Advanced Materials for Fusion Technology

S.J. Zinkle$^1$ and A. Kohyama$^2$

$^1$Oak Ridge National Laboratory, Oak Ridge, TN USA
$^2$Institute of Advanced Energy, Kyoto University, Kyoto, Japan

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OUTLINE

• Development of Improved Materials
  – Advanced steels, including Nanocomposited ferritic steel
  – Refractory alloys (V, Mo, W alloys)
  – New welding technology
  – Ceramic composites

• Brief comments on prospects for improved Cu alloys and nonstructural materials
INTRODUCTION

• Major design criteria for structural alloys include
  – Resistance to He embrittlement & swelling from \((n,\alpha)\) reactions
  – High temperature strength
  – Low temperature radiation embrittlement resistance
  – Safety and environmental (disposal) issues

• Major design criteria for ceramic composites include
  – Thermal conductivity degradation
  – Reduced-cost fabrication and joining techniques
  – Safety and environmental (disposal) issues
Low uniform elongations occur in many BCC and FCC metals after low-dose irradiation at low temperature.

Uniform elongation of neutron-irradiated V-4Cr-4Ti

Uniform elongation of neutron-irradiated GlidCop Al25 and CuCrZr

Damage Level (dpa)

Test Temperature is Irradiation Temperature

Unirradiated elongations

Fabritsiev et al (1996)
Heinisch et al (1992)
Singh et al. (1995)
Irradiated Materials Suffer Plastic Instability due to Dislocation Channeling
Radiation-induced Tensile "Embrittlement" does not Necessarily Produce Fracture Toughness Embrittlement
Application of Thermal Defect Resistance Model to Predict Conductivity of Irradiated SiC

\[
\left[ K(T) \right]^{-1} = \left[ \frac{1}{K_u(T)} + \frac{1}{K_{gb}(T)} + \frac{1}{K_{d0}} + \frac{1}{K_{rd}} \right]
\]

- Maximum irradiated thermal conductivity for SiC is estimated to be ~ 10 W/m-K at 500°C, ~37 W/m-K at 700°C.
Current Alloy Systems Have Key Limitations

- **V-4Cr-4Ti Alloy**
  - Thermal creep limits
  - Low temperature radiation hardening
  - Possible He embrittlement
  - Requires MHD coating
  - Poor oxidation resistance

- **Ferritic/Martensitic Steels**
  - Low temperature radiation hardening
  - Thermal creep limits
  - Possible He embrittlement

- **Refractory Alloys**

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Operational Temperature Windows

S.J. Zinkle and N.M. Ghoniem (2000)
Key Feasibility Issues for Ferritic Steels

- Verify ferromagnetic structures are acceptable for MFE
- Expand low temperature operating limit (experiments and physical modeling, master curve methodology)
  - Development of alloys with improved resistance to low temperature (<350°C) embrittlement
- Expand high-temperature and dose limits
  - Alloy development, including dispersion strengthened alloys
  - Effect of He on creep rupture
- Resolve system-specific compatibility issues (T barrier development, etc.)

IEA-integrated worldwide ferritic steel program is examining items 2 & 3 (items 1&4 are being addressed by JA and EU programs)

Note: reduced-activation grades of Fe-9Cr ferritic/martensitic steels (e.g., F82H) have been developed with superior properties compared to commercial steels (e.g., HT9)
Void Swelling of Ferritic Steels is Low up to ~100 dpa (~10 MW-yr/m²), Although Further Work is Needed to Examine He Effects

HFIR irradiation at 400°C to 51 dpa

F82H (36 appm He)  

10B-doped F82H (330 appm He)
Potential New Ferritic/Martensitic Alloy

• Dispersion-Strengthened Fe-9.5Cr-3Co-1Ni-0.6Mo-0.3Ti-0.07C steel
  – High number density of nano-size TiC precipitates

• Superior elevated-temperature strength and impact properties compared to conventional 9-12Cr steels

• Advantage over ODS steels: produced by conventional steel processing techniques

• Present composition not for nuclear applications; processing technique applicable to reduced-activation compositions

Oxide Dispersion Strengthening Approach

• **Perspective**
  • +50 year history

• **Benefits**
  • any desired combination of matrix composition and dispersoid
  • significant improvements in high temperature mechanical properties

• **Problems**
  • time-consuming & expensive
  • often produces materials with:
    - anisotropic properties
    - coarse particles with non-uniform size and spatial distribution
  • joining and fabrication
  • lack of understanding - experiment, theory, and modeling

Fe-13Cr-3W-0.4Ti + 0.25Y$_2$O$_3$

Fe-9Cr-2W + 0.33TiO$_2$ + 0.67Y$_2$O$_3$
Comparison of Tensile strength of New 12YWT Nanocomposited Ferritic Steel vs. other ODS steels

![Graph showing comparison of tensile strength](image)

**Axes:**
- **Y-axis:** Stress/MPa
- **X-axis:** Temperature K

**Legend:**
- 12YWT
- ODS (SUMITOMO)
- MA956
- 9Cr-2WVTa
- ODS(ZrO$_2$)
- ODS(TiO$_2$)
- ODS(MgO)
- ODS(Al$_2$O$_3$)
Recently Developed Isotropic Oxide Dispersion Strengthened Steels Offer Potential for Improved Performance

- Thermal creep temperature limit for martensitic Fe-8Cr steel is \(~550^\circ C\) (vs. \(>700^\circ C\) for several grades of ODS steel, including Kobe Fe-12Cr-3W-0.4Ti-0.25Y_2O_3)
Nanocomposited 12YWT Ferritic Steel Exhibits Excellent High Temperature Creep Strength

- Time to failure is increased by several orders of magnitude
- Potential for increasing the upper operating temperature of iron based alloys by ~200°C

800°C and 138 MPa
- minimum creep rate $2.13 \times 10^{-10}s^{-1}$
- total strain 2.03%

• 650°C, 1080h, Rupt.
• 600°C, 17000h
• 650°C, 13000h
• 800°C, 817h
• 700°C, 530h, Stop
• 900°C, 1104h
• 800°C, 931h, Stop

Strain (%) vs. Time (h)

LMP T(25+logt), (K-h)

Stress (MPa)

450
400
350
300
250
200
150
100
50
0
22000 24000 26000 28000 30000 32000 34000 36000

14,235h

Oak Ridge National Laboratory
U.S. Department of Energy
Atom Probe Reveals the Presence of Nanoclusters in the Mechanically Alloyed 12YWT Ferritic Alloy

3-DAP Atomic Coordinates

Isocompositional Surface

• Nano-size clusters
  – Average composition (at.%):
    \[\text{O} - 23.6 \pm 10.6\]
    \[\text{Ti} - 19.9 \pm 8.7\]
    \[\text{Y} - 9.2 \pm 7.8\]
  – Size: \(r_g = 2.0 \pm 0.8\) nm
  – Number Density: \(n_v = 1.4 \times 10^{24}/\text{m}^3\)

• Original \(\sim 30\) nm \(\text{Y}_2\text{O}_3\) particles evolve to \((\text{Y},\text{Ti},\text{O})\) enriched nanoclusters

• Nanoclusters not present in ODS Fe-13Cr + 0.25\(\text{Y}_2\text{O}_3\) alloy
Refractory Alloys

- Attractive Thermal/Physical Properties
  - high temperature capabilities
  - thermal stress figure of merit
  - liquid metal compatibility (Li)

- Main problems
  - low fracture toughness
  - low oxidation resistance
  - poor mechanical properties of weldments
Recent research offers promise for developing refractory alloys with improved ductility

• Controlled (50-1600 appm) additions of Zr, B, C to molybdenum increases room temperature ductility of weldments from nearly zero to $e_{\text{tot}} \sim 20\%$ (M.K. Miller and A.J. Bryhan, 2001)

• Mechanically alloyed W-0.3wt%Ti-0.05wt%C (H. Kurishita et al., ICFRM-10, Baden-Baden, Oct. 2001)
  – Avoid (W,Ti)$_2$C brittle phase by limiting max concentration of carbon
  – Small grain size (~2 µm) helps to dilute harmful oxygen grain boundary segregation
  – TiC dispersed particles provide increased toughness (appropriate fracture mechanics tests needed to verify preliminary smooth bend bar results)
HAZ HAZWeld
Fracture occurred predominantly in the heat affected zones
The fracture mode was transgranular cleavage with only small regions of intergranular fracture. This contrasts the intergranular fracture typically found in commercial Mo welds.

ALLOY COMPOSITION

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (appm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zr</td>
<td>1600</td>
</tr>
<tr>
<td>C</td>
<td>96</td>
</tr>
<tr>
<td>B</td>
<td>53</td>
</tr>
<tr>
<td>O</td>
<td>250</td>
</tr>
<tr>
<td>N</td>
<td>178</td>
</tr>
<tr>
<td>Mo</td>
<td>balance</td>
</tr>
</tbody>
</table>

MECHANICAL PROPERTIES

- Strain rate: $8.3 \times 10^{-4} \text{ s}^{-1}$
- Mo- 30% Re filler
- Ductility: 19.5%
- Yield Stress: 481 MPa
- UTS: 544 MPa

Commercial Mo weld: 3% Ductility

Research performed by
M. K. Miller, Oak Ridge National Laboratory and A. J. Bryhan, Applied Materials
ATOM PROBE TOMOGRAPHY REVEALS Zr, B and C SEGREGATION TO THE GRAIN BOUNDARIES

- Base metal: Zr, C and B enrichments and O depletion
- HAZ: Heavy B and moderate Zr enrichments

GIE (atoms m\(^{-2}\))

**Base Metal**
- Zr: \(7.6 \times 10^{13}\)
- B: \(7.3 \times 10^{12}\)
- C: \(1.1 \times 10^{13}\)
- O: \(-3.9 \times 10^{12}\)

**Heat Affected Zone**
- Zr: \(1.3 \times 10^{13}\)
- B: \(9.9 \times 10^{14}\)
- C: \(9.9 \times 10^{11}\)
- O: \(1.1 \times 10^{13}\)

Research performed by M. K. Miller, Oak Ridge National Laboratory and A. J. Bryhan, Applied Materials
Three-point bending stress-strain curves for pure tungsten and developed alloys A and B. Example of the as-rolled sheets.

Comparison of test temperature dependence of total absorbed energy and maximum strength among alloy A, W-0.2wt%TiC and pure tungsten (un-notched bend bar impact tests).

Mechanically alloyed W-0.3wt%Ti-0.05wt%C exhibits good low temperature ductility

H. Kurishita et al., 2001
Comparison of Tensile (Red. in area) and Charpy Impact Ductile-Brittle Transition Behavior of Mo-0.5Ti

- The DBTT is dependent on numerous factors, including strain rate and notch acuity ("tensile DBTT" is not a meaningful design parameter)

J.A. Houck, DMIC Report 140 (1960)
The fusion materials welding program has successfully resolved one of the key feasibility issues for V alloys. 

Success is due to simultaneous control of impurity pickup, grain size

- Results are applicable to other Group V refractory alloys (Nb, Ta)
- Use of ultra-high purity weld wire may reduce atmospheric purity requirements
Motivation for pursuing Friction Stir Welding (FSW)

• A solid-state joining process such as FSW may enable field welding of refractory alloys (V, Mo, W), due to reduced pickup of atmospheric contaminants.

• Irradiated materials with He contents above ~1 appm cannot be fusion-welded due to cracking associated with He bubble growth; the lower temperatures associated with FSW may allow repair joining of irradiated materials.

![Graph showing calculated size of He bubbles at grain boundaries in 316 SS.](image)
Advanced materials can be successfully joined with friction stir welding (FSW) process

- Metal matrix composites (MMC) and oxide dispersion strengthened (ODS) alloys are difficult to join using conventional fusion welding processes.
  - Particle / fiber reinforcement deteriorate in MMCs due to melting.
  - In Al-SiC MMC laser welds, SiC decomposes and forms Al$_4$C$_3$ carbides.

- Friction stir welding (FSW) uses plastic deformation to join materials.
  - Homogeneous microstructure and properties are achieved.
  - SiC fibers were uniformly distributed.

Sponsor: DOE Office of Transportation Technologies
Office of Heavy Vehicle Technologies
Silicon Carbide Composite Development

Silicon carbide composite is the least-developed of the 3 main structural materials being studied in the Fusion Materials Program, but it has the greatest potential for Very Low Radioactivation - Very High Temperature Use.

Areas being actively studied:

- Radiation Hardened Composite Development
- Effects of Helium on Mechanical Properties
- Radiation Degradation of Thermal Conductivity
- Swelling, Amorphization and Defect Fundamentals

Matrix

Fiber

Interphase

Matrix
Development of Radiation-Resistant SiC Composites

Until recently, SiC/SiC composites exhibited significant degradation in mechanical properties due to non-SiC impurities in fibers causing interfacial debonding.

Upon irradiation, if fibers densify, fiber/matrix interfaces debonds --> strength degrades
We Now Have First Radiation-Resistant SiC Composite

Bend strength of irradiated “advanced” composites show no degradation up to 10 dpa

1st- and 2nd generation irradiated SiC/SiC composites show large strength loss after doses >1 dpa
High Thermal Conductivity SiC/G Composites offer potential for improved thermomechanical performance

<table>
<thead>
<tr>
<th>Fiber</th>
<th>K-1100</th>
<th>P-55</th>
<th>Nicalon</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type-S</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Kth (W/m-K@RT)</td>
<td>~950</td>
<td>120</td>
<td>15</td>
</tr>
<tr>
<td>Diameter (micron)</td>
<td>10</td>
<td>10</td>
<td>13</td>
</tr>
<tr>
<td>Tensile Strength (GPa)</td>
<td>3.1</td>
<td>1.9</td>
<td>2.6</td>
</tr>
<tr>
<td>Tensile Modulus (GPa)</td>
<td>965</td>
<td>379</td>
<td>420</td>
</tr>
<tr>
<td>Density (g/cc)</td>
<td>2.2</td>
<td>2.0</td>
<td>3.2</td>
</tr>
</tbody>
</table>

Predicted irradiated conductivity of SiC/G is higher than monolithic SiC
New Developments in SiC/SiC Fabrication: Nano-Powder Infiltration and Transient Eutectoid (NITE) Process

Institute of Advanced Energy, Kyoto University

- **Reinforcement**
  - Tyranno™-SA grade-3 (Ube Industries, Ltd.)
  - Uni-directional
  - PyC coating, 800nm-thick nominal

- **Matrix raw materials**
  - Beta-SiC Nano-powder (110m²/g) and submicron (~40m²/g) powders
  - Al₂O₃-Y₂O₃ complex as sintering additive
  - Pre-ceramic polymer inclusion for intra-bundle densification

- **Transient eutectoid process**
  - Uni-axial hot-pressing
  - \( T_p \leq 1800°C \)
  - \( P_p \leq 20\text{MPa} \)
Flow Chart of the "NITE" Process

Institute of Advanced Energy, Kyoto University

Carbon coated Tyranno SA fiber tows

Pre-forming through winding

Pre-treatment by PIP

Matrix coating by mixed slurry

Drying and stacking

Hot pressing

Characterization
“NITE”-Fabricated SiC/SiC

Institute of Advanced Energy, Kyoto University
Microstructure of “NITE”-Fabricated SiC/SiC

Institute of Advanced Energy, Kyoto University
Fabrication Cost of SiC/SiC by Various Processes

- Estimated Mass-production Fiber cost Tyranno™-SA
- Fiber cost As of Yr.2001 Nicalon™-CG
- Fiber cost As of Yr.2001 Tyranno™-SA

- NITE Mass-prod.
  - NITE (Kyoto U.)
- CVI (Mass-Prod.)
  - CVI (Lab-grade)
- Adv. RS/MI (Mass-prod.)
- Adv. RS/MI (Lab-grade)
- Adv. PIP (Mass-prod.)
- PIP
- RS/MI
- Conventional

Production cost (k$/kg) vs. Total Performance

Institute of Advanced Energy, Kyoto University
Permeability of He in SiC/SiC

Institute of Advanced Energy, Kyoto University

Conventional SiC/SiC and Bonded-SiC-Fiber Ceramics

Tubes by LPS (Kyoto University)
Comparison of Thermal Conductivity of SiC/SiC

Institute of Advanced Energy, Kyoto University

Thermal conductivity / W/m-K

Temperature / C

TySA/PyC/NITE-SiC (1780/20)
TySA/PyC/NITE-SiC (1800/15)
Monolithic-SiC (1780/15*)

CVI-SiC/SiC
Mechanical behavior of a wide range of copper alloys has been investigated vs. strain rate and temperature (constitutive equations for deformation and fracture)

- CuNiBe has superior properties below 100°C; CuCrZr and Cu-Al₂O₃ have best properties at intermediate temperatures
- high temperature limits in CuNiBe and Cu-Al₂O₃ alloys are associated with grain boundary phenomena
Mechanical behavior of copper alloys can be understood on the basis of current materials science models of deformation.

Applications to US industry (e.g., USCAR) as well as fusion energy sciences program.
## Optical fibers for ITER round robin irradiation tests

<table>
<thead>
<tr>
<th>Team</th>
<th>Fiber Type</th>
<th>Supplier</th>
<th>Diameter(µm)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>RF</td>
<td>KS-4V</td>
<td>FORC</td>
<td>200</td>
<td>Pure silica core, OH &amp; Cl free</td>
</tr>
<tr>
<td></td>
<td>KU-1</td>
<td>FORC</td>
<td>200</td>
<td>Original, OH:800ppm</td>
</tr>
<tr>
<td></td>
<td>KU-H2G</td>
<td>FORC</td>
<td>200</td>
<td>Improved, Hydrogen treated OH:800ppm</td>
</tr>
<tr>
<td>JPN</td>
<td>F F</td>
<td>Fujikura Ltd.</td>
<td>200</td>
<td>Fluorine doped silica core, OH-free</td>
</tr>
<tr>
<td></td>
<td>M F</td>
<td>Mitsubishi Cable</td>
<td>200</td>
<td>Fluorine doped silica core, OH-free</td>
</tr>
</tbody>
</table>

*T. Kakuta et al., ICFRM11 (2001)*
Comparison of increased absorption in ITER round robin fibers at gamma-ray irradiation of 1.9e6 Gy.

T. Kakuta et al., ICFRM11 (2001)
High loss in MF fiber may be due to neutron radiation-induced microcracking

T. Kakuta et al., ICFRM11 (2001)
Conclusions

• There is a strong prospect for improvements in the capabilities of fusion structural materials based on ongoing research
  – Nanocomposited ferritic steels
  – Ductile Mo and W alloys
  – Hermetic, high conductivity, radiation-resistant, lower cost SiC composites
• Improved joining techniques are being applied to fusion materials
  – Gas tungsten arc welding of V alloys
  – Friction stir welding
• Materials which can be categorized as “reduced-activation” have properties comparable or superior to their commercial (high-activation) counterparts
  – e.g., Fe-9Cr ferritic/martensitic steels developed for fusion
• Additional screening studies are needed to identify the most promising nonstructural materials for fusion (organic insulators, optical materials, etc.)