EFFECT OF MATERIALS HETEROGENEITIES ON MECHANICAL PROPERTIES AT INITIAL STATE

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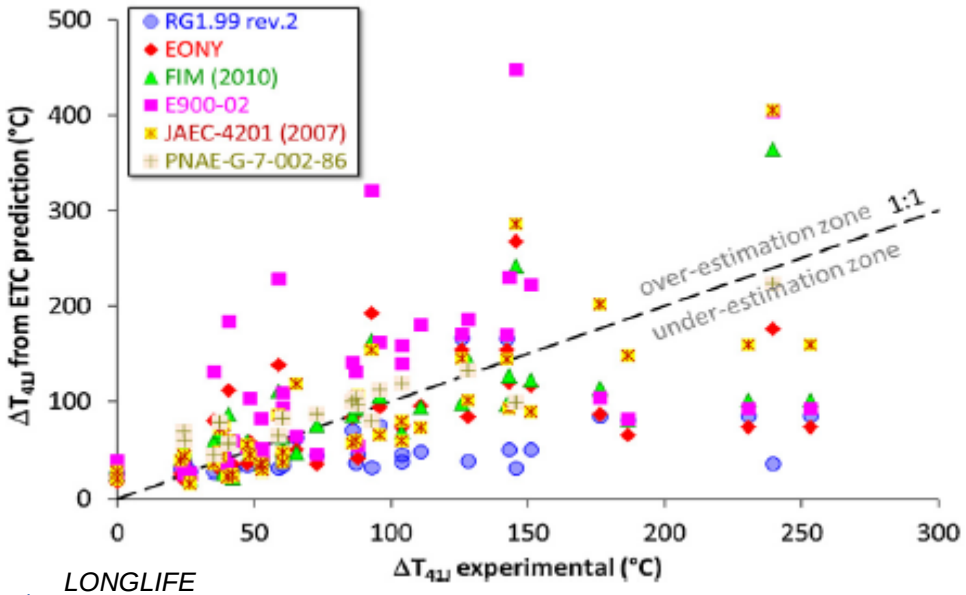
Contents

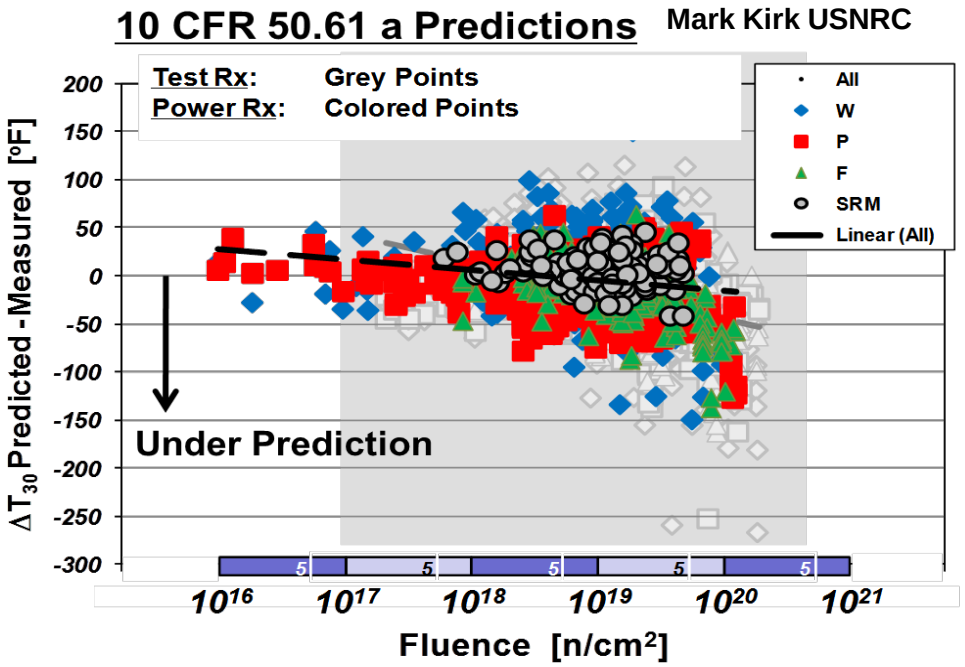
- Introduction
- SOTERIA related activities
  - Inhomogeneities in terms of mechanical properties
  - Inhomogeneities in terms of composition and microstructure
  - Fractographic analysis
- Summary

Introduction

- As a result of the analysis of embrittlement trend curves (ETCs) performed within LONGLIFE 7FWP none of the ETCs could predict the behaviour of the entirety of the LONGLIFE materials sufficiently
- 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

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

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

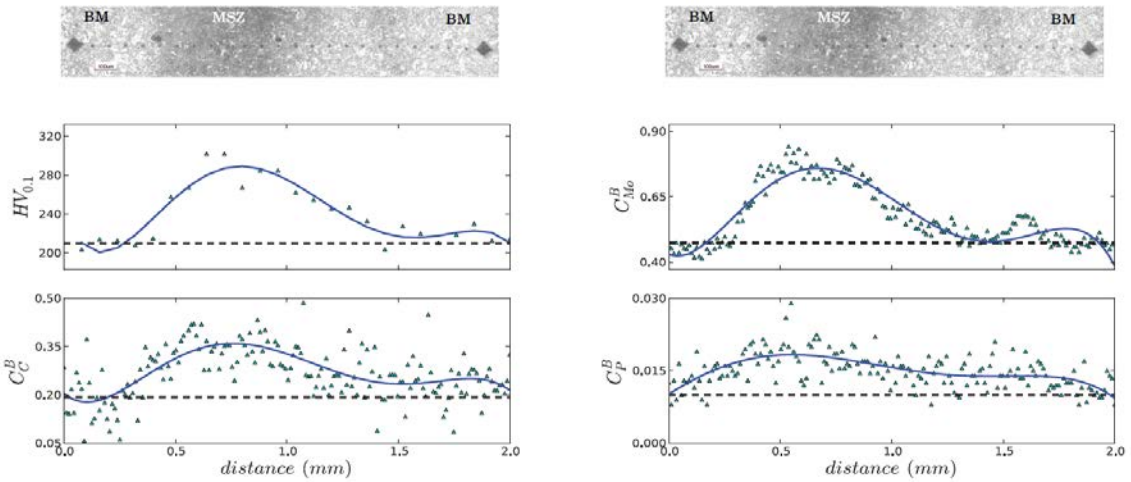
Unnecessary conservatism can be reduced when the accuracy of the mean condition is increased and the contributions to the total margin are properly understood

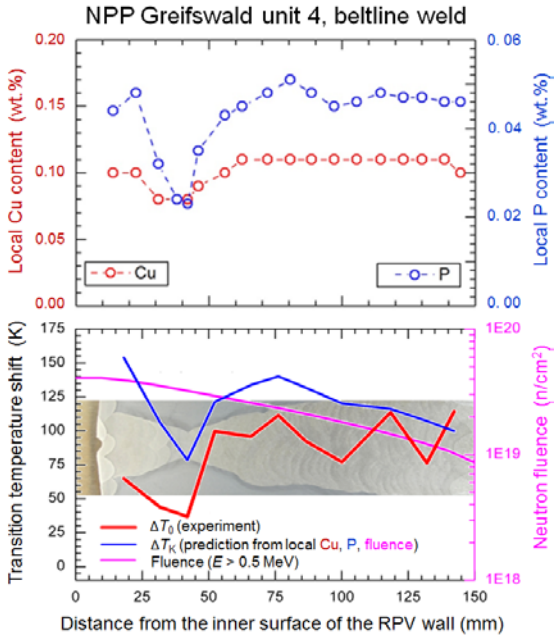
→ This presentation is focused on the **initial state**

Introduction

- Main sources of scatter in mechanical properties at initial state
  - Macro-segregations play on an important role
  - Multilayer welding seams
- The resulting material inhomogeneity involves place-to-place variations in matrix chemistry, variations in precipitate and inclusion sizes and densities and, in some cases, variations in the matrix phase
- Attenuation of the mechanical properties through thickness is strongly influenced by the variation in the start-of-life properties

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





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LONGLIFE

Introduction

- Work performed within SOTERIA
  - Inhomogeneities in terms of mechanical properties
    - Tensile, charpy and fracture toughness
  - Inhomogeneities in terms of composition and microstructure
    - Grain structure and precipitate distribution.
    - Micro- and mesoscopic segregations.
    - Chemical composition
  - Fractographic analysis: Primary initiation site
    - identification by SEM
    - Local chemical analysis
    - Microstructural and chemical characterizations of cross-sections

Materials

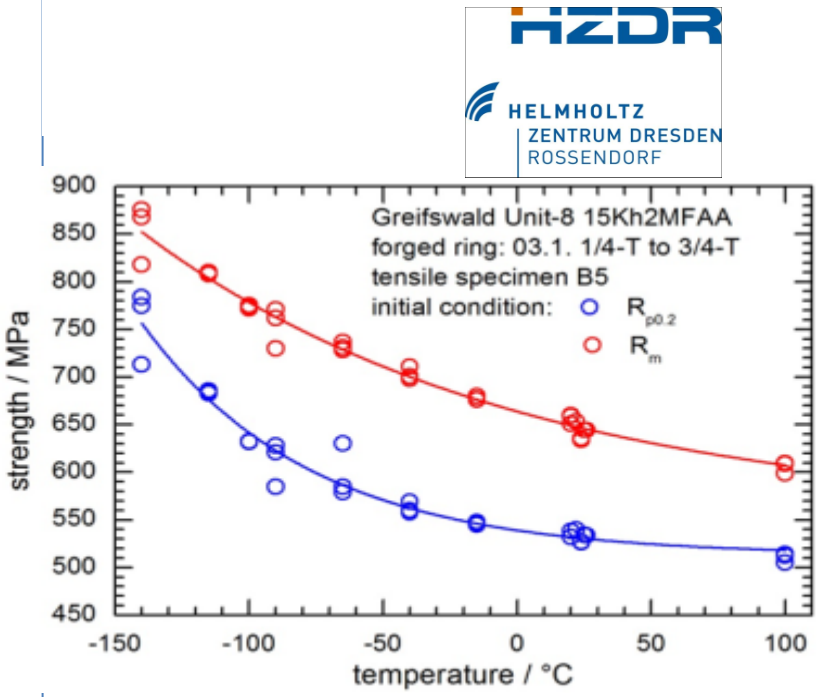
- FZD-4: Base metal 15Kh2MFAA – Trepan from Greifswald NPP Unit 8 (GR-8)
- CIE-1: Base metal A508 Class 3 – Zorita RPV head
- ANP-4: Base metal 22NiMoCr3-7
- EDF-4 16MND5 base metal
- VTT-1: VVER-440 type 10KhMFT surveillance weld material from the Loviisa 2 reactor
- VTT-MW1: a VVER-440 type 10KhMFT weld mock-up for the Loviisa surveillance testing program
- ANP-2: Weld metal S3NiMo1/OP41TT

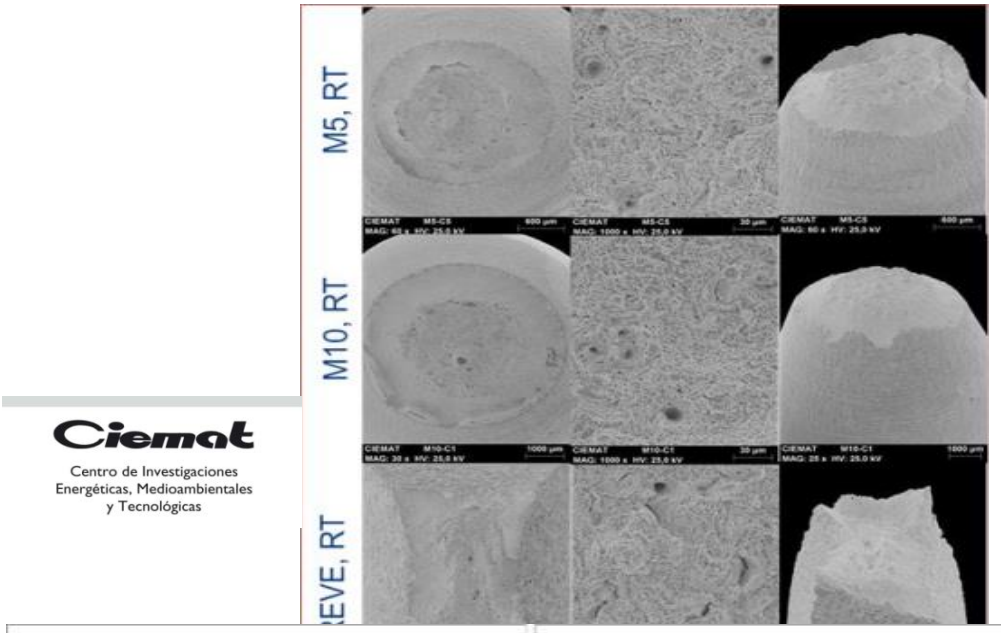
Material	C	Si	Mn	Cr	Ni	Mo	Cu	P	V	S
FZD-4 (BM)	0.15	0.30	0.45	2.86	0.10	0.79	0.05	0.008	0.31	0.002
CIE-1 (BM)	0.20	0.24	1.37	0.16	0.96	0.52	0.04	0.008	<0.01	
ANP-2 (W)	0.06	0.08	1.05	0.04	1.02	0.63	0.03	0.017	<0.01	
EDF4	0.16	0.24	1.31	0.18	0.7	0.5	0.08	0.011	0.006	

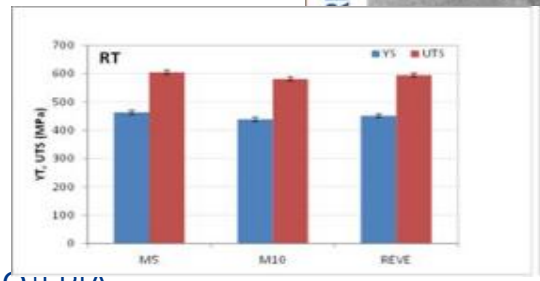
Mechanical properties

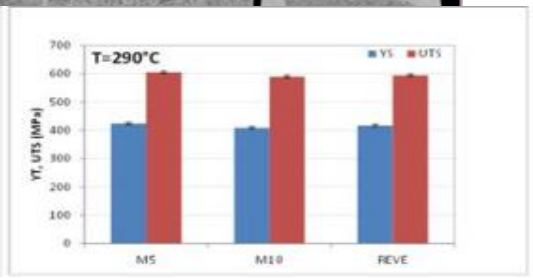
- In general tensile properties of studied material do not show a significant scatter; even when different specimen geometry is tested

CIE-1 BM A508 C3





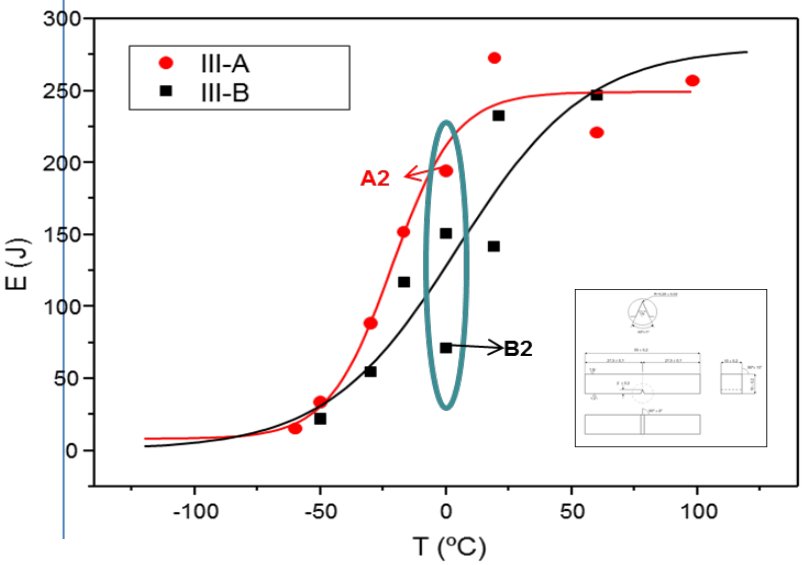


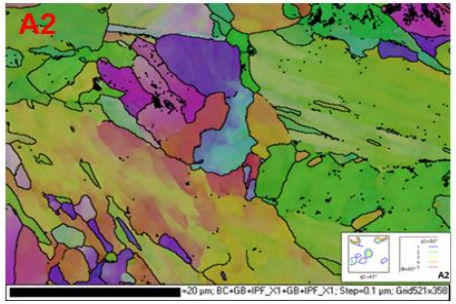


Mechanical properties

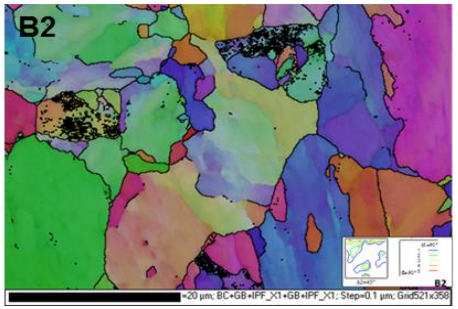
- Impact tests show a dependence of the absorbed energy with the location of the specimens in CIE-1 material

CIE-1 BM A508 C3





High impact energy



Low impact energy

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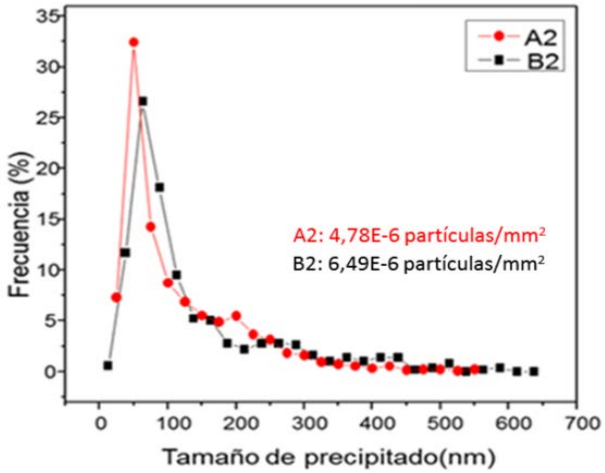
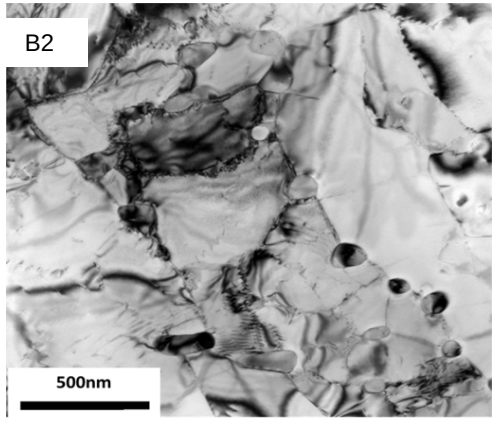
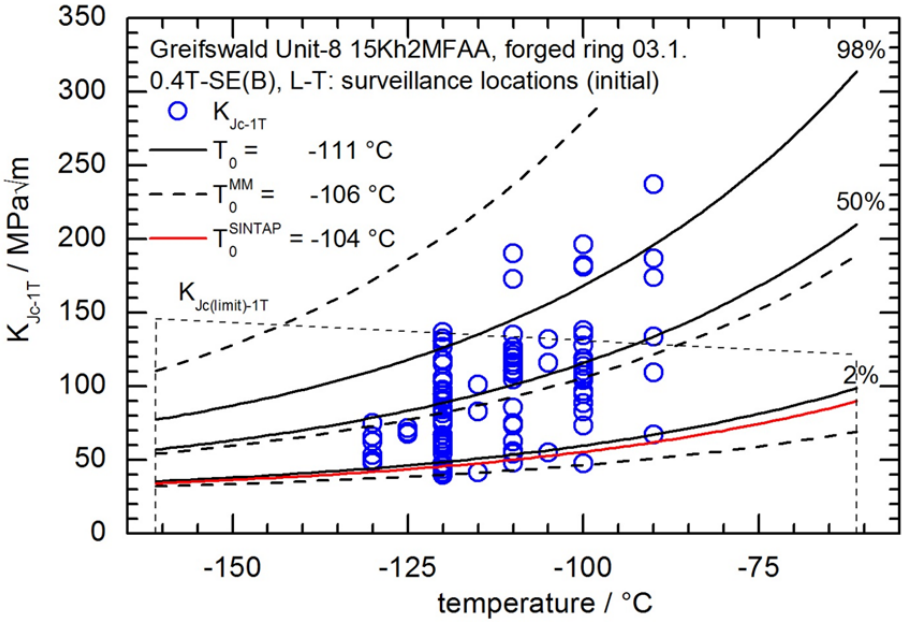


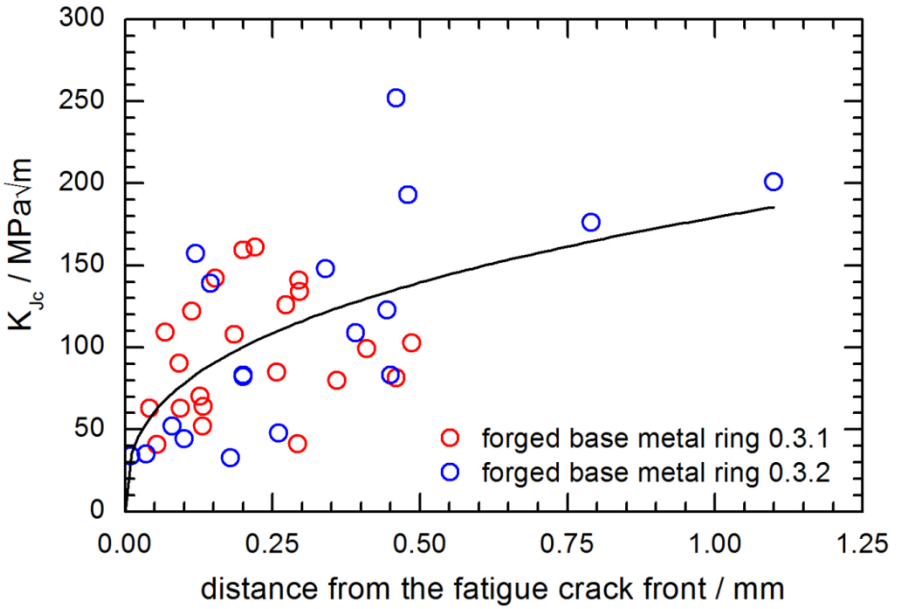


Mechanical properties

- Fracture toughness tests show larger scatter than expected for FZD-4 material that could be attributed to the presence of intergranular fracture areas.

FZD-4 BM 15Kh2MFAA



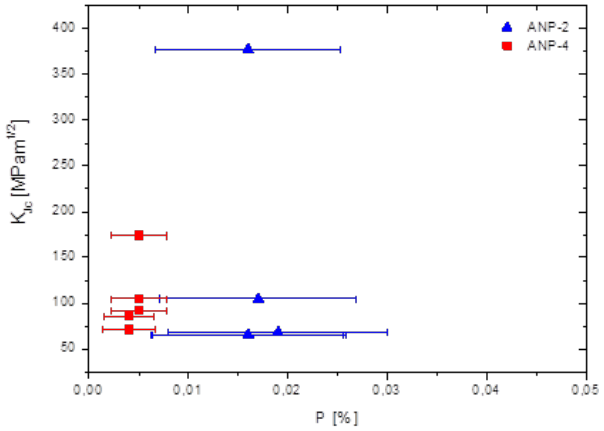


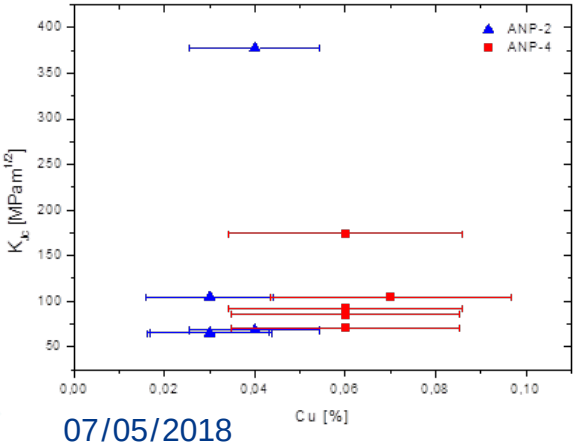
Chemical composition

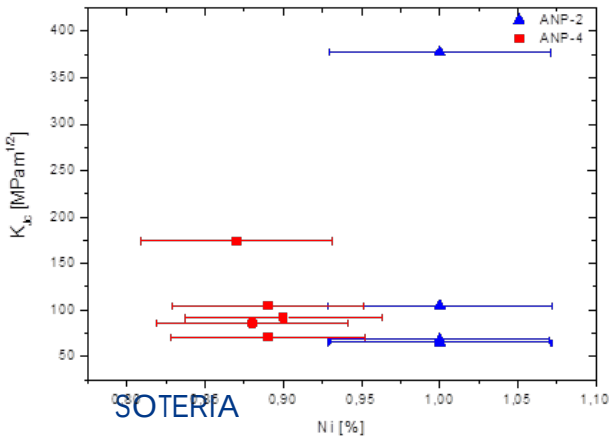
- Based on the chemical analyses performed on unirradiated ANP-2 and ANP-4 materials, using OES method a non-negligible uncertainty in weight % was found for some chemical elements, such as C, P, S, Cu and Ni (in high Ni weld) playing an important role in aging mechanisms of RPV steels.
- Fracture toughness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

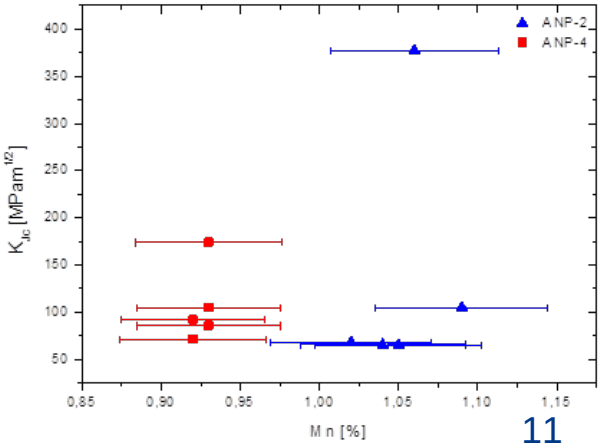
Material	Specimen designation	$K_{JC(1T)}$ in MPa√m	T in °C	Cu	Ni	P	Mn	Mo	Cr	C
				in %						
ANP-2	CACS2	65,1	-20	46	7,2	61	5	9,9	8,2	14
	CACS3	68,3	-30	36	7	58	5	10	8,5	13
	CACS4	65,4	-20	44	7,1	60	5	9,8	8,5	14
	CACS5	377,4	-10	36	7,1	58	5	10	8,5	13
	CACS6	104,8	-25	47	7,2	58	5	9,9	8,5	14
ANP-4	TL21	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









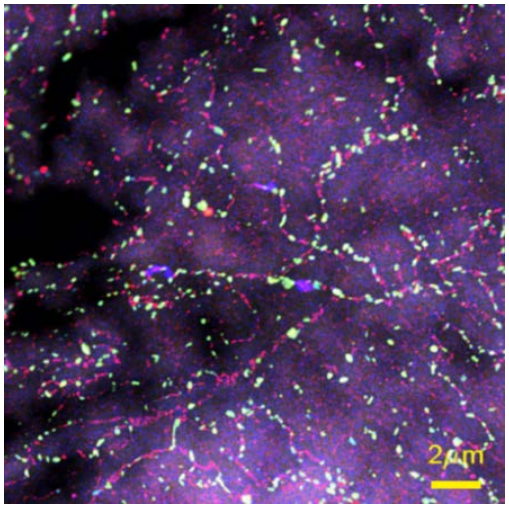
Microstructure

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

Figures and text replaced by Table

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



STEM-EDX elemental mapping of FZD-4

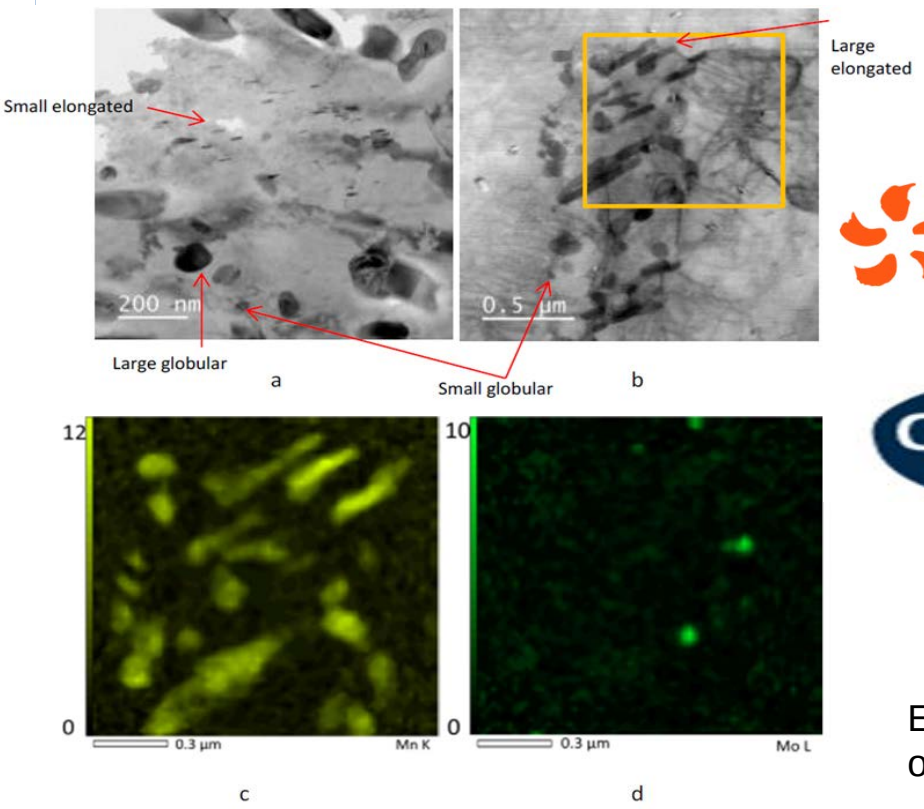
red: V (Kα)
green: Cr (Kα)
blue: Mo (Lα)

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.

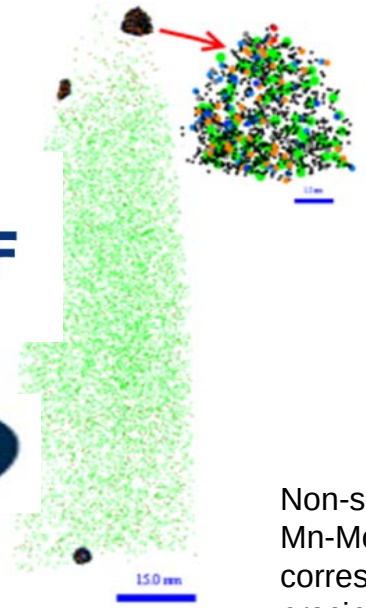
Microstructure

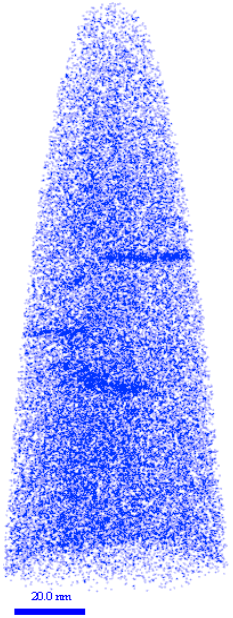
❑ EDF-4 material show micro- and mesoscopic segregations

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.





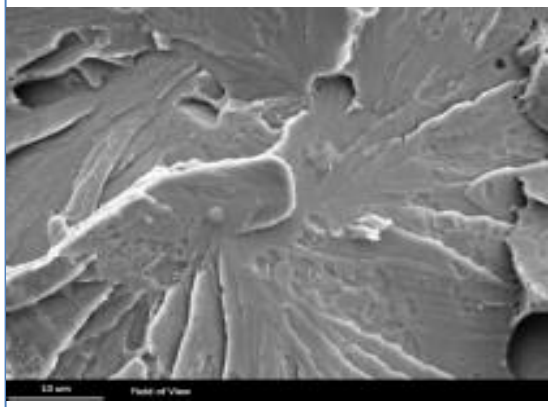
Non-segregated area
Mn-Mo-Cr-C carbides with corresponding Si-Ni-rich precipitates

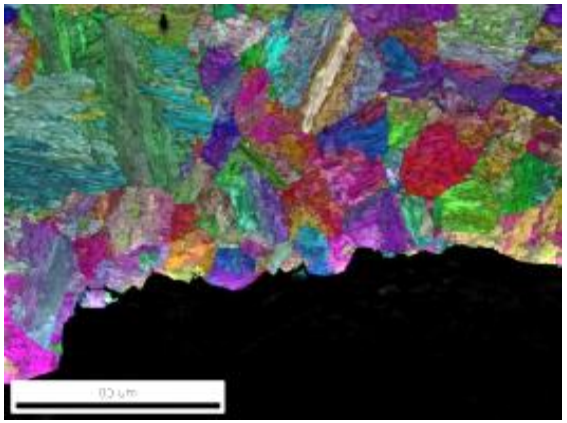
EDF4 samples do not contain characteristic ghost lines, but only micro- and mesoscopic segregations

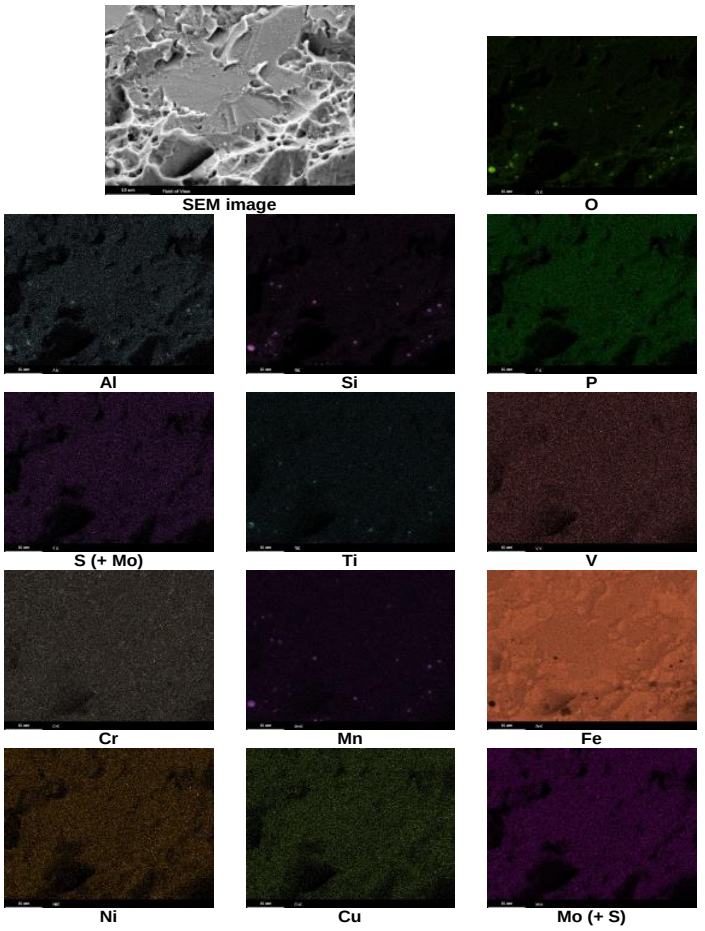
Fracture initiation sites

- Fracture initiation sites (VTT): Exhaustive characterization of primary initiation sites for ANP-3, ANP-4 (cont. ANP), VTT-1 and VTT-MW1

ANP-2





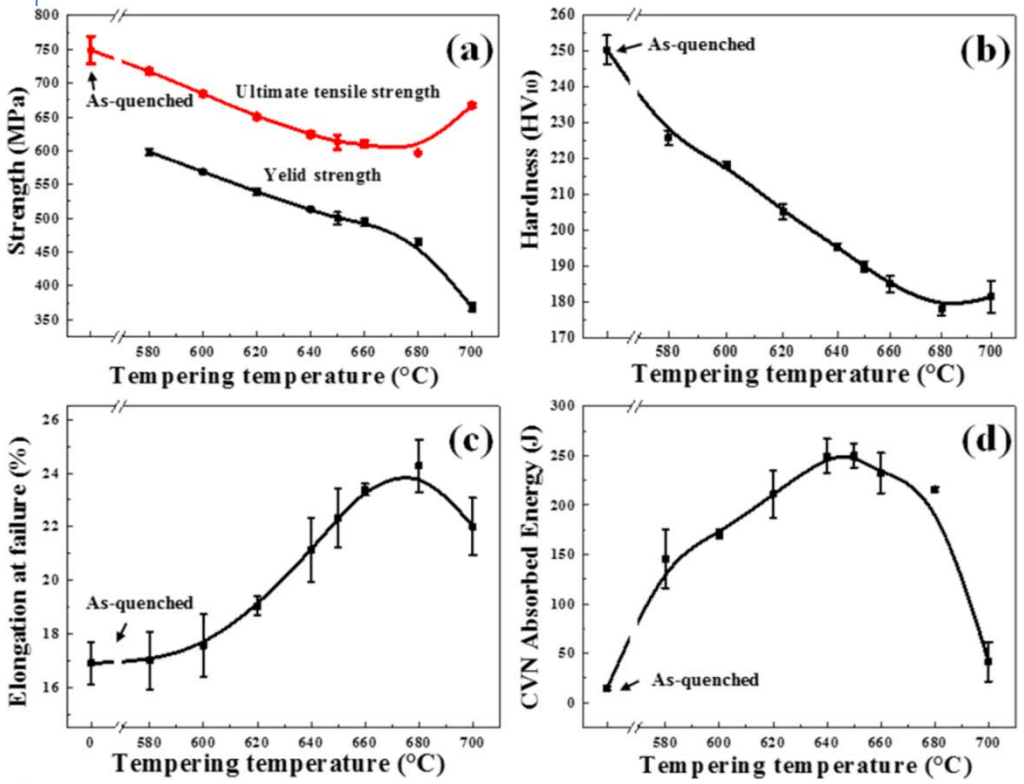


the primary initiation site is not characterized by a specific microstructural feature, such a precipitate or inclusion, at which brittle fracture would have initiated
Two halves should be analyzed

Sources of uncertainties

- Chemical inhomogeneities associated with the solidification of large ingots

Effect of tempering on final microstructure and mechanical properties



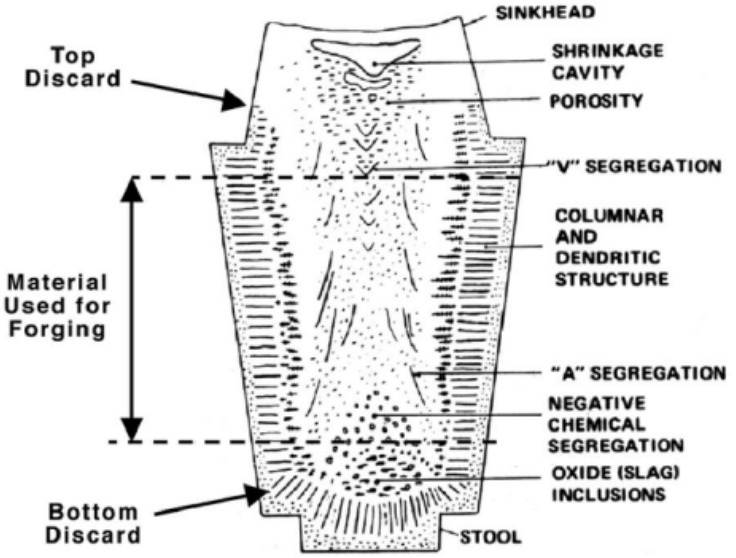
C.W. Li et al JNM (2016)

- Carbon macrosegregation
- Inhomogeneous distribution of alloying elements Mn, Ni, and Mo

- Slight or not effect on tensile
- Significant effect on hardness
- Significant effect in fracture (impact and fracture toughness)

Sources of uncertainties

- Macro-segregation in large ingots
  - Segregation of alloying elements in an ingot over large length scales (cm or m)
  - Occurs during solidification of an ingot
- Cause heterogeneity in a forging
  - As carbon is increased, strength increases but toughness decreases
  - Available data in literature suggest that increasing C by 0.1 wt.% increases RT_{NDT} by ~32-35°C
- The presence of small areas of low toughness, referred to as ghost lines, in ferritic thick components, may be an important source of scatter in fracture toughness values – well known since 80`s
  - These lines were identified as segregated zones containing increased amounts of impurities (P, S, etc.) and alloying elements (C, Mn, Ni, Mo, etc.) in the literature
- Carbon macrosegregation is a long-studied phenomenon



Several studies have shown no detrimental levels RPV shell forging

T. Hardin (EPRI 2017)

Sources of uncertainties

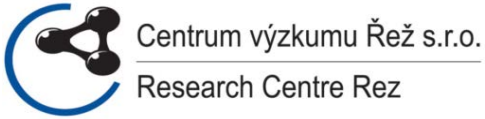
- The absence of gosh lines is not an indication of homogeneity
  - EDF4 material do not contain characteristic ghost lines, but micro- and mesoscopic segregations
  - FZD4 show indications of inhomogeneity at different length scales, The microstructure with respect to both grain structure and precipitate distribution is largely hierarchically
- The scatter in cleavage stress measurements is well known since a long time -> Kim Wallin Master Curve in the 80`s
  - However, the presence of intergranular areas gives large scatter that the predicted by the Master Curve
- Although the microstructure can be well defined (precipitated, inclusions, etc..) primary initiation site is not characterized by a specific microstructural feature
  - Need to examine both broken halves
- Different authors report that the complicated microstructural state of RPV steels in the unirradiated condition are unchanged after irradiation.

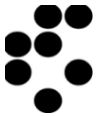
Summary

- Impact and fracture properties of tested material show large scatter as expected possible related to the location of the specimens.
- Microstructural examination by using combined techniques as STEM-EDX, STEM-HAADF SEM-EDS, SEM-EBSD and APT is needed to explain the mechanical behaviour of inhomogeneous RPV materials.
- Regarding chemical analysis a non-negligible uncertainty in weight % was found for some chemical elements, such as C, P, S, Cu and Ni playing an important role in aging mechanisms of RPV steels.
- The primary crack initiation site is not characterized by a specific microstructural feature, such as precipitate or inclusion.

Details on microstructure at the initial state is available for modelling activities

The SOTERIA Consortium





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