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)
• CO₂ coolant + solid graphite moderator
• Hollow ceramic UO₂ 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
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
  - $M_6C/M_{23}C_6$, G phase ($Ni_{16}Nb_6Si_7$), complex carbonitrides, silicides
    - Only observable using electron microscopy

- Sigma (σ) phase formation also occurs (triple points)
  - σ phase can be used to indicate operational T
CAGR Cladding Material

Cladding:
• Radiation induced segregation (RIS)

• Result of irradiation at low temperatures (<550 °C)
• 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
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  $\sim 400 \leq 700^\circ C$
- Carbon dioxide (CO$_2$) atmosphere
- $\sim 30$GWd/tU burn-up
- $\sim 2500$ days in core (60,000 hrs)
CAGR Microstructures

Cladding:
- Sigma (σ) phase
- Nb(C) precipitates
CAGR Features

Outer Cladding Surface:
- Protective layer Cr$_2$O$_3$ [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]
Cladding to fuel interface:

Fe, Cr and U
Cladding Study

Cladding grain boundaries:

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