

#### Brittle fracture mechanisms of three model low alloy steels chemically representative of macrosegregated forgings

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# Brittle fracture mechanisms of three model low alloy steels chemically representative of macrosegregated forgings

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### **Outline of the presentation**

- 1. Introduction
- 2. Materials and methods
- 3. Results and discussion
- 4. Conclusions



## 

### **Industrial context**

- According to IAEA, ~78% of all operational reactors are Pressurized Water Reactors (PWR)
- PWR feature very large components
  - Reactor pressure vessel
  - Steam generator
- Made from low-alloyed steels (RPV steels), usually forged from heavy ingots

#### Hinkley Point C EPR reactor pressure vessel (UK)



### **Industrial context**

- Heavy ingots feature zones of positive macrosegregation
  - Richer in carbon, alloying elements and impurities
  - Limited segregations (%Cmax = 0.22%wt in France) can still be present in the final component
- Macrosegregation of the pressure vessel of Flamanville 3 EPR



(Rapport au Groupe permanent d'experts pour les équipements sous pression nucléaires, 2017)

### **Industrial context**

- Positive segregation associated with the embrittlement of RPV steels
- Destructive material characterizations necessary
  - Ensure structural integrity against brittle failure
  - Expensive and time-consuming



(From the data published by Rapport au Groupe permanent d'experts pour les équipements sous pression nucléaires, 2017)



### **Objectives**

- Understand the link between macrosegregation, microstructure and brittle failure of RPV steels
  - Material models representing the chemical composition of macrosegregated zones
  - Microstructural characterisation
  - Mechanical characterisation

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#### **Materials**

• Three **model materials** representing the chemical composition of macrosegregated zones

- Non-segregated/nominal composition (0,18%C), similar to 18MND5/ASME A508 Gr3 Cl2
- Moderate macrosegregation (0,29%C)
- Intense macrosegregation (0,38%C)
- Alloying elements content increases linearly with carbon content
- Casting of small ingots (~ 50 kg)



### **Materials**

- Homogenisation heat treatment carried out before forging (1200 °C, 24h)
  - Separate the effects of macro and microsegregation
  - Not commonly used for industrial components
- Forging at 1180 °C
- Heat treatment simulating the one used for industrial components



### **Microstructural characterisation**

- Microstructural evolution quantified based on misorientation angles obtained via EBSD
  - Lower bainitic/martensitic microstructures feature a high frequency of 50-60° misorientation angles (Gourgues, 2000; Zajac et al., 2005)



### **Microstructural characterisation**

- Microstructural evolution quantified based on misorientation angles obtained via EBSD
  - Lower bainitic/martensitic microstructures feature a high frequency of 50-60° misorientation angles (Gourgues, 2000; Zajac et al., 2005)
- Microstructural parameter  $\mu$  calculated as the cumulative frequency between 50-60°
  - Increase in µ indicates evolution towards lower bainitic/martensitic microstructure



# 

### **Mechanical tests**

- Charpy and fracture toughness tests
  - Standard V-Charpy specimens
  - CT specimens (B = 12.5 mm)
- Temperatures  $T_{56J}$  and  $T_0$  from Master curve (ASTM E1921) used to quantify embrittlement
  - Increase in  $T_{56J}/T_0 \rightarrow \text{embrittlement}$



### **Fractographic analyses**

- Carried out for both Charpy and CT specimens at the ductile-to-brittle transition
  - Non-segregated (0,18%C)
    - 5 specimens (2 Charpy + 3 CT)
  - Moderate macrosegregation (0,29%C)
    - 6 specimens (2 Charpy + 5 CT)
  - Intense macrosegregation (0,38%C)
    - 6 specimens (2 Charpy + 4 CT)
- Type of brittle fracture (cleavage or intergranular)
- Initiation site of brittle fracture
  - Determined based on cleavage river marks
  - Qualitative analyses of chemical composition via EDX





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### **Mechanical tests**

- Non-linear evolution of  $T_{56J}$  (Charpy) and  $T_0$  (fracture toughness)
  - Similar temperatures for **non-segregated** and **moderately macrosegregated** materials
  - Intensely macrosegregated material has the lowest temperatures (least brittle)



#### **Microstructural evolution**

- Microstructure depends on the macrosegregation level
  - Usual bainitic microstructure for **non-segregated** and **moderately macrosegregated** materials
  - Intensely macrosegregated material is lower bainitic/martensitic



#### Moderate macrosegregation



#### Intense macrosegregation



### **Microstructural evolution**

- Increase in  $\mu \rightarrow$  microstructure evolves towards lower bainite/martensite
- Consistent with the increase in hardenability with the increase in alloying elements
- Martensite can be more resistant to cleavage than bainite for RPV steels (Marini et al., 2015)



#### **Failure mode**

- Failure mode depends on the macrosegregation level
  - Cleavage for non-segregated and moderately macrosegregated materials
  - Intensely macrosegregated material feature both cleavage and intergranular failure
    - Intergranular failure possibly due to increase in the cleavage resistance

#### **Non-segregated**



#### Moderate macrosegregation



#### Intense macrosegregation



### **Initiation sites**

- Determination possible only for non-segregated and moderately macrosegregation
- Carbides are the dominant microstructural feature at the origin of failure (7/11 cases)
  - Mostly Mo-enriched (5/11)
  - Always found near grain boundaries



#### С Fe Мо Mn 0.012 10 keV Matrix Precipitate 0.010 Normalized Counts [1/Total Counts] 0.008 0.006 0.004 0.002 Fe Fe 0.000 100 $10^{-1}$ $10^{1}$ log Energy [keV]

## Hypothesis for the evolution of temperatures

• Presence of two effects associated with macrosegregation



- For moderately macrosegregated material, both effects have similar magnitudes
  - Temperatures T<sub>56J</sub> and T<sub>0</sub> similar to those of **non-segregated** material
- For **intensely macrosegregated** material, the strengthening effect is dominant
  - Lowest temperatures  $T_{56J}$  and  $T_0$
  - Likely associated with homogenisation heat treatment
    - Less microsegregation  $\rightarrow$  homogeneous chemical composition  $\rightarrow$  microstructure globally more hardenable

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#### Conclusions



- Unexpectedly, the **intensely macrosegregated** material is the least brittle
  - Similar results for **non-segregated** and **moderately macrosegregated** materials
- Macrosegregation has two different effects
  - Embrittlement due to the increase in the carbide population
  - Strengthening due to the evolution of the microstructure towards lower bainite/martensite
- Unexpected evolution of  $T_{56J}$  and  $T_0$  explained by the competition between those effects
  - For moderately macrosegregated material, effects cancel out
  - For **intensely macrosegregated** material, strengthening more pronounced than embrittlement
- Analyses limited to homogenised model materials, **do not represent industrial materials** 
  - Different thermomechanical history, absence of homogenisation, ingot size, etc.

## 

#### **Future works**

- Similar analyses on industrial materials
  - Specimens machined from macrosegregated steam generator (sacrificial component)
- Modelling of the effects of microsegregation based on the local approach to fracture



#### References

- Rapport au Groupe permanent d'experts pour les équipements sous pression nucléaires. "Analyse des conséquences de l'anomalie des calottes de la cuve du réacteur EPR de Flamanville sur leur aptitude au service " (2017)
- Gourgues A.-F., et al. "Electron backscattering diffraction study of acicular ferrite, bainite, and martensite steel microstructures." *Materials Science and Technology 16.1 (2000): 26-40.*
- Zajac et al. "Characterisation and quantification of complex bainitic microstructures in high and ultrahigh strength linepipe steels." *Materials Science Forum. Vol. 500. Trans Tech Publications Ltd, 2005.*
- ASTM. "Standard Test Method for Determination of Reference Temperature, To, for Ferritic Steels in the Transition Range" (2019)
- Marini et al. "Effect of the bainitic and martensitic microstructures on the hardening and embrittlement under neutron irradiation of a reactor pressure vessel steel." *Journal of Nuclear Materials 465 (2015)*: 20-27



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Hinkley Point C reactor pressure vessel, © EDF Energy

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