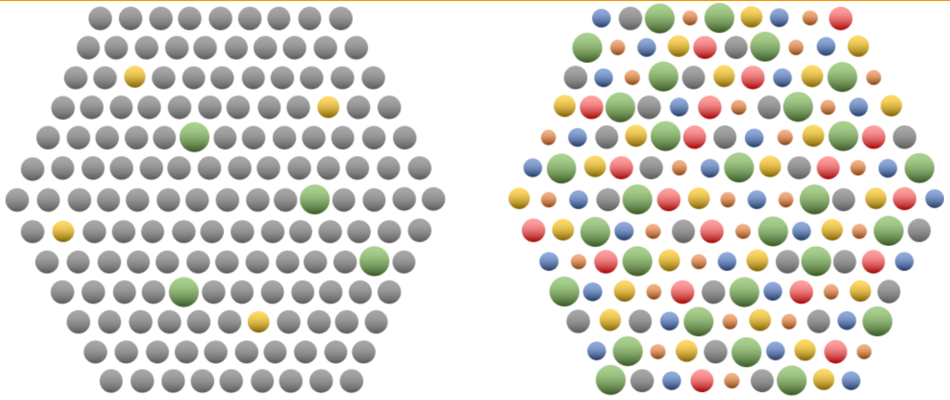
VTT

The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N° 958374.

High Entropy Alloys (HEAs)

Class of materials containing more than 4-5 principal elements that have a mixture of simple FCC, BCC, and HCP structures.

- They can appear in different phases : solid solution (SS), intermetallic (IM), amorphous (AM), or a mix of them.



Many HEAs have higher strength than traditional alloys even in elevated temperatures

- The BCC metals often have very high yield strengths with limited ductility.
- The FCC metals have high ductility but low strength

→ The mixture of BCC + FCC is expected to possess balanced mechanical properties.

Based on high entropy concept

$$\Delta S = -R \sum_{i=1}^{n} c_i \ln c_i$$

↓

$$\Delta G = \Delta H - T\Delta S$$

n	1	2	3	4	5	6	7	8	9	10	11	12	13
ΔS_{conf}	0	0.69R	1.1R	1.39R	1.61R	1.79R	1.95R	2.08R	2.2R	2.3R	2.4R	2.49R	2.57R

low | medium | high entropy →

1.0R 1.5R

The project has received funding from the European Union’s Horizon 2020 research and innovation program under Grant Agreement N° 958374.

Challenges in HEA design

Hit and trial method

- No phase diagram
- Does not follow traditional (emperical) rules
- Huge number of combinations

Ab initio simulation

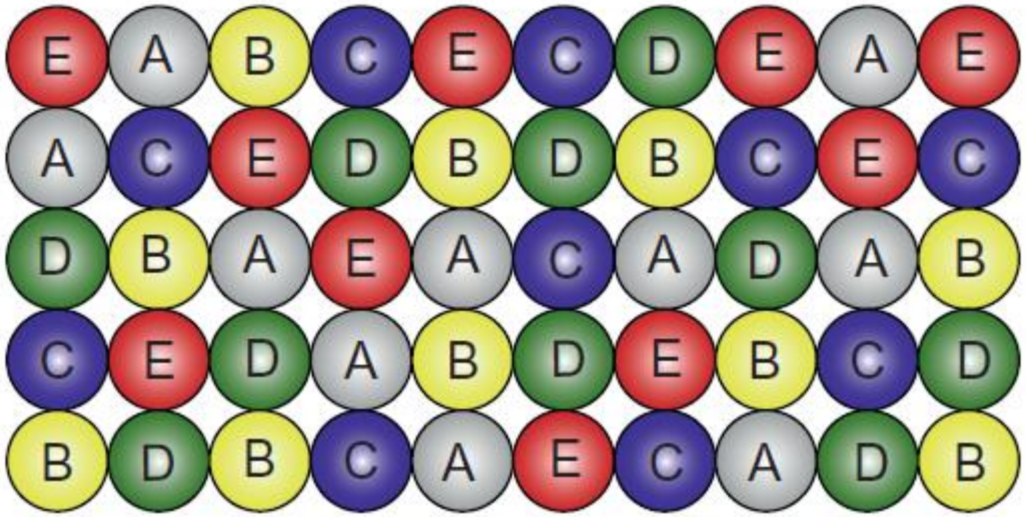
- Availability of suitable potentials?
- Huge computing power and time required

Parametric approach

- Relatively new material group
- Not enough scientific background

CALPHAD

- Limited reliable databases
- Time and cost effective



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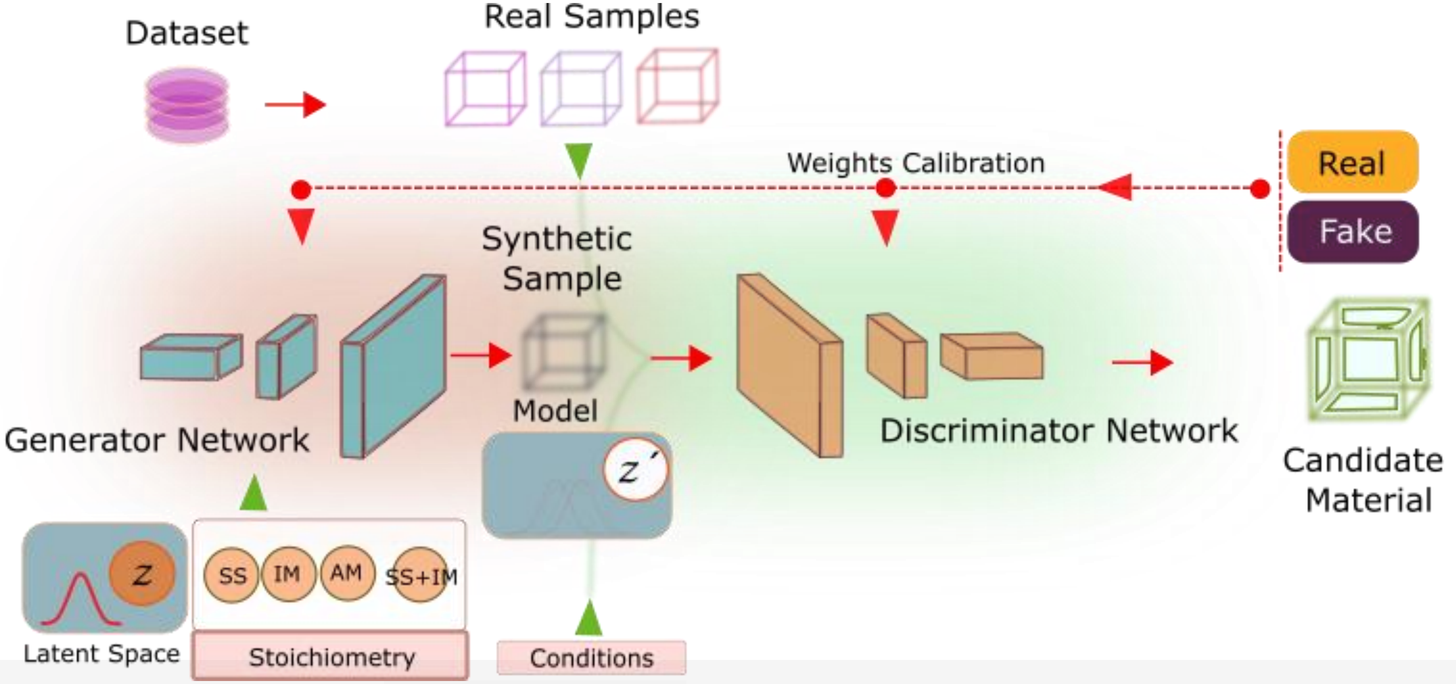
Workflow for generating new alloy candidates

- The used approach is based on Generative Adversarial Networks (GAN).
- Datasets have been developed, containing data from real samples, and used as a training data.
- After trained, the model can generate synthetic samples, based on the features (design parameters) learned from the real samples, giving as output a candidate.



OQMD
NOMAD
AFLOW

1. Developed dataset
2. Fed neural Network with the dataset
3. Generate synthetic candidates
4. Validate candidates

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High entropy alloys design parameters:

Based on literature a dataset was created.

1. 15 features (in the table)
2. Number of elements
3. Phase (amorphous, intermetallic, solid solution, and a mix of intermetallic + solid solution)
4. 78 chemical elements with their corresponding concentration

Features	Equations
Mean atomic radius	$a = \sum_i c_i \cdot r_i$
Atomic size difference	$\delta = \sqrt{\sum_i \left(c_i \cdot \left(1 - \frac{r_i}{\sum_i c_i \cdot r_i}\right)^2\right)}$
Average melting temperature	$T_m = \sum_i c_i \cdot T_{mi}$
Standard deviation of melting temperature	$\sigma_T = \sqrt{\sum_i c_i \cdot \left(1 - \frac{T_{mi}}{T_m}\right)^2}$
Mixing enthalpy	$\Delta H_{mix} = 4\sum_{i \neq j} c_i \cdot c_j \cdot H_{ij}$
Standard deviation of mixing enthalpy	$\sigma_{\Delta H} = \sqrt{\sum_{i \neq j} c_i \cdot c_j \cdot (H_{ij} - \Delta H_{mix})^2}$
Mixing entropy	$\Delta S_{mix} = -R\sum_i c_i \cdot \log c_i$
Electronegativity	$\chi = \sum_i c_i \cdot \chi_i$
Standard deviation of electronegativity	$\Delta\chi = \sqrt{\sum_i \left(c_i (\chi_i - \chi)^2\right)}$
Valence electron concentration	$VEC = \sum_i c_i \cdot VEC_i$
Standard deviation of VEC	$\sigma_{VEC} = \sqrt{\sum_i \left(c_i \cdot (VEC_i - VEC)^2\right)}$
Mean bulk modulus	$K = \sum_i c_i \cdot K_i$
Standard deviation of bulk modulus	$\sigma_K = \sqrt{\sum_i c_i \cdot (K_i - K)^2}$
Young modulus	$E = \sum_i c_i E_i$
Shear modulus	$G = \sum_i c_i G_i$

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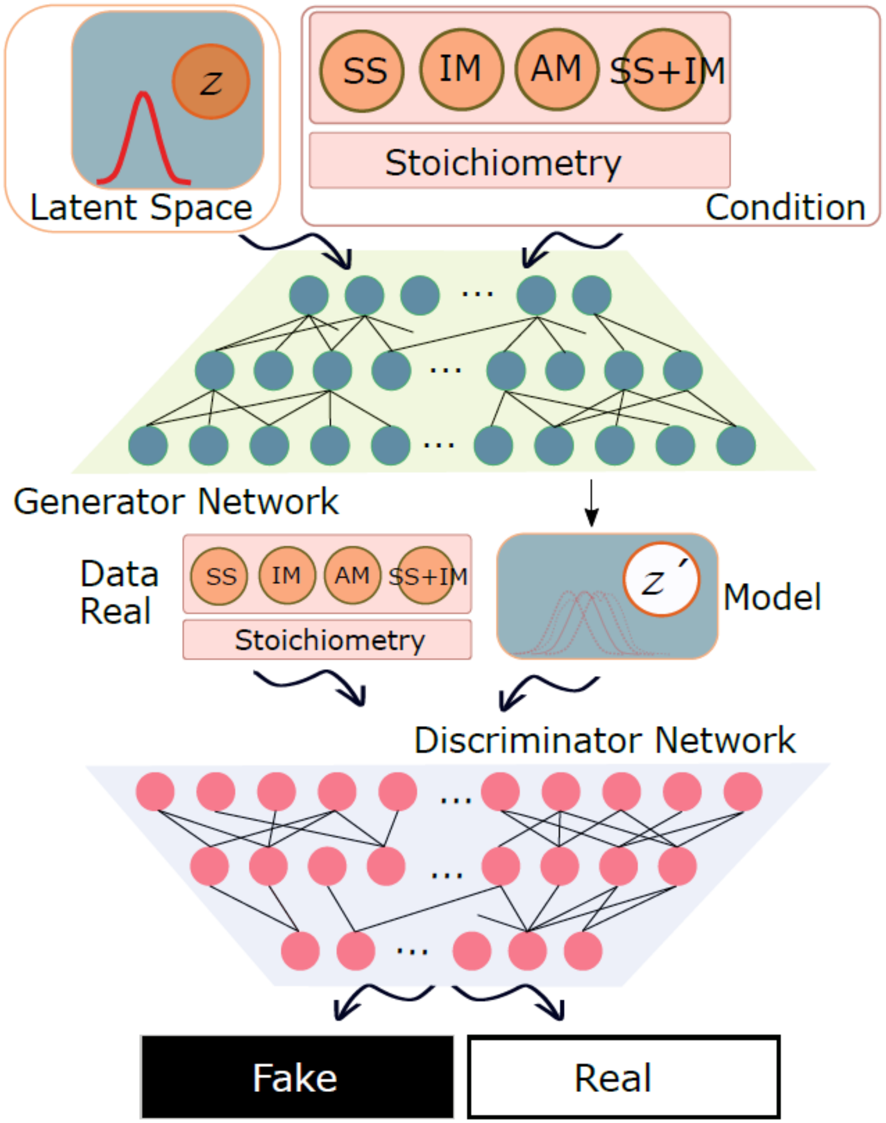
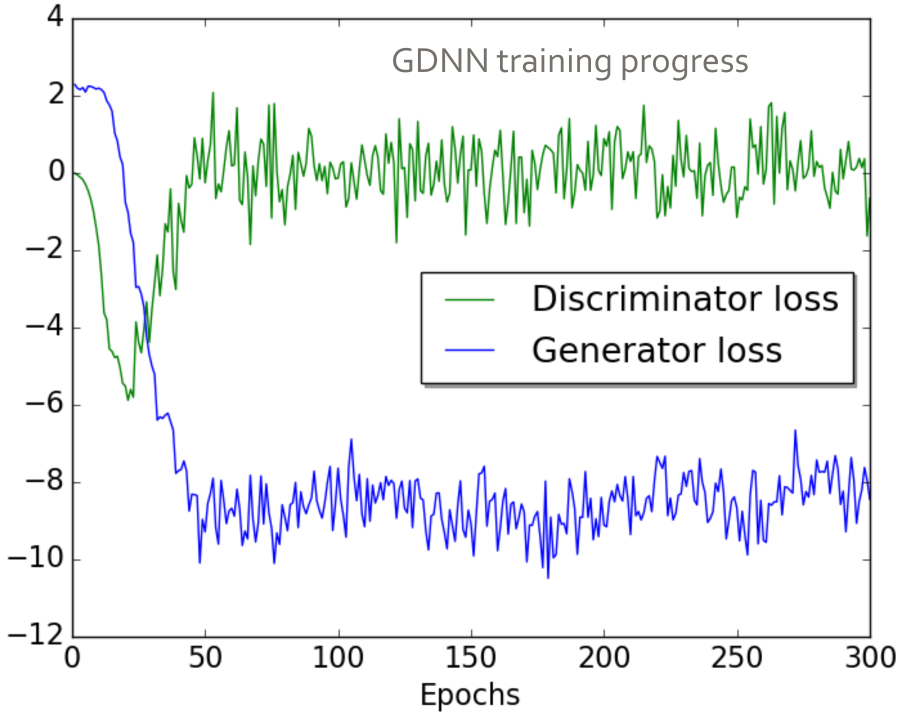
Training the model



	Generator		Discriminator	
Layer	Type	Dimension	Type	Dimension
Input	Latent + Cond.	90	Features + Cond	71
Hidden 1	Dense layer	256	Dense layer	256
Hidden 2	Dense layer	128	Dense layer	128
Output	Dense layer	71	Dense layer	1



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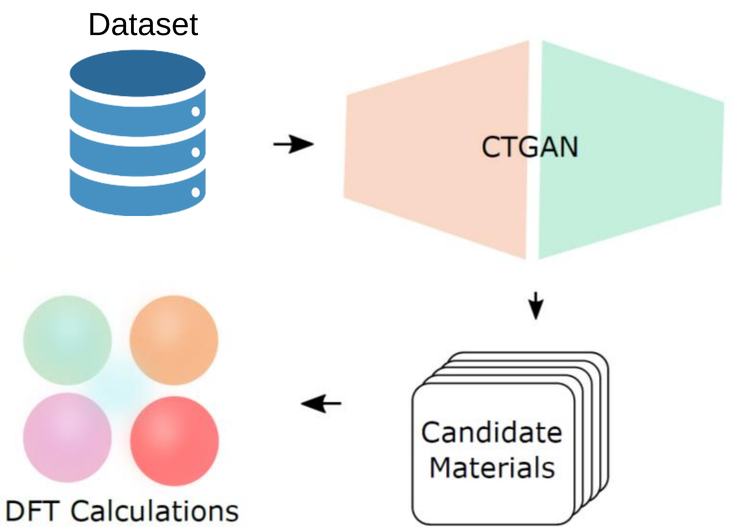
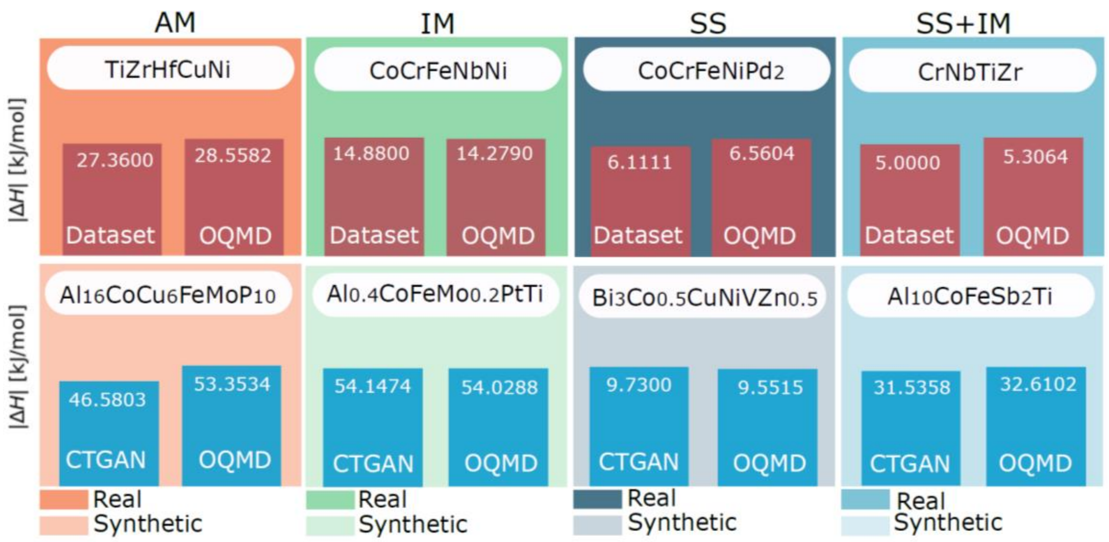
Validation of GAN

➢Validation methods

- Verification of generated samples which were not included at the training dataset
- Comparison between DFT-based calculation and NN enthalpy for HEA.





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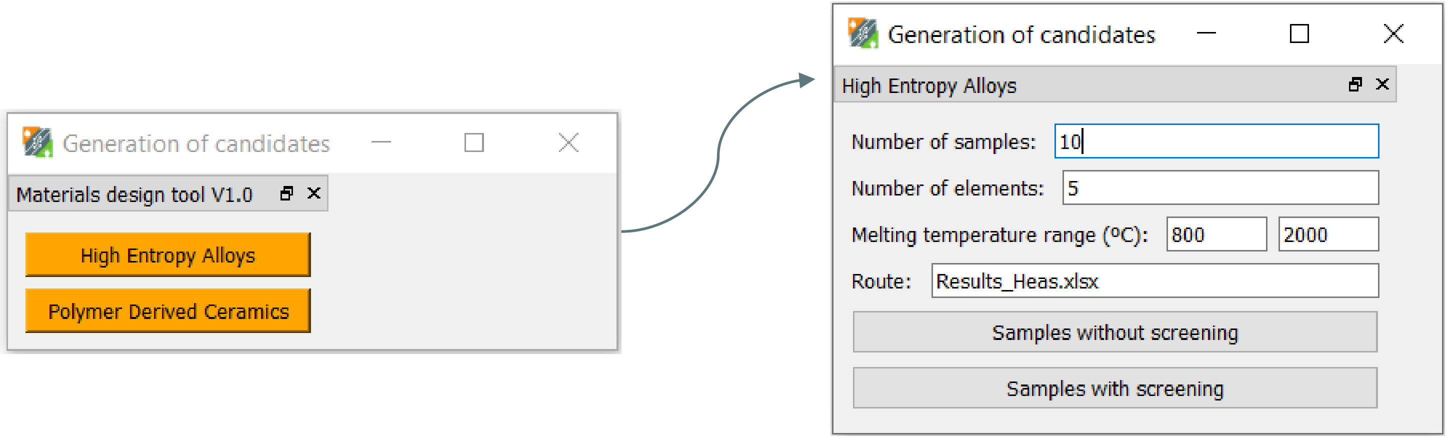
Materials Design Tool

Tool for generating high entropy alloys based on this methodology.



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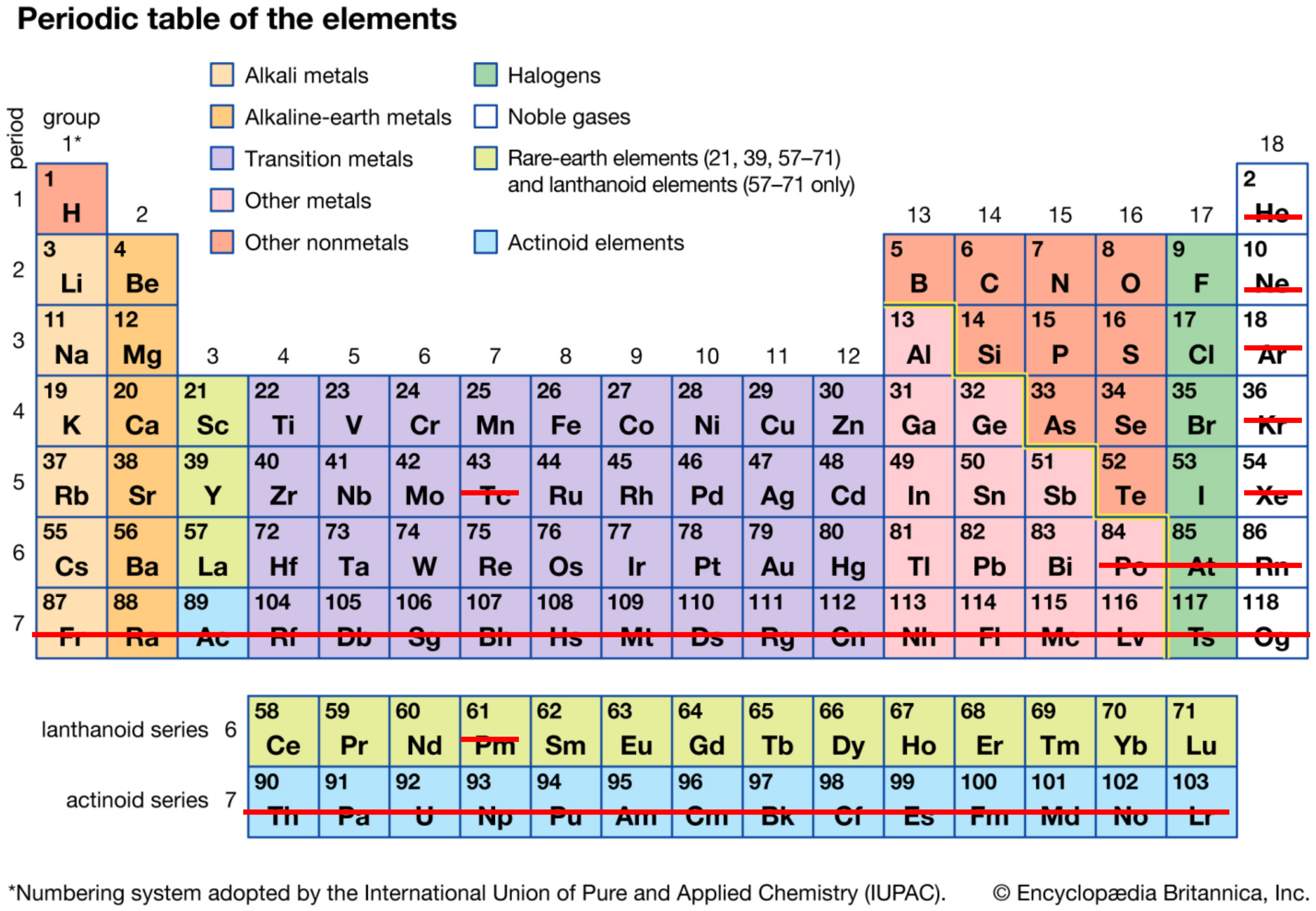
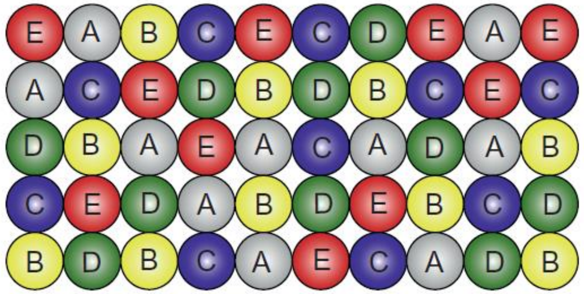
Screening for simulation



- **Initial approach:**
- Noble gases and radioactive elements removed
- A total of 78 chemical elements remained



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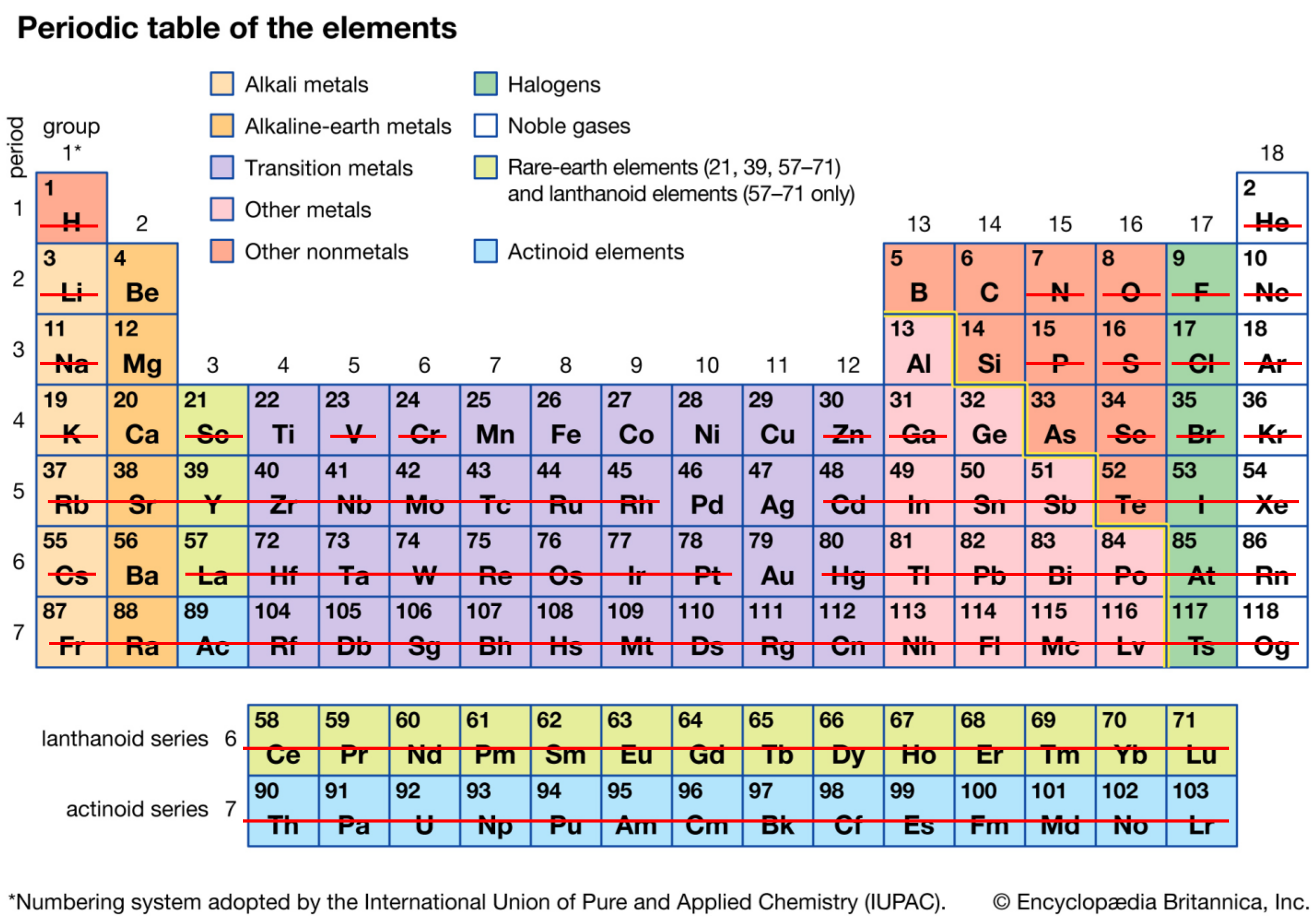
Screening for simulation



- Noble gases removed.
- Radioactive elements removed.
- Rare-earth elements removed.
- Toxic elements removed.
- Expensive elements removed.
- Elements with T_m > 2000 °C removed.
- A total of 19 chemical elements remained.

$$\binom{19}{5} = 11\,628 \qquad \binom{19}{6} = 27\,132$$

$$\binom{19}{7} = 50\,388 \qquad \binom{19}{8} = 75\,582$$

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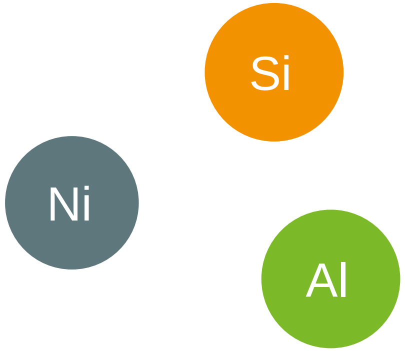
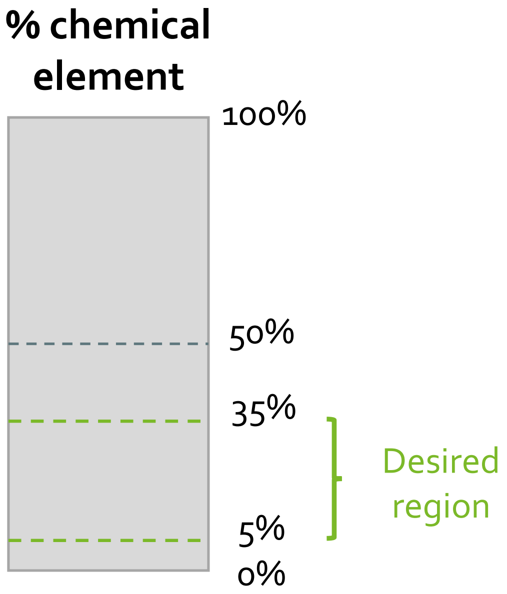
Screening for simulation

- Following VTT’s experience on stability analysis, compounds that include Ni, Al and Si were promoted.
- No more than 35% per chemical element.





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HESA Materials

The project has received funding from the European Union’s Horizon 2020 research and innovation program under Grant Agreement N° 958374.

Why HEAs-Basic data

- Introduced by Cantor in 2004
- At least 5 main elements (5-35% at) equal or near equiatomic proportion
- Based on High Entropy Concept

$$\Delta S_{\text{conf}} = -R \sum_{i=1}^{n} X_i \ln X_i$$

- More constitutional elements–> Higher configurational entropy

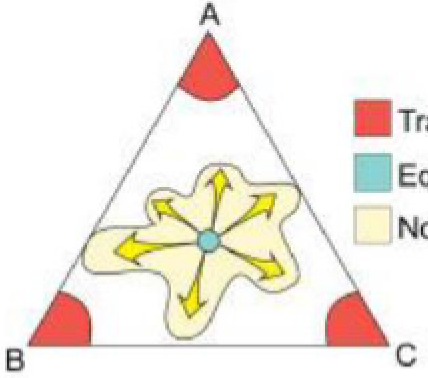
n	1	2	3	4	5	6	7	8	9	10	11	12	13
ΔS_{conf}	0	0.69R	1.1R	1.39R	1.61R	1.79R	1.95R	2.08R	2.2R	2.3R	2.4R	2.49R	2.57R

low | medium | high entropy →

1.0R 1.5R

- Higher entropy → Higher thermal stability

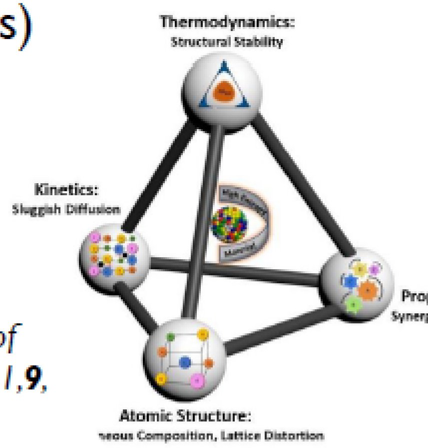
$$\Delta G_{\text{mix}} = \Delta H_{\text{mix}} - T\Delta S_{\text{mix}}$$



*Pradeep et al., **Mater. Sci. Eng. A 648,** 2015*

Four core effects of HEAs

1. High-entropy effect (thermodynamic)
2. Severe lattice distortion effect (structure)
3. Sluggish diffusion effect (kinetics)
4. Cocktail effect (properties)



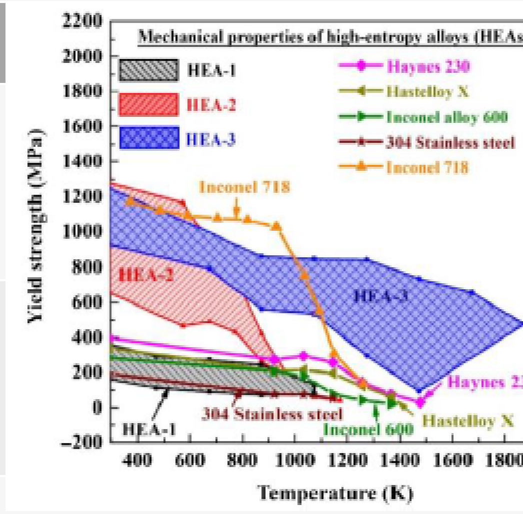
*Schematic showcases the four core effects of HEMs. A Amiri et al. **J. Mater. Chem. A**, 2021, **9**, 782-823*

HEAs for HT Applications

- Potential to replace Superalloys as the next generation high-temperature materials

1. **Refractory HEAs (RHEAs):** TiZrNbHfTa, AlMo0,5NbTa0,5TiZr, Ta-Mo-Cr-Ti-Al
2. **High entropy superalloys (HESAs):** $Alx\text{-}Tiy\text{-}Co_ACr_BFe_CNi_D$
3. **Eutectic High Entropy Alloys (EHEAs):** AlCoCrFeNi2.1 , CuFeNiTi, Co-Fe-Mn-Ni-Ti

HEAs	HESAs	EHEAs
perior HT strength through dual-ase nanostructure A2B2 (bcc)	HT strength due to Y/Y´structure through precipitation hardening	One hard phase on a ductile Solid Solution (SS) one (lamellar morphology) Excellent both strength and ductili
gher operating Ts>900°C	Operating temperatures up to 900°C	Good compromise between high strength and ductility up to 700°C
CHALLENGES		
mited processability/getting ements; Costly	Coarsening of precipitates at Grain Boundaries (GBs) promotes embrittlement	Designing of EHEAs
estricted RT ductility	HT strength need to be improved compared with State of the Art (SoA) Ni-based Superalloys (SA)	
wer oxidation resistance at HT		



Challenges in HEA design

Hit and trial method

- No phase diagram
- Does not follow Hume-Rothery rules
- Very large number of combinations

Ab initio simulation

- No potential available
- Huge computing power and time required

Parametric approach

- No scientific background (empirical rule)

CALPHAD

- Limited reliable databases
- Time and cost effective

In ACHIEF a combination of AI+ physical modelling is pursued for fast screening of HT HESA candidates

PROCESSING ROUTES IN HEAS

- Casting (arc-melting)
- Powder metallurgy route (mechanical alloying)
- In situ deposition routes (Laser directed energy deposition)
- Carbothermal shock synthesis (HEAs nanoparticles)
- Combinatorial materials synthesis (chemically graded HEAs)

In ACHIEF, atomization + powder-Additive Manufacturing route have been selected to have enough quantity for industrial application and quality powder specific for AM routes.

PDC coating developments with improved high temperature corrosion and erosion resistance

Sébastien Vry – CEA Liten, Grenoble, France

The project has received funding from the European Union’s Horizon 2020 research and innovation program under Grant Agreement N° 958374.

Objectives and applications

Development of a “easily applicable and inexpensive” anti-corrosive coating based on PDC and fillers for industrial user case

Objectives
O3.1. To assess the thermodynamically characteristics for optimum selection the nature of charges and pre-ceramic polymer for improved high-temperature corrosion and wear resistance. O3.2. To select the appropriate formulation and determine processing parameters for coatings development. O3.3. To Identify the processing-composition-properties relationship that control high-temperature characteristics against aluminium attack.

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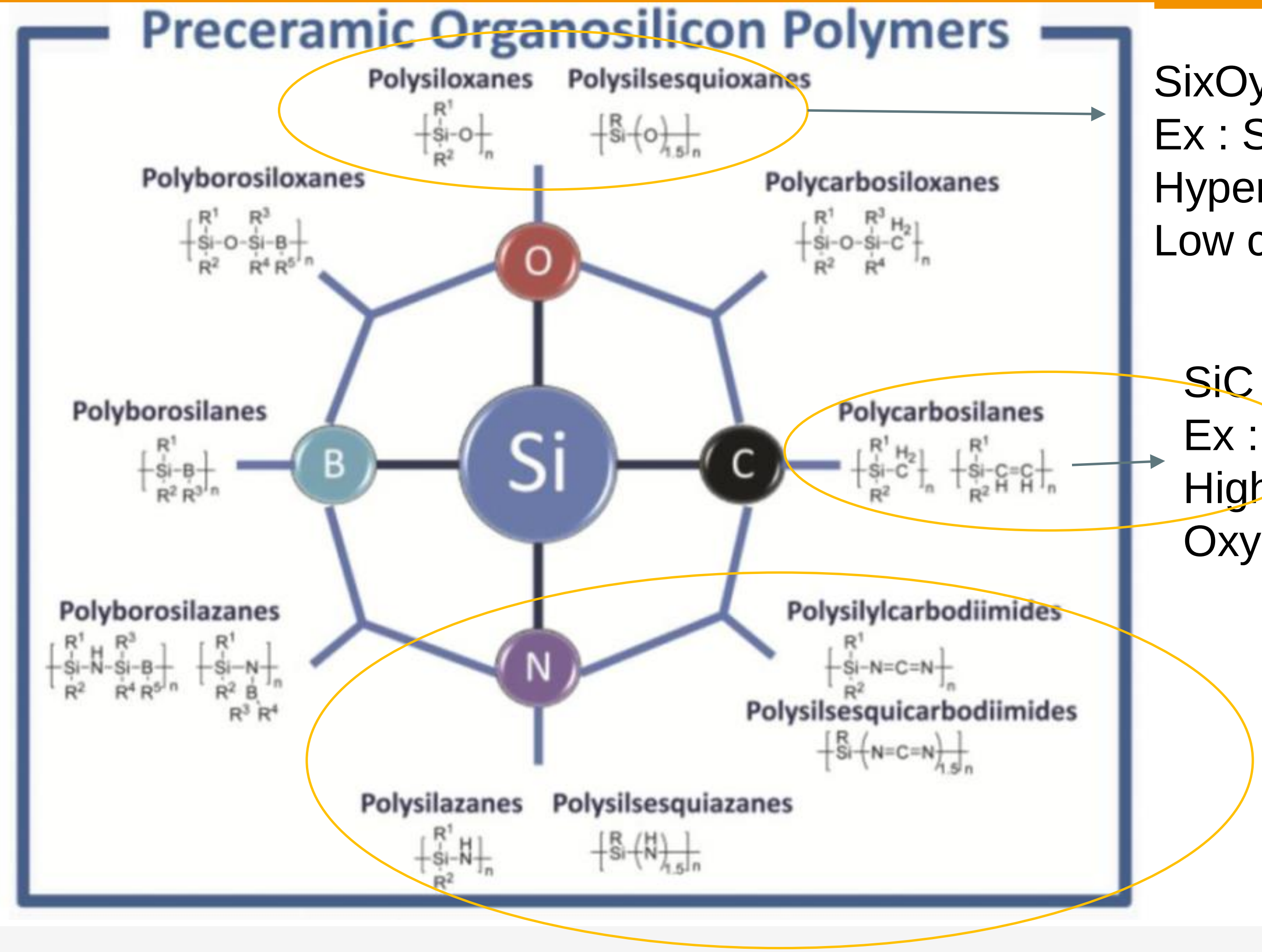
Pre-ceramic polymer Family / To remind !



SixOyCz
Ex : Silrès MK
Hyper-branched
Low cost

SiC
Ex : SMP10
Higher cost
Oxydation

SixCyNz

Organosilicon type pre-ceramic polymers make it possible to produce silicon-based ceramics by applying heat treatments to them in air or in inert atmospheres. It is possible to modulate the properties of the final ceramic by controlling the chemistry of the precursors and the associated heat treatments.

P Colombo, et al., Polymer-Derived Ceramics: 40 Years of Research and Innovation in Advanced Ceramics, J. Am. Ceram. Soc., 93 [7] 1805–1837 (2010)

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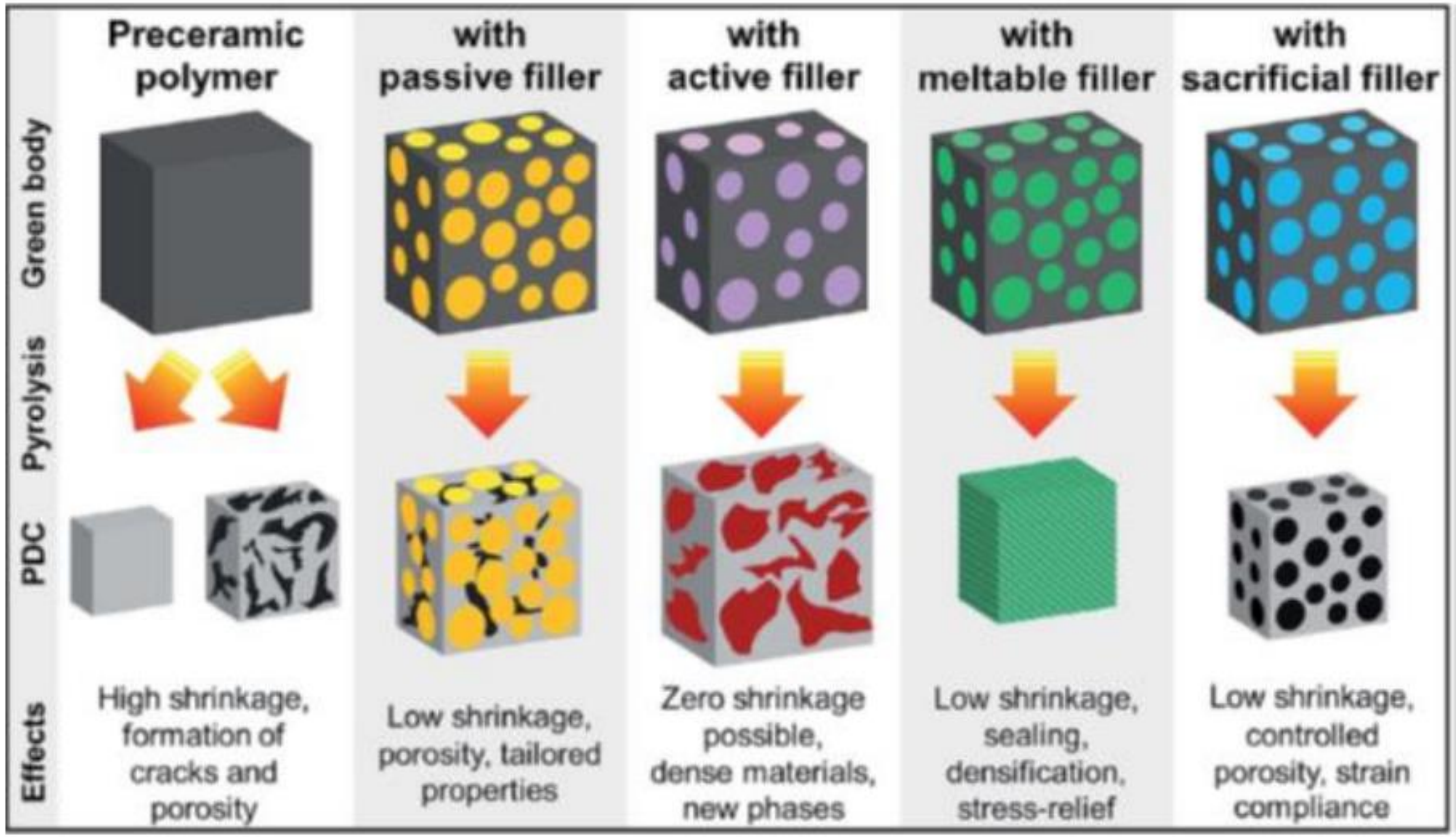
Différent types of fillers



G. Barroso, Q. Li, R. K. Bordia, and G. Motz, 'Polymeric and ceramic silicon-based coatings – a review', J. Mater. Chem. A, vol. 7, no. 5, pp. 1936–1963, 2019, doi: 10.1039/C8TA09054H.

The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N° 958374.

PDC Properties

Depending on the mixture and addition of metallic or ceramic fillers:

- Mechanical properties: high hardness, modulus and strength.
- Stability in extreme environments: good thermostability, high oxidation and corrosion resistance.
- Adhesion properties: high adhesive attraction and low surface tension.
- Hydrophobicity (non wetting)
- Durability: wear resistance, anti-fouling and anti-biofilm formation properties. Low toxicity and biocompatibility.

The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N° 958374.

Approach

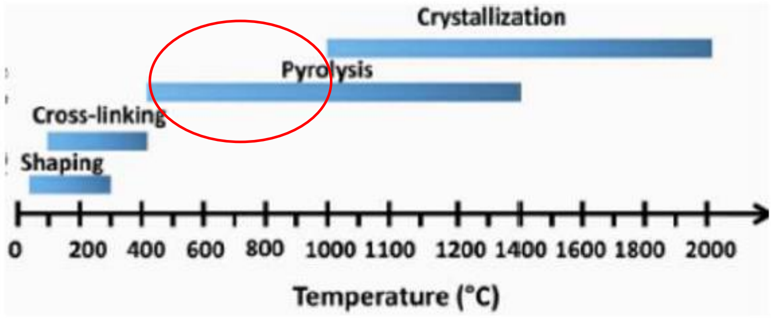
→ **Development of a coating based on**

- Preceramic polymer :
  - ✓ Low cost, easily processable
  - ✓ Tailor ceramic at molecular scale
  - ✓ Low temperature conversion into silicon based ceramics
  - ✗ High weight loss and shrinkage

Polysiloxanes → ΔT → SiO_xC_y

Polycarbosilanes → ΔT → SiC

- Ceramic fillers:
  - chemical barrier
  - anti-wetting
  - thermal & mechanical properties



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User cases

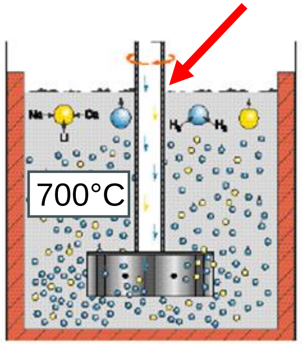
Constellium

→ Components of Aluminum foundry casting

- ❑ Bricks (refractory)
- ❑ Rotors (graphite)

→ Difficulties

- Adhesion, wearing and diffusion of Aluminum into refractory
- Oxidation in air of graphite



Objectives :

- ❖ Reduce defects for the rotor
- ❖ Reduce of 25% the replacing frequency
- ❖ Increase durability at least 20%

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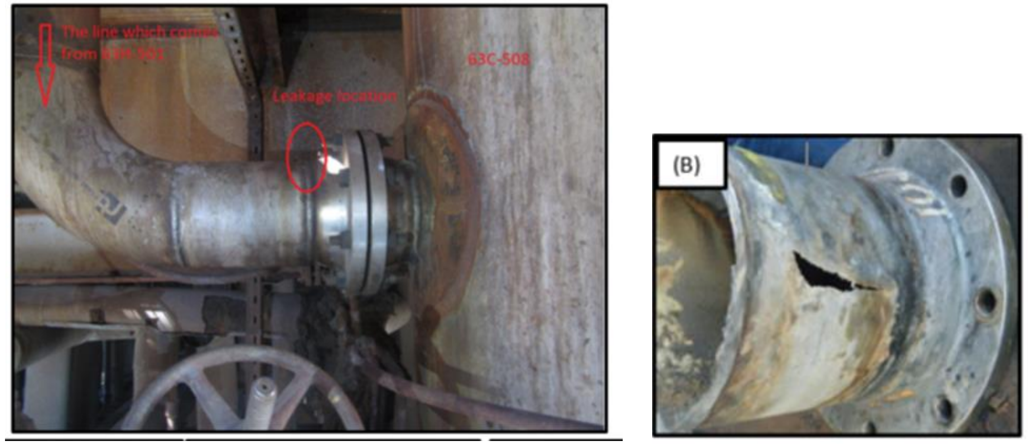
User cases

Tüpras
Turkish Petroleum Refineries Corp.

→ Continous Catalyc Cracking unit venting pipeline (*converts naphta into high octane reformate*)

- Corrosion and leakage damage on SS321 pipes in HCl environment



Objectives :

- Decrease corrosion by 40% (6 → 3.6 mm/year)
- Decrease replacing frequency of the pipelines
- Increase the maintenance interval (>12 month).

→ Saves: cost production, energy consumption
→ Decrease the risk on the process safety

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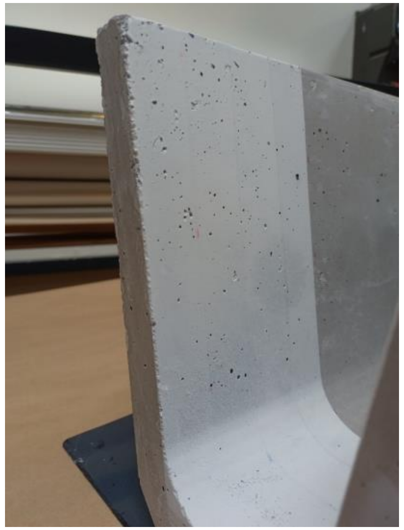
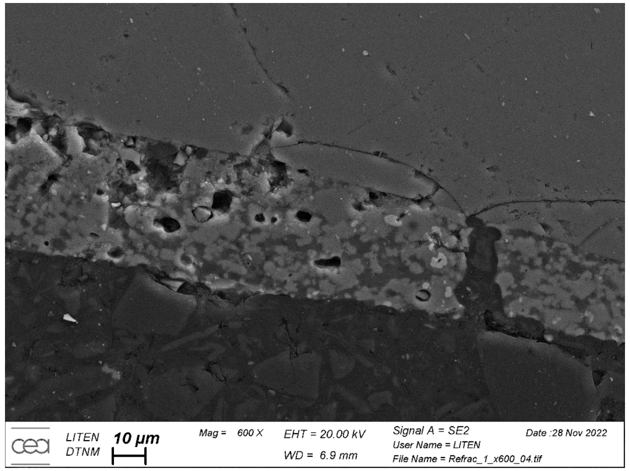
Scaling up the method of deposition

Robotic Arm deposition

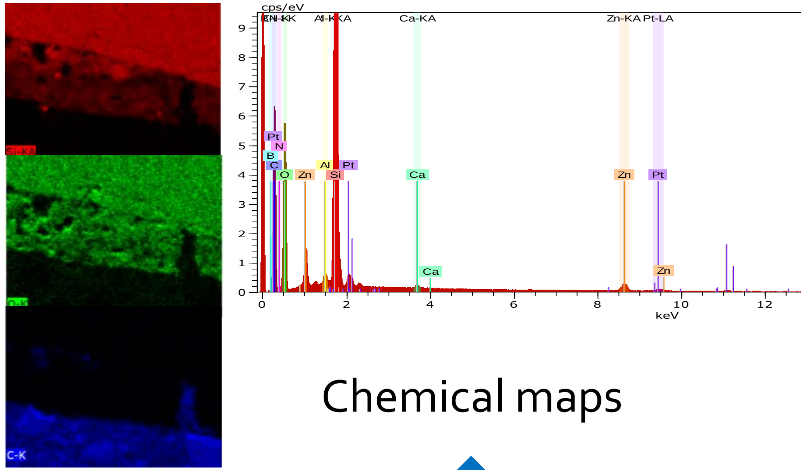
Creation of trajectory



Evaluation of aspect and thickness with spraying parameters (20-30 µm)



Thermal treatment and evaluation of coating homogeneity



Chemical maps

Computation modelling of thermomechanical properties

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Work plan

Formulate materials
→ Target properties

Feed
Knowledge

Computation modelling and AI
→ List of spec

Objectives:
- Good adhesion on surface (nature, cleaning, rugosity)
- No delamination during cycles → durability
- Continuous layer with no cracks (porosity, shrinking)
- Coefficient of Thermal Expansion close to the substrate
- Low surface free energy / wettability
- Inertness with liquid phase (chemical reaction, diffusion)

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ceramics

an Open Access Journal by MDPI

Silicon Carbide Precursor: Structure Analysis and Thermal Behavior from Polymer Cross-Linking to Pyrolyzed Ceramics

Sébastien Vry; Marilyne Roumanie; Pierre-Alain Bayle; Sébastien Rolère; Guillaume Bernard-Granger

Ceramics **2022**, Volume 5, Issue 4, 1066-1083

The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N° 958374.

Optimized Cr-steels for Creep Resistance

Lorena M. Callejo - TECNALIA

The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N° 958374.

OUTLINE

- WHAT IS CREEP?
- THE ROLE OF THERMAL TREATMENTS
  - Austenitization
  - Annealing
  - Normalizing
  - Quenching
  - Tempering
- HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS
  - Requirements
  - Definition
  - Feasibility
  - Pre-industrialization
  - Industrialization
  - Characterization

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WHAT IS CREEP?

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CREEP

- In many applications, alloys work at high temperatures under static loads (steam generators, turbine blades, etc)
- In such conditions, after time, materials suffer from permanent deformation although the load keeps constant
- This phenomenon is known as creep, and it becomes relevant in alloys when exceeding 0.5 Tm (K)

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CREEP

A creep test involves a tensile specimen under a **constant load** maintained at a **constant T. Measurements of strain are then recorded over a period of time.**

- **The Yield Point** is the **stress** an alloy can resist **without breaking** within a certain temperature range indefinitely
- **Components are designed** so as not to be strained more than acceptable or not to be broken for an established period of time which must be longer than the component lifetime

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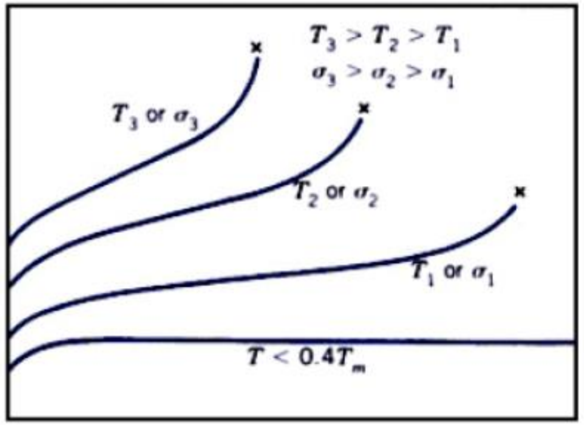
o CREEP TEST

1. Cylindrical test samples are tested under constant stress and constant temperature. The test sample inside the furnace is tensile tested under constant load for a defined period of time (from 1.000 to 10.000 hours), and finally the strain/elongation is measured as a function of time

The result is the lifetime of the test sample:

Time for rupture / time to reach the defined strain (for example 1%).

Creep strain vs Time:



2. The tests are repeated at different temperatures and for different stress values

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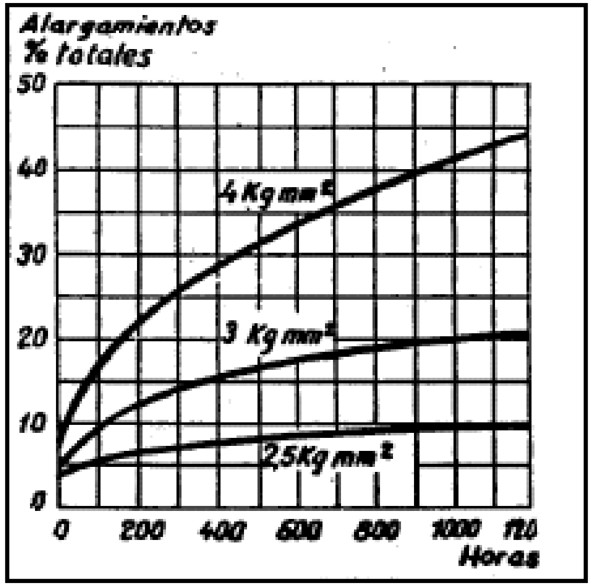
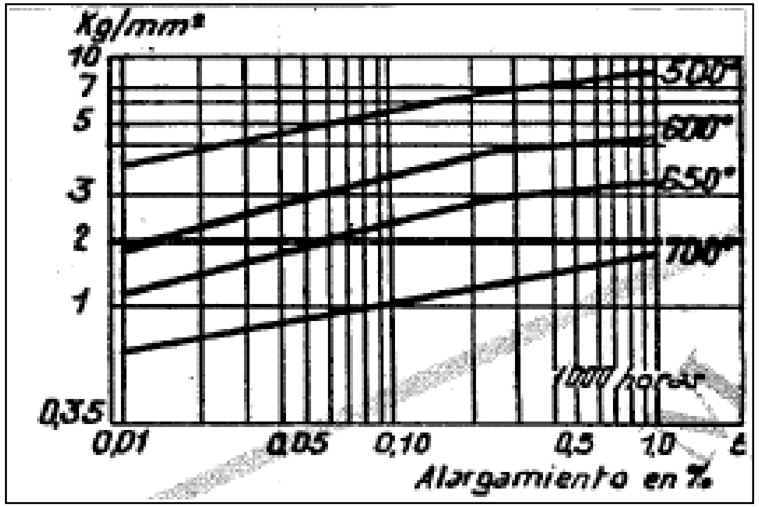
Elongation/strain values obtained for different stress values at different times



Constant Temperature

Elongation/strain values obtained for different stress values at different temperatures



Constant Time

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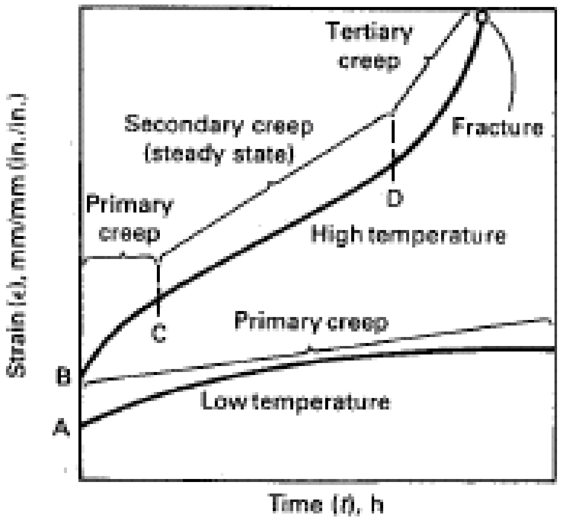
o Different types of creep



- **At low T**, **T < 0,3T$_m$**, logarithmic creep
- Creep rate decreases with time
  - The strain is always small
  - The stress is higher than the yield strength

- At **high temperature**, **T > 0,3T$_m$**, recovery creep. 3 zones can be distinguished:
  - Primary creep: where the strain rate decreases
  - Secondary creep: where the strain rate keeps constant
  - Tertiary creep: where the strain rate increases until rupture

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THE ROLE OF THERMAL TREATMENTS

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THE ROLE OF THERMAL TREATMENTS

THERMAL TREATMENTS ARE APPLIED TO:

Provide the alloy with the properties required.
Main factors to take into account: **Time & Temperature**

Can change:

1. The nature of the constituents preserving the chemical composition
2. The microstructure
3. The internal tensions

The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N° 958374.

THE ROLE OF THERMAL TREATMENTS

THERMAL TREATMENTS ARE APPLIED TO:

Provide the alloy with the properties required.
Main factors to take into account: **Time & Temperature**

USUAL THERMAL TREATMENTS	SURFACE THERMAL TREATMENTS
Austenitization	Carburising
Annealing	Nitriding
Normalizing	Carbonitriding
Quenching	Surface quenching
Tempering	

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USUAL THERMAL TREATMENTS

Austenitization
Annealing
Normalizing
Quenching
Tempering

SURFACE THERMAL TREATMENTS

Carburising
Nitriding
Carbonitriding
Surface quenching

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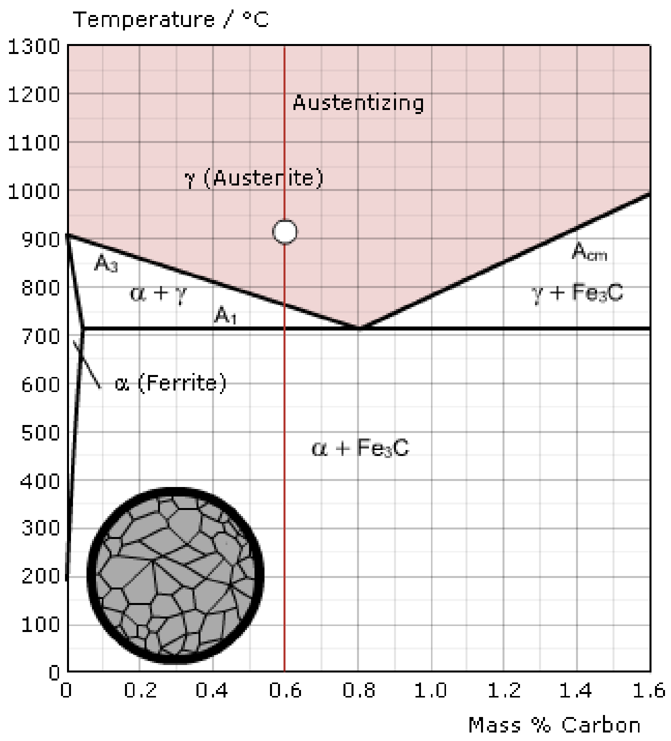
- Austenitization

The steel grade **is heated over A3/Acm (critical temperature) to obtain a fully austenitic** microstructure

- The previous thermal history and microstructure of the material are removed



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THE ROLE OF THERMAL TREATMENTS

- Annealing = Heating + holding + slow control cooling down to RT

There are several types of annealing that can be applied as a function of the final objective:

- **To remove previous structures resulting from forging/rolling/thermal treatment: microstructure regeneration**
- **To soften the steel**
- **To promote structures suitable to ulterior processes/thermal treatments**
- **To remove internal and surface tensions**
- **To homogenize the chemical composition (eliminate segregations), promote diffusion**

Types of annealing

Of Total Austenitization

$T > Ac_3 / A_{cm}$

- Steel softening & Structure regeneration

Subcritical annealing

$T < Ac_1$

- Remove tensions & Increase ductility

Of Partial Austenitization

$Ac_1 < T < Ac_3 / A_{cm}$

- Steel softening & Improve machinability

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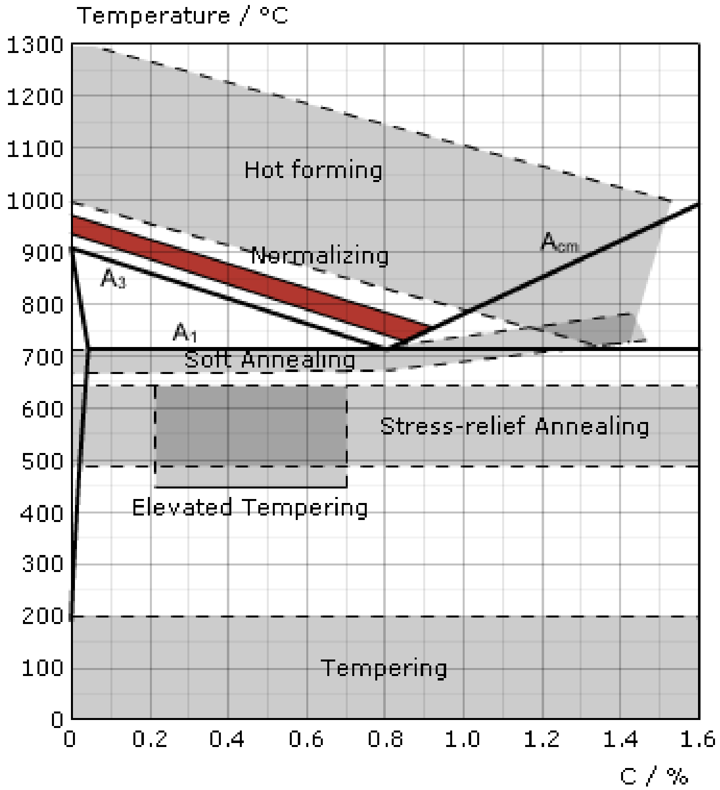
- Normalizing

Heating at a **T** > Ac_3 / A_{cm} (~ 50º-70ºC higher)
& Air cooling (CR < quenching & > annealing)



Objective:

- Remove internal tensions
- Refinement & homogenisation of the grain size
- Improve the mechanical properties

It is commonly applied to steels that have been subjected to hot/cold working, irregular coolings or supercoolings

Or to remove the effects of a previous treatment

- It is only used for Carbon steels or low alloyed steels (0.15-0.40%C)

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THE ROLE OF THERMAL TREATMENTS

- Quenching

To Harden and strengthen the steel through the formation of martensitic structures (at least 50% of martensite in the core)

- **Uniform Heating >**Ac_3 (40º-60ºC higher)
- **Holding at such T**
- **Rapid cooling** (in water, oil, etc) depending on the steel grade

Hardenability: ability of the steel grade to form martensite from austenite on cooling

It is determined by the depth reached by the martensitic transformation in the steel, and consequently the hardness distribution (from the surface to the core) inside the steel component

Factors influencing the quenching process and CCR: chemical composition; grain size; component size, quenching conditions

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- Tempering
  - Commonly applied after quenching to decrease the hardness & strength of the steel and increase the toughness
    - **Heating to a T <** Ac_1
    - **Holding at that T**
    - **Air cooling (conditions & time according to the requirements of the component)**

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HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS

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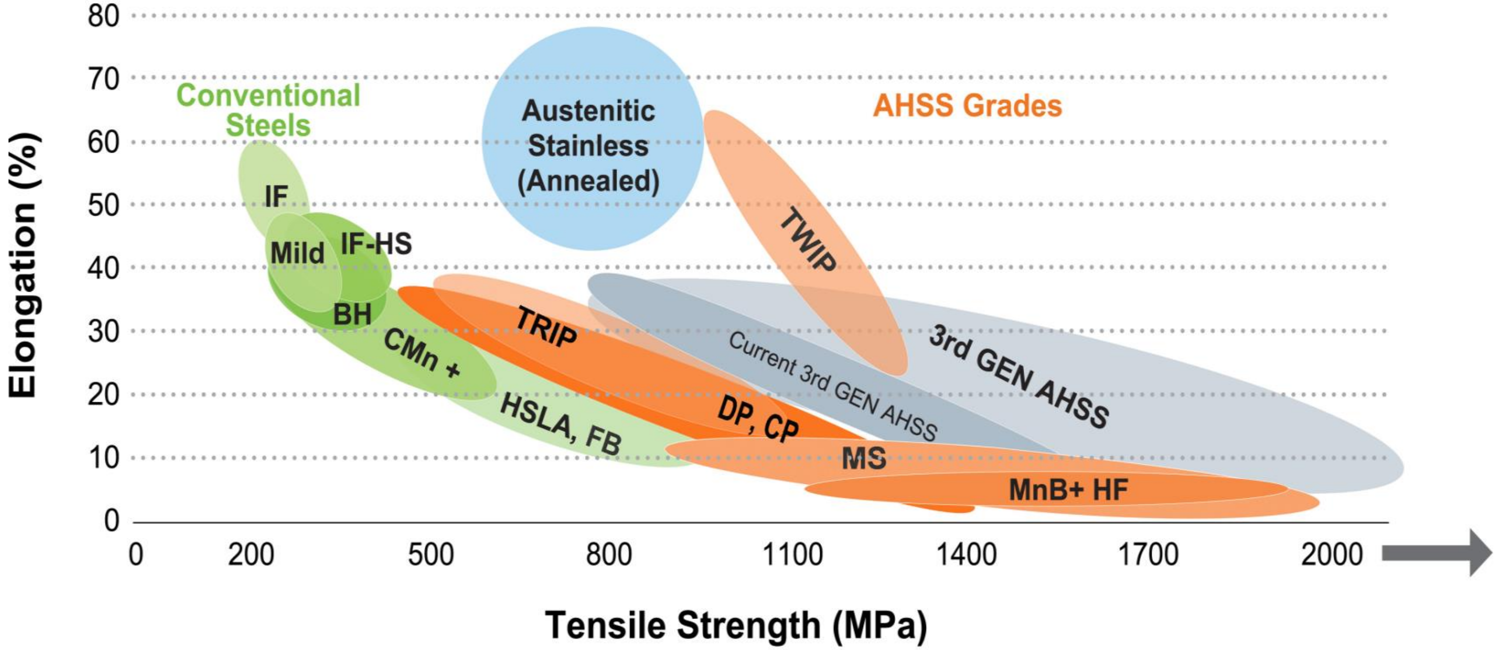
HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS



Source: WorldAutoSteel

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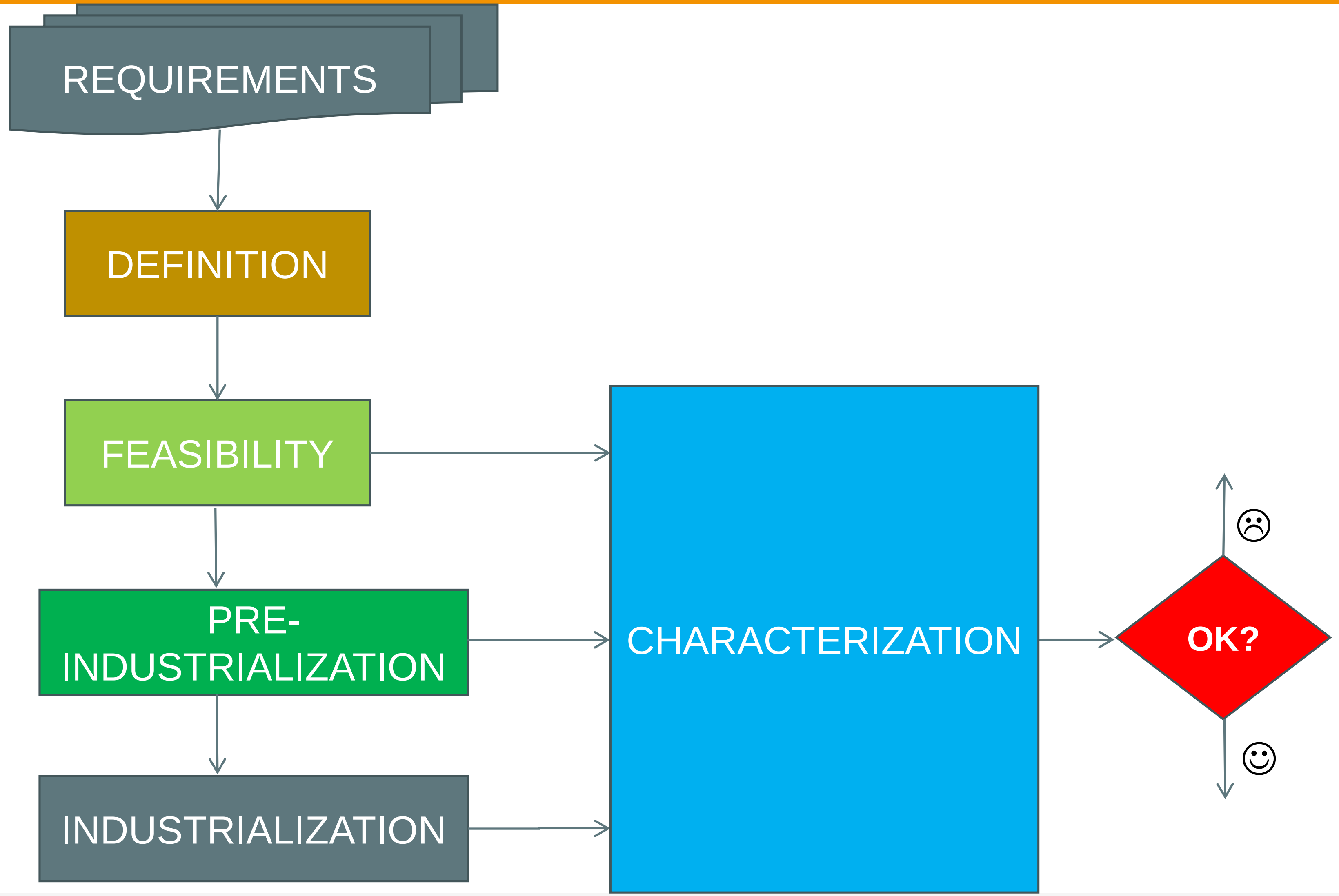
HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS



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HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS

REQUIREMENTS

Requirements / Objectives pursued

- **15% improved Creep strength** in comparison to T115 grade
- **Corrosion resistance** of T115 preserved
- **Weldability**

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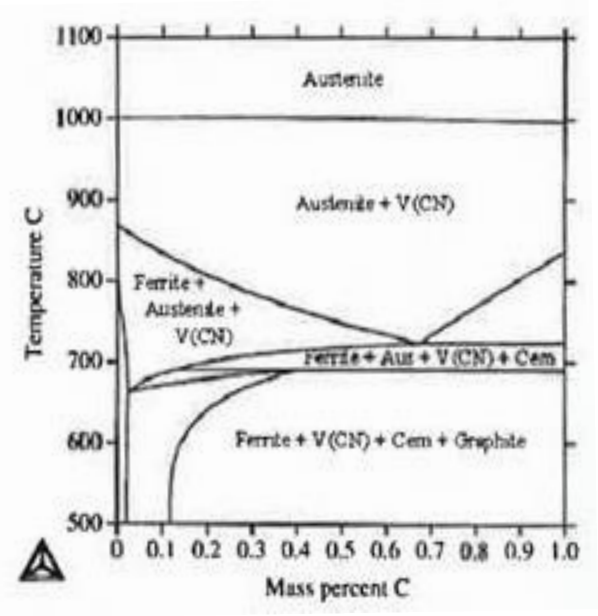
HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS

DEFINITION

- ❑ Advanced thermodynamic calculations
- ❑ Equilibrium phase diagrams
- ❑ Solidificación simulation
- ❑ Calculation of properties
- ❑ Diffusion calculations

Design of chemical composition and/or thermal treatment by modelling

- ➢ Advanced modelling software (ThermoCalc, DICTRA, JMatPro, etc.)
- ➢ **In-house** models (analytic and AI techniques based models)
- ➢ **Optimization** tools
- ➢ **Study the effects of the elements/precipitates** on desired properties



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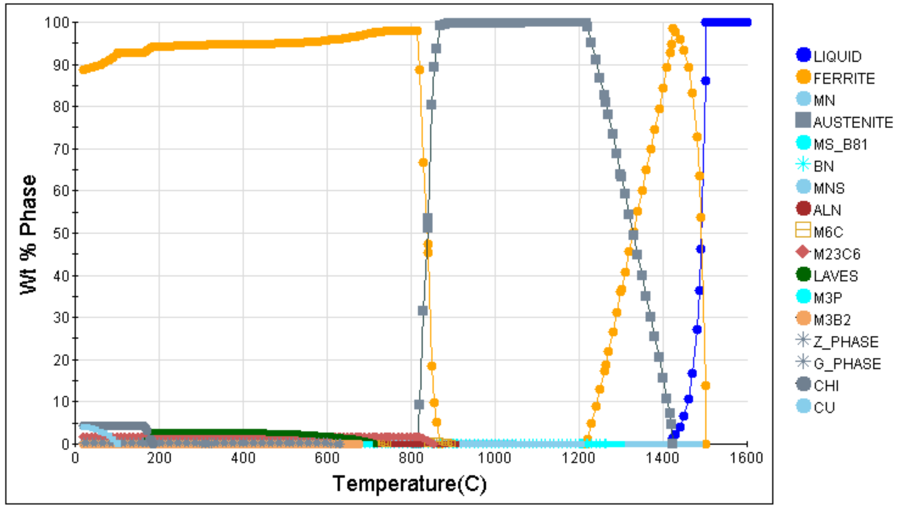
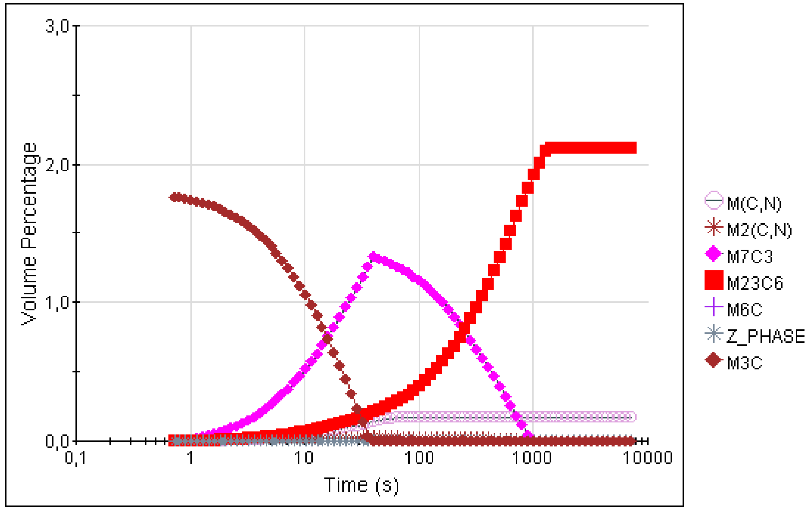
HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS

DEFINITION

Equilibrium phase diagram

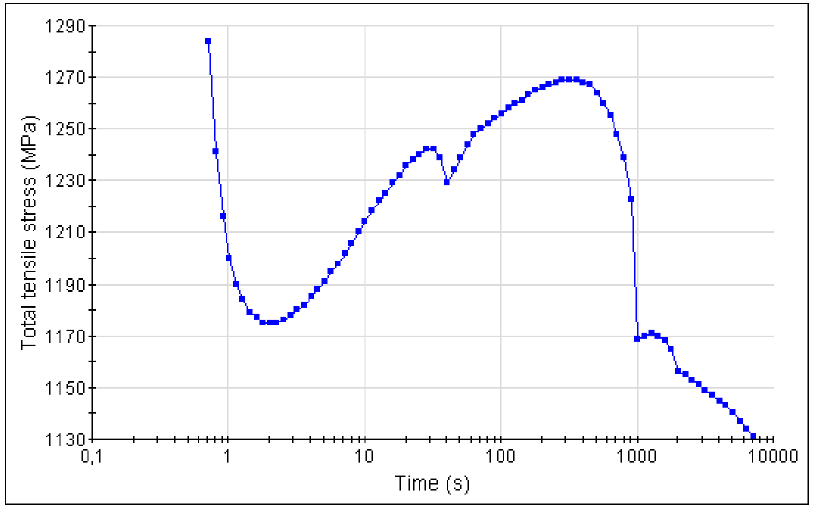


Precipitation during tempering



Solution temperature (C) : 1060.0
Tempering : at 765.0C for 7200.0 s

Evolution of mechanical properties during tempering



Solution temperature (C) : 1060.0
Tempering : at 765.0C for 7200.0 s

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HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS

FEASIBILITY

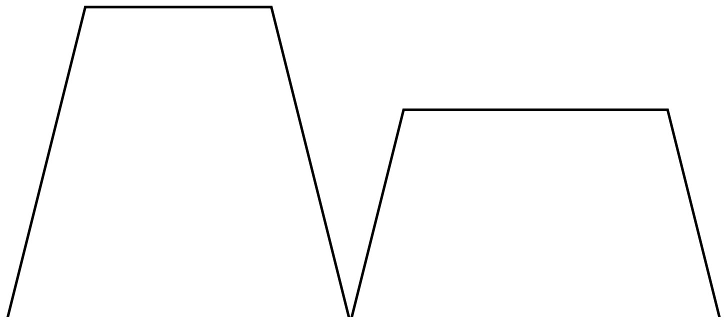
LAB SCALE: Casting, Forging, Thermal Treatment

Melting & Casting facilities for the new alloys (from 1 Kg – 20 Kg)

- Ar/air system, vacuum furnace, vacuum levitation furnace equipped with a Copper mold-casting system for rapid cooling

Improvement of thermo-mechanical and thermal treatments through design in experimental equipment

- Dilatometer (to analyse the material behaviour and **understand the microstructure evolution** during thermal treatment)
- Gleeble 3800 machine (thermo-mechanical equipment for **physical simulation** of transformation processes and thermal treatments of metallic materials)
- Furnaces and **salt baths** (to apply the thermal treatments)



DESIGN THERMAL TREATMENT
TEMPERATURES TIMES HEATING & COOLING RATES STEPS

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HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS

EQUIPMENT

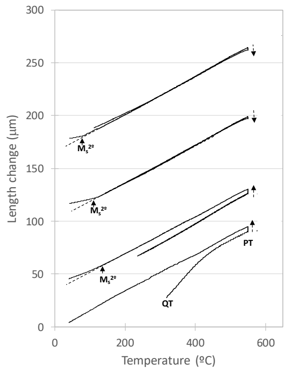
FEASIBILITY

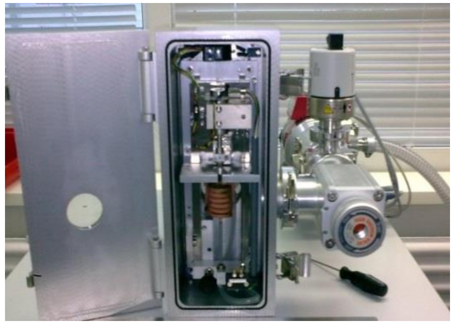
LAB SCALE: Casting, Forging, Thermal Treatment

Vacuum levitation furnace
Cu mold casting
(rapid solidification)
(1 Kg)

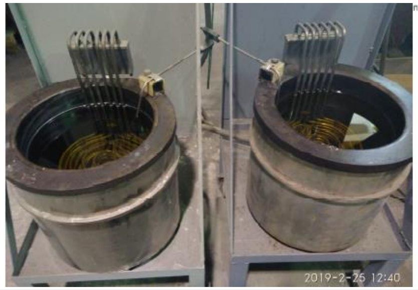
Induction furnace Air/Ar
(10 Kg)

Vacuum induction furnace (20 Kg)





Dilatometer



Salt bath installation

Gleeble 3800 System

Muffle-type furnaces

ACHIEF
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HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS

PRE-INDUSTRIALIZATION

PREINDUSTRIAL SCALE: Casting (Preindustrial level), Rolling into tubes, Thermal Treatment

Melting & Casting facilities for the new alloys (up to 1 T)

- **Pilot plant** equipped with induction furnaces to melt & cast steel up to 1 T

Improvement of thermo-mechanical and thermal treatments through design in experimental equipment

- Furnaces and **salt baths** (to apply the thermal treatments)

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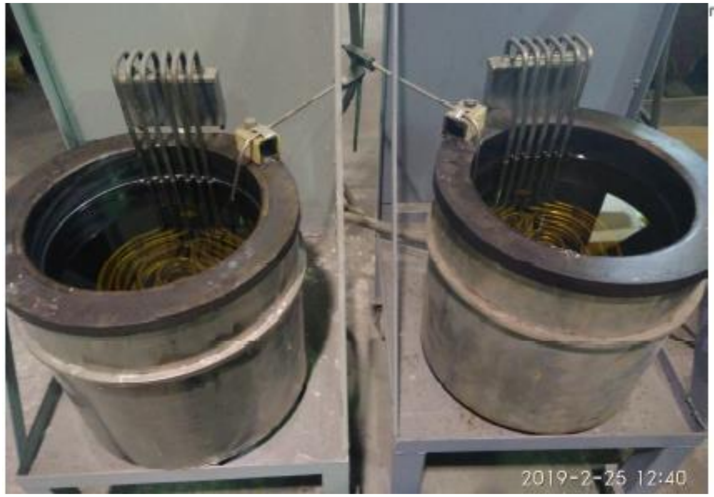
EQUIPMENT

PRE-
INDUSTRIALIZATION

PREINDUSTRIAL SCALE: Casting (Preindustrial level), Rolling into tubes, Thermal Treatment

Preindustrial / Pilot Plant (1 T)



Salt bath installation

Muffle-type furnaces

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HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS

INDUSTRIALIZATION

INDUSTRIAL SCALE: Casting (Industrial level), Rolling into tubes, Thermal Treatment (Industrial furnaces)

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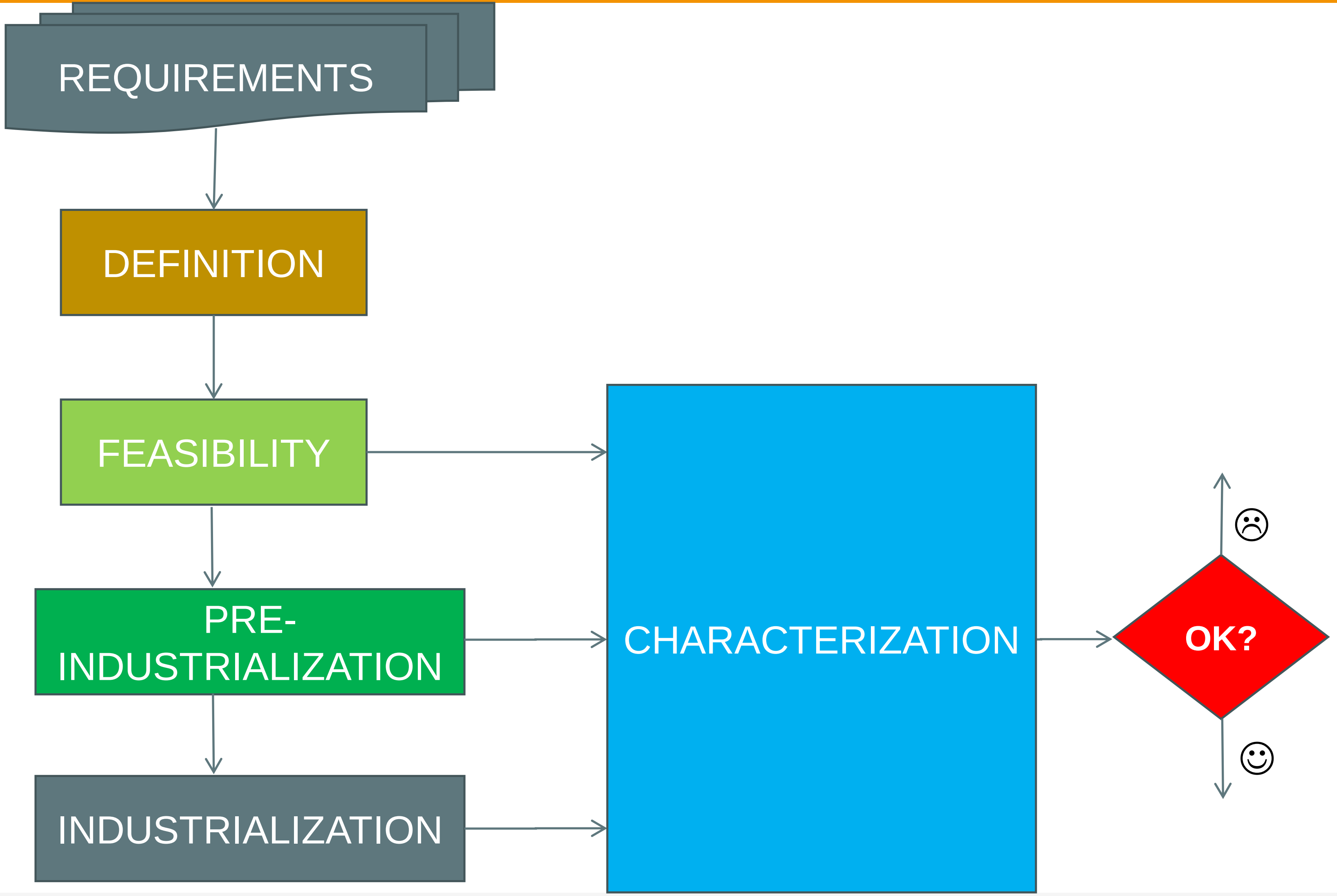
HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS



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HOW TO DESIGN AN ALLOY FOR SPECIFIC APPLICATIONS

CHARACTERIZATION →

Composition

Microstructure

Mechanical properties

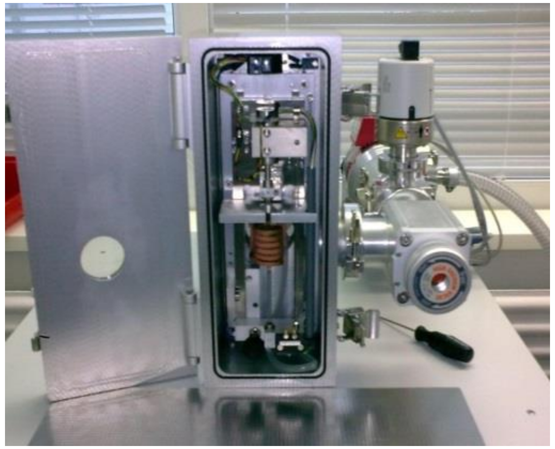
Creep Strength

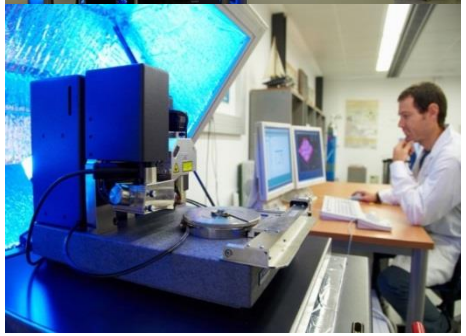
Corrosion resistance

Weldability

Explore Design Validate treatments

Tests design





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CHARACTERIZATION →

DESIGN CREEP TESTS

Define a constant value for Stress & Temperature according to the application targeted
Collect the variation of Strain with Time produced during the test

ZwickRoell

The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N° 958374.

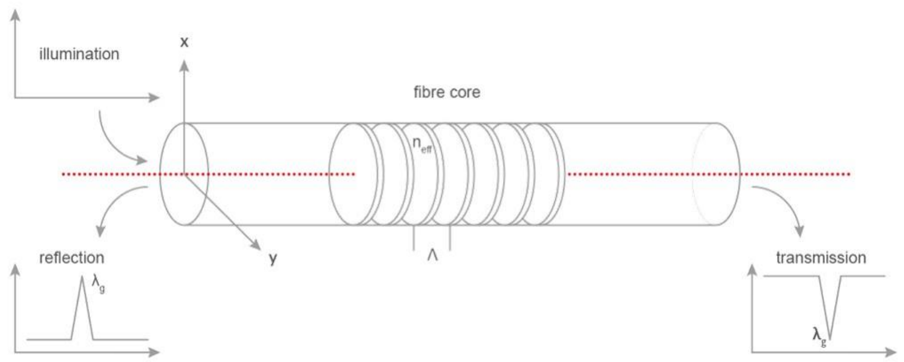
Sensors Development with the Ability to Withstand Harsh Environments

Dr. Andreas Pohlkötter (engionic, AIMEN)

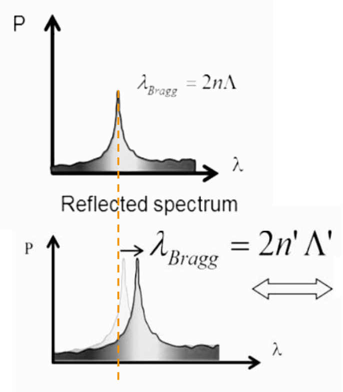
The project has received funding from the European Union’s Horizon 2020 research and innovation program under Grant Agreement N° 958374.

engionic Fiber Optics:
Point-by-point inscription of FBGs in glass fibers

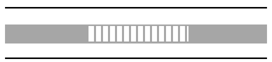
Principle of the Fiber Bragg grating sensor



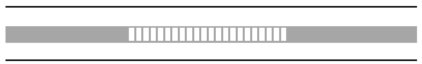
Details on measurement principle



FBG Λ



Strained/heated FBG



- Fiber Bragg Gratings (FBGs) are **optical reflection gratings** that are inscribed in optical fibers
- A **periodic refractive index change** of the fiber core with the distance of Λ leads to a formation of a wavelength selective mirror at $\lambda=2*n*\Lambda$ in the fiber core
- Strain and temperature changes cause a **change of the grating period** resulting in a change of the wavelength Δλ which is quasi linear over a large range
- Whatever physical quantity impacts the fiber expansion can be measured

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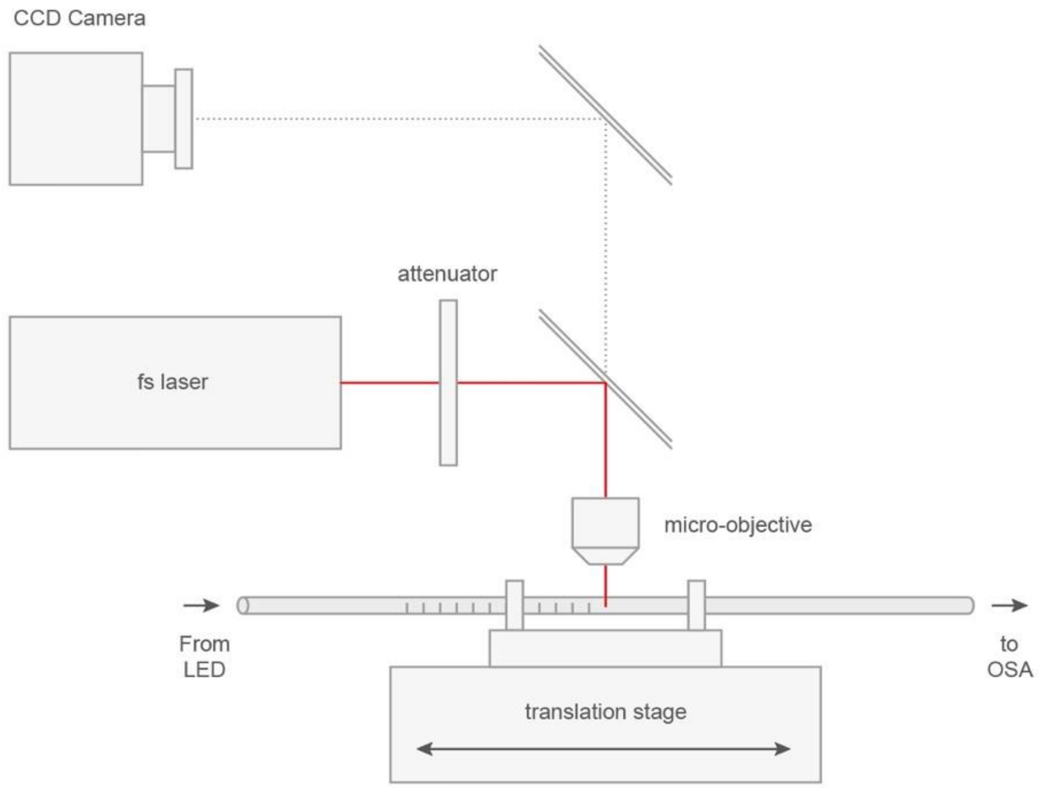
engionic Fiber Optics:
Point-by-point inscription of FBGs in glass fibers



Schematic of point-by-point FBG inscription setup

- Highly flexible **point-by-point inscription** without phase mask allows writing of any wavelength
- Writing **through the coating** is possible due to high transmission of typical coatings for IR light and low Laser intensity at coating
- Highly flexible **array configurations** with **distances** between a few mm and several km in **customized** fibers are possible

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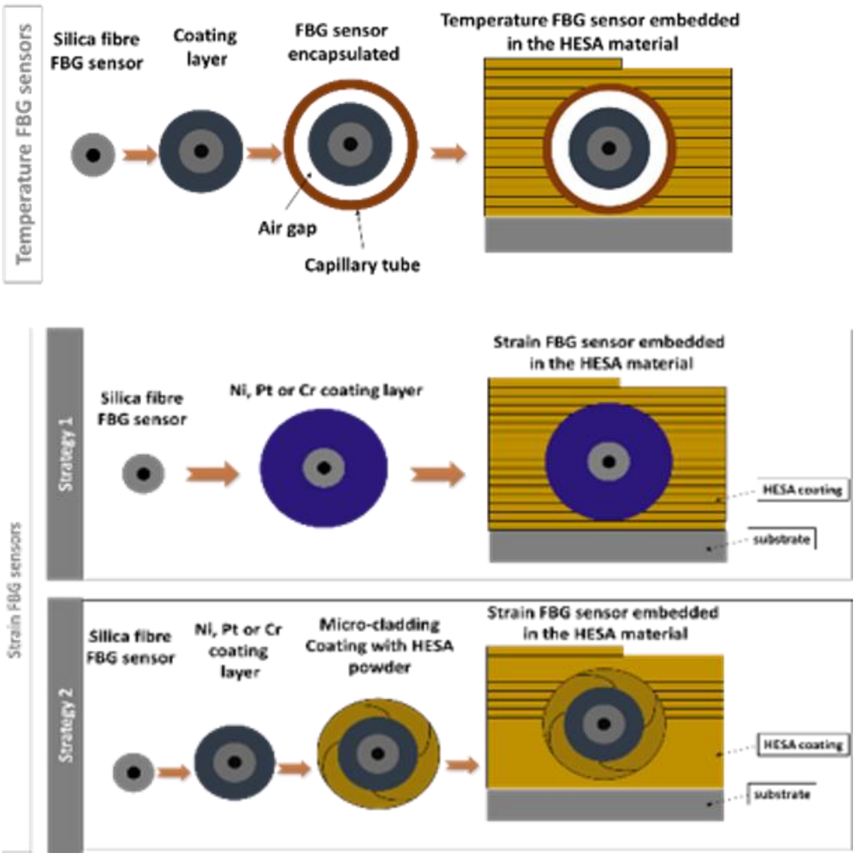
AIMEN:
Embedding glass fibres in HESA material

To use the FBG sensor in high temperature industrial environments a **robust mounting system** is needed.

Concept:

1. Removal of fiber polymer coating
2. Inscription of FBGs
3. Coating with metal
4. Embedding within High Entropy Super Alloy (HESA) material

For **temperature sensing**: optical fibre **loosely** mounted in tube
For **strain sensing**: optical fibre **mechanically bonded** to measuring object



Schematic of sensor mounting in HESA material

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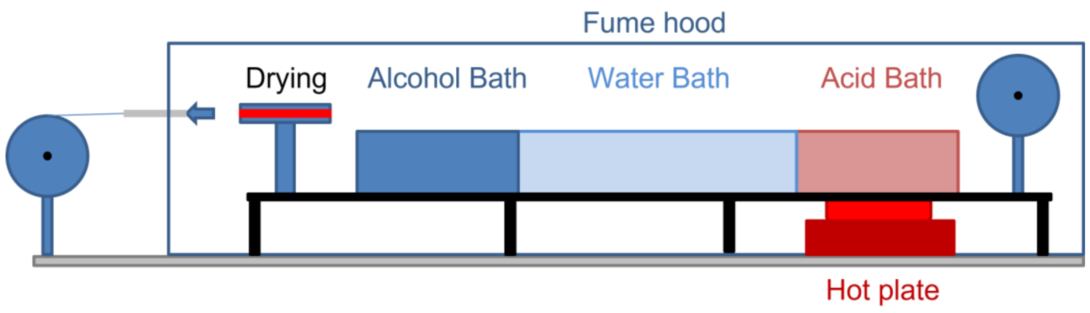
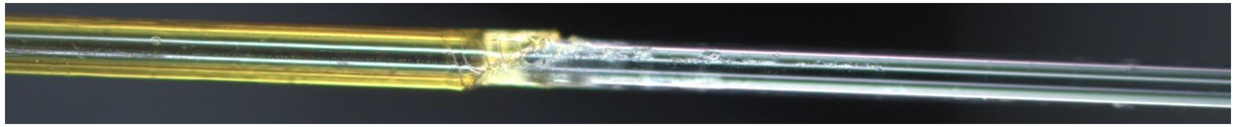
1. Removal of fiber polymer coating over long lenths



- Decoating of optical fibers over long length is difficult as the **fiber is brittle without coating**
- An apparatus for decoating fibers by **wet etching** was developed and set up
- First tests with polyimide-coated fibers were successfull
- The apparatus can be used for different types of coatings including metal-coated fibers with different acids for decoating



Schematic of fiber decoating setup, microscopic image of processed vs. coated fiber

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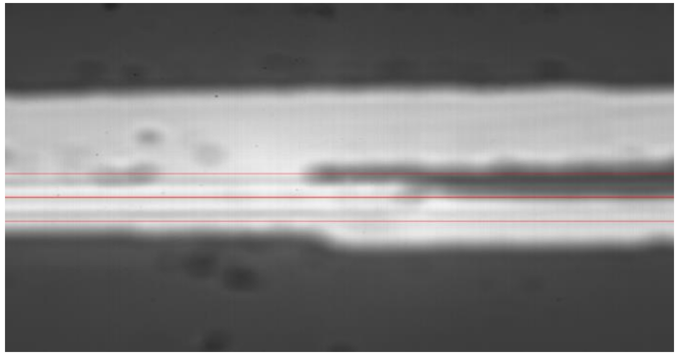
2. Point-by-point inscription of FBGs in carbon- and metal-coated glass fibers

Microscope image of fibre core

Photo of fibre with coating

Polymer-coated fiber | Carbon-coated fiber | Copper-coated fiber

- FBGs can be inscribed in polymer- and carbon-coated fibers without preparation
- Carbon-coated fibers absorb much more light, finding the core can be challenging
- Carbon coating may be damaged during inscription
- FBGs cannot be inscribed directly in metal-coated fibers, coating removal (etching) is required



Damage of carbon coating after FBG-inscription

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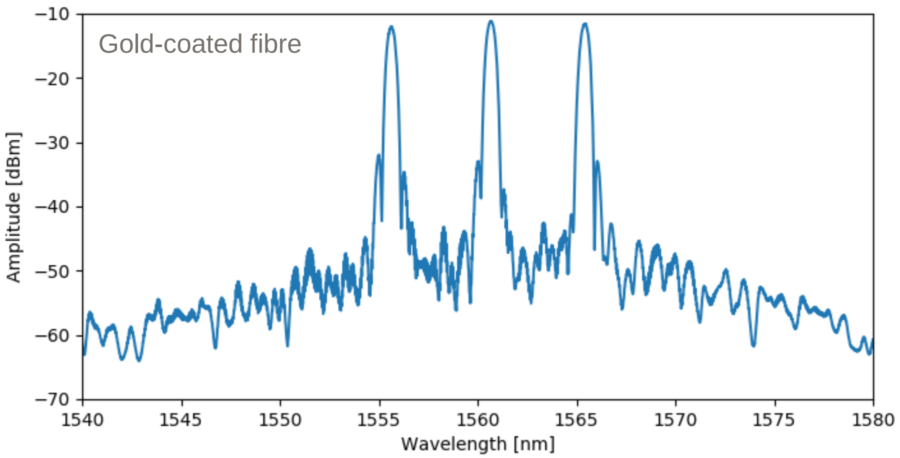
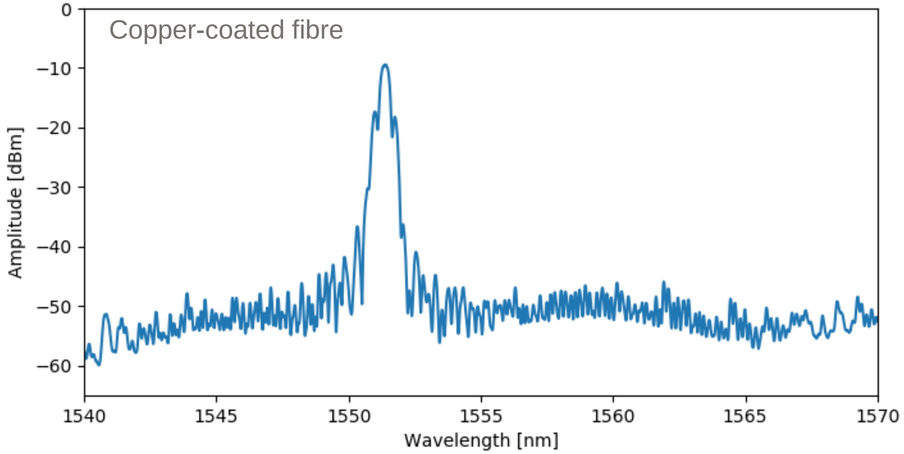
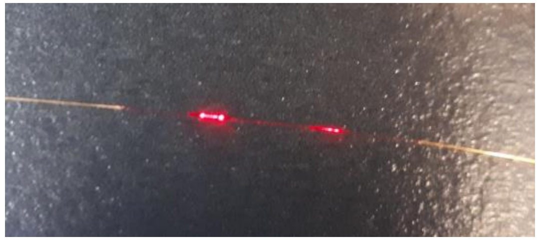
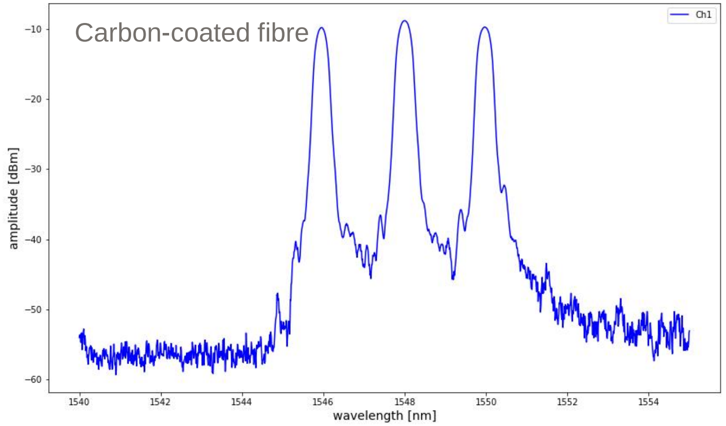
2. Point-by-point inscription of FBGs in carbon- and metal-coated glass fibers

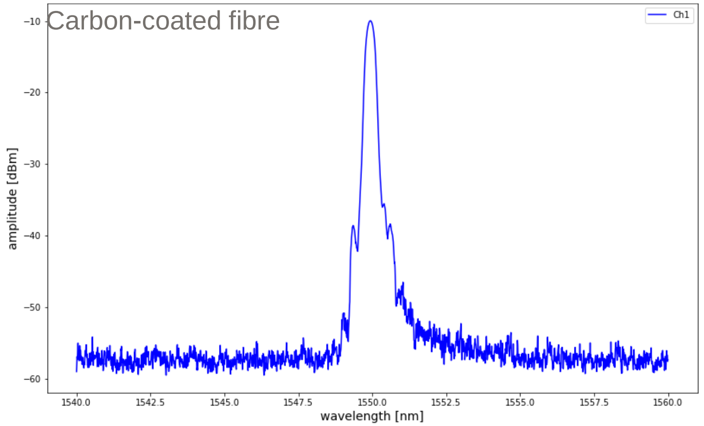




- FBG inscription in carbon-coated fibers is working well
- Inscription in metal-coated fibers is more difficult and time consuming
- Results show both coating materials are suitable for sensing applications







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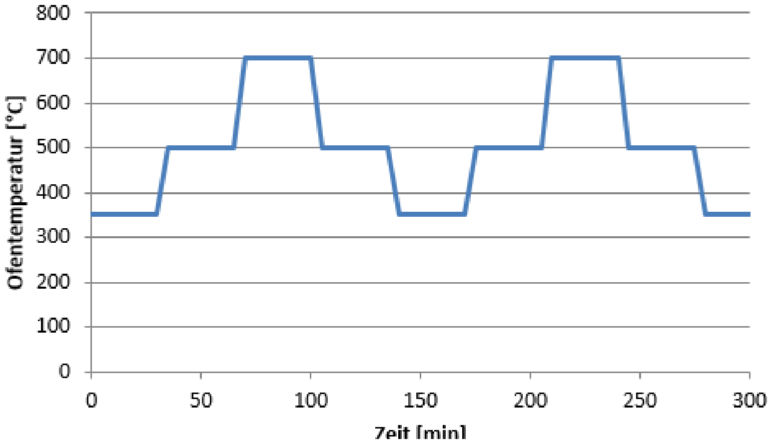
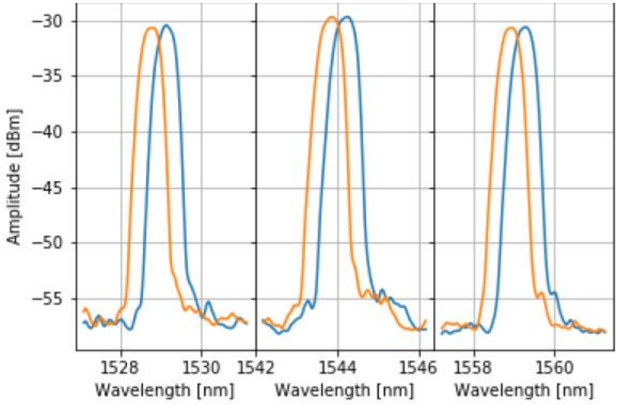
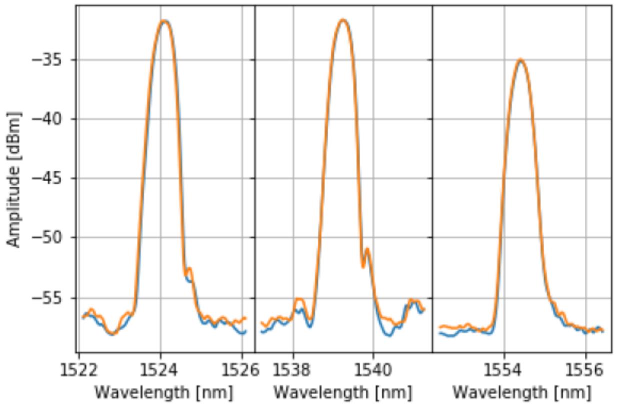
Sensor characterisation

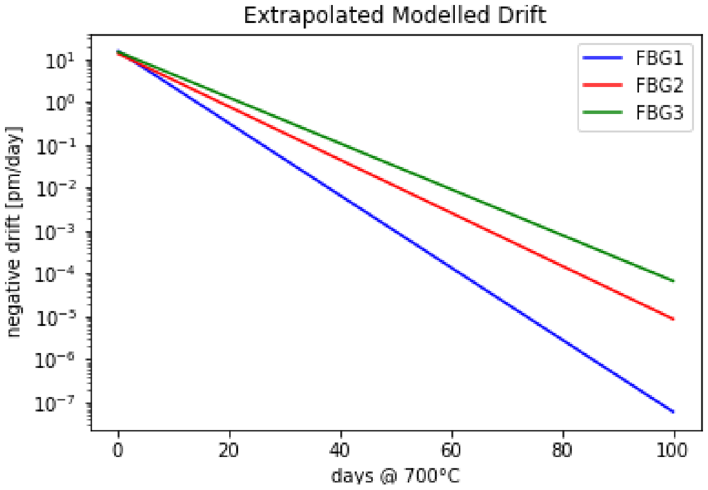
- ✓ FBGs are tested and optimised under high temperature conditions
- ✓ Measurements show a good thermal stability at high temperatures
- ✓ Biggest issue still not resolved: Drift at high temperatures that depends on:
  - fibre type
  - Inscription and annealing conditions

-> Can potentially be modelled to compensate drift









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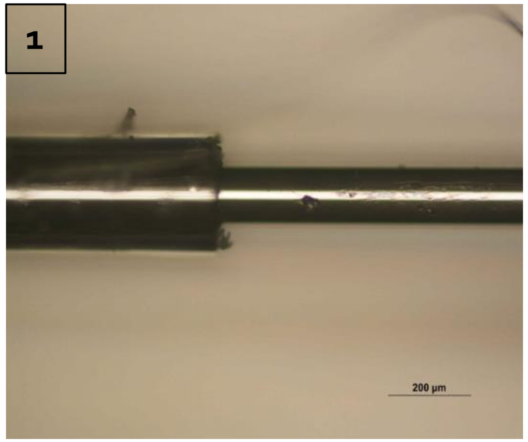
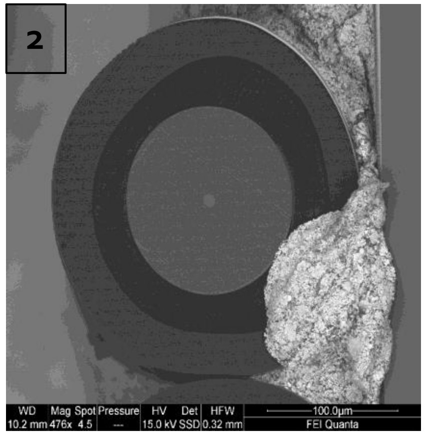
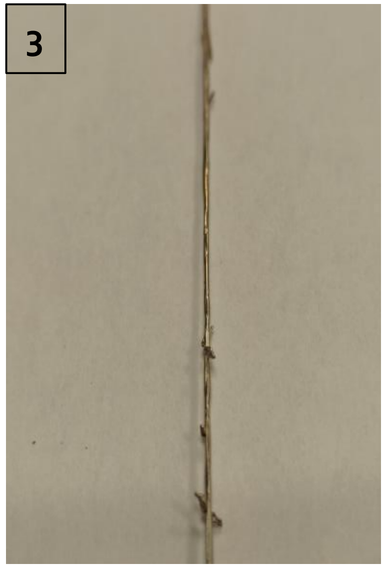
3. Coating with metal

The commercial C-coated optical fiber has a coating layer of a few nm of thickness protected by a polymer. The polymer doesn’t resist T > 350°C, and the C-coating layer is not resistant enough in an industrial environment (very fragile). A metallic coating is needed to protect the (hermetic) C-coating layer. Ni is (a-priori) the best metal to re-coat optical fibers.







Images of commercial C-coated optical fibres.

1. fibre with and without polymer coating.
2. SEM picture of cross section.
3. Carbon fiber with Ni-coating.

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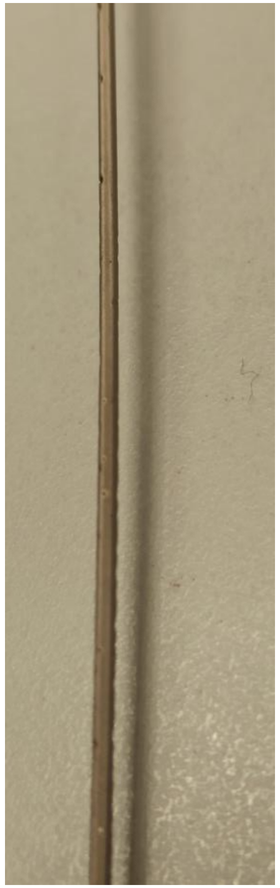
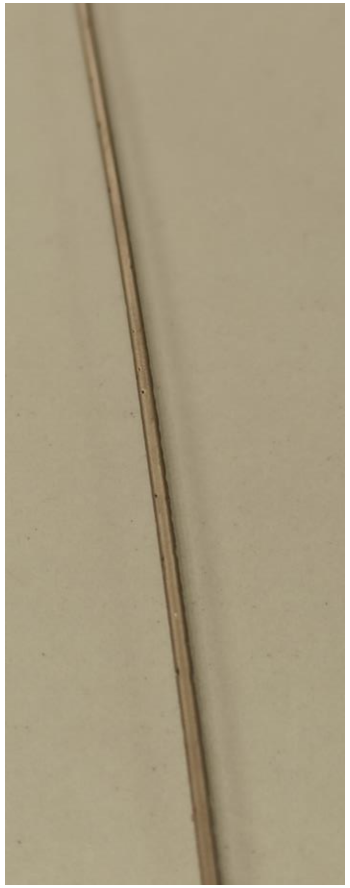
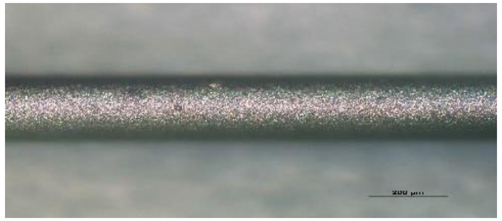
3. Coating with metal

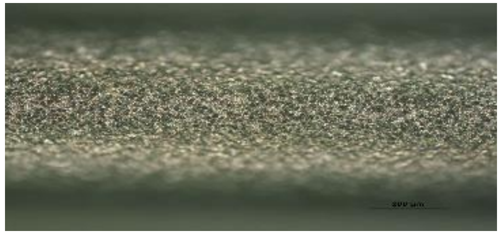
Metallic coating

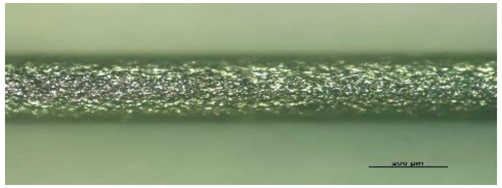
- A metallic nickel coating is applied to the FBG sensors using the electrodeposition technique
- FBG sensors are coated with different thicknesses between 500 µm and 750 µm, to withstand high temperatures
- Pictures on the right show examples of FBG sensors and fiber optics metallized and some images taken with magnification of their surface finish











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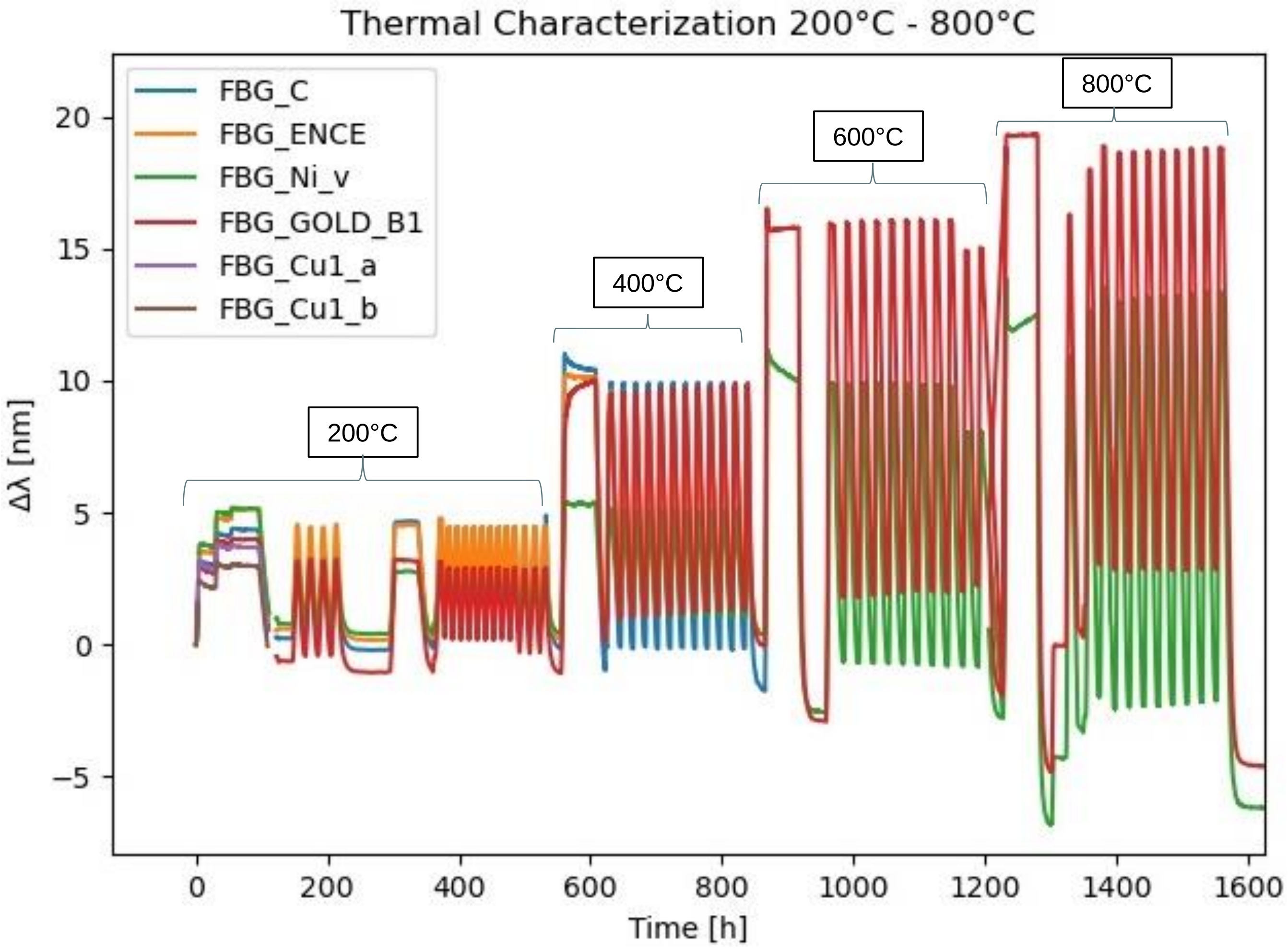
Sensor characterisation

Thermal fatigue tests

- An annealing process was applied above the cycle temperature and then cycled at the indicated temperature.
- Thermal test with the metal-coated FBGs were completed:
  - Fatigue tests at 200/ 400/ 600 / 800ºC
  - 66 days under thermal fatigue, 40 days under stable T
  - The thermocouple showed noise for T > 650ºC
- FBGs manufactured in Au-coated optical fibers as well as splicing standard FBGs to Cu-coated optical fibres showed more stable and reproducible responses

-> A new and improved batch of metallic coated FBGs will be thermally tested to compare results. New C-coated optical fibers will be tested.



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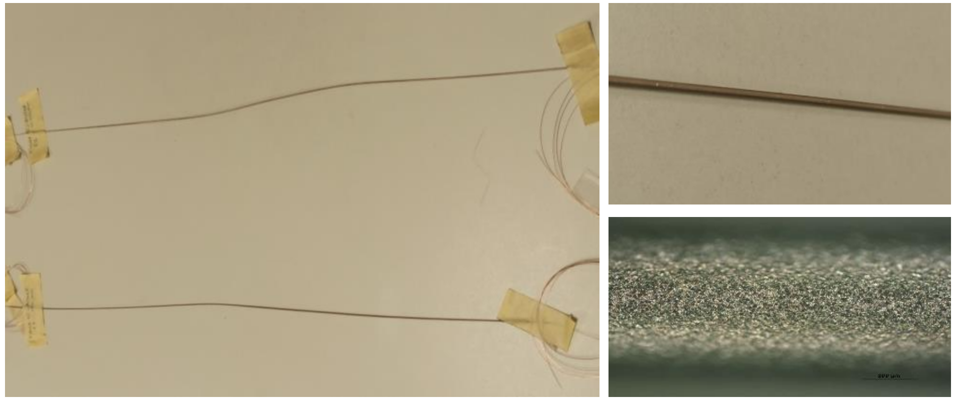
Outlook: 4. Sensor embedding

Concept

- Laser Additive Manufacturing (AM) of the metallic-coated FBG sensors
  - Embedding trials are in progress employing optical fibres with different Ni coating thicknesses



Some fibers with Ni-coating to be embedded by AM.

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