The Effect of Neutron Irradiation on Candidate First Wall and Diverter Materials

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Plasma Surface Interaction

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Thanks to: Yuri Osetskii (ORNL)
Maria Caturla (Universidad de Alicante)
Meimei Li (ANL)
Vertical Target
- PFC: either W or Carbon Fiber Composite
- Heat Sink: CuCrZr alloy
- Structure: 316 LN(I)-G steel alloy

Dome
- PFC: W
- Heat Sink: CuCrZr alloy
- Structure: 316 LN(I)-G steel alloy

Tile
- Pure Beryllium
Neutron Cascade Damage and Defect Evolution

neutron-
copper
collision
Before we proceed ……a definition……
DPA = displacement per atom
Typical reactor application = 1-10 DPA / year
1 DPA Ceramic ~ 1 x 10^{25} n/m^2 E>0.1 MeV

Lattice Displacement Energies

<table>
<thead>
<tr>
<th>Material</th>
<th>Energy (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Graphite</td>
<td>25</td>
</tr>
<tr>
<td>Be</td>
<td>25</td>
</tr>
<tr>
<td>Iron</td>
<td>40</td>
</tr>
<tr>
<td>Tungsten</td>
<td>80</td>
</tr>
</tbody>
</table>

A 100 keV imparted energy
Will produce thousands of displaced atoms!
Neutron Irradiation Damage in Fusion

High energy neutron interaction with structural materials in fusion reactors have two broad effects:

1) Creation of displacement "cascades" (very high temperature, radical mixing of atoms) which happen very quickly. This has a range of effects microstructure and mechanical properties of materials (not very good.)

2) Creation of transmutant elements (Helium, lithium, hydrogen…) which also don’t help material performance

MD simulation of He-bubble formation in tungsten at 600 K (T. Suzuda, JAEA)

Helium Bubbles in Tungsten Matrix

$T_{irr} = 550 \, ^\circ\text{C}, 1 \, \text{dpa}, 5000 \, \text{appm} \, \text{He}$

Helium Bubbles in Tungsten Matrix

Bubbles at grain boundary

P. Jung, FZJ
Five Evils of Radiation Damage (in Metals)

- Radiation hardening & embrittlement
  \((<0.4 \ T_M, >0.1 \ dpa)\)

- Phase instabilities from radiation-induced precipitation
  \((0.3-0.6 \ T_M, >10 \ dpa)\)

- High temperature He embrittlement
  \((>0.5 \ T_M, >10 \ dpa)\)

- Volumetric swelling from void formation
  \((0.3-0.6 \ T_M, >10 \ dpa)\)

- Irradiation creep
  \((<0.45 \ T_M, >10 \ dpa)\)
Evils of Radiation Damage (in Ceramics…)

Graphite Sublimes at > 3000 K

- Point Defect Swelling and Amorphization
  ($<0.3 \ T_M, < 5 \ dpa$)

- Radiation Induced Thermal Conductivity Degradation
  ($<0.4 \ T_M, < 5 \ dpa$)

- Volumetric swelling from void formation
  ($0.4-0.6 \ T_M, > 4 \ dpa$)

- Irradiation creep ($<0.45 \ T_M, > 10 \ dpa$)
Operating Range, Irradiated Structural Materials

- C/C: thermal conductivity limited
- SiC/SiC: thermal conductivity limited
- Tungsten: Limited by embrittlement
- Molybdenum: Limited by embrittlement
- ODS Ferritic: Limited by embrittlement
- F/M Steel: Limited by embrittlement
- 316 Stainless: Limited by embrittlement
- Alloy 718: Limited by embrittlement
- CuCrZr: Questionable
- Beryllium: Reasonable

Operating Temperature (°C)

0 200 400 600 800 1000 1200 1400 1600
Copper for Nuclear Application

- Copper has been used extensively in tokamak application for heat sink application.

- Annealed, pure copper, is extremely weak at both room and elevated temperature. Solid solution, precipitation hardened, or dispersion strengthened copper alloys are used to increase strength.

- Neutron irradiation-produced defects cause significant embrittlement in copper.
Defect Production and Annihilation in Irradiated Materials

- strong function of material, time, and temperature...

Legend:
- Displaced atoms
- Vacant sites

Copper 25keV:
- Cube faces (100)
- Cube size = 25nm
- 2,048,000 atoms
- Temperature = 100K
- time = 0.005ps
- NFP = 1

- vacancy
- interstitial
A large non-perfect SFT and several SIA clusters/loops are formed.
Defect cluster microstructure in Cu irradiated to 1 dpa at low temperature (~90°C)

Dislocation dynamics and in-situ TEM of dislocation SFT interaction

Yuri Osetskii, ORNL

Yoshi Matsukawa, ORNL (now U. mich)
Irradiation Hardening/Embrittlement
- copper -

![Graph showing stress-strain relationship for non-irradiated and irradiated copper.](image)

- Stress vs. temperature (K)
- Strain vs. temperature (K)

4.1nm SFT-edge
- Apex up
- Apex down
- 3nm void

CuCrZr SAA

- Irrad
- 0.14 dpa
- 1.5 dpa

Temperature: $T_{irr} = 80^\circ C$, $T_{test} = 20^\circ C$

Micrograph of copper at 0.01 dpa with i-loops at 25 nm scale.
Irradiation Hardening and Embrittlement

• Irradiation produced defects serve as “road blocks” to the dislocation motion required for deformation (plasticity.)

• Defects can be formed either directly within the cascade or can develop (mature) upon diffusion of interstitial/vacancy species following cascade:

  SFT in Cu
  “in cascade”

• Immobile defects formed directly within the cascade are extremely troublesome, as we have no “metallurgically fix”.

  - typically, higher Z metals have higher fraction of “in cascade defects”

• However, migrating defects (or transmutants such as He) can be dealt with by various means (alloying, dispersion strengthening, etc.)
CuCrZr for ITER Application

- Copper alloys have significantly higher strength than pure copper, and still retain good conductivity

- Three copper alloys were considered:
  - Precipitation-hardened CuCrZr
  - Precipitation-hardened CuNiBe
  - Oxide-dispersion-strengthened (GlideCop Al-25)

- CuCrZr was selected due to its high fracture toughness, availability and low cost.

- ITER grade CuCrZr:
  - Cu – (0.60-0.90%)Cr – (0.07-0.15%) Zr
CuCrZr -vs- Pure Copper

- Precipitation hardening of CuCrZr alloy extends useful life of copper. Embrittlement and “plastic instability” stills occurs, but at a much higher level of dose

Channels formed in CuCuZr similar to the case in OFHC Cu, except that CuCrZr channels were not free of dislocations and defects

CuCrZr Tensile Properties Under Irradiation

- CuCrZr loses its uniform elongation rapidly under irradiation
“Elevated” Temperature Irradiation Results for CuCrZr

- for irradiation in the 0.3-0.6 Tm range, copper embrittlement becomes less of an issue, however, alloy has little strength at these temperatures.
Effect of Neutron Irradiation on Fracture Toughness

- Neutron irradiation effect on fracture toughness is small; Fracture toughness was reduced slightly after irradiation.
- No correlation between tensile ductility loss and fracture toughness

Fracture toughness of CuCrZr remained high up to 1.5 dpa ($J_Q > 200 \text{ kJ/m}^2$)

Biased Summary Comments on Copper Alloys

• Copper and copper alloys has been very well studied, both the fundamentals of the alloys and their irradiation effects. Improvements in both non-irradiated and as-irradiated performance will be incremental.

• Copper alloys are inherently limited in elevated temperature strength. For this reason they may not be attractive alloys beyond ITER.
Tungsten for Nuclear Application

- Currently, tungsten is utilized as a plasma facing “tile” material with only moderate structural requirements. For ITER the requirement for ductility and irradiation resistance is limited.

- As discussed already in this school, the primary advantage of tungsten lies in its good resistance to sputtering (high mass) and high melting temperature (3695 K). However, both of these benefits argue in direct opposition to good irradiation resistance:
  - high mass means higher fraction of defects formed directly within cascade (limiting metallurgical tools to mitigate irradiation effects)
  - the application temperature (<1000°C) is < 0.4 Tm puts the material within the low temperature embrittlement regime

- Currently, the manufacturing technology for tungsten and tungsten alloys has struggled to produced “structural” tungsten
Manufacturing of Present Day Tungsten

Sintering & Forming
- + Mass Production
  + Density
- -/+ Specific, anisotropic microstructure

Powder Metallurgy
- Mechanical Alloying & HIP
  - + Fine Particles
    + Homogenous Microstructure
  - Small Quantities
  - Porosity
  - Brittleness?
- Injection Molding & Sintering/HIP
  - + Mass Production
    + Nearly Finished Products
    + Homogenous Microstructure
  - Porosity
  - Severe Brittleness

Melting & Forming
- + Real Alloying
- Expensive
  (EB, Arc, Vacuum, ...)

Texture of Microstructure Highly Dependent on Process

RODS

<table>
<thead>
<tr>
<th>Material</th>
<th>Diameter</th>
<th>Percent</th>
<th>Process</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>Ø6.9 mm</td>
<td>91%</td>
<td>Rolling</td>
</tr>
<tr>
<td>WL10</td>
<td>Ø6.9 mm</td>
<td>91%</td>
<td>Rolling</td>
</tr>
<tr>
<td>WL10opt</td>
<td>Ø6.9 mm</td>
<td>94%</td>
<td>Swaging</td>
</tr>
<tr>
<td>WVM</td>
<td>Ø16 mm</td>
<td>91%</td>
<td>Rolling</td>
</tr>
<tr>
<td>PW</td>
<td>Ø20 mm</td>
<td>93%</td>
<td>Swaging</td>
</tr>
<tr>
<td>PWL</td>
<td>Ø20 mm</td>
<td>93%</td>
<td>Swaging</td>
</tr>
<tr>
<td>W1Re1La2O3</td>
<td>Ø10 mm</td>
<td>81%</td>
<td>Rolling</td>
</tr>
<tr>
<td>TZM</td>
<td>Ø7 mm</td>
<td>91%</td>
<td>Sw+Rol</td>
</tr>
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50 μm

RD

L-R
Properties Can be Highly Non-Uniform
process ductility has been gained by using additions of materials such as $\text{La}_2\text{O}_3$, which resides at grain boundaries.
Fracture Characteristics of W Rod Materials

- **Rod**: Transition from brittle to ductile behavior for powder metallurgy processed materials, in the non-irradiated condition, is above room temperature. Upon irradiation, the transition temperature would further increase.
- **Plate**: Same situation, though properties are worse.
• Irradiation effects data on tungsten is rather limited, and typically on powder metallurgy samples. Results indicate severe embrittlement at low temperature.
Upon irradiation, the transition temperature for which tungsten goes from brittle to ductile behavior increased to well above room temperature.
Importance of Brittle to Ductile Transition Temperature

The RMS Titanic

<table>
<thead>
<tr>
<th>Composition</th>
<th>Titanic hull plate</th>
<th>A36 modern structural steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.21</td>
<td>0.20</td>
</tr>
<tr>
<td>Mn</td>
<td>0.47</td>
<td>0.55</td>
</tr>
<tr>
<td>P</td>
<td>0.045</td>
<td>0.012</td>
</tr>
<tr>
<td>S</td>
<td>0.069</td>
<td>0.01 to 0.04</td>
</tr>
<tr>
<td>Si</td>
<td>0.017</td>
<td>0.007</td>
</tr>
<tr>
<td>Cu</td>
<td>0.024</td>
<td>0.01</td>
</tr>
<tr>
<td>O</td>
<td>0.013</td>
<td>-</td>
</tr>
<tr>
<td>N</td>
<td>0.0035</td>
<td>0.0032</td>
</tr>
<tr>
<td>Mn:S Ratio</td>
<td>7:1</td>
<td>15:1 (typical)</td>
</tr>
</tbody>
</table>

K. Felkins, H. P. Leighly, and A. Jankovic. JOM, 50(1), 1998, 12-18
Importance of Brittle to Ductile Transition Temperature

Fracture surface of Charpy specimens from Titanic plate

Longitudinal direction

At 120 °C, ductile fracture

At -32 °C, brittle fracture
Effect of temperature on toughness

H. P. Leighly, B. L. Bramfitt, and S. J. Lawrence. Practical Failure Analysis, 1(2), 2001
• Present forms of tungsten undergo significant embrittlement following low dose irradiation. It currently appears that for end of life ITER and higher dose fusion reactors, tungsten alloys will be fully brittle.
Biased Summary Comment on Tungsten

• Tungsten is potentially a very high performance material. However, its current structural applications (any application) are very limited due to its extreme difficulty in fabrication and limited ductility.

• The poor ductility of tungsten is an issue of both the nature of tungsten crystal, and the nature of the processing used (which controls the grain boundary.) Improvements are possible, and the current understanding of how alloying may improve tungsten ductility, is not well understood.

• Irradiation will make the ductility situation even worse. What is currently needed is a combination of fundamental studies on the irradiation effects
  - are in-cascade formed defects a fatal issue for tungsten?
  - can be use solid solution alloying to enhance ductility?
  - are the current nano-composited tungsten alloys going to be effective
Beryllium as an Engineering Material

- Be has only two operating slip planes \{0001\} & \{1010\} significantly limiting the ductility of the metal.
  - $T < 200^\circ\text{C}$, no \{1010\} slip, Ductility $\sim 5\%$
  - $200 < T < 500$, \{1010\} & \{0001\}, Ductility to 50\%
  - $T > 500^\circ\text{C}$, grain boundary failure, Ductility $< 20\%$

- For this reason key engineering properties such as fracture toughness, ultimate strength, and plastic elongation are poor.

- For the non-irradiated case, and especially in the neutron irradiated case, fabrication and design must consider working with a “brittle” material.

Hexagonal Close Packed Crystal

$T_{\text{melt}} = 1560^\circ\text{C}$
SP-100 Design Utilized Fairly Complex Beryllium Shapes with Structural and Non-Structural Functions
Effect of Neutron Irradiation on Be Metal

Defects Produced

- Single vacancy
- Di-vacancy
- Nanovoids (5 nm)
- 5% Li/He conc.

- following cascade, most helium are in the “interstitial” positions, with time the helium finds static vacancies, forming stable complexes and eventually He bubbles.

Troev Res. Let
Phys 2008
Effect of Neutron Irradiation on Be Metal

The primary effects of neutron irradiation on metallic Be: swelling and embrittlement
Effect of Neutron Irradiation on Be Metal

The primary effects of neutron irradiation on metallic Be: swelling and embrittlement

\[
\begin{align*}
T_{\text{irr}} & = 372 \text{ K} \\
10^{22} \text{ n/cm}^2, E > 1 \text{ MeV}
\end{align*}
\]

\[
\begin{align*}
T_{\text{irr}} & = 672 \text{ K} \\
10^{22} \text{ n/cm}^2, E > 1 \text{ MeV}
\end{align*}
\]

Low Temperature Swelling due to Defect Accumulation

High Temperature Swelling due to He Accumulation:
\[ \rightarrow B^9(n, \text{He}) \text{ reaction.} \]
Defining an Operating Window for Structural Use of Be

- Operating windows are typically defined using following properties:
  - Creep
  - Fracture toughness
  - Fatigue
  - Ductility limits (plasticity, elongation)
  - Environmental effects

- There are no creep data on modern-day Be, though Larson Miller data exist to estimate the rupture lifetime.

- Design Space Assumptions:
  - Upper stress using extrapolated Larson Miller data.
  - Assume lower bound set by required elongation of 1% total.
Example Fracture Behavior of Beryllium: Fracture Surface

- At elevated temperature, neutron irradiation causes He bubbles to form and embrittle grain boundaries.

- Formation of bubbles and their catastrophic effect on mechanical properties have been slightly improved by material purification, but significant embrittlement is unavoidable.

- Example of Improvement:

  1960’s vintage: $1 \times 10^{24} \text{n/m}^2 \text{E}>0.1 \text{MeV}$
  total elongation < 0.2%

  1990’s vintage: $1 \times 10^{24} \text{n/m}^2 \text{E}>0.1 \text{MeV}$
  total elongation few %
With Neutron Irradiation

- 0.1 dpa
  - $T_{\text{irr}} = 100^\circ\text{C}$
  - $1 \times 10^{24} \text{n/m}^2; E>0.1 \text{ MeV}$
  - 250 appm He

- 2 dpa
  - $T_{\text{irr}} = 400^\circ\text{C}$
  - $2 \times 10^{25} \text{n/m}^2; E>1 \text{ MeV}$
  - ~700 appm

<table>
<thead>
<tr>
<th>DPA Level</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 0.001 dpa</td>
<td>Essentially non-irradiated</td>
</tr>
<tr>
<td>0.01-1 dpa</td>
<td>Embrittlement a factor</td>
</tr>
<tr>
<td>&gt;1 dpa</td>
<td>Embrittlement and Swelling Unavoidable</td>
</tr>
<tr>
<td>- ITER -</td>
<td>- beyond ITER -</td>
</tr>
</tbody>
</table>

Rupture Stress (MPa) vs. Temperature (C)

- 1 year
- 20 year

Tensile Elongation %

1022 n/cm², E>1 MeV
Biased Summary Comments on Beryllium

• Beryllium has been well studied and its use as a plasma facing material requires very high purity materials. Alloying of beryllium will certainly improve mechanical properties, but the consequences on the plasma will likely be unacceptable.

• Improvement in recent years have been largely in the improvement in the grain boundary integrity (removal of oxygen.) Further, incremental improvement is possible, but low temperature embrittlement and helium swelling due to neutron irradiation is unavoidable.
The Graphite Crystal

• The graphite crystal is an interpenetrating hexagonal “benzene” ring structure.

• Very weak bonding between planes, strong bonding in planes.

• Extraordinary in-plane properties, drastically different out-of-plane.

<table>
<thead>
<tr>
<th></th>
<th>In-plane</th>
<th>Out-plane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal Conductivity W/m-K</td>
<td>&gt;2200 W</td>
<td>20</td>
</tr>
<tr>
<td>Thermal Expansion</td>
<td>0.5</td>
<td>6.5</td>
</tr>
<tr>
<td>Strength (MPa)</td>
<td>&gt;1000</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Elastic Modulus(GPa)</td>
<td>20</td>
<td>&lt;1</td>
</tr>
</tbody>
</table>

van der Walls

\[ c = 6.7\text{Å} \]

a=b = 2.45\text{Å}

covalent bonding
Irradiated Graphite Crystal

- Interstitials mobile $> 70$ K, move within the basal plane.

- Vacancies mobile $> 1000$ K, move freely between the basal plane.

- Interstitial-vacancy recombination barrier $< 1$ eV ($< 400$ K).
Simple Defect Recombination in Graphite: Low Temperature

As irradiation temperature exceeds activation energy for recombination, vacancy concentration and stored energy is reduced.
Simple Defect Recombination in Graphite: Low Temperature Stored Energy Release (cal/g-C)

Annealing Temperature (°C)

- 4.2 \times 10^{20} (nv_0)t
- 2.6 \times 10^{20} (nv_0)t
- 1.6 \times 10^{20} (nv_0)t
- 7.7 \times 10^{20} (nv_0)t
- 1.3 \times 10^{20} (nv_0)t

Specific heat of unirradiated graphite

Windscale
- Air cooled, Graphite Moderated
- Operating Temperature ~ 250°C
- Core burned to > 1300°C for five days

The Windscale Pile reactors
Dimensional Change in Graphite Crystal
Low Temperature

C-axis growth
formation of between plane interstitial clusters

Swelling ( % )

Neutron Dose (10^{25} n/m^2)

Overall Large Volume Increase

Kelly 1986

0 5 10 15 20 25 30

150 °C

170 °C

200 °C

250 °C

0 2 4 6 8 10 12 14 16

a - axis

C = 0.67 nm

A-axis shrinkage
Dimensional Change in Graphite Crystal
Low Temperature

**c - axis**

- 150 °C
- 170 °C
- 200 °C
- 250 °C

**Swelling (%)**

**Neutron Dose (10^{25} n/m^2)**

- 2-4 interstitial cluster
- dose increase
- interstitial loop
- migrating interstitial
- single vacancy
- di-vacancy
- vacancy line

**Graphite Crystal**

- Kelly 1986

**Notes:**

- Kelly 1986
- Neutron Dose
- Interstitial cluster
- Dose increase
- Interstitial loop
- Migrating interstitial
- Single vacancy
- Di-vacancy
- Vacancy line
Above ~1000 K both vacancy and interstitials are mobile.

Dimensional change occurs to high dose conserving volume.

Higher crystal perfection material suffers less dimensional change due to reduced pinning centers for migrating defects.
Pyrolytic Graphite: Property Changes Under Irradiation

- Both strength and elastic modulus increases pyrolytic graphite due to defect pinning.
Nuclear Graphite

H-451 Extruded Nuclear Grade Graphite

Graphite
Coal-tar pitch
Pitch coke
Petroleum binder

IG-11 Isomolded Nuclear Grade Graphite

$\rho = 1.76 \text{ g/cc}

79 \% \text{ TD}$
H-451 Nuclear Graphite

\[ T_{\text{irr}} = 600^\circ \text{C} \]

\[ T_{\text{irr}} = 900^\circ \text{C} \]
H-451 Nuclear Graphite

Pyrolytic Graphite

- **c - axis**

- **a - axis**

![Graph showing swelling and dimensional change](image)

- Neutron Dose (n/cm² x 10²² [E>50KeV])
- Dimensional Change, %

- T<sub>irr</sub> = 600°C
- T<sub>irr</sub> = 900°C

- Kelly 1986

- Neutron Damage Dose, n/cm² x 10²² [E>50KeV]

- Swelling (%)
Effect of Temperature and Swelling of Nuclear Graphite

- Initial swelling accommodated by closure of intrinsic porosity.
- Once porosity filled, swelling can begin.
- Less initial porosity for higher initial temperature (closure of intrinsic porosity.)
Effect of Irradiation on Strength of Nuclear Graphite

Fracture Stress (MPa) vs. Neutron Fluence ($10^{25} \text{ n/m}^2; E>0.5 \text{ MeV}$)

- Grade TSX
- HTC1 Data – 620°C
- HTU1 Data – 575°C
- HTK3 Data – 620°C
- Controls

10 dpa, 20 dpa, 30 dpa
Effect of Irradiation on Strength of Nuclear Graphite

Fracture Stress (MPa) vs. Neutron Fluence ($10^{25}$ n/m$^2$; $E>0.5$ MeV)

- Grade TSX
- HTC1 Data – 620°C
- HTU1 Data – 575°C
- HTK3 Data – 620°C
- Controls

Return to “zero” Volume change
Effect of Irradiation on Elastic Modulus of Nuclear Graphite

Return to “zero” Volume change
Irradiation-Degraded Thermal Conductivity - Graphite -

Degradation in High Conductivity Graphite Composite as a Function of Radiation Damage and Temperature

- Thermal conductivity in graphite is dominated by phonon transport.
- Vacancy complexes formed during irradiation are extremely effective at scattering phonons and degrading thermal conductivity.
Comparison of Thermal Conductivity Degradation

Thermal Conductivity (W/m-K) vs. Time in Reactor (hours)

MKC 1-PH; $T_{irr} \approx 430^\circ$C

FMI-222; $T_{irr} \approx 310^\circ$C

H451; $T_{irr} \approx 430^\circ$C

2 dpa
Thermal Conductivity of Irradiated Ceramics

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

Specific Heat

Umklapp

Irradiation Defects

Grain Boundaries

Thermal Defect Resistance

Temperature (C)

Specific Heat

Umklapp

Irradiation Defects

Grain Boundaries

Thermal Defect Resistance

Temperature (C)
Thermal Defect Resistance

The main motivation for using thermal defect resistance is that radiation-induced defects, such as vacancies and clusters, have resistances proportional (or square root dependent) to their concentration and are additive. This gives an easy way to compare stability of ceramics under irradiation.
Graphite Perfection and Thermal Defect Resistance

- Larger, more perfect crystallites accumulate phonon scattering defects at a lower rate in graphite (lower vacancy production.)
  
  \[ \text{---}> \text{higher initial conductivity will always have higher irradiated conductivity} \]
Yield Strength of Various Structural Materials

- Superalloy
- C/C Composite
- SiC/SiC
- 800H
- Zircaloy
- Carbon Steel
- Stainless Steel
- Graphite
Carbon Fiber Composites

• Composites as being defined here are technically “continuous fiber reinforced composites,” the two most mature of which are Carbon(graphite) Fiber Composites (CFC’s) and Silicon Carbide fiber composites (SiC/SiC.)

• Of the two, the CFC is the more mature system, though they are similar in terms of processing status and cost.
Reinforced Fired Adobe Composite

Inca city ~ 1500 AD

Present Day

 LOAD

0

0.5

1

1.5

2

2.5

3

3.5

Compressive Strength (Kg/cm²)

-0.5

0

0.5

1

1.5

2

2.5

3

3.5

% Straw or Grass

Andes Straw

Ichu grass

J. Vargas Data

Crack arrest

Crack

Clay

Straw

% Straw or Grass
Fort Paramonga  Chimu civilization ~1300 AD
Puye Cliff Dwelling
Anasazae Indians
1100-1580 AD
Short History of Materials

Date

Relative Importance

10000 bc 5000 bc 0 1000 1500 1600 1900 1940 1960 1980 1990 2000

CERAMICS/ GLASSES
COMPOSITES
STRAW-BRICK
POLYMERS/ ELASTOMER
GOLD COPPER
IRON BRONZE
STEELS
CAST IRON
LIGHT ALLOYS
SUPER ALLOYS
TITANIUM, ZIRCONIUM
GLASSY METALS
etc. ALLOYS
HIGH TEMPERATURE POLYMERS
HORSEHAIR PLASTER
BAKELITE
GLASS CEMENT
PORTLAND CEMENT
CEMENT
REFRACTORIES
FUSED SILICA
GFRE C/C METAL MATRIX
CERAMIC MATRIX
METAL MATRIX
PYROLITIC CERAMICS
TOUGHENED CERAMICS
HIGH MODULUS POLYMERS
POLYESTERS
EPOXIES
ACRYLICS
P. E.
NYLON
PMMA
POLYMERS
BLUES
RUBBER
CERAMICS/ GLASSES
METALS
CEMENT
GLASS
POTTERY
POTTERY
FLINT
STONE
WOOD
SKIN
FIBERS
P. E.
NYLON
GLUES
TITANIUM, ZIRCONIUM
GLASSY METALS
TITANIUM, ZIRCONIUM
GLASSY METALS
Fabrication of C/C Composites

Carbon Fiber:
- PAN (polyacrylonitrile) based carbon fiber
  - Commercial use for general purpose.
  - Varieties: high strength, high modulus, long elongation, ...
- Pitch based carbon fiber
  - High performance carbon fiber: Anisotropic, high graphitization.
    Tensile strength: 2.3~4.0GPa, Tensile modulus: 400~900GPa
  - General purpose (low cost) carbon fiber: Isotropic microstructure.
    Tensile strength: 0.6~1.0GPa, Tensile modulus: 30~60GPa

Carbon Matrix:
- Chemical vapor deposition (CVD)
- Impregnation and pyrolysis using resin or pitch.

Environmental Barrier Coating:
Concern about high reactivity to oxidative products.
- Boron based glasses (<1000°C)
- Silicon carbide (<1500°C)
Graphitization of carbon to graphite involves a process where the material transforms into a more perfect crystal structure with increasing temperature. This transformation is illustrated in the diagram, showing the progression from low perfection to high perfection. The key stages include:

- **Low Perfection**: The initial state with less ordered structure.
- **“Graphitization”**: The intermediate stage showing the early stages of transformation.
- **High Perfection**: The final state with a highly ordered graphite structure.

The temperature axis indicates the progressive change at different temperature levels (1600 K, 1700 K, 2000 K), highlighting how the transformation occurs with increasing temperature.
Divertor Mock-Ups Using C/C Composites

Full-scale vertical target armored mock-up uses a pure Cu clad DS-Cu tube armored with saddle-block C/C and CVD-W armors. (Hitachi Ltd., Japan)

Pure Cu clad DS-Cu tube armored with C/C monoblocks. (Kawasaki Heavy Industries, Japan)
### Candidate Nuclear Graphite Compared to CFC

#### Poco Graphite to ~ 8 dpa

<table>
<thead>
<tr>
<th></th>
<th>Virgin</th>
<th>500°C (6 \times 10^{25} \text{n/m}^2)</th>
<th>800°C (7.7 \times 10^{25} \text{n/m}^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Strength</strong></td>
<td>113±20</td>
<td>107 ±7 ((-5%))</td>
<td>98 ±11 ((-13%))</td>
</tr>
<tr>
<td><strong>MPa</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Length Change</strong></td>
<td>-</td>
<td>0.1 %</td>
<td>1.11 %</td>
</tr>
</tbody>
</table>

#### FMI-222 Composite to 8 dpa

<table>
<thead>
<tr>
<th></th>
<th>Virgin</th>
<th>500°C (6 \times 10^{25} \text{n/m}^2)</th>
<th>800°C (7.7 \times 10^{25} \text{n/m}^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Strength</strong></td>
<td>176</td>
<td>286 ±25 (+63%)</td>
<td>241±22 (+37%)</td>
</tr>
<tr>
<td><strong>MPa</strong></td>
<td>±20</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Length Change</strong></td>
<td>-</td>
<td>- 1.5 %</td>
<td>- 3.6 %</td>
</tr>
</tbody>
</table>

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Snead, JNM 2004
CFC’s Under Irradiation
(HFIR, 600°C)

- composite irradiation-induced dimensional changes explained by classic graphite model...

- limited data suggests that Pitch based fibers undergo lower irradiation-induced densification...

1-D Fiber Composite (UFC)

Burchell 1996

Dimensional Change (%)

Dose (dpa)

3D Balanced Weave

Pitch Fibers

PAN Fibers

Dimensional Change (%)

Dose (dpa)
Elevated temperature irradiation begins to show severe degradation.
Dimensional Change in 1-D Composite

1-Dimensional Composite

2 dpa

Diameter Change (Perp. Axis)

Length Change (Parallel to Axis)

Fiber axis

Dimensional Change (%)

Irradiation Temperature (°C)

800 900 1000 1100 1200 1300 1400 1500

MKC 1-PH 2 dpa

Length Change (Parallel to Axis)

Dimensional Change (Parallel to Axis)
Dimensional Change in 3-D Composite

A Balanced 3-D Composite

Dimensional Change (%)

Irradiation Temperature (°C)

Length Change (Parallel to Axis)

2 dpa

Fiber axis
Dimensional Change in FMI-222 Composite

A Balanced 3-D Composite

- FMI-222 2 dpa
- Snead (03) ~7 dpa
- Burchell (96) Same fluence

Dimensions:
- L: 30 mm
- W: 6 mm
- T: 2.5 mm

Composite unit cell: ~3 cells (~35 cells)

Irradiation Temperature (°C)

Dimensional Change (%)
• Recent, high-temperature studies have shown that the dimensional change in high-quality graphite fiber composite is far more serious than previously thought.
Biased Summary Comments on Graphite and CFC’s

- Graphite, like beryllium (and perhaps tungsten) suffers from “crystal-driven irradiation-induced changes. The effects can be somewhat mitigated, but strict lifetimes exist.

- CFC irradiation damage follows the same principals as for graphite.
  - Swelling perpendicular to basal planes, shrinkage within planes
  - Increase in strength and modulus up to composite lifetime.

- The lifetime of the composite will depend sensitively on the irradiation temperature and dose. It appears that, due to the inherent perfection of graphite fibers, the lifetime of the composite may be lower than that of nuclear graphite, especially at high temperatures.

- The CFC materials studied to date have been selected based on high intrinsic perfection (thermal conductivity.) This selection may lead to lower lifetime. --> we may do much better with poorer materials…
Questions ???
CFC’s Under Irradiation : Tritium Retention

- T-3 attaches to basal plane edges and highly defected structure. More perfect material and/or high temperature allows less retention.

![Graph showing Tritium Retention vs. Irradiation Temperature](image_url)

- Intermediate Quality Irradiated Graphite (Causey, Snead)
- High Quality Irradiated CFC (Causey, Snead)
- Non-irradiated, infinite charge time
- Non-Irradiated 1 hr Charge Time

NRL IFE 2/2001
Operating Window

- Normalized Shear Stress, $\tau/\mu(20^\circ C)$
- Normalized Temperature, $T/T_M$
- Elastic regime ($d\epsilon/dt<10^{-8}\, s^{-1}$)
- Coble creep
- Dislocation creep
- Dislocation glide
- N-H creep

- Uniaxial Tensile Stress, MPa

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- Ultimate Tensile Strength (MPa)
- Temperature (°C)

- Fracture Toughness $K_{IO}$ (MPa-m$^{1/2}$)
- Test Temperature (°C)
- Uniform elongation (%)
Effect of Thermo-mechanical Treatment

- Properties of CuCrZr strongly depend on thermo-mechanical treatment
- Optimal heat treatment produces a high number density of fine precipitates, giving rise to high strength

\[\text{CuCrZr SAA}\]

\[\text{CuCrZr SCA}\]

\text{ITER reference heat treatment, SAA: solution anneal at 980-1000°C/0.5-1h, WQ, aged at 460-500°C/2-4 h}\]

\text{Heat treatment simulated the manufacturing cycle for large components, SCA: HIPped, SA 980°C/0.5 h, slow cooled 50-80°C/min, aged 560 °C/2 h}\]

## Composition

<table>
<thead>
<tr>
<th></th>
<th>Titanic hull plate</th>
<th>A36 modern structural steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.21</td>
<td>0.20</td>
</tr>
<tr>
<td>Mn</td>
<td>0.47</td>
<td>0.55</td>
</tr>
<tr>
<td>P</td>
<td>0.045</td>
<td>0.012</td>
</tr>
<tr>
<td>S</td>
<td>0.069</td>
<td>0.01 to 0.04</td>
</tr>
<tr>
<td>Si</td>
<td>0.017</td>
<td>0.007</td>
</tr>
<tr>
<td>Cu</td>
<td>0.024</td>
<td>0.01</td>
</tr>
<tr>
<td>O</td>
<td>0.013</td>
<td>-</td>
</tr>
<tr>
<td>N</td>
<td>0.0035</td>
<td>0.0032</td>
</tr>
<tr>
<td>Mn:S Ratio</td>
<td>7:1</td>
<td>15:1 (typical)</td>
</tr>
</tbody>
</table>

K. Felkins, H. P. Leighly, and A. Jankovic. JOM, 50(1), 1998, 12-15