R&D on graphite-based oxidation resistant materials and radiation resistant tungsten

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Graphite-based oxidation resistant materials
  • Graphite & Silicon carbide
  • Oxidation resistant tests of SiC coated graphite
  • Irradiation tests at BLIP and PIE tests at PNNL
Ductile, radiation-resistant tungsten materials
Summary
Many thanks to everyone, (^_^)/

Collaborators

- Muon Section, J-PARC: S Makimura, S Matoba, N Kawamura, K Shimomura

**SiC coated graphite**

supported by RaDIATE & US-JP collaboration

- T Ishida, T. Nakadaira, E. Wakai, M. Teshi J-PARC, T2K, JAEA
- P. G. Hurh, K. Ammigan, D. Senor, A. Casella, N. Simos, and RaDIATE (FNAL, BNL, PNNL,"
- M. Calviani, A. P. Marcone, C.L.T. Martin, E. Fornasiere and CERN

**Ductile Tungsten alloy**, supported by Industry and Nuclear Fusion Field

- H. Kurishita, KEK
- Kinzoku Giken (Metal Technology Co., LTD) Collaborative research
- Ito Seisakujyo Co., LTD
- H. Noto, National Institute for Fusion Science Collaborative research
R&D on Graphite-based oxidation resistant materials (SiC coated graphite)
Graphite Target

Muon Rotating target at MLF
3 GeV, 1 MW, thickness 20 mm
Thermal radiation in vac.
950 Kelvins @ 1MW

Neutrino target at J-PARC
30 GeV, 750 kW, thickness 900 mm
He-cooling: 1010 Kelvins @ 750 kW

Graphite target IG-430U
26mm x ~900mm
Inner tube (graphite)

Outer tube / beam window (Ti-6Al-4V)

MuSIC target at RCNP
400 MeV, 400 W, thickness 200 mm
Thermal rad. In vac. 600 K

Pion Production Target

Pion Capture Solenoid

E target (PSI)
600 MeV, 1.2 MW,
thickness 60 mm
Thermal rad. In vac.
1700 K

COMET target P1 at J-PARC
8 GeV, 4 kW, thickness 400 mm
Thermal rad. In vac. 500 K

NUMI, MSU, ISIS, GSI,

Pion Production Target
Polycrystalline graphite

King of Low-Z target material, especially for muon/pion production

**PROS**
- **High thermal resistance** (1600 degC @Vac.)
- Mech. properties (Low Young’s modulus, Low thermal expansion, **High resistance to thermal shock**)
- Experience as irradiation material (Nuclear fission reactor)

**CONS**
- **Easy oxidation at high temperature**
  - For use in vacuum, Unexpected air introduction
  - For use in He-cooling, Loss of target material through O₂, impurity during normal beam operation
- **Low density** (Volume of muon/pion source should be small for efficient transport.)
SiC coated graphite

- Commercially available at Graphite manufacturers (Toyotanso, Ibiden, ADMAP, ...)
- CVD-SiC coating (Dense coating)
- Study for fission nuclear reactor with higher oxidation resistance
Oxidation resistance of SiC

- Accidental Loss of Vacuum during beam operation
- Loss of target material through O₂, impurity during normal beam operation

Oxidation behavior depends on temperature and partial oxygen pressure. Passive vs Active

Scope
- J-PARC/MLF/Muon
  700 degC in vacuum
  Accidental loss of Vac.
- J-PARC/Neutrino
  800 degC in He
  Loss of target material by oxidation during normal operation

Research of CVD-SiC for fusion nuclear reactor
Oxidation tests of SiC coated graphite

- SiC-coated graphite (IG-610U, IG-110U)
- Graphite (IG-430U)

Diameter: 10 mm, thickness: 1 mm
Thickness of Coating: 0.1 mm

- 800 deg C
- Dried air (N₂ + O₂ 21 %): 200 cc/min.

The experiment was performed by using Tube furnace at the CROSS-Tokai user laboratories.

Volume loss was observed on graphite and not observed SiC coated graphite.
Weight variation of the oxidation tests

Weight loss of Graphite is large. Oxidation resistance of SiC-coating is very high.
Irradiation tests at BLIP and PIE tests at PNNL
Under RaDIATE collaboration
Purpose: Investigation of irradiation effects.
Irradiation at BLIP facility at BNL is ongoing.

- SiC coated graphite is included at CERN capsule.
- Confirmation through Microstructural analyses at PNNL whether exfoliation will be conducted.
- Comparison of three kinds of graphite.

Precious opportunity for high-energy proton irradiation.
Thermal Analysis – Si Capsule

Temperatures

- Max T Si samples: 216 °C
- Max T Graph/SiC: 220-240 °C
- Max T Sigraflex: 193 °C
- Max T SS window: 71 °C
- Max HF SS window-Water: 28 W/cm²

Thermal Expansions:

Initial lateral gaps Samples-Fillers = 0.1 mm
Initial lateral gap Fillers-SS capsule = 0.2 mm

- Remaining gap Graph/SiC –Filler: 94 um
- Remaining gap Si samples – Si Filler: 80 um
- Remaining Fillers– SS Capsule: 200 um

(remains the same)

*TCC in Back-up slides

By Claudio. L.T. Martin at CERN
Specimens of SiC-coated graphite for BLIP irradiation at BNL

- Specimens were assembled in Si capsule of CERN.
- BLIP irradiation has been conducted at BNL.
- Microstructural analysis will be conducted at PNNL.

Thickness measurement, Optical Microscopy, SEM, EDX, TEM to make sure the effect of gas production.
R&D on
Ductile, radiation-resistant tungsten materials
Tungsten as high-power target material

**Advantageous material properties**
- High melting point, Low coefficient of thermal expansion, Low vapor pressure, High thermal conductivity
- High density, Large mass number, Low solubility of hydrogen isotopes

**Tungsten as high-power target material**
- Tungsten rotating target at European spallation source, He cooling
- Spallation Neutron Source, 2nd target station at ORNL, Water cooling
- Mu2e target at FNAL, COMET phase 2 target at J-PARC, thermal radiation
- Candidates of MLF 2nd target station at J-PARC, ??

**Critical issue of tungsten: Brittleness**
- Limitation against design and lifetime
- Enhanced by p-irradiation or under high temperature

**Ductile, radiation-resistant tungsten materials, TFGR**
Ductile, radiation-resistant tungsten materials
(Toughened, fine-grained, recrystallized (TFGR) W-1.1%TiC)
e.g. H. Kurishita et al. Mater. Trans. 54 (2013) 456.

- Originally developed for diverter material of nuclear fusion reactor by Prof. Kurishita at Tohoku Univ.
- Very high performance
- But further development is required. In particular, method to manufacture large material.
- KEK will turn over the activities under collaboration with industries and Prof. Kurishita.

In this presentation
- Review of TFGR development by Prof. Kurishita
- Present status and prospect of our activities
Recrystallization embrittlement of tungsten

Current structure modification to mitigate GB fracture

- Fiber grain structure by heavy plastic working
- Equiaxed, especially after recrystallization
- Recrystallized grain structure by heating at and above $T_r$

GB fracture: brittle

The use of W and Mo is limited below $T_r$ ($T_r : \sim 0.4T_m$ for stress relieved pure W)
($T_m : \sim 3700\text{K}$)

Radiation embrittlement and its mitigation

- Radiation embrittlement: caused by radiation induced lattice defects which impede the movement of dislocations (radiation hardening)

- Suppression of accumulation of radiation induced point defects by introducing sinks: Dispersoids (precipitates) and GBs
RT strength & ductility of Pure tungsten & TFGR

Ductility is improved by heavy plastic working with orientation. But, Brittle after anneal.

TFGR~W-1.1%TiC

1920 K GSMM
Fracture strength: 3200 MPa

W plate (hot rolling)
Thermal shock response in TFGR W-1.1TiC

E-Beam by JUDITHs (120kV, 275mA): $A = 4 \times 4 \text{ mm}^2$, $t = 1 \text{ ms}$, $P = 1.1 \text{ GW/m}^2$ (HF = 35 MWm$^{-2}s^{1/2}$, $\Delta T \approx 2000^\circ\text{C}$), $T_{\text{base}} = 100^\circ\text{C}$, $n = 100$

MA-HIPed W-0.5TiC/H-170ppmO TFGR W-1.1TiC/H-160ppmO

Repeated dynamic heating and cooling

During heating

Compressive stresses

During cooling

Tensile stresses

Cracking occurs during cooling ($T < DBTT$)

Thermal fatigue response in TFGR W-1.1TiC/H

E-beam: ~1700°C x 3 min, then 2 sec-heating / 7.5 sec-off (resulting in variation of 1250°C ⇔ < 450°C) with 380 cycles

Stress relieved pure W
Significant surface roughening and GB cracking

TFGR W-1.1TiC/H
No appreciable roughening and cracking

• Suppression of plastic deformation
  \( \sigma_y \): SR pure W << TFGR W-1.1TiC
  • Grain size strengthening
  • Dispersion strengthening

• Suppression of crack formation
  \( \sigma_f \): SR pure W << TFGR W-1.1TiC
  • Reinforcement of recrystallized GBs

Manufacturing of Ductile tungsten, TFGR
Present status and prospect of our activities

Glove box
Purified H₂ or Ar
W- X wt%TMC
Powder handling

The powder with hard balls in a vessel (TZM)
Mechanical Alloying with high energy ball milling

GB Sliding based Microstructural Modification at ~1700°C
TFGR sample

Funding was approved and fabrication will be completed this December.

Small specimens can be made by an ordinal hot press next year.

Collaborative research with National Institute for Fusion Science

Applying for funding to manufacture large specimens
Summary and Acknowledgement

- R&D on SiC coated graphite for target material is on going.
  - Oxidation tests was conducted successfully.
  - Irradiation tests at BLIP and PIE tests at PNNL

- R&D on Ductile, radiation-resistant tungsten materials, TFGR has been initiated.

I want to express my gratitude to DOE understanding for US-JP collaboration.

Thank you for your attention.