Yann De CARLAN
CEA (France)

Relationship between Microstructure and Mechanical Properties in ODS Materials for Nuclear Application

Workshop organized by:
Christophe GALLÉ, CEA/MINOS, Saclay – christophe.galle@cea.fr
Constantin MEIS, CEA/INSTN, Saclay – constantin.meis@cea.fr
Relationship between Microstructure and Mechanical Properties in ODS Materials for Nuclear Application

Yann De CARLAN

1CEA-DEN-DMN, Service de Recherches Métallurgiques Appliquées, SRMA (Saclay, France)

Oxide Dispersion Strengthened ferritic/martensitic alloys are developed as prospective cladding materials for future Sodium-Cooled-Fast-Reactors (GEN IV) [1]. These advanced alloys present a good resistance to irradiation and a high creep rupture strength due to a reinforcement by the homogeneous dispersion of hard nano-sized particles (such as Y$_2$O$_3$ or YTiO). ODS alloys are elaborated by powder metallurgy, consolidated by hot extrusion and manufactured into cladding tube using the pilger cold-rolling process [2, 3]. ODS alloys present usually low ductility and high hardness. The aim of this talk is to present the specificity of the metallurgy of ODS materials in relationship with the main mechanical properties (tensile and creep properties, toughness, transition temperature…). Two types of alloys will be presented: Fe-9Cr martensitic ODS and Fe-14Cr ferritic ODS alloys. Mechanical properties of the materials depend on the metallurgical state (fine grains, recrystallized, martensitic) and very different behaviors are observed as a function of final microstructure. For example, for a Fe-9CrODS alloy, tempered martensite lets obtaining material with high strength whereas softened ferrite see figure 1 [4] tolerates high deformation levels.

Fig. 1: Electron Backscatter Diffraction maps for a Fe-9Cr ODS associated with the tensile properties at room temperature.

This is an Open Access article distributed under the terms of the Creative Commons Attribution License 2.0, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.
References


RELATIONSHIP BETWEEN MICROSTRUCTURE AND MECHANICAL PROPERTIES IN ODS MATERIALS FOR NUCLEAR APPLICATION

Yann de Carlan et al., CEA, DEN, SRMA, 91191 GIF/YVETTE FRANCE
Beginning of the studies on the Fast Sodium Reactors to produce electricity

1960
1967
1973
1985
1987
2000

Materials
First ODS (SCK-CEN)
First irradiated tubes
Industrial Production (DourMetal)
ODS tubes in Phenix reactor

Need of optimized materials is still relevant!

Optimization of the austenitic steels
New reinforced ferritic alloys: ODS alloys

L. Toualbi
Why ODS materials for Nuclear Applications?

ODS materials
(Oxide Dispersion Strengthened)

- Limited swelling under irradiation
- Low thermal creep
- Nano dispersion

ODS included

SFR
Dose > 150 dpa
Stress ~ 100 MPa
Temperatures: 400°C – 670°C

Use limit for Phenix reactor
Complex manufacturing

- ODS steels present low ductility and limited consolidation at room temperature
- Low deformability
- Intermediate heat treatments needed for stress relief purpose
Ex: ODS steel
18 Cr-1W Ti  0.5 Y₂O₃
Uniform distribution of nano oxides

Homogeneous microstructure
Fine ferritic grains
elongated in extrusion direction
- Ferritic grades
  Fe-12/20Cr

- Martensitic grades
  Fe-9Cr

Large differences between both type of alloys as a function of their final microstructure but generic creep behavior is noticed.
METALLURGY OF ODS MATERIALS

Typical microstructures

Ferritic Fe-14Cr ODS:
Extruded at 1100°C + 1h at 1050°C (very difficult to recrystallize)

L. Toualbi, Phd Thesis

Martensitic Fe-9Cr ODS:
Tempered martensite
(very high critical cooling rates)
Martensitic grade: Fe-9Cr-1W-0.2Ti-0.3Y₂O₃

- CCT diagram
  - Transformation temperatures
  - Critical cooling rates ~ 1°C/s

L. Toualbi, Phd Thesis
Tensile properties
Creep properties
Impact properties
Ferritic (Fe-14 Cr ODS)

Extruded and annealed ferritic ODS (Fe-18Cr), tensile test at 650°C in the transvers direction, large decrease of the ductility when the deformation rate is decreasing.

M. Ratti, PhD thesis
TENSILE PROPERTIES OF ODS ALLOYS

- Martensitic (Fe-9Cr ODS)

Tensile properties in different metallurgical state:
- J95-M3, tempered martensite
- J95- M5, Softened ferrite

L. Toualbi, Phd Thesis
Ferritic (Fe-18 Cr ODS)

ODS plate
Extruded and annealed

Extrusion direction

Bamboo like microstructure

M. Ratti, Phd Thesis
Ferritic Fe-18 Cr ODS at 650°C 250 MPa

- Very low deformation rates (Long-250 MPa : $7 \times 10^{-8}$ h$^{-1}$).
- Almost no tertiary domain.
- Different order of magnitude on the rupture time as a function of the orientation of the sample / texture of the microstructure.

Transvers

Long (in progress)

M. Ratti, Phd Thesis
Grain Morphology Anisotropy Results in Poor Creep Rupture Strength under Internal Pressure

Finely elongated grain structure along rolling direction results in
- degradation of internal creep rupture strength and
- significant loss of ductility around 673 K

Inoué et al.
Ferritic ODS

Higher performances of Fe-14Cr ODS alloys (fine grains) in the longitudinal direction compared to Fe-9Cr ODS (softened ferrite)
CREEP PROPERTIES OF ODS ALLOYS

M. Praud, J. Malaplate et al., creep 2012
In situ relaxation tests at 550°C
- Creation of cavities
- Decohesion along the grain boundaries

**INTERGRANULAR** mechanism
Consistent with the macroscopic observations

M. Praud, J. Malaplate et al., creep 2012
Ferritic Fe-14 Cr ODS alloys

Ferritic Fe-14 Cr ODS alloys

- **anisotropic** impact behaviour for the **rod**: higher USE and lower DBTT under LT loading

<table>
<thead>
<tr>
<th>Diameter</th>
<th>LT</th>
<th>TL</th>
</tr>
</thead>
<tbody>
<tr>
<td>rod Ø36</td>
<td>-29</td>
<td>108</td>
</tr>
</tbody>
</table>

Fe-14Cr 1W ODS
“Fine grain microstructure”

diameter: 36mm

AL Rouffié et al. http://dx.doi.org/10.1016/j.jnucmat.2012.08.050
IMPACT PROPERTIES OF ODS ALLOYS

- Ferritic Fe-14 Cr ODS alloys

- Anisotropic impact behaviour for the rod: higher USE and lower DBTT under LT loading

<table>
<thead>
<tr>
<th>DBTT (°C)</th>
<th>USE (J)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LT</td>
<td>TL</td>
</tr>
<tr>
<td>rod Ø36</td>
<td>-29</td>
</tr>
</tbody>
</table>

For different samples, tendency to delamination

Effective propagation plane

Expected propagation plane

Notch

ED

AL Rouffié et al. http://dx.doi.org/10.1016/j.jnucmat.2012.08.050
- Ferritic Fe-9Cr ODS alloys (thick plate)

Quasi-isotropic behaviour
The mechanical properties of nuclear F/M alloys depend strongly on the chemical composition and the microstructure of the product.

- **Tensile properties**
  - Large dependence on the grain size / morphology / dislocation density / nano-precipitation
  - Collapse of the strength from 400°C
  - Strain rate effect on the ductility

- **Creep properties**
  - No tertiary creep but high performances
  - Reduced fracture strain
  - Damage more severe at high temperature low strain rate / low stress => **Intergranular mechanism of failure**

- **Impact properties**
  - Low toughness and in general “low” Upper Shelf Energy and sometimes tendency to delamination
  - Very important impact of the microstructure (morphological texture) on the impact properties

The fabrication route of ODS materials with the appropriate microstructure is a key issue for the nuclear applications.