

Electron and in-cell optical microscopy study of irradiated 20%Cr: 25%Ni: Nb stainless steel

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#### Introduction to CAGR



- UK 9 sites- 7 CAGR, 1 Magnox, 1 PWR
- Commercial Advanced Gas-cooled Reactors (CAGR)
- CO<sub>2</sub> coolant + solid graphite moderator
- Hollow ceramic UO<sub>2</sub> pellets clad with 20Cr/25Ni:Nb stainless steel alloy
- Each fuel pin is 1m length
- Pins grouped in bundles of 36, in 3 rings= 1 Fuel Element
- Graphite "sleeve"
- 8 Elements make up 1 stringer (articulated)
- High temperature cladding in comparison to LWR

### PIE Techniques- NNL



#### Non destructive:

- Visual examination
- Dimensional Measurement
- Profilometry
- Gamma scanning (isotopic and total)

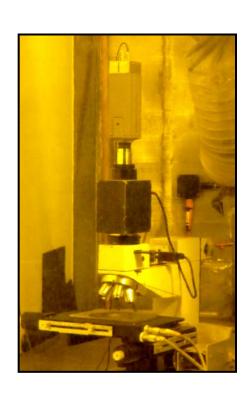
#### **Destructive:**

- Puncturing for fission gas sampling and analysis
- Fission gas analysis (mass spectrometry)
- Density
- Raman spectroscopy
- Thermal properties
- Optical microscopy- polarised, fluorescent and BF
- Electron optical examination (SEM & TEM)
- Micro cXRT (FIB)

# **Optical Microscopy**



# Modified components coloured red





Ergolux

Leica DMI5000

## Scanning Electron Microscopy



• FEI Quanta 200 FEG SEM with Oxford Instruments SDD EDX detector



### **CAGR Cladding Material**



### Cladding:

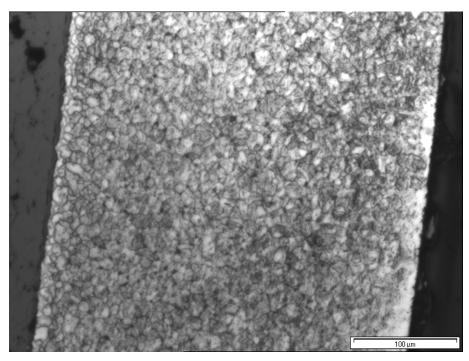
- 20%Cr: 25%Ni: Nb stainless steel (austenitic polycrystalline face centred cubic (fcc) solid solution alloy)
- Contains dispersed Nb(C) precipitates
  - In reactor (with increased T) these precipitates coarsen
  - M<sub>6</sub>C/M<sub>23</sub>C<sub>6</sub>, G phase (Ni<sub>16</sub>Nb<sub>6</sub>Si<sub>7</sub>), complex carbonitrides, silicides
    - Only observable using electron microscopy
- Sigma (o) phase formation also occurs (triple points)
  - o phase can be used to indicate operational T

### **CAGR Cladding Material**



#### Cladding:

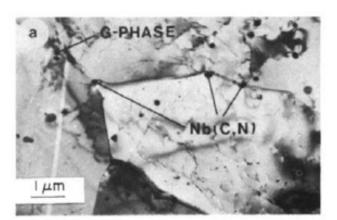
- Radiation induced segregation (RIS)
  - Result of irradiation at low temperatures (<550°C)</li>
  - Migration of Cr away from the grain boundaries-sensitisation (similar to thermally aged steels).
  - (Ni,Si)-rich precipitates form (10nm to 60nm) similar to the formation of γ' in other hi Ni alloys
  - Manifests as optically irresolvable 'dark' phase
  - Can also be used to estimate operational temperatures

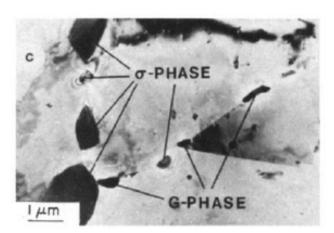


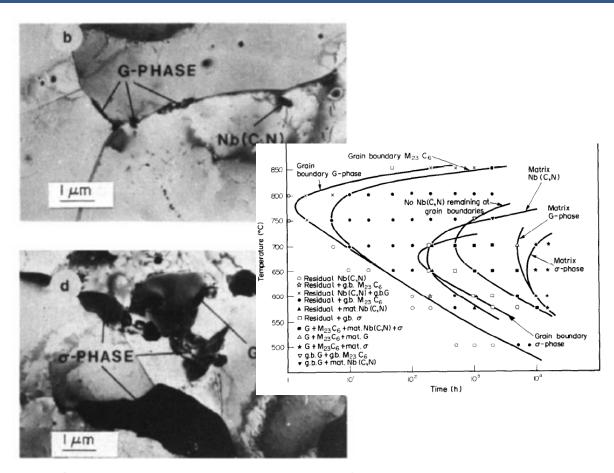
LOM image

#### Previous work









Transmission electron micrographs showing the microstructural changes as a function of time at 650°C. (a) 100 h, (b) 500 h, (c) 5000 h, (d) 15,000 h.

TEM images- unirradiated steels

#### Current work

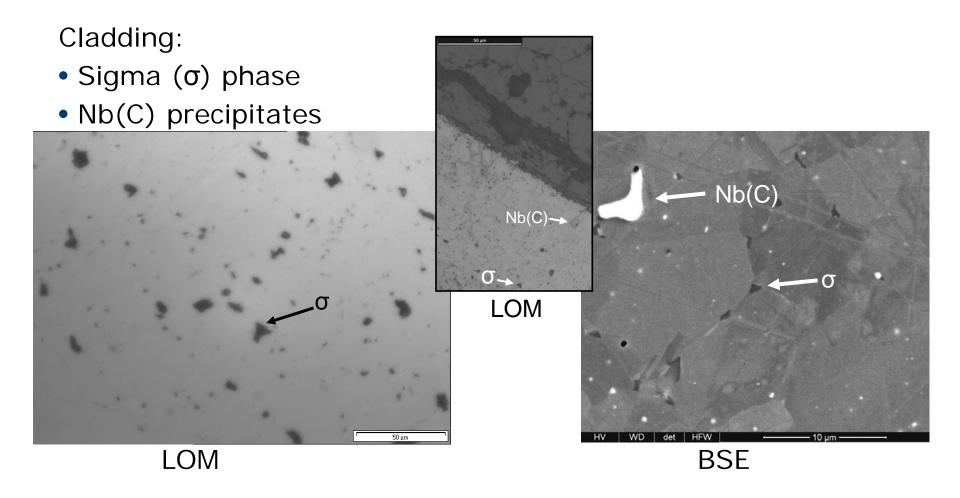


### Reactor irradiated CAGR cladding

- 20Cr: 25Ni: Nb
- In-reactor Temperature range ~400≤700°C
- Carbon dioxide (CO<sub>2</sub>) atmosphere
- ~30GWd/tU burn-up
- ~2500 days in core (60,000hrs)

### **CAGR Microstructures**



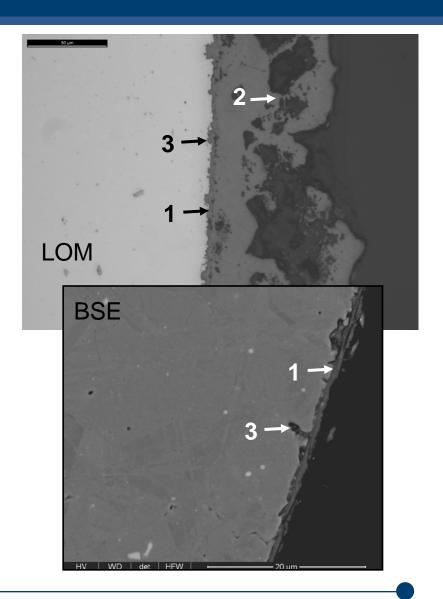


#### **CAGR** Features



#### **Outer Cladding Surface:**

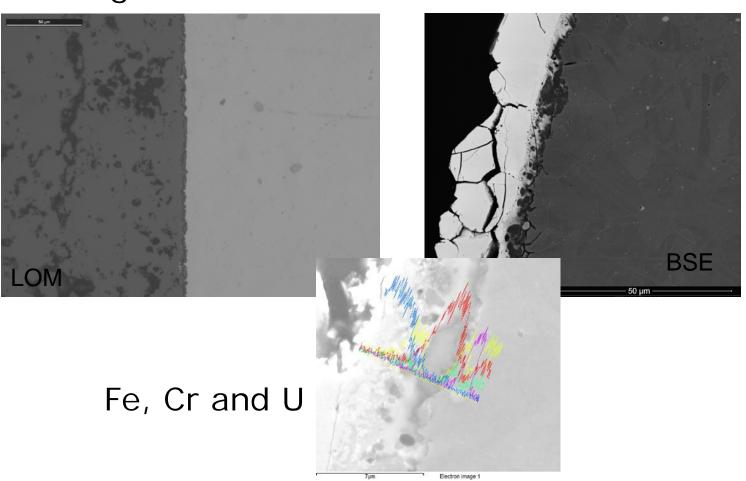
- Protective layer Cr<sub>2</sub>O<sub>3</sub> [1]
  - Between 2-8 μm
  - Thin, adherent
  - May contain
    - silica, oxide, spinel
- Carbonaceous deposition [2]
  - Coolant additives CH4 and CO breakdown
  - Deposit builds on some can surfaces
  - Impairment of heat transfer from fuel to coolant
  - May nucleate on small surface oxide pits [3]



### **CAGR Microstructures**



### Cladding to fuel interface:

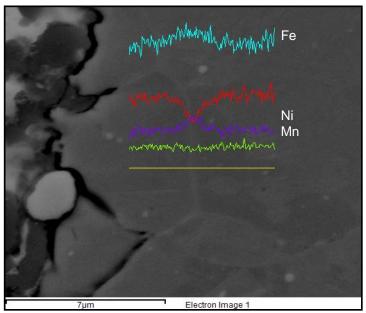


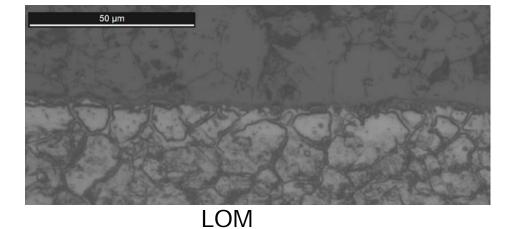
## Cladding Study



#### Cladding grain boundaries:

- Some localised enrichment at clad-fuel interface
- Uniform through bulk





**BSE** 

#### Further work



- WDS on clad-fuel interface- comparison with EDX to eliminate contribution of Fe-55 to Mn
- Quantitative TEM on smaller samples
- Non-active TEM
- Non-active FIB

### Acknowledgements



EDF Energy Generation Ltd (UK)

Ray Graham and Gary Blacklock (PIE technicians)