EFFECT OF MATERIALS HETEROGENEITIES ON MECHANICAL PROPERTIES AT INITIAL STATE

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- Introduction
- SOTERIA results
  - Microstructure & Chemical composition
  - Tensile
  - Impact
  - Fracture toughness – SEM initiation

SOTERIA

D3.1 Microstructural inhomogeneities of RPV steels and their impact on mechanical properties at initial state

Grant agreement number:	661913	Due date of Deliverable	31 August 2017
Start date of the project:	1 September 2015	Actual submission date	11 October 2017
Duration:	48 months	Deliverable approved by the WPL/CO	☒

Lead Beneficiary : CIEMAT
Contributing beneficiaries : CIEMAT, AR-G, HZDR, VTT, UJV, CNRS

SOTERIA

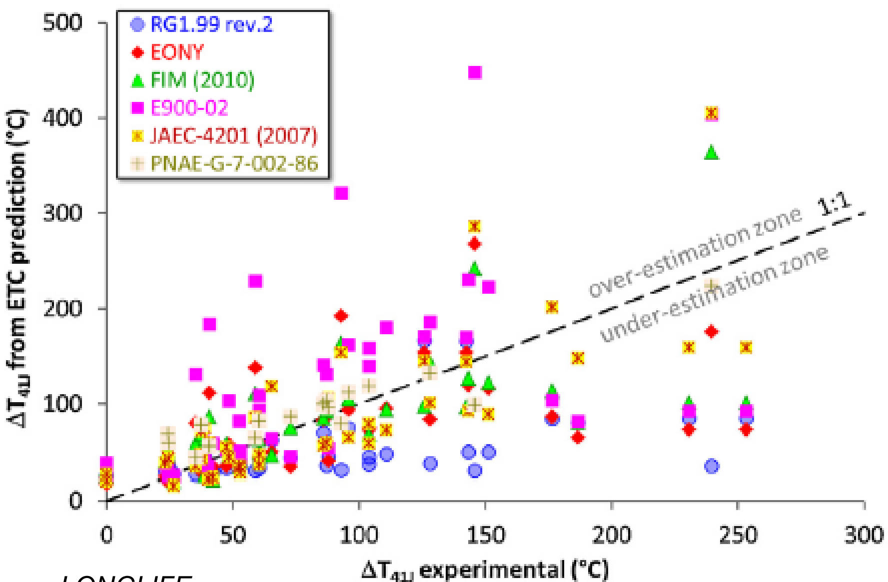
D3.4 – Assessment of uncertainties in RPV irradiation behaviour with respect to initial microstructure, material variability and other influencing factors

Grant agreement number:	661913	Due date of Deliverable:	[28 February 2019]
Start date of the project:	1 September 2015	Actual submission date:	[18 April 2019]
Duration:	48 months	Deliverable approved by the WPL/CO : ☒	

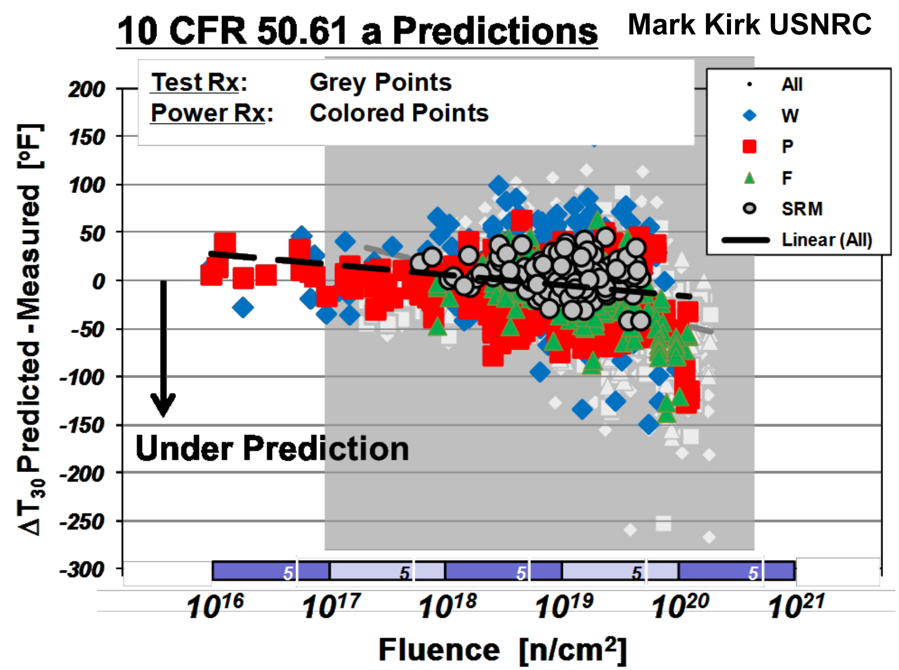
Lead beneficiary : AR-G
Contributing beneficiaries : CEA, EDF, CIEMAT, CNRS, HZDR, UJV, VTT

Introduction

- Existing ETC do are not accurate to predict embrittlement at high fluences and/or low Cu materials



LONGLIFE
E. Altstadt et al. / NED 278 (2014) 753–757



Introduction

- The scatter of the measured shift could come from
  - A inaccurate prediction of the ETC
  - Uncertainty of irradiation effects
  - Uncertainty on initial properties
- Included on RT_{NDT} shift as margin

$$ART = RT_{NDT(u)} + \Delta RT_{NDT} + Margin$$

$$Margin = 2\sqrt{\sigma_I^2 + \sigma_\Delta^2}$$

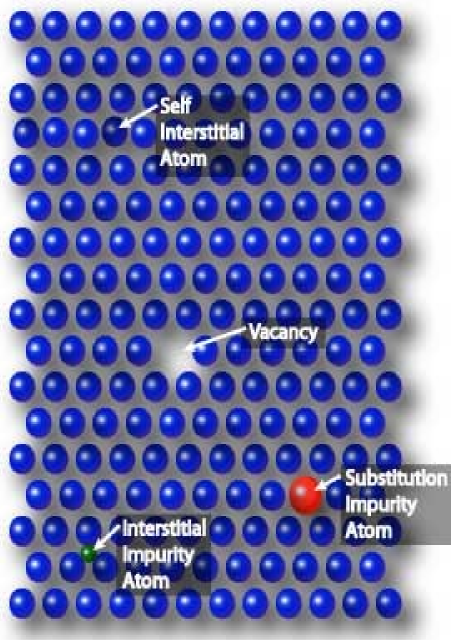
σ_I is the standard deviation for the initial RT_{NDT} (T_{41J})
σ_Δ is the standard deviation of ΔRT_{NDT}:
28ºF for welds
17ºF for base metal

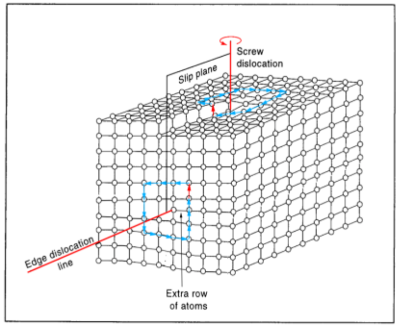
Unnecessary conservatism can be reduced when the accuracy of the mean condition is increased and the contributions to the total margin are properly understood

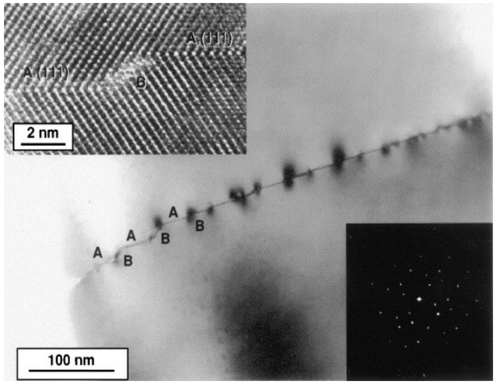
This presentation is focused on the **initial state**

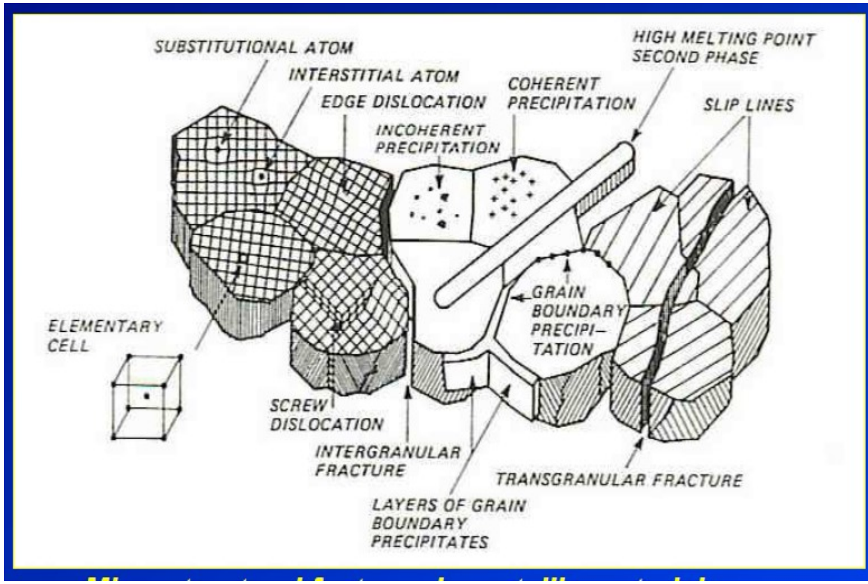
Introduction

- Most engineering materials are inherently inhomogeneous in their processing, internal structure, properties, and performance.
- Their properties are therefore statistical rather than deterministic
- These inhomogeneities manifest across multiple length





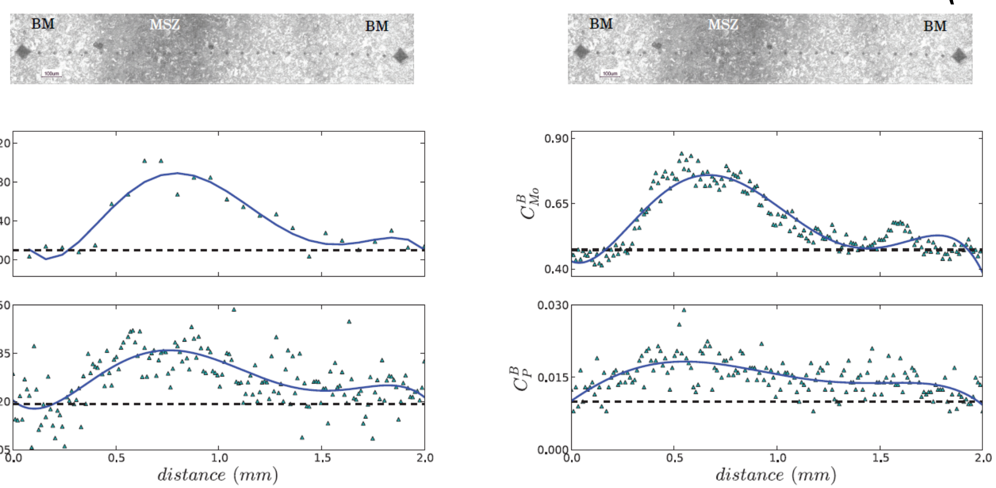


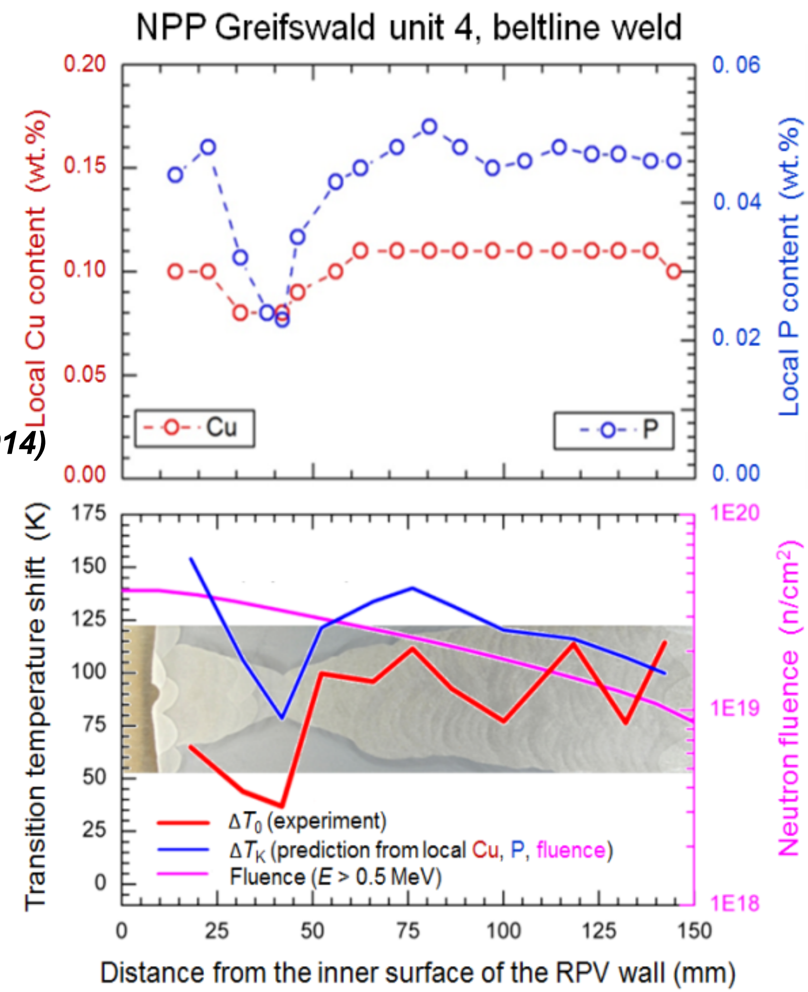


Introduction

- Inhomogeneity usually comes from the fabrication procedure
  - Casting - Forging
  - Welding
  - Surface finishing
  - Final heat treatment

Antoine Andrieu et al. / Procedia Materials Science 3 (2014)

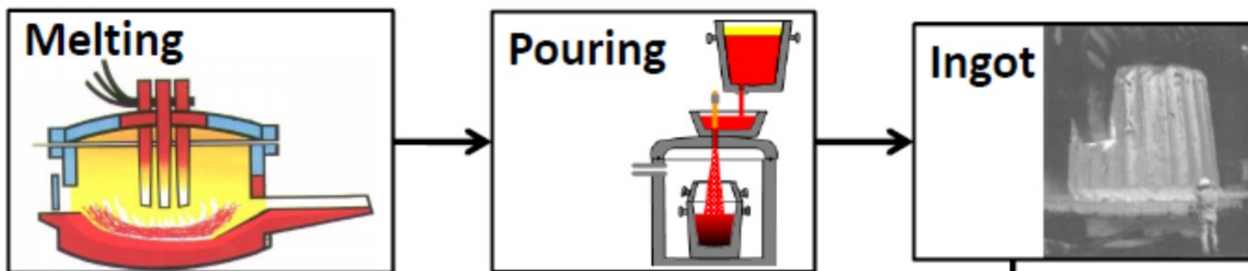


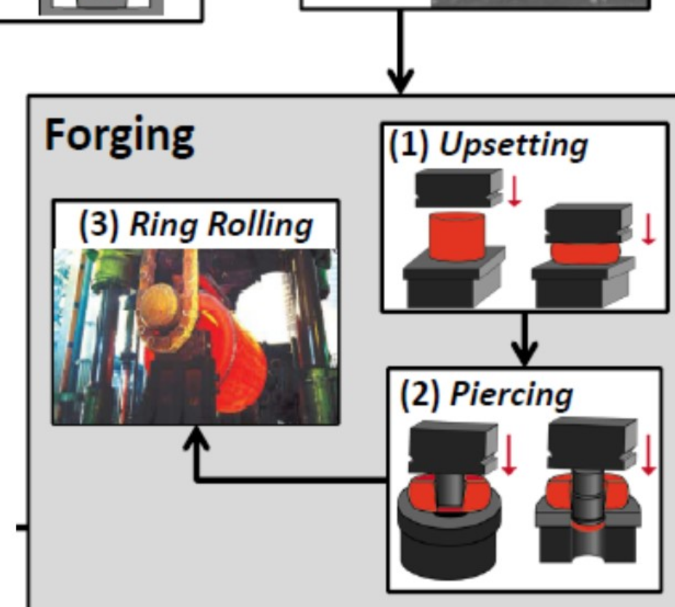


H.-W. Viehrig (HZDR) LONGLIFE

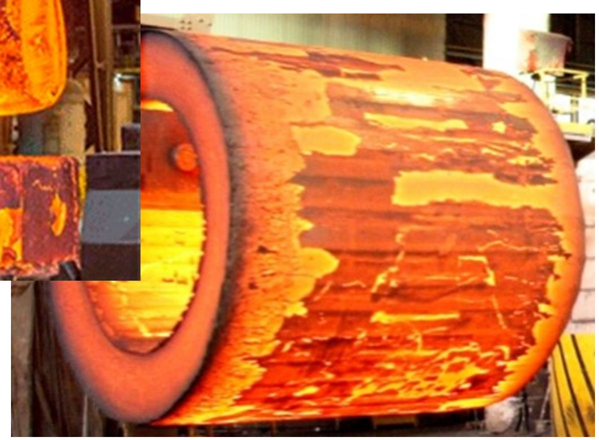
Forgings

- Forgings
  - Origin of scatter comes from the fabrication procedure



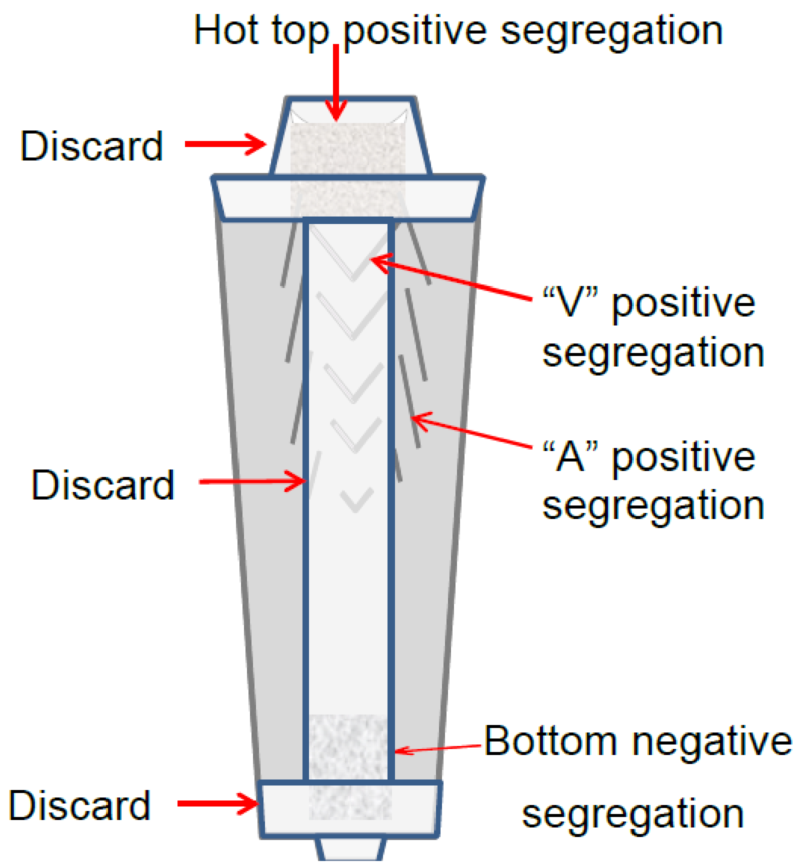


Creusot-Forge



Forgings

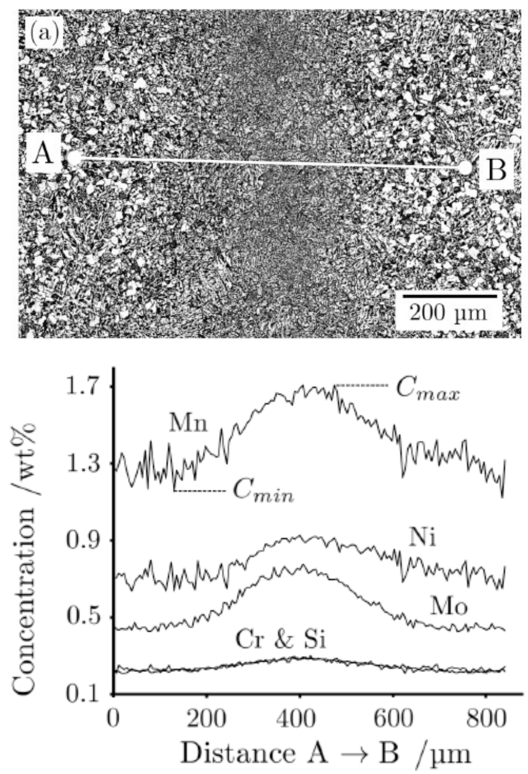
- If a ring is to be forged, then trepan forging is used to remove a core of material from the center of the forging; this process removes most of the "V" macrosegregation that is present in the center of the forging.
- Some of the "A" macrosegregation then will remain and extend along the length of the forging both at the surface and embedded in the wall. The portions of the ingot that are discarded and the carbon macrosegregation that is removed by the cropping and trepan forging operations
- If a head is to be forged, then the centre is not trepanned, and the "A" and "V" segregates not removed by cropping the top of the ingot will remain in the forged component.



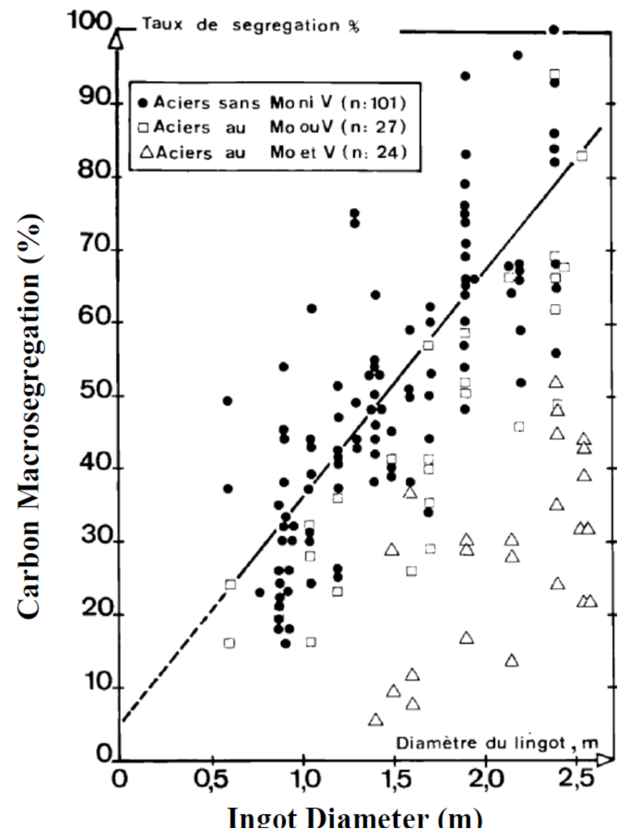
EPRI MRP 414

Forgings

- ❑ Carbon segregation
  - Typically, the nominal carbon content in the large forgings used in nuclear reactors ranges from 0.15 wt.% to 0.2 wt.%, while areas of positive carbon segregation can have 0.25 wt.% or more

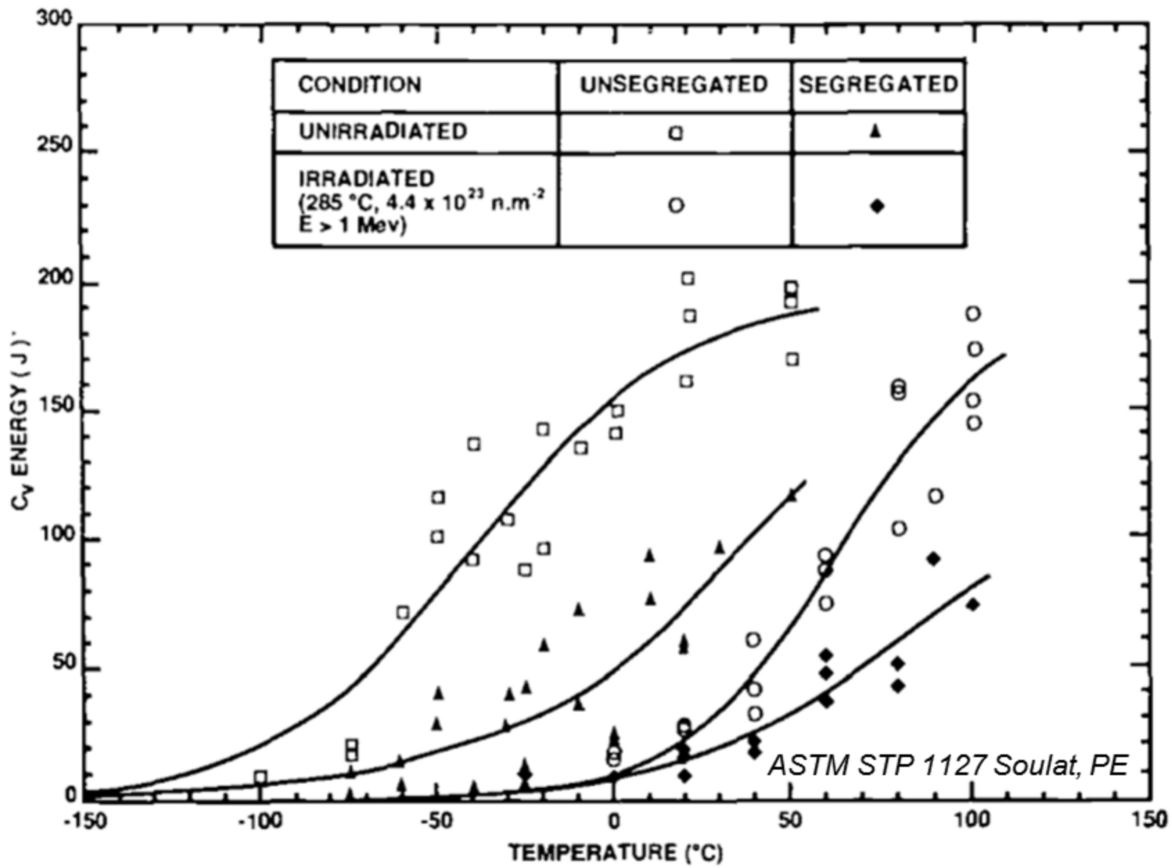


E.J. Pickering



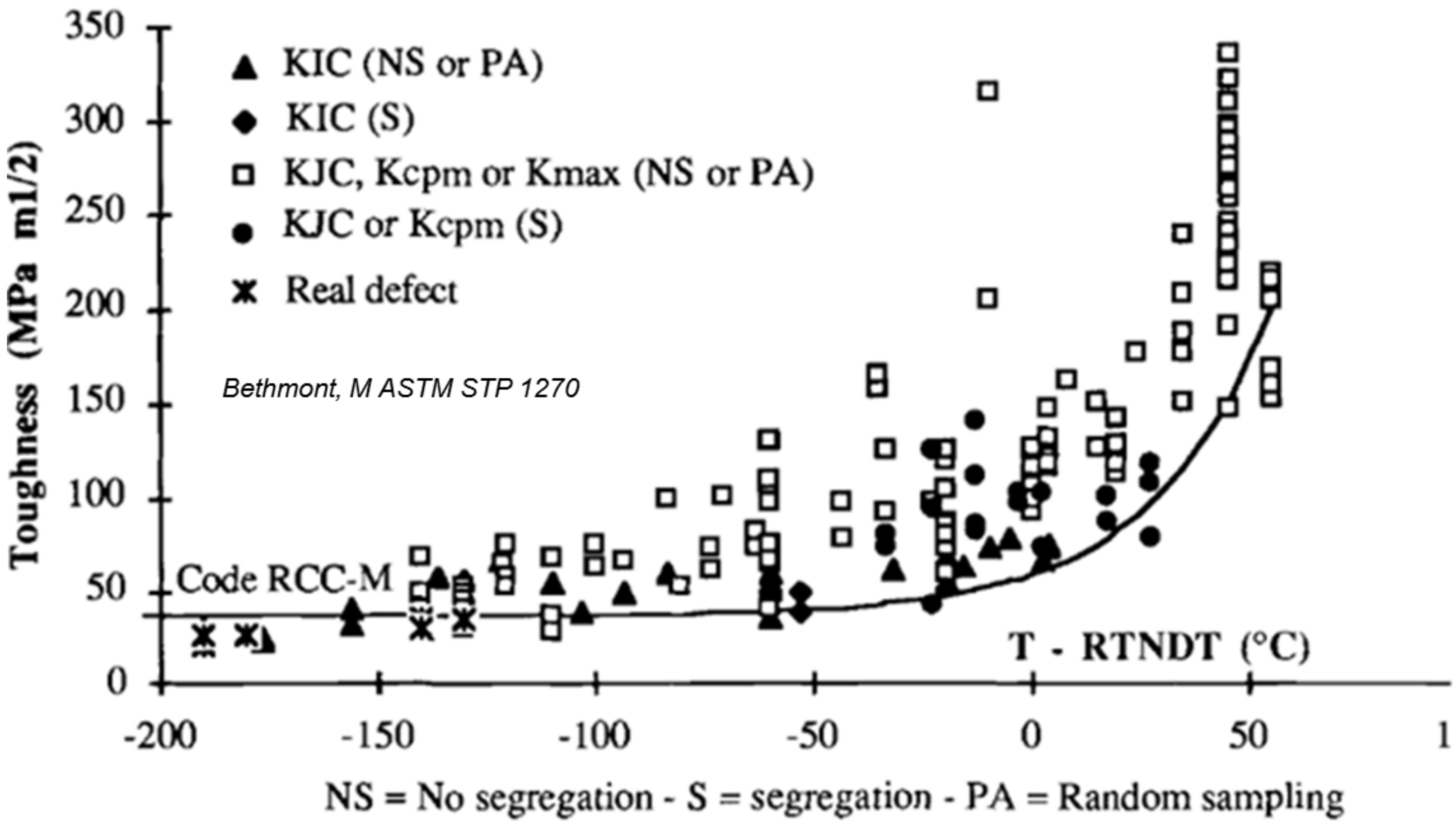
Forgings

- CVN energy (CV) plotted against test temperate for RPV material with and without regions of positive channel segregation
- The data exhibits a 75°C shift in the unirradiated RT_{NDT}, at the 68J energy level, for the segregated material.



Forgings

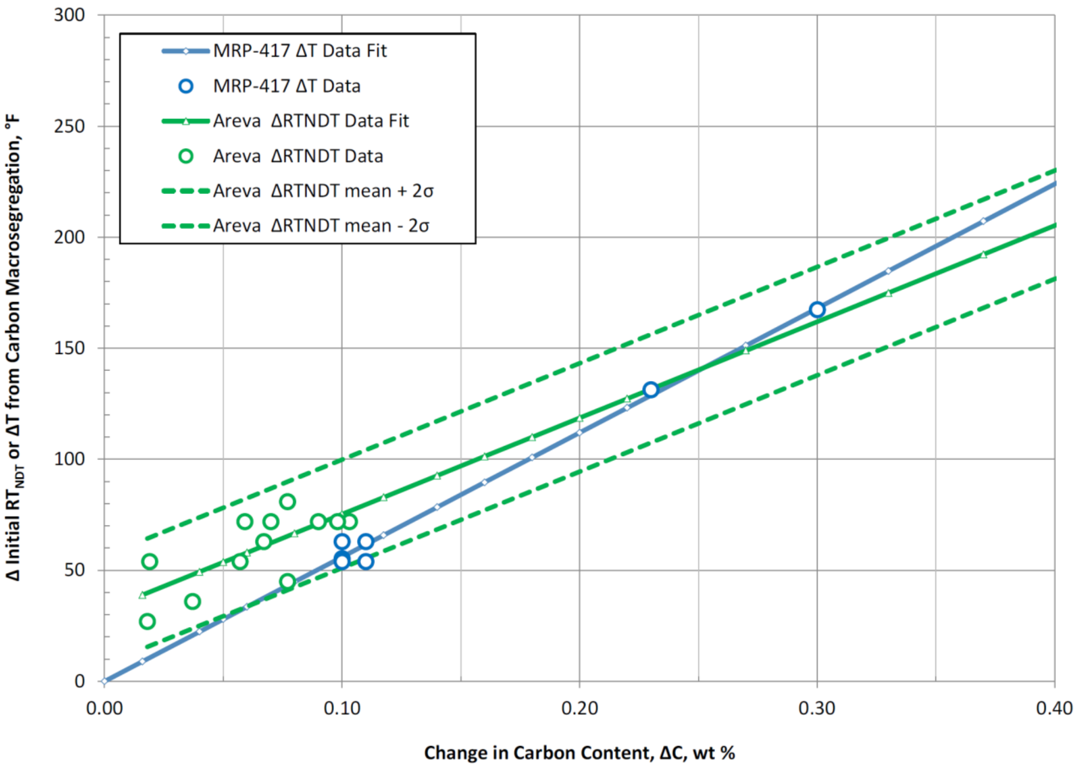
- Fracture toughness plotted against the indexed temperature for RPV steel with and without regions of positive channel segregation



Carbon macrosegregation

- EPRI assumed the RT_{NDT} changes per the following expression (MRP-471)
- $RT_{NDT(U)} = RT_{NDT(U0)} + \Delta RTNDT(U)$
  - $RT_{NDT(Uo)}$) is the unirradiated reference temperature for the material with carbon content equal to the nominal value in the ingot, Co.
  - ΔRTNDT(U) is the change in $RT_{NDT(U)}$) as a function the change in carbon content, °F (°C)/wt. % C, and is determined from experimental data.

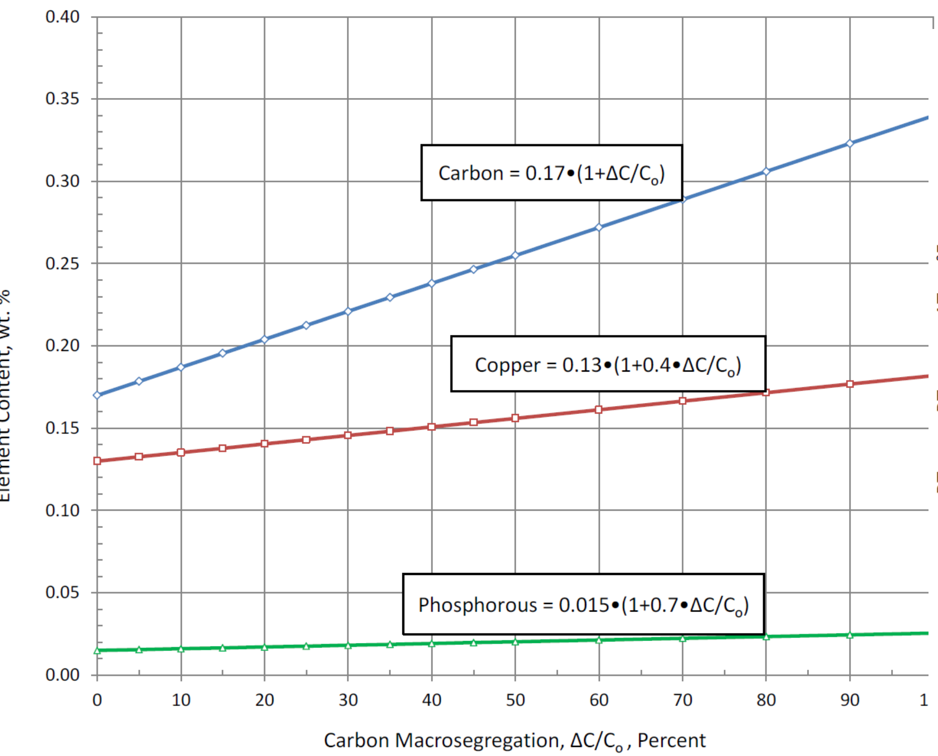
$\Delta RT_{NDT(U)}$ (°C) = 241.01 (°C/wt. % C) • ΔC (wt. %) + 17.83 (°C).

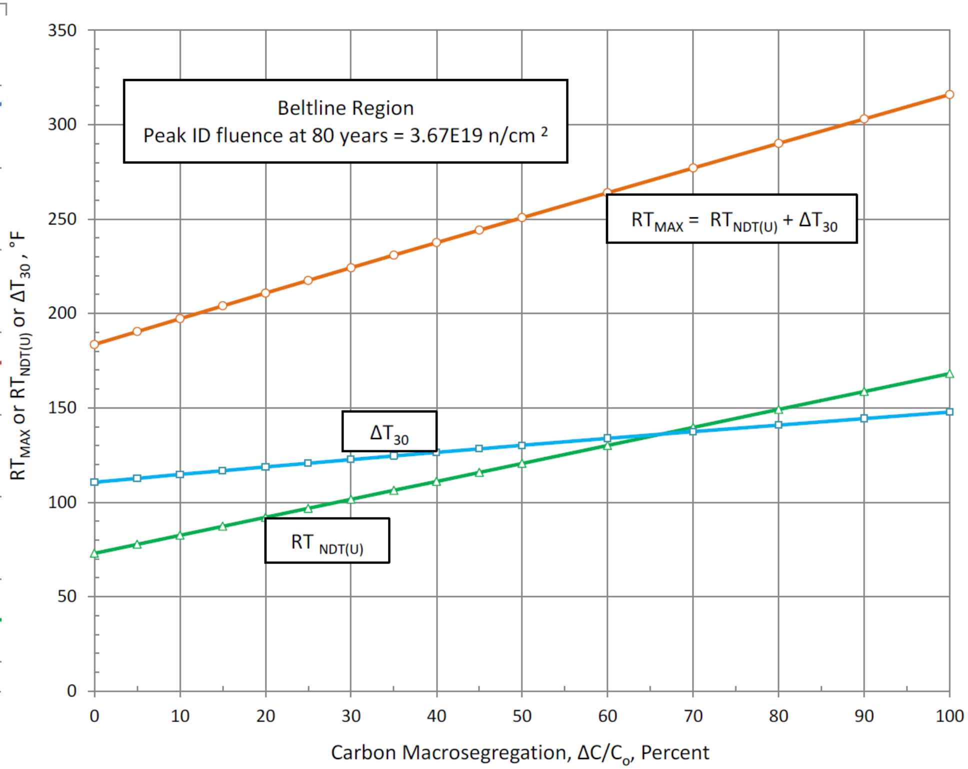


EPRI MRP-417

Carbon macrosegregartion

- When carbon macrosegregation occurs in large forgings it is accompanied by copper and phosphorus macrosegregation with increased **copper** and phosphorous





EPRI MRP-417

Non-Metallic inclusions

- UT indications found in 2015 in Beznau RPV Base material were identified as non-metallic aluminium oxide inclusions.
  - Fracture mechanics testing on specimen from replica ring C did not show an influence of these inclusions.
  - Fractographic analyses never showed crack initiation at Al_2O_3-Inclusions.
  - Inclusions do not have a negative effect on fracture toughness.

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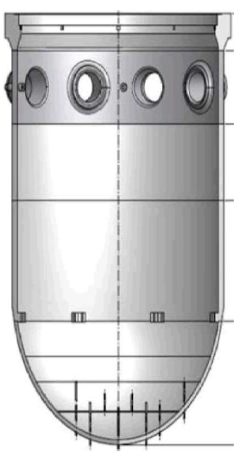
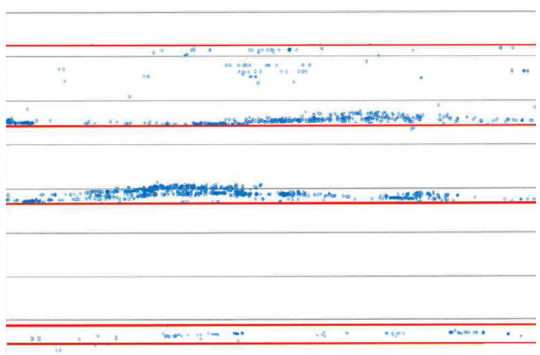
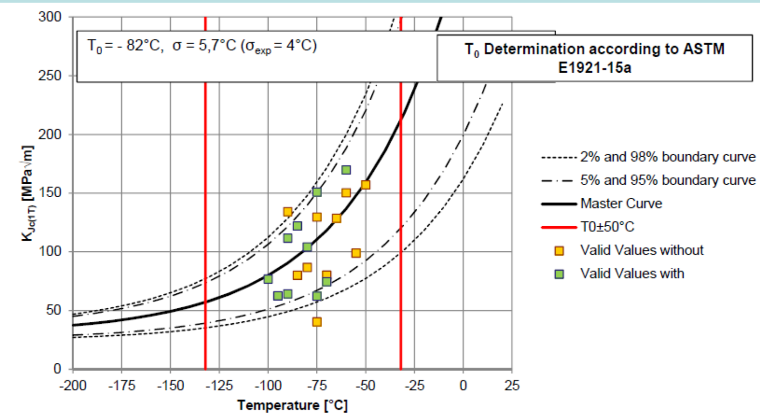
KKB 1, UT Indications in 2015



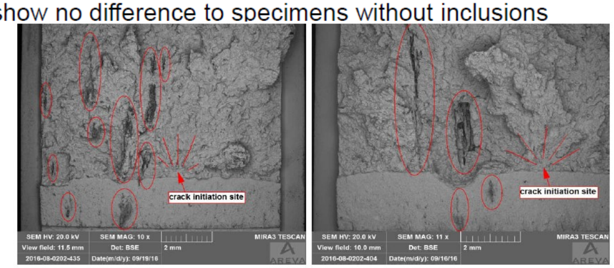


- Indications identified as Al2O3-Inclusions
- Not Ageing, manufacturing problem (insufficient discard rate from ingot)

KKB 1, Master Curve by 1 CT-Specimen from Replica



Master Curve Testing of the replica shell C material: Specimens with inclusions show no difference to specimens without inclusions



Fracture surface (SEM) of broken CT-specimens, replica C material
Result: No influence of Al_2O_3 inclusions on crack initiation

ENSI Review of the Axpo Power AG Safety Case for the Reactor Pressure Vessel of the Beznau NPP Unit 1

Uncertainties Non Irradiated

Relevant SOTERIA Results

- Materials investigated:
  - Forgings (A508 Class 3, 16MND5, 15Kh2NMFAA, 15Kh2MFAA, 22NiMoCr3-7)
  - Welds , weld metal S3NiMo1/OP41TT
  - Plates (JRQ)
- Inhomogeneities in terms of composition and microstructure
- Inhomogeneity in terms of mechanical properties
  - Tensile properties
  - Impact properties
  - Fracture toughness
- Fractographic analysis

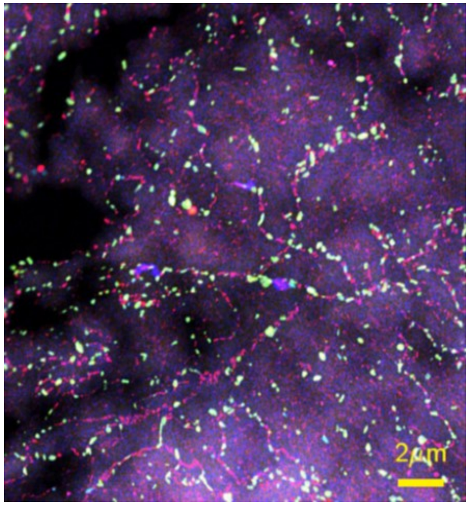
SOTERIA RESULTS

- Microstructural examination of FZD-4 (NPP Greifswald, Unit 8 15Kh2MFAA) samples: SEM/EBSD, STEM-HAADF, STEM-EDX

Difficult to include on embrittlement prediction

FZD-4 BM 15Kh2MFAA

Type of precipitates	Mean size (nm)	Number density (cm^3)
V-rich	140	0.38E+14
V-rich (small)	39	0.96E+14
V-rich (very small)	17	2.74E+14
Cr-rich	200	0.10E+14
Carbides	230	0.07E+14
Mo-rich	~500	4.53E+05



HELMHOLTZ ZENTRUM DRESDEN ROSSENDORF

STEM-EDX elemental mapping of FZD-4

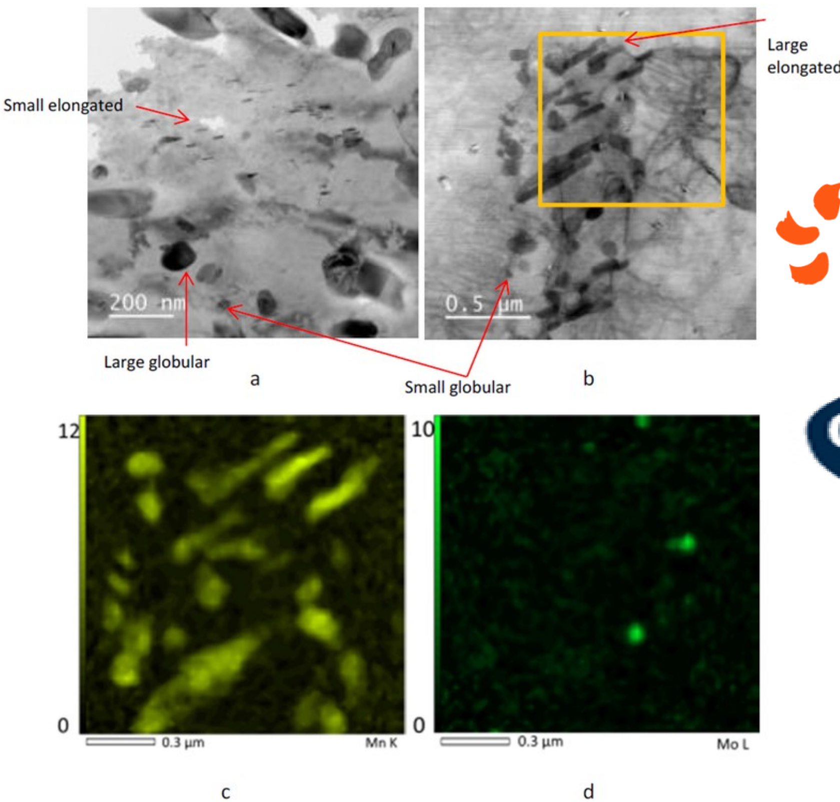
red: V (Kα)
green: Cr (Kα)
blue: Mo (Lα)

There are indications of inhomogeneity at different length scales. Phosphorous tends to segregate at grain boundaries. Some kinds of precipitates are also preferentially located at grain boundaries.

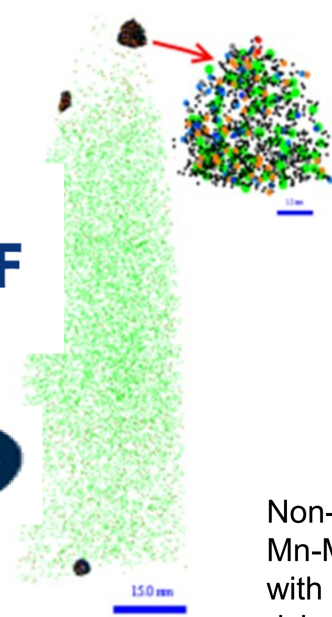
SOTERIA RESULTS

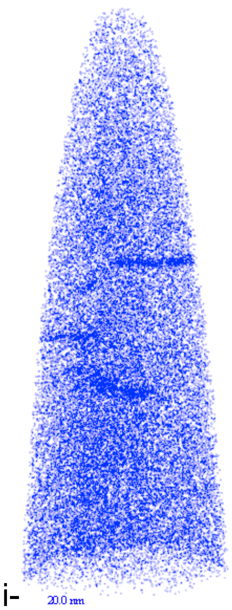
- After inspection using conventional light microscopy, it was established, that 16MND5 samples do not contain characteristic ghost lines, but only micro- and mesoscopic segregations

The distribution of precipitates (carbides) is not homogenous



Formation of Mo, Mn, C - rich clusters was clearly observed on the dislocations in the non-segregated material.





Non-segregated area Mn-Mo-Cr-C carbides with corresponding Si-Ni-rich precipitates

EDF4 samples do not contain characteristic ghost lines, but only micro- and mesoscopic segregations

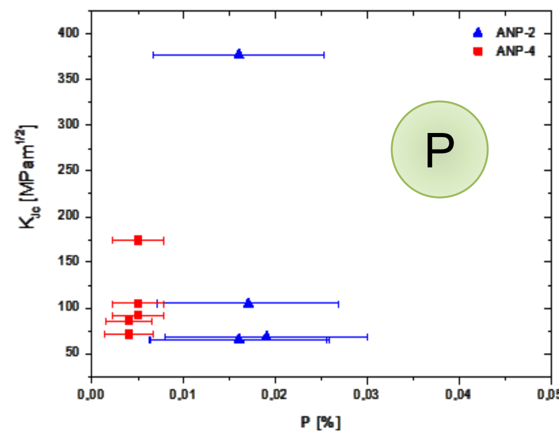
SOTERIA RESULTS

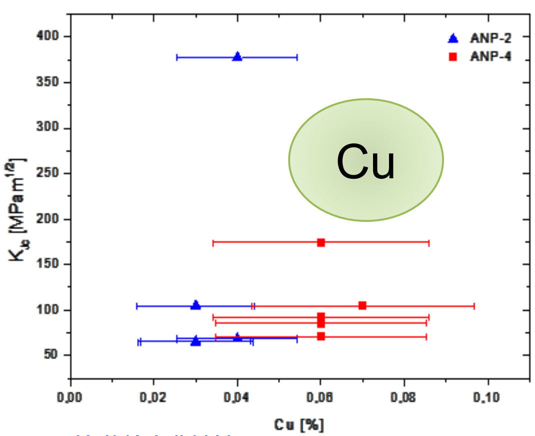
- Based on the chemical analyses performed on unirradiated ANP-2 and ANP-4 materials, using OES method a non-negligible uncertainty in weight % was found for some chemical elements, such as C, P, S, Cu and Ni (in high Ni weld) playing an important role in aging mechanisms of RPV steels.
- Fracture toughness show a dependence on the chemical composition, mainly Mn, Mo, Cr, C and P content

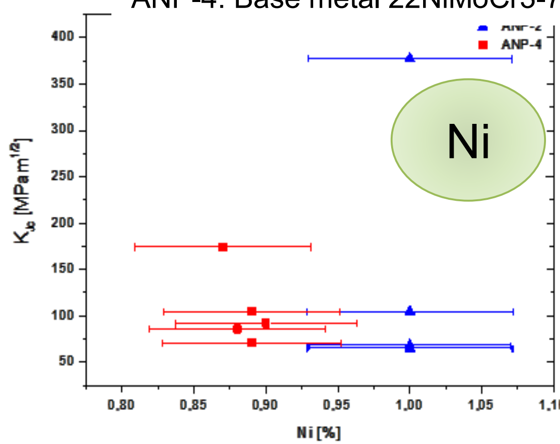
framatome

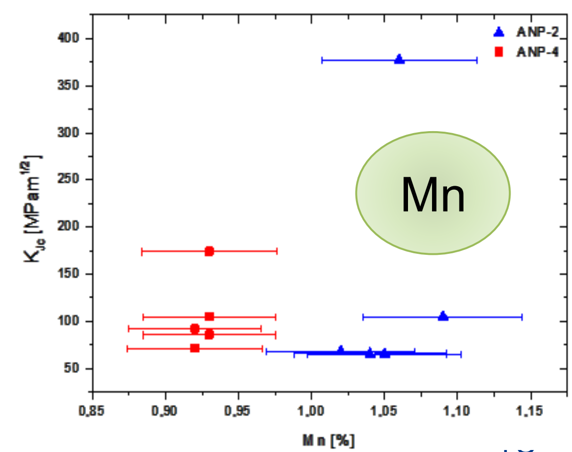
Material	Specimen designation	$K_{JC(1T)}$ in MPa√m	T in °C	Cu	Ni	P	Mn	Mo	Cr	C
				in %						
ANP-2	CACS2	65,1	-20	46	7,2	61	5	9,9	8,2	14
	CACS3	68,3	-30	36	7	58	5	10	8,5	13
	CACS4	65,4	-20	44	7,1	60	5	9,8	8,5	14
	CACS5	377,4	-10	36	7,1	58	5	10	8,5	13
	CACS6	104,8	-25	47	7,2	58	5	9,9	8,5	14
ANP-4	TL21	92	-100	43	7	56	4,9	10	8,9	14
	TL22	174,1	-110	43	7	56	5	9,9	9	13
	TL24	85,6	-100	42	7	62	4,9	9,9	9,1	14
	TL28	104,9	-115	38	6,9	56	4,9	9,9	8,9	13
	TL30	71,2	-120	42	7					

ANP-2: Weld metal S3NiMo1/OP41TT
ANP-4: Base metal 22NiMoCr3-7







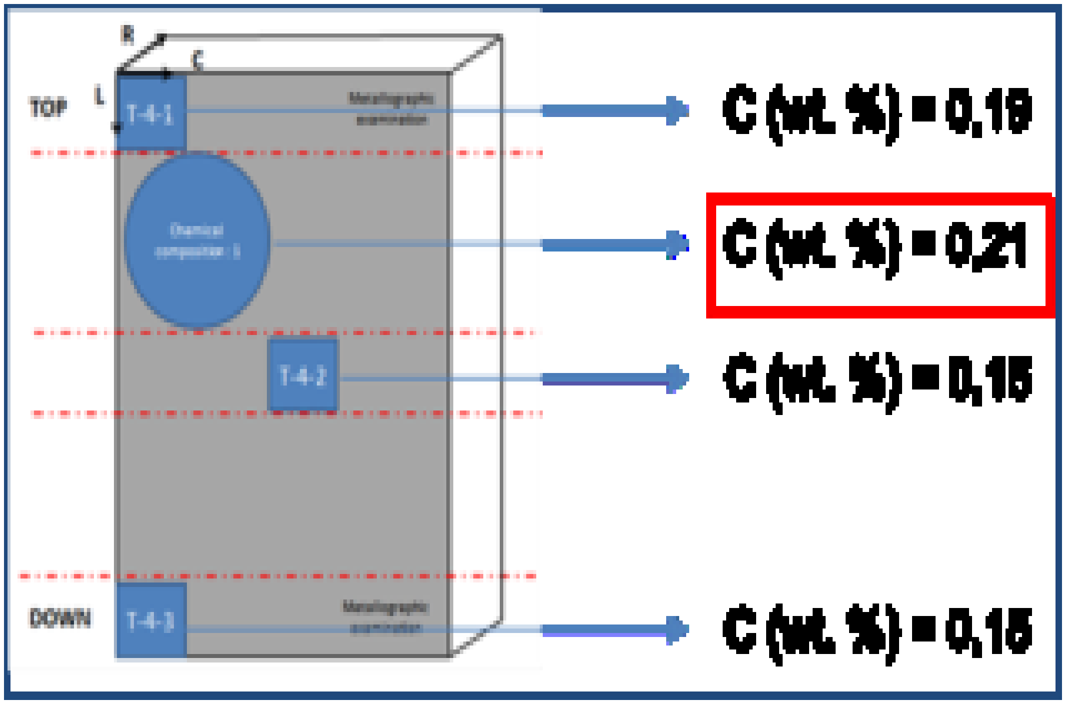


Uncertainty – Chemical composition

SOTERIA

- Carbon content on CIE-1 forging

Ciemat



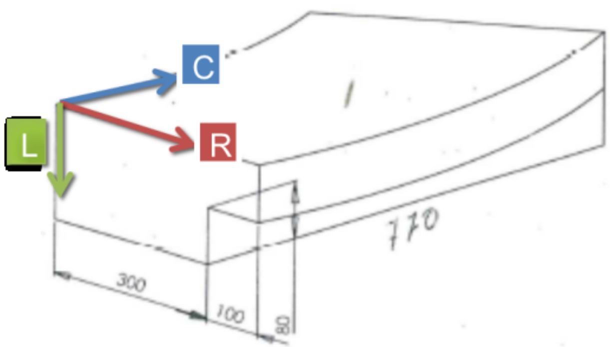
Difficult to include on embrittlement prediction

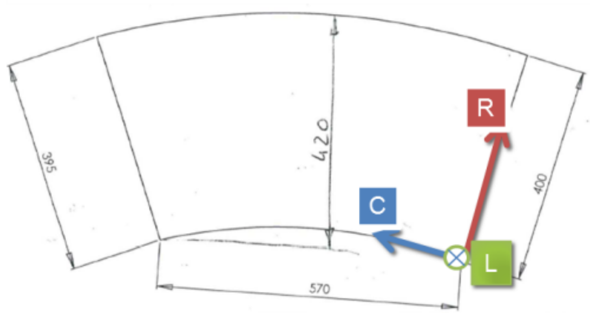
Uncertainty – Micro & Chemical composition

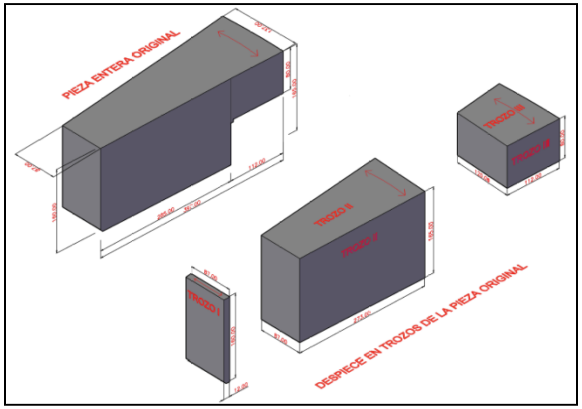
- WWER Forging
  - Microstructural inhomogeneities is observed at the nm, mm- and cm-length scales respect to both grain structure and precipitate distribution
- French forging
  - Micro- and mesoscopic segregations are observed but not characteristic ghost lines
  - The observed small differences in chemical composition between the non-segregated and segregated zones are due to poor enrichment of the interdendritic spaces by alloying elements during the solidification process.
- German forging & weld:
  - A non-negligible uncertainty in weight % was found for some chemical elements, such as C, P, S, Cu and Ni (in high Ni weld) playing an important role in aging mechanisms of RPV steels.
- A508 Class 3 forging:
  - Differences in carbon content

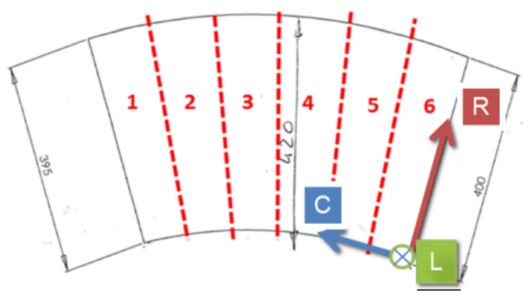
Uncertainties - Tensile

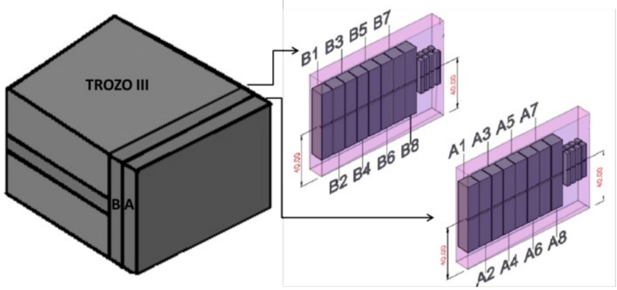
- CIE-1 (A508 Class 3): Specimen tracking in three orientations

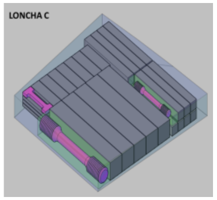








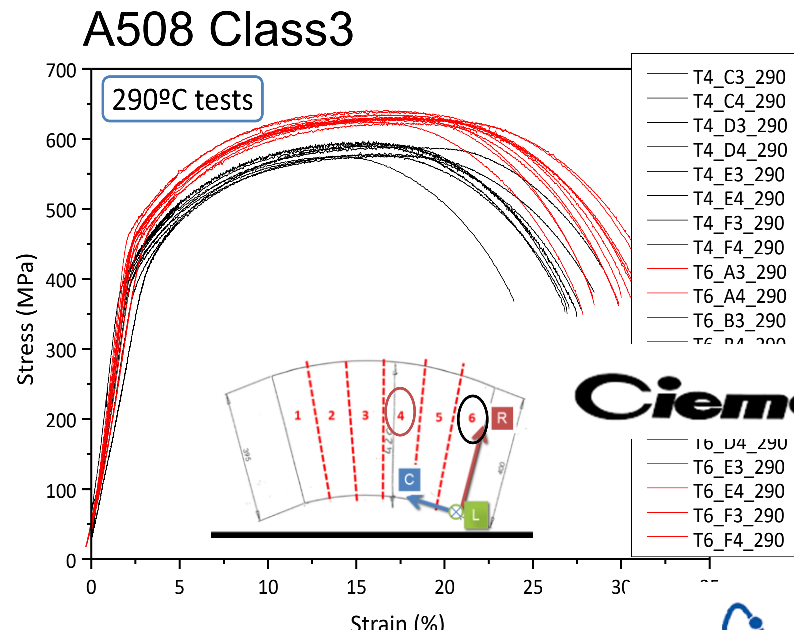


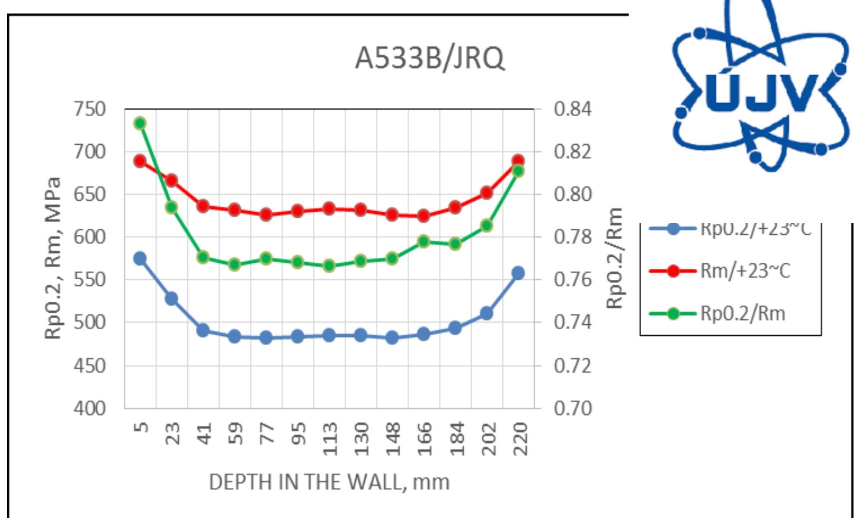


Uncertainties - Tensile

- Material tested at different locations

Material	Nº specimens	0.2% Yield Strength (MPa) [ASTM E8 error= 2.32%]		
		Average	SD	Error
JRQ RT	13	503	31.0	6%
UJV-2 RT	11	479	5.5	1%
CIE-1 T=-100ºC	7	563	25.0	4%
CIE-1 T=-50ºC	8	514	11.3	2%
CIE-1 T=0ºC	8	495	16.4	3%
CIE-1 RT	18	485	40.2	8%
CIE-1 290	20	434	26.5	6%
FZD-4 T=100ºC	3	511	5	1%
FZD-4 RT	8	534	5.0	1%
FZD-4 T=-15ºC	3	546	1.5	0%
FZD-4 T=-40ºC	3	562	5.9	1%
FZD-4 T=-65ºC	3	598	27.9	5%
FZD-4 T=-90ºC	3	611	23.1	4%
FZD-4 T=-100ºC	3	632	0.5	0%
FZD-4 T=-115ºC	3	685	1.5	0%
FZD-4 T=-140ºC	3	757	38.7	5%



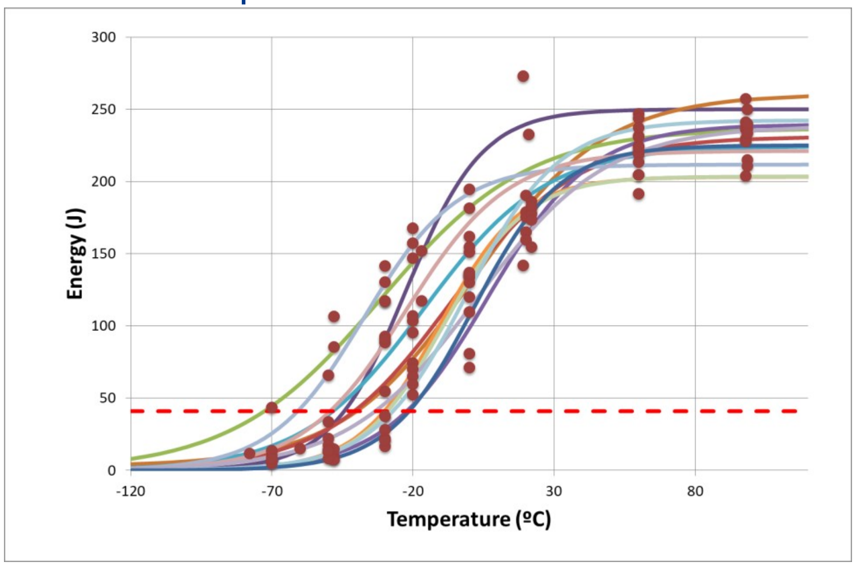


Uncertainties - Tensile

- The tensile properties are dependent on the circumferential location as observed for tensile strength in a A508C3 forging (CIE-1).
- A533B1 plate
  - Tensile properties (yield strength, Rp0.2, ultimate tensile strength, Rm, and fracture stress, Ru) may depend on the RPV wall depth as observed for a A533B1 material (UJV-1) with practically constant properties in the inner half of the thickness whereas tensile properties are increasing toward the surface.
  - On the contrary, plasticity properties (elongation, A and reduction of area, Z) are practically constant through the whole thickness.
- 15Kh2NMFAA forging
  - Tensile properties practically do not depend on the depth in the wall, only a slight tendency of decreasing tensile properties from one surface to the other can be observed – this effect could be connected with the different quenching rates on both cylindrical ring surfaces.
  - No effect of specimen location was also observed for FZD-4
- The error of the tensile results for some conditions are larger than the ones indicated in ASTM E8 standard -> depends on number of speciemns tested

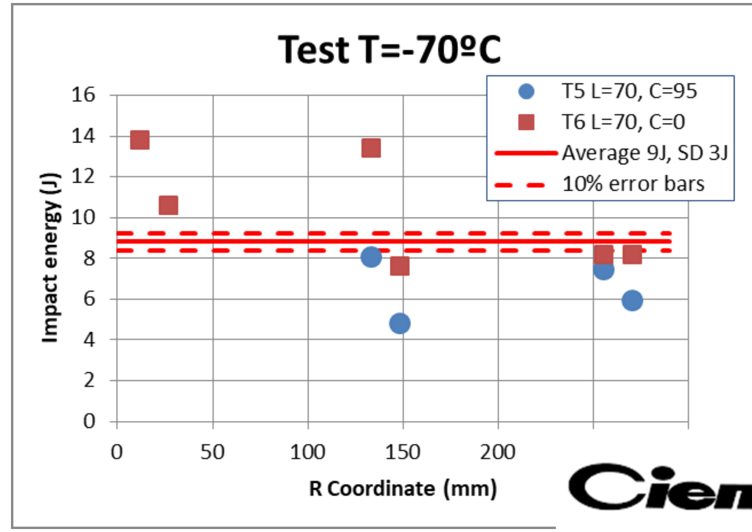
Uncertainty impact

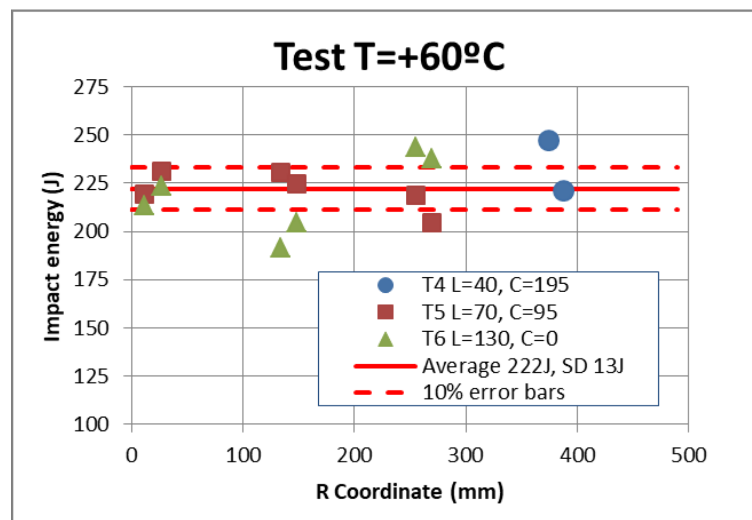
- CIE-1 data
  - Statistical analysis per testing temperature



	T41J	USE
Average	-41	230
SD	14.44	16.64
Error	35%	7%

Larger than precision of ASTM E23



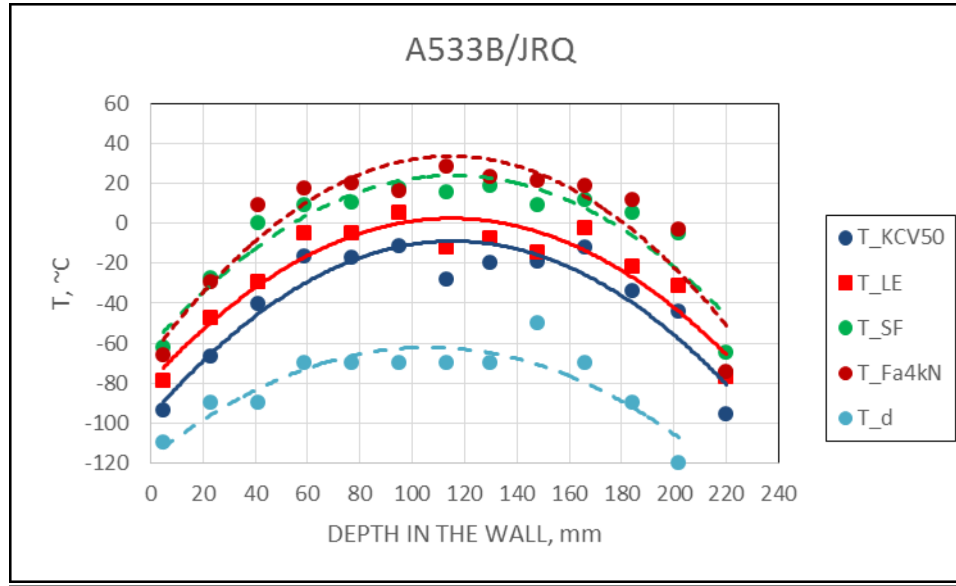


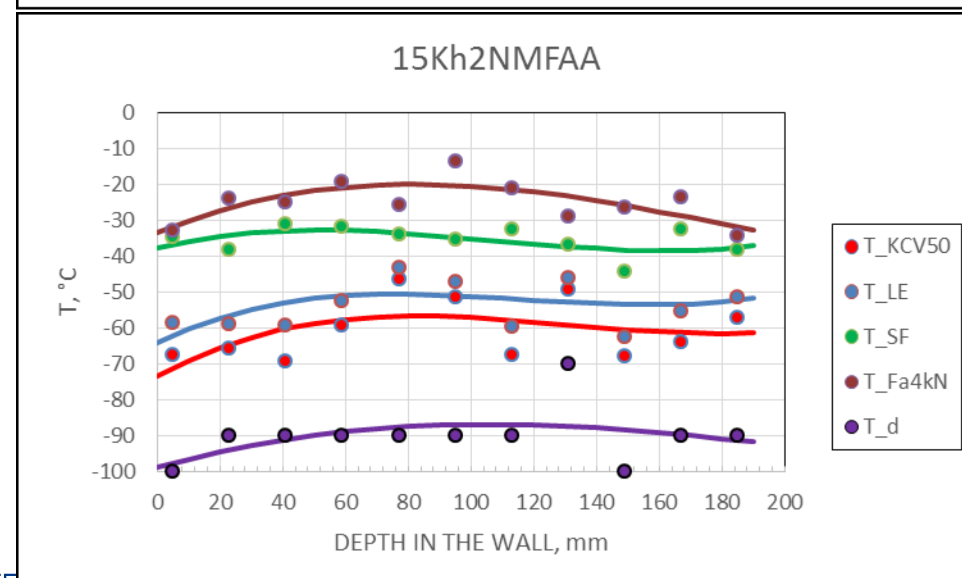
Uncertainty impact

- UJV data

JRQ	TT_KCV50
Average	-38.44
SD	29.36
Error	76%

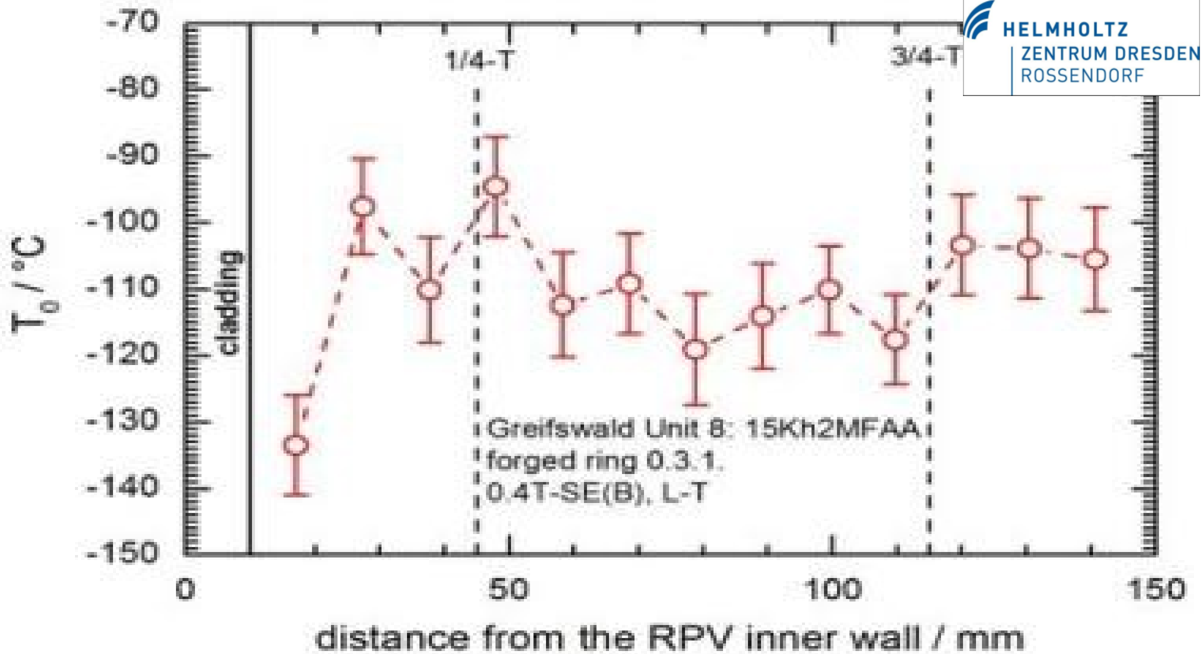
15Kh2NMFAA	TT_KCV50
Average	-60.50
SD	8.19
Error	14%





Uncertainty – T0

- HZDR – FZD-4 BM 15Kh2MFAA
  - T0 standard deviation larger to T0 uncertainty E1921 (aprox. 7.8°C)
  - ASTM E1921 of specimens located between ¼ to ¾ **- SD = 4.5°C**



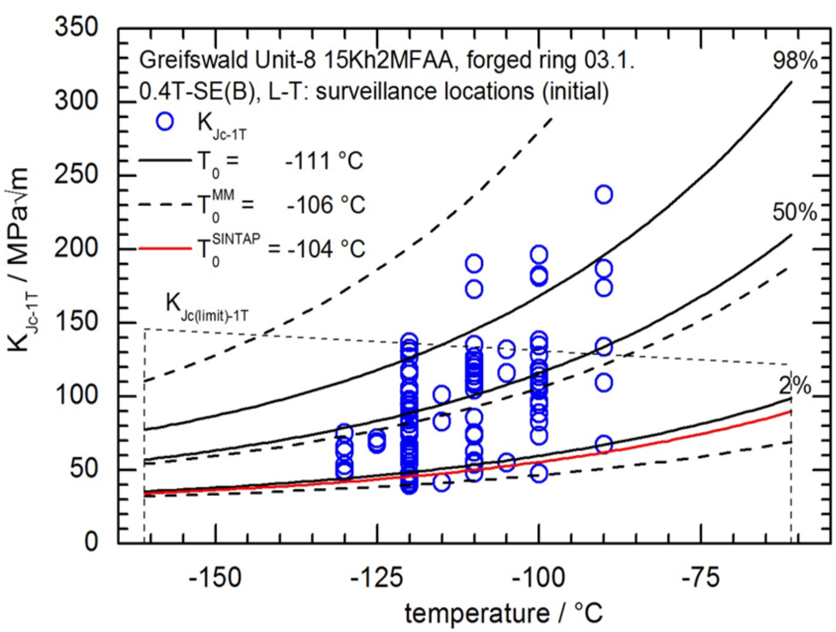
FDZ-4

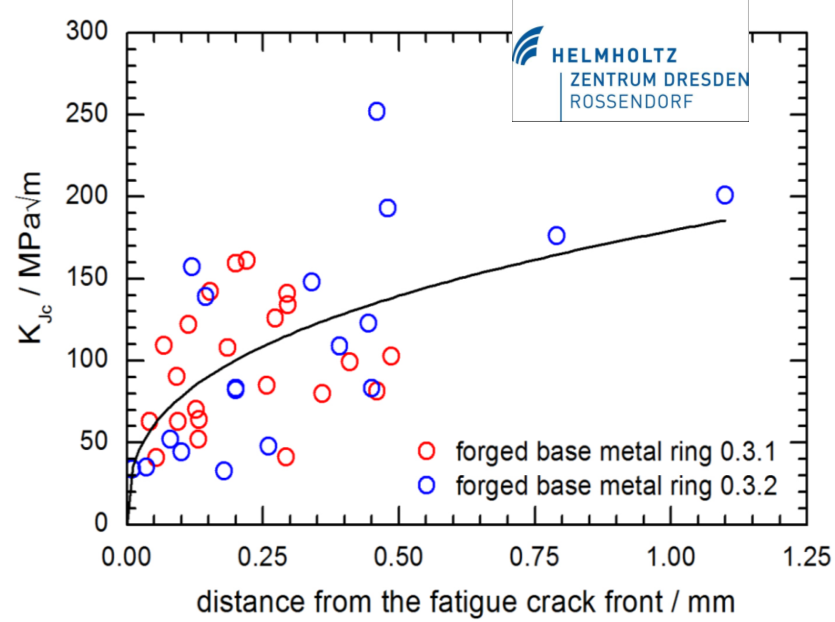
Average	-110.31
SD	**10.17**
Error	9%

Uncertainty – T0

- There is a strong indication of random inhomogeneity, for samples mechanized between ¼ and ¾ thickness
- The fractographs display intergranular areas below and above the marked fatigue crack front -> No clear initiators
- Although IGF was found on the fracture surface, it can be assumed that the Weibull weakest link mechanism is governing failure mechanism and that the MC can be applied to this material .

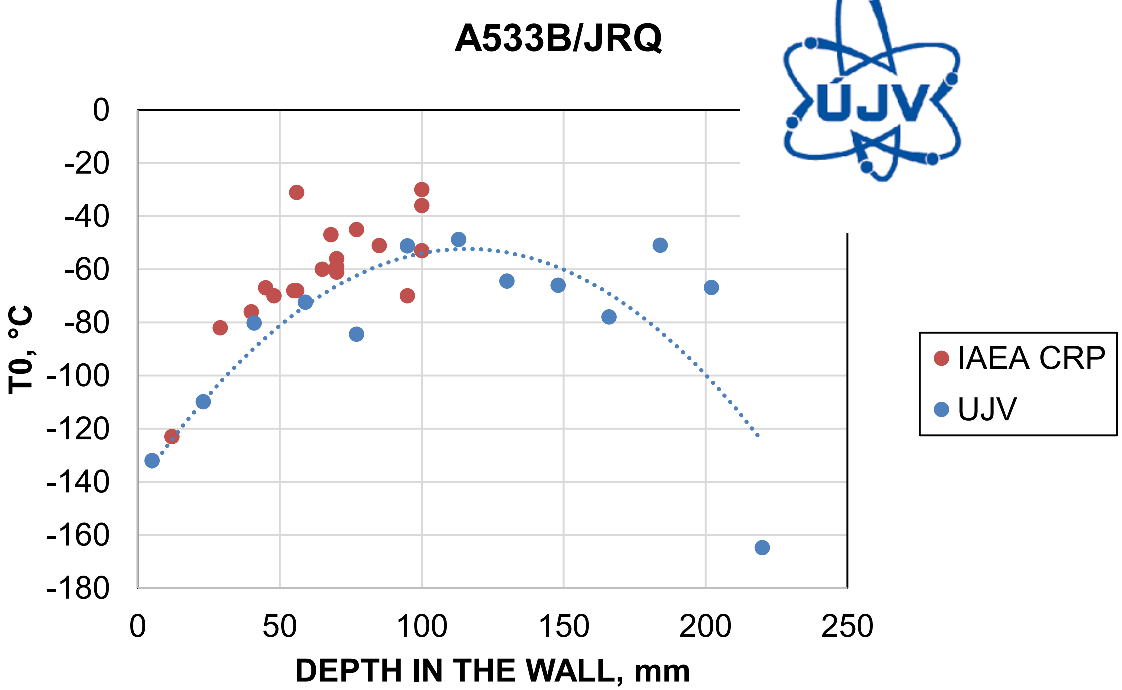
FZD-4 BM 15Kh2MFAA





Uncertainty – T0

- UJV - JRQ
  - T0 standard deviation larger to T0 uncertainty E1921 (aprox. 7.8°C)



JRQ

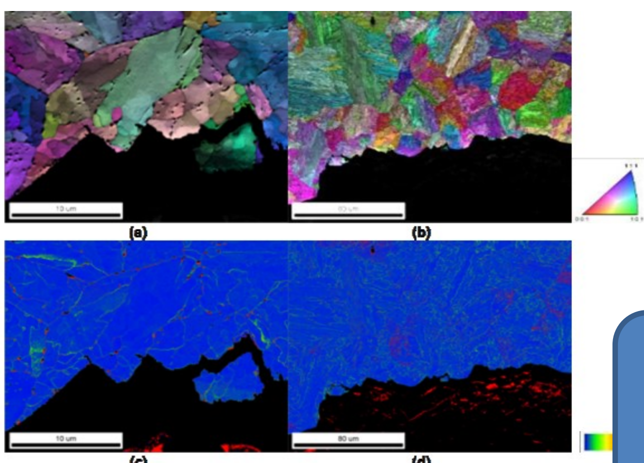
Average	-82.28
SD	**34.23**
Error	42%

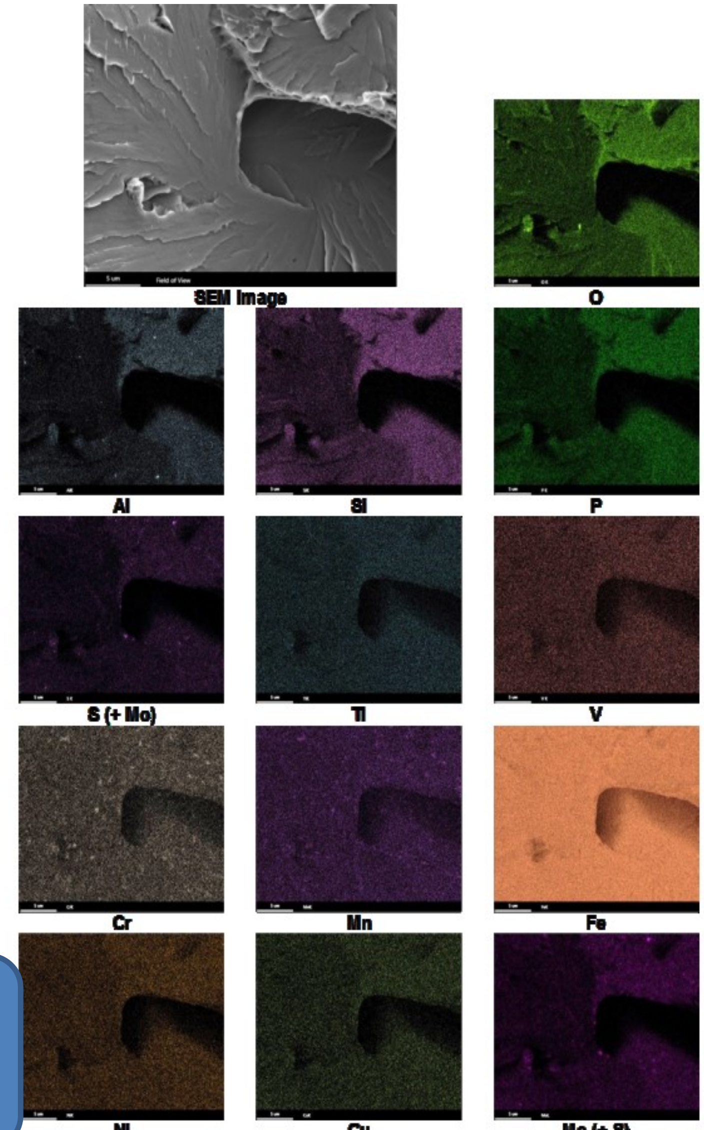
Fracture initiation sites

❑ 22NiMoCr3-7 PWR forged

- Low Cu/P/Ni base materials show similar microstructural features in the initiation site and the bulk, even if slightly higher contents of Cr, Mn, Ni or Mo in the bulk may occur.
- The primary initiation site is not characterized by a specific microstructural feature, such a precipitate or inclusion, at which brittle fracture initiated

Cross section



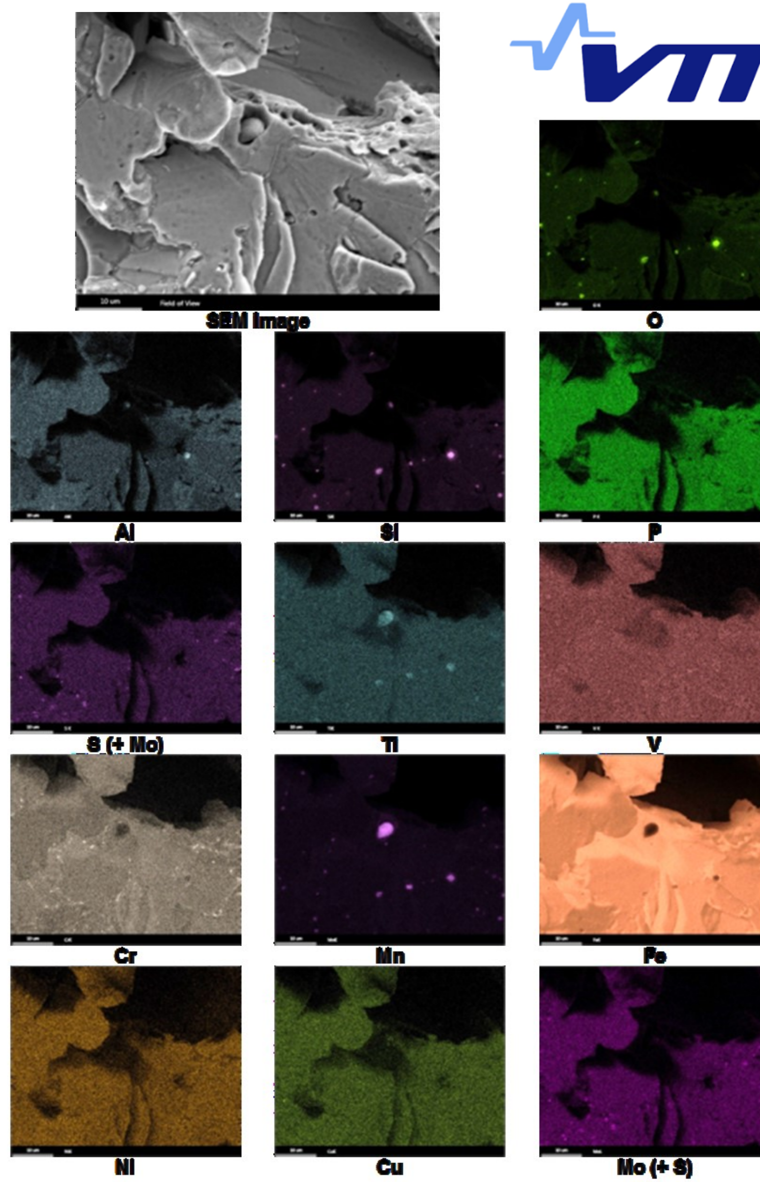


Two halves has to be examined

Fracture initiation sites

❑ VTT-1 weld

- Specific microstructural feature such a precipitate or inclusion, at which brittle fracture initiated, was not observed.
- Al, Si, Mo, S, Ti and Mn [oxide] particles, Mn and S(+Mo) rich [oxide] particle and Cr-rich particles decorating the material near the fracture surface and primary initiation site can exist



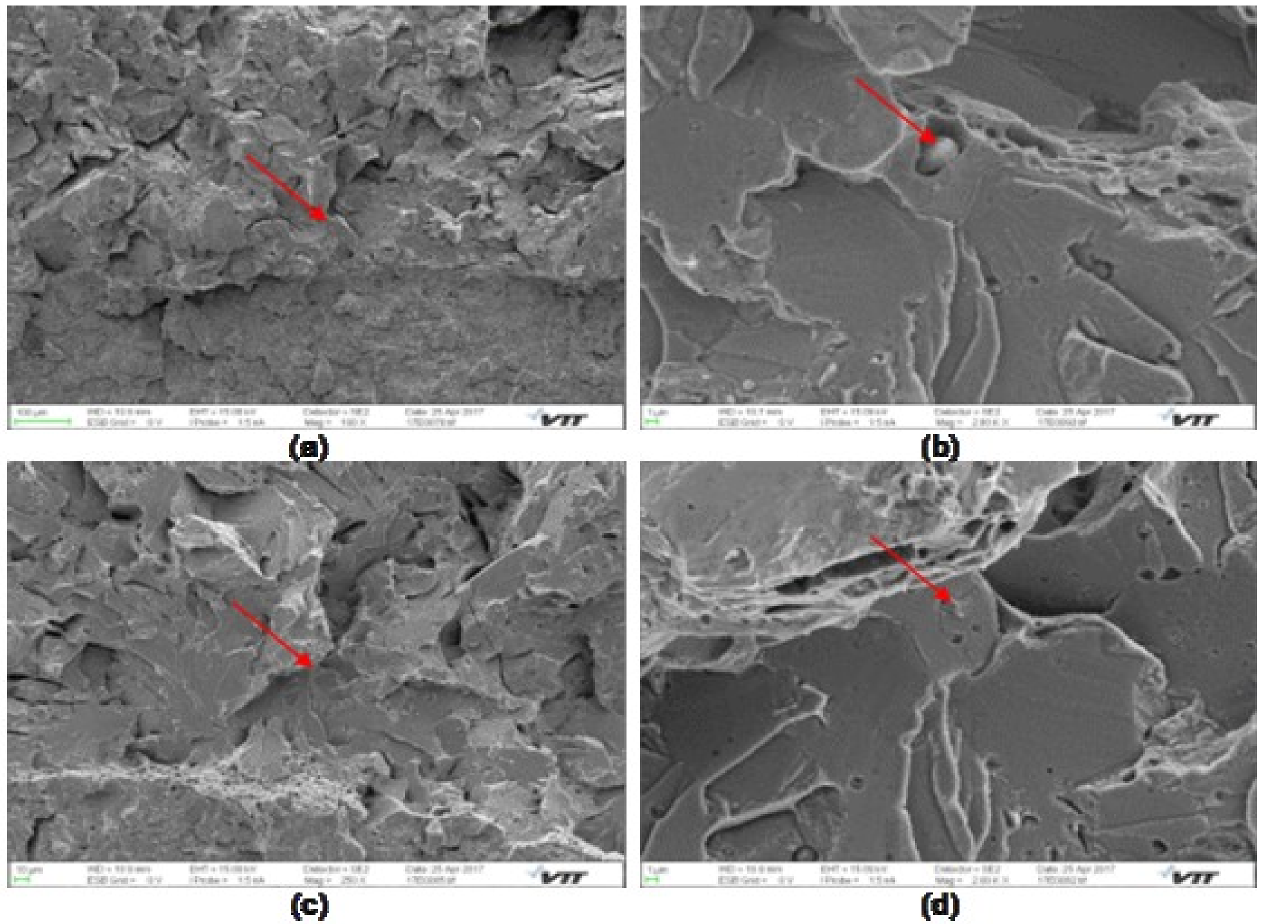
Two halves has to be examined

Fracture initiation sites

- VTT-1 weld: Both halves has to be examined

Half 1 the particle is round and ductile

Half 2 the particle is brittle that is the actuator expected



Summary

- Inhomogeneity on the as received material has been analysed
  - Inhomogeneity at microstructural level were found at all scales -> difficult to quantify
  - Precipitates nature on segregrated and non-segreated areas
  - Relevant scatter on chemical composition
  - Scatter on tensile properties larger than expected
  - Impact properties depends on specimen location -> effect of the thermal treatment, welds,…
  - Typical standard deviation for T_0 is less than 10 °C, specimen location can result in an additional standard deviation for T_0 of about 10 °C.
  - Requirement of the RPV surveillance standards to take the surveillance specimens in the ¼ to ¾ thickness range was confirmed by the test results
  - Fracture initiation site are not conclusive -> both halves should be examined

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