ODS Steels: Recent Progress and Justification for Significant In-vessel Applications

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Key Issues Towards Practical Applications

Contents

1. Fracture toughness:
   1) Improvement
   2) Phase decomposition

2. Strong texture:
   1) Recrystallization
   2) Anisotropy

3. Radiation tolerance:
   1) Tensile properties
   2) Swelling
   3) Nanostructure

4. Welding/joining:
   1) FSW
   2) etc.

5. Infrastructure:
   1) Massive production
   2) Homogenization

6. Accident tolerant fuel cladding:
   1) Alloy design, Cr/Al
   2) Assessment
Fracture Toughness Improvement


- 9Cr-ODS
- Bend specimens
- 2.5 x 5 (3.5) x 15 mm
- With notch
- TL direction

Fracture toughness of a martensitic ODS steel was improved to the level of HT-9.

\[ \rightarrow \] Ferritic ODS steel?
FCRD NFA-1

- LANL-ORNL-UCSB larger US 14YWT heat of Y-Ti-O added Fe14Cr ATI powders Zoz milled 40h, ORNL extruded at 850°C annealed 1 hr and cross rolled at 1000°C.
- UCSB studies show excellent properties - very low DBTT (≈ -185°C), high ≈ isotropic strength*, ductility* and toughness
- Stable crack growth initiation at general yield loads and graceful ductile failure behavior.
- High toughness is due to delamination converting thick section plane strain to thin section plain stress crack tip fields.

Fracture toughness of a ferritic ODS steel shows very low DBTT. → How about anisotropy?

Fracture toughness of a ferritic ODS steel shows very low DBTT. → How about anisotropy?

- Pre-cracked bend bars Averages
- Atypical ≈ isotropic toughness
- Averages

3PB NFA1

K_C (MPa m^-1/2)

120
100
80
60
40
20
0
-200 -150 -100 -50 0 50

Temperature (°C)

0 200 400 600 800 1000

Load, N

Displacement (mm)

Stress (MPa)

Averages

RT

Crack deviated by ~90°
Delamination and Microcracking

- Delamination from texturing and brittle (001)<110> cleavage systems formed by deformation, also leading to internal stress driven microcracks parallel to the plate surfaces. RT tensile test cleavage in S-thickness direction but BDT at higher temperature.

- Strain self-assembled dislocations/sub-boundaries crack paths & local stresses

- Cracking in processing products (e.g. tubes)
Tubes of FCRD-NFA1 NFA

LANL-ORNL-UCSB
larger US 14YWT heat of Y-Ti-O added Fe14Cr ATI

- Tube was EDMed from FCRD-NFA1 plate (0.288 in. OD x 0.25 in. ID x 0.75 in. length - wall thickness = 0.038 in.)
- Tube was extruded at 815ºC with 4:1 ratio with no cracking at Case Western Reserve University

- Final dimensions of tube was 0.14 in. OD x 0.12 in. ID x 2.85 in. length – wall thickness = 0.01 in. (0.25 mm)
- Additional tubes are being produced through hydrostatic extrusion and other processing methods such as pilger processing.
Hydrostatic Extrusion Processing Path

LANL-ORNL-UCSB

larger US 14YWT heat of Y-Ti-O added Fe14Cr ATI

- Hydrostatic extrusion shear dominated processing alters texture ($\alpha \rightarrow \gamma$), suppresses brittle crystallography & microcracking!

14Cr-Ferritic ODS steel (US 14YWT) tube is now available.
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Age-hardening Ratio ($\Delta \sigma_y / \sigma_0$)

D.S. Chen, A. Kimura, IAE, Kyoto U.

- Age-hardening ratio, $\Delta \sigma_y / \sigma_0$:
  1) SUS430 (Fe-16Cr-0.74Mn) shows a remarkable age-hardening.
  2) ODS steels show very small age-hardening.

- There is a linear relationship between age-hardening and ductility loss in SUS430, while no such a relationship in ODS steels.
TEM-EELS for Cr-rich Phase Identification

D.S. Chen, A. Kimura, IAE, Kyoto U.

- Oxide particles are quite stable during thermal aging at ~ 475 °C.
- The α’ precipitates can be finely characterized by TEM/EELS, while the oxide particles can also be distinguished.

### Table: Grain size, µm

<table>
<thead>
<tr>
<th>Grain size, µm</th>
<th>Shorter</th>
<th>Longer</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.2</td>
<td>0.7</td>
<td>0.45</td>
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</tbody>
</table>

15Cr-ODS, aged at 475 °C for 10,000 h

(a) Cr, pre-edge image
(b) Cr M\textsubscript{2,3} element map
(c) Ti L\textsubscript{2,3} element map
(d) Y M\textsubscript{4,5} element map
Large oxide particles have a shell at their peripheral, which mainly contains Cr atoms.

The interfaces of oxides/matrix provide precipitation sites for Cr and reduce the potential of age-hardening.
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pilgering processes 2 way studied

**VMR:**

**Industrial rolling device (VN)**

- Non-axisymmetric grooves
- Axisymmetric conical mandrel

**Dies require special expertise and equipment**

**HPTTR:**

**Laboratory rolling device (CEA)**

- Axisymmetric grooves
- Axisymmetric cylindrical mandrel

**Dies are relatively simple to design and build**

(VMR: Vertikaler Massenausgleich Ringwalze in german or mil offsetting vertical grooves of the masses)

(HPTTR: High Pressure Tube Reduction)
2°) Few results on ODS alloys – tube fabrication

**Fabrication route for ferritic ODS**

- Efficiency of the very high temperature heat treatment
  - Higher cold deformability (lower hardness)
  - Easier manufacturing, but small deformation rate
- **BUT** anisotropic microstructure even after heat treatment
  - Difficult to trigger the recrystallization
  - Grains elongated in the rolling direction

<table>
<thead>
<tr>
<th>Fabrication route</th>
<th>Hardness HV1</th>
<th>Mother Tube</th>
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</thead>
<tbody>
<tr>
<td>IHT</td>
<td>~20%</td>
<td>CR</td>
</tr>
<tr>
<td>HT</td>
<td>~20%</td>
<td>CR</td>
</tr>
<tr>
<td>HT</td>
<td>~20%</td>
<td>CR</td>
</tr>
<tr>
<td>CR</td>
<td>~50%</td>
<td>FHT</td>
</tr>
</tbody>
</table>

IHT: Initial Heat Treatment 1250°C/30min
HT: Intermediate Heat Treatment 1200°C/1h
Slow cooling rate (80°C/h)
FHT: Final Heat Treatment 750°C/30min

*Microstructural and mechanical characterization of the cladding tube*
2°) Few results on ODS alloys – tube fabrication

- Fabrication route for Martensitic ODS
  - Cold rolling passes punctuated by intermediate heat treatments performed in the austenitic domain

Safe manufacturing:
No risk of damage during fabrication route

<table>
<thead>
<tr>
<th>Hardness HV</th>
<th>Mother Tube</th>
<th>HT</th>
<th>HT</th>
<th>FHT</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>300</td>
<td></td>
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</tr>
<tr>
<td>400</td>
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<tr>
<td>500</td>
<td></td>
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</table>

- Hot extruded mother tube annealed at 1050°C/1h
- After ~40% cold rolling
- Phase transformation \( \alpha / \gamma \)
- After intermediate heat treatment performed at 1050°C (in the austenitic domain)

Effective softening
Higher deformation rates of CR
Recrystallization Behavior

Y.S. Ha, A. Kimura, IAE, Kyoto U. (Po 2-17)

Recrystallization behavior is influenced by:

- Grain size
- Dislocation density
- Particle size

Microstructure/hardness correlation

![Graphs and images showing hardness vs. temperature for Al-free and Al-added 15Cr ODSS with 0, 20%, 40%, and 80% cold work.](image-url)

<table>
<thead>
<tr>
<th></th>
<th>As-received</th>
<th>80% Cold Work</th>
</tr>
</thead>
<tbody>
<tr>
<td>No heat</td>
<td>Recrys. (1350°C)</td>
<td>No heat</td>
</tr>
</tbody>
</table>

**Oxide Particles**

- Size: 20nm
- No heat
- Recrys. (1350°C)
- Recrys. (850°C)

**Dislocations**

- Size: 20nm
- No heat
- Recrys. (1350°C)
- Recrys. (850°C)
The contribution of Orowan strengthening factor is estimated rather large on the basis of a simple summation of each factor.

The grain size dependence of the total stress, $\sigma_y$, reflects the change in both the grain size and the distribution morphology of oxide particles.
Recrystallization Effect on Irr. Hardening

Y.S. Ha, A. Kimura, IAE, Kyoto U.

15Cr-2W-3Al ODSS

Ion-irradiation (DuET)
- Dual ions: 1.7 MV Fe^{3+} + 1 MV He^+
- Damage: 30 dpa at 600 nm
- Temperature: 470°C
- He injection: 450 appm
- Flux: 4 x 10^{-4} dpa/s

- Recrystallization induced a larger irradiation hardening, but the following cold work reduced the hardening.

As-received
Recrystallized
Recrystallized and CW

Displacement (nm)

0 50 100 150 200 250 300 350 400 450 500

Hir-Hunir

0 0.5 1.0 1.5 2.0 2.5

dpa
Recrystallization Effect on Microstructure

Y.S. Ha, A. Kimura, IAE, Kyoto U.

<table>
<thead>
<tr>
<th></th>
<th>Dislocations</th>
<th>Oxide particles</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>As-received</strong></td>
<td><img src="487x421" alt="Image" /></td>
<td><img src="481x312" alt="Image" /></td>
</tr>
<tr>
<td></td>
<td>100 nm</td>
<td>100 nm</td>
</tr>
<tr>
<td><strong>Recrystallized</strong></td>
<td><img src="498x438" alt="Image" /></td>
<td><img src="438x356" alt="Image" /></td>
</tr>
<tr>
<td></td>
<td>100 nm</td>
<td>100 nm</td>
</tr>
<tr>
<td><strong>Recry. + cold rolled</strong></td>
<td><img src="700x146" alt="Image" /></td>
<td><img src="3x13" alt="Image" /></td>
</tr>
<tr>
<td></td>
<td>100 nm</td>
<td>100 nm</td>
</tr>
</tbody>
</table>

**As-received ODSS**
- Interfaces of matrix/oxide particles are trapping sites for He bubble formation.
- No cavity was observed.

**Recrystallized ODSS**
- Grain boundary area and interfaces of matrix/oxide particles (trapping sites) is reduced.
- Oxide particles trap He bubbles. Some of the large cavities are spherical as well as having faceted shapes.

**Recrystallization Effect on Microstructure**

Grain boundaries play a role to trap vacancies and suppress swelling.

**Recry. + cold rolled ODSS**
- Oxide particles and bubbles distribution is similar with that of recrystallized ODSS.
- Dislocations suppress bubble formation.
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Neutron Irradiation Hardening

RAFS: 9Cr-2W F/M steel (FFTF/MOTA irradiation, PNNL/USA)
ODSS: 9/12Cr-2W steels (JOYO irradiation, JAEA/Japan)
ODS EUROFER after Neutron irradiation: Substantial improvement of tensile properties

**RAFM Steel**

- Substantial irradiation hardening
- Early strain localization due to dislocation channeling $\rightarrow A_u \sim 0.3\%$

**RAFM-ODS Steel**

- Still work hardening $\rightarrow$ almost no loss of uniform elongation ($A_u \sim 7\%$)

![Graphs showing stress-strain curves for Eurofer 97 and ODS-Eurofer HIP steel.](image)
Mechanism of No-Loss-of-Elongation (NLE)

Ferritic/Martensitic steels
Hardening and loss of elongation

1) Absorption of defects
2) Softening
3) Continuous activation of source 1
4) Channeling
   → Localized deformation
   → Loss of elongation

ODS steels
Hardening and no loss of elongation

1) Absorption of defects
2) Dislocation pile-up at oxide particles
3) Suppress the activation of source 1
4) Activate another source 2, 3, ⋯ n
   → Homogeneous deformation
   → No loss of elongation

Oxide particle suppress dislocation channeling, resultantly loss of elongation.
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Steady Swelling Rate in Ferritic Steel

Voyevodin, Garner and Maloy, et al.

Like austenitic steels, a ferritic steel also shows a rather large swelling rate above 150 dpa.

Void swelling is larger in ferrite grain than sorbite grain.

→ How about in ODS steels (Martensitic ODS, ferritic ODSS)

Duplex FM alloy EP-450, 300 DPA, 480°C, 1.8 MeV Cr⁺
Small Void Swelling

by Voyevodin (KIPT), Garner, et al.

Fine grains in ODSS

Void Swelling (%) vs Dose (dpa)

- MA957 ODSS-1
- 14YWT ODSS-2

0.2% / dpa

Ferritic ODSS

Fine grains in ODSS
Elongated grains were produced by drawing.
Swelling resistance arises primarily from dispersoid pinning of grain boundaries, leading to stable denuded zones.
Oxide particles suppress grain growth and supply a number of grain boundaries that absorb vacancies.

Large grain boundary area of ODS steel is effective to trap vacancies up to 500 dpa.

Swelling of MA-957 after 1.8 MeV Cr ion irradiation at 450°C and 500 dpa (KIPT-Ukraine)

Toloczko, Garner et al. JNM 453, 2014
Martensite shows higher resistance than ferrite, which would be due to difference in the coherency of P/M.
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Phase Stability under Ion-irradiation


Fe18Cr-Y2O3 ODS alloy
- sizes >2 nm: pyrochlore-type
- 500 °C, 150 dpa
→ Ostwald ripening

- 500 ºC up to 74.4 dpa
- 500 °C up to 150 dpa

Slight change
Stability of the nano-phases under irradiation
Neutron irradiation

The increase in size and a decrease in density of the finely dispersed \( \text{Y}_2\text{Ti}_2\text{O}_7 \) nanoparticles are observed under ion and neutron irradiation.

\[ \rightarrow \text{Radiation-induced Ostwald ripening} \]

It is so slow under neutron irradiation that nano-phases between 400°C and 650°C can be considered as stable for the new generation of ODS.

\[ \text{Y}_2\text{Ti}_2\text{O}_7 \text{ grows a little under high dose irradiation like Ostwald ripening.} \]
HR-TEM/FFT of 15Cr-4Al-0.6Zr-ODSS

T. Takayama, A. Kimura, IAE, Kyoto U. (Po 2-19)
Hydrogen promotes de-cohesion at the oxide particle/matrix interface under the action of mechanical loading.

Trapping is similar to the carbides in Cr-Mo steels.

Hydrogen attack would be critical if high content of hydrogen exists under high applied stress or strain.

Hydrogen promotes de-cohesion at the oxide particle/matrix interface under the action of mechanical loading.

Trapping is similar to the carbides in Cr-Mo steels.
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**ARROS: Advanced Radiation Resistant ODS Steel**

- **Fe**: Bal.
- **C**: 0.12
- **Cr**: 10
- **Mn**: 0.6
- **Mo**: 1
- **V**: 0.2
- **Ti**: 0.25
- **Y$_2$O$_3$**: 0.35

### Properties

- **FM phase**
- **High temp. stability**
- **Chemical resistance**
- **S. Solution**
- **High temp. strength**
- **Fine complex oxides**
- **Dispersion strengthening**
- **High temp. strength**

**10Cr-1Mo ODS steel**

**Homogenous nano-oxide particle distribution in martensitic matrix**
Fabrication of ODS steel tubes

Pre-alloyed powder
Fe-10Cr-1Mo-0.12C-TiMnV

+ Y2O3 powder

High energy horizontal mechanical alloying (Simoloyer CM20)

Uniform and fine MA powder

Hot extruded ODS rods and mother tubes

Hot extrusion process
Creep properties of ODS alloy

- **FM steel (Gr. 91, Gr. 92, HT9)**
  - Good swelling resistance
  - Poor creep strength

- **Austenitic steel**
  - Good creep strength
  - Severe irradiation swelling

- **FM ODS steel**
  - 10Cr-1Mo-MnVCTiY$_2$O$_3$
  - Superior creep resistance
  - 700°C/120MPa, 10,000h evaluation on-going
Candidates for joining of ODS steels

Magnetic Pulse Welding

- **Joining condition**
  - Capacitance: 826 µF
  - Voltage: max. 15 kV
  - Electric energy: max. 60 kJ

  ![Joining condition diagram](image)

- **Tube**: FM ODS steel
- **End plug**: FM steel
- **FSW tool material**: Inconel 718
- **FSW tool size**: 3.5 mm
- **Tool rotation speed**: 1200 rpm
- **Jig rotation speed**: 1.5 rpm

Friction Stir Welding

- **Joining condition**
  - Tube: FM ODS steel
  - End plug: FM steel
  - FSW tool material: Inconel 718
  - FSW tool size: 3.5 mm
  - Tool rotation speed: 1200 rpm
  - Jig rotation speed: 1.5 rpm

  ![Joining condition diagram](image)

- **Hoop strength (at room temperature)**
  - Joint: 740 MPa (66% of tube)
FSW R&D of 15Cr-ODS Steel

W.T. Han, IAE, Kyoto U., H. Serizawa, IWJ, Osaka U.

FSW of 1.5 mmt plates

FSW resulted in the grain growth but not significant changes in particle size, resulting a softening at RT but not elevated temperatures.

Creep tests are required.
The stir tool should be plugged into F82H to achieve better joining.

**FSW, Dissimilar 15Cr-ODSS/F82H**

W.T. Han, IAE, Kyoto U., H. Serizawa, IWJ, Osaka U.

**Specimens: 35 x 10 x 1.5mm**

12 mm shoulder dia., 4 mm pin dia., 1.3 mm pin leng.

<table>
<thead>
<tr>
<th>FSW 1</th>
<th>WC based</th>
<th>Tilted angle: 3°</th>
</tr>
</thead>
<tbody>
<tr>
<td>200rpm In ODS</td>
<td></td>
<td></td>
</tr>
<tr>
<td>FSW 2</td>
<td></td>
<td></td>
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<tr>
<td>200rpm In F82H</td>
<td></td>
<td></td>
</tr>
<tr>
<td>FSW 3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>250rpm In ODS</td>
<td></td>
<td></td>
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<tr>
<td>FSW 4</td>
<td></td>
<td></td>
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<tr>
<td>250rpm In F82H</td>
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</table>

W.T. Han, IAE, Kyoto U., H. Serizawa, IWJ, Osaka U.
FSW, Dissimilar 15Cr-ODSS/F82H

W.T. Han, IAE, Kyoto U., H. Serizawa, IWJ, Osaka U.

FSW of ODS steel and dissimilar joint with F82H are high in feasibility.

F82H: $AC_1 \approx 840 \, ^\circ C$; $840 \, ^\circ C < T_{peak} < 970 \, ^\circ C$
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Hydrogen Explosion

Reaction with high temperature steam
\[ \text{Zr} + 2\text{H}_2\text{O} = \text{ZrO}_2 + 2\text{H}_2 + 587\text{kJ/mol} \]
\[ \text{Fe} + 1.5\text{H}_2\text{O} = 0.5\text{Fe}_2\text{O}_3 + 1.5\text{H}_2 + 27\text{kJ/mol} \]

Source of hydrogen:
1) Water decomposition by the reaction of zircaloy, SS, Inconel, UO\textsubscript{2}, B\textsubscript{4}C with water (severe accident)
2) Radiolysis of water and coating/zinc plating/Al-water reactions in containment
Why strengthening?

Reaction cross section of Fe is not small like Zr → Needs to be thinner than Zr

ATF cladding requires high strength and high oxidation resistance.
Conclusion

1. Fracture toughness of ODS steel has been improved by thermo-mechanical treatment.

2. Aging embrittlement of ODS ferritic steel is much less than non-ODS ferritic steel with same amount of Cr.

3. High dose irradiation experiments have rather confirmed radiation tolerance in ODS steels.

4. Welding technology of ODS steels is developing in France, Korea and Japan with showing signs of high feasibility.

5. Recrystallization control is critical for ODS ferric steels.

6. Hydrogen embrittlement is another issue.

Several critical issues of ODS steels have been solved, while new issues are arising. Powder metallurgy still has high potential of innovation in materials science and technology.
Conclusion

Present study includes the result of “Super ODS steels R&D towards highly efficient nuclear systems” entrusted to “Kyoto University” by the Ministry of Education, Culture, Sports, Science and Technology of Japan (MEXT).

Present study includes the result of “Development of corrosion resistant ODS steels for high burn-up fuel claddings” entrusted to “Kyoto University” by the Ministry of Economy, Trade and Industry (METI).

Present study includes the result of “Accident Tolerant Fuel Claddings R&D with high Aluminum” entrusted to “Hokkaido” by the Ministry of Education, Culture, Sports, Science and Technology of Japan (MEXT).
Comparison of H₂O Decomposition Behavior between Zircalloy and SS

\[
\begin{align*}
\text{Zr} + 2\text{H}_2\text{O} &= \text{ZrO}_2 + 2\text{H}_2 + 587\text{kJ/mol} \\
\text{Fe} + 1.5\text{H}_2\text{O} &= 0.5\text{Fe}_2\text{O}_3 + 1.5\text{H}_2 + 27\text{kJ/mol}
\end{align*}
\]

Substitution of Zr with Fe causes the reductions of
1) Heat of reaction
2) Hydrogen generation

APMT (FeCrAlMo alloy) → Reduction of 3 orders of magnitude

→ FeCrAl-ODSS (ATF cladding)
  - Reduction of H₂
  - Elevation of strength