

# **RECENT ACCOMPLISHMENTS AND FUTURE PROSPECTS OF MATERIALS R & D IN JAPAN**

*Institute of Advanced Energy*

*Kyoto University*

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**3: National Institute for Fusion Science, Toki, Gifu, Japan,**

**4: JAERI, Tokai Establishment, Tokai, Ibaraki, Japan**

# AGENDA

*Institute of Advanced Energy*

*Kyoto University*

**1: Japanese Fusion Materials R & D Activities and Strategy**

**2: Research Emphasis and Status**

**a: Reduced Activation Ferritic/Martensitic Steels**

**b: Vanadium Alloys**

**c: SiC/SiC Composite Materials**

*d: Refractory Alloys*

*e: Modeling and Mechanistic Studies*

**3: Key Issues for Materials Development**

**4: Summary**

# Materials R & D Strategy (1)

- Mission-oriented and time-driven to meet ITER and DEMO schedules
- Long-range fundamental studies and development of advanced materials are also promoted
  - Long lead time necessary for their development
- Planned to proceed with IFMIF and ITER in a coordinated way
  - IFMIF as a crucial facility for materials qualification
  - ITER blanket module tests as an important milestone for technology integration

# Materials R & D Strategy (2)

- Categorized into reference and advanced materials
  - **Reference materials** for timely realization of DEMO and first generation power plant:  
*Reduced Activation Ferritic Martensitic Steels*
    - Having the most matured industrial infrastructure and database
    - Use of Oxide Dispersion Strengthened (ODS) RAF(M) considered as a method to increase the operation temperature
  - **Advanced materials** for increasing attractiveness of the power plant (COE, environmental benignity):  
*V alloys and SiC/SiC Composites*
    - Having the very rapid progress under the very extensive R & D efforts

# Materials R & D Strategy (3)

**JUPITER-II: Japan/US collaborative program (FY2001-2006)**

*-Materials integration utilizing reactor irradiation and related basic research for advanced blankets-*

Emphasizing the importance of strong tie of “Materials” with “Blanket Engineering”.

- concentrating

*1: Self-cooled Liquid Blanket with V (RAF, SiC/SiC)*

*2: He Cooled Solid Blanket with SiC/SiC*

*3: Modeling and Mechanistic Studies*

**JAERI/ORNL Phase IV and V: (FY1999-2003, 2004-2008)**

Emphasizing the importance to establish

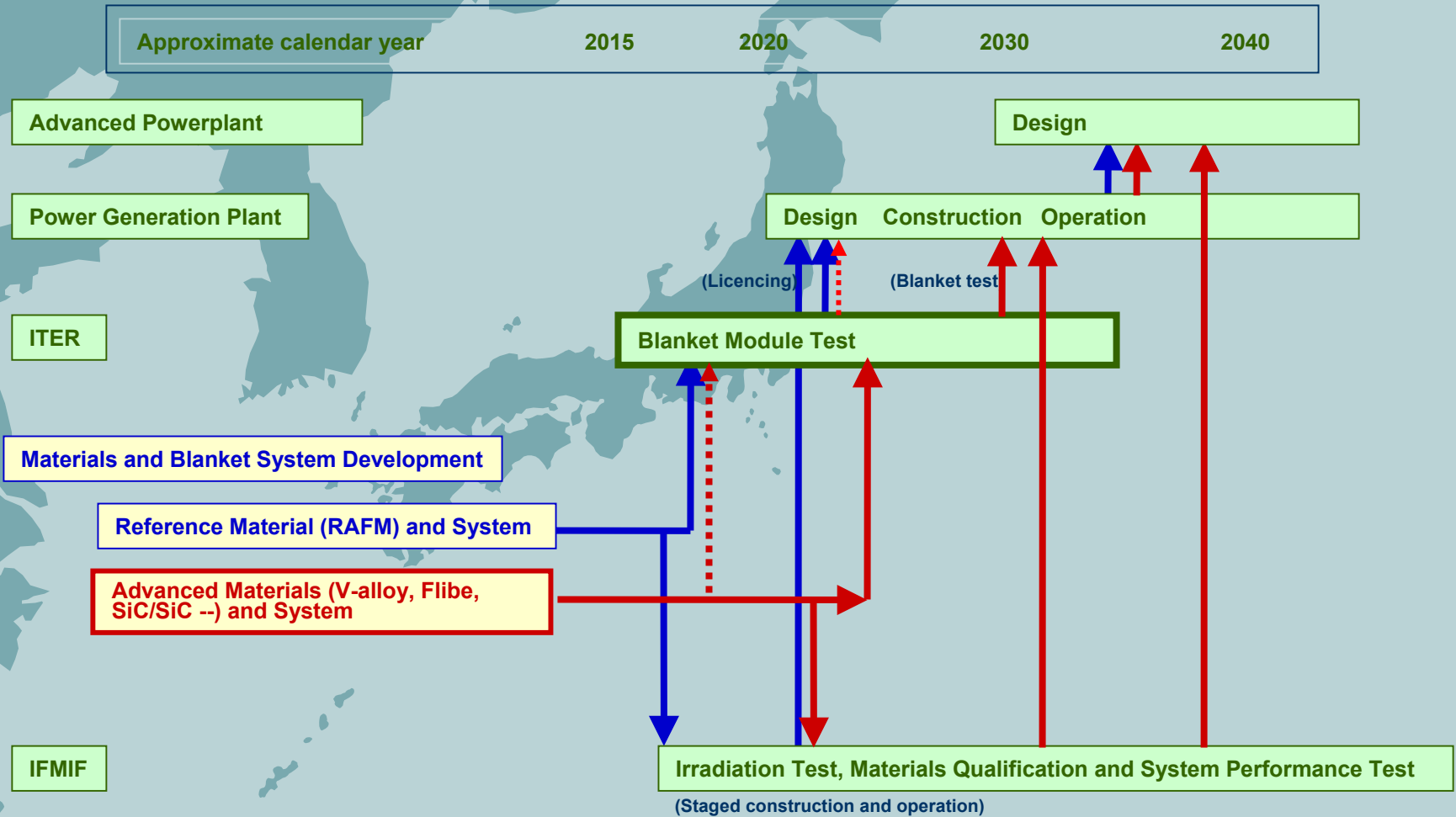
Clear understandings and Basis for blanket design from “Neutron Radiation Effects up to High DPA”



- concentrating

*1: water cooling ceramic breeder/Be blanket with RAF*

*2: He cooling solid breeding blanket with RAF*

# Roadmap for Materials and Blanket Development in Japan



-  Reference Blanket (under JAERI responsibility)
-  Advanced option (under NIFS/University responsibility)

## Advanced Blanket Concepts of Concern:

1. Liquid Lithium( or FLiBe)/Vanadium-Alloy
2. He cooled Solid Breeder/SiC/SiC

A: LiPb/He Dual Coolant with SiC/SiC (+RAF)

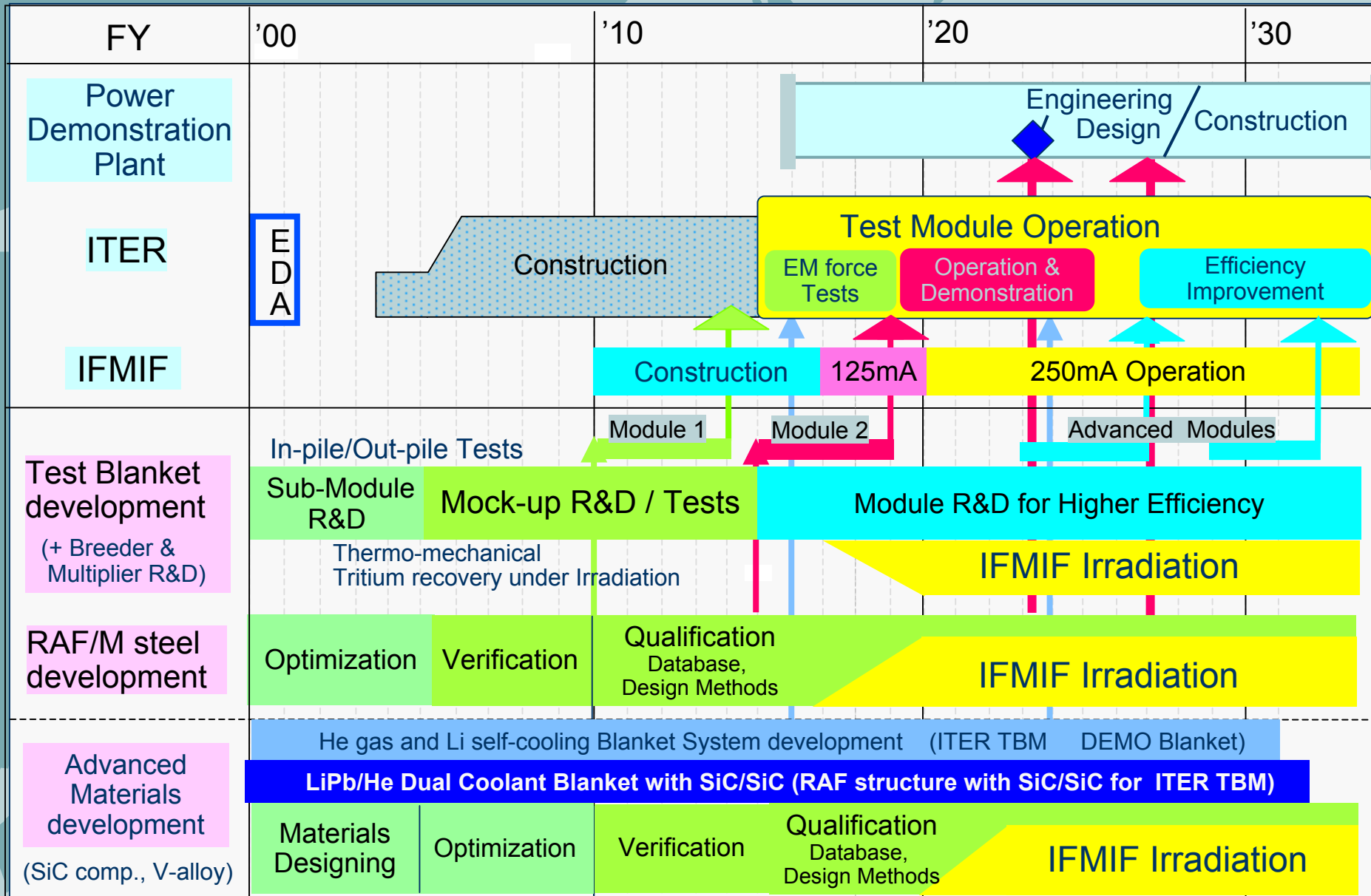
B: LiPb as Breeding Material and Coolant

1 and 2 are being conducted in **Universities and NIFS**  
as the activities of Fusion Network and JUPITER-II

A and B are new activities in **Universities**

**R & D strategy is under extensive discussion  
including the planning as a  
candidate for the post JUPITER-II (2007- )**

# Blankets and Structural Materials Development



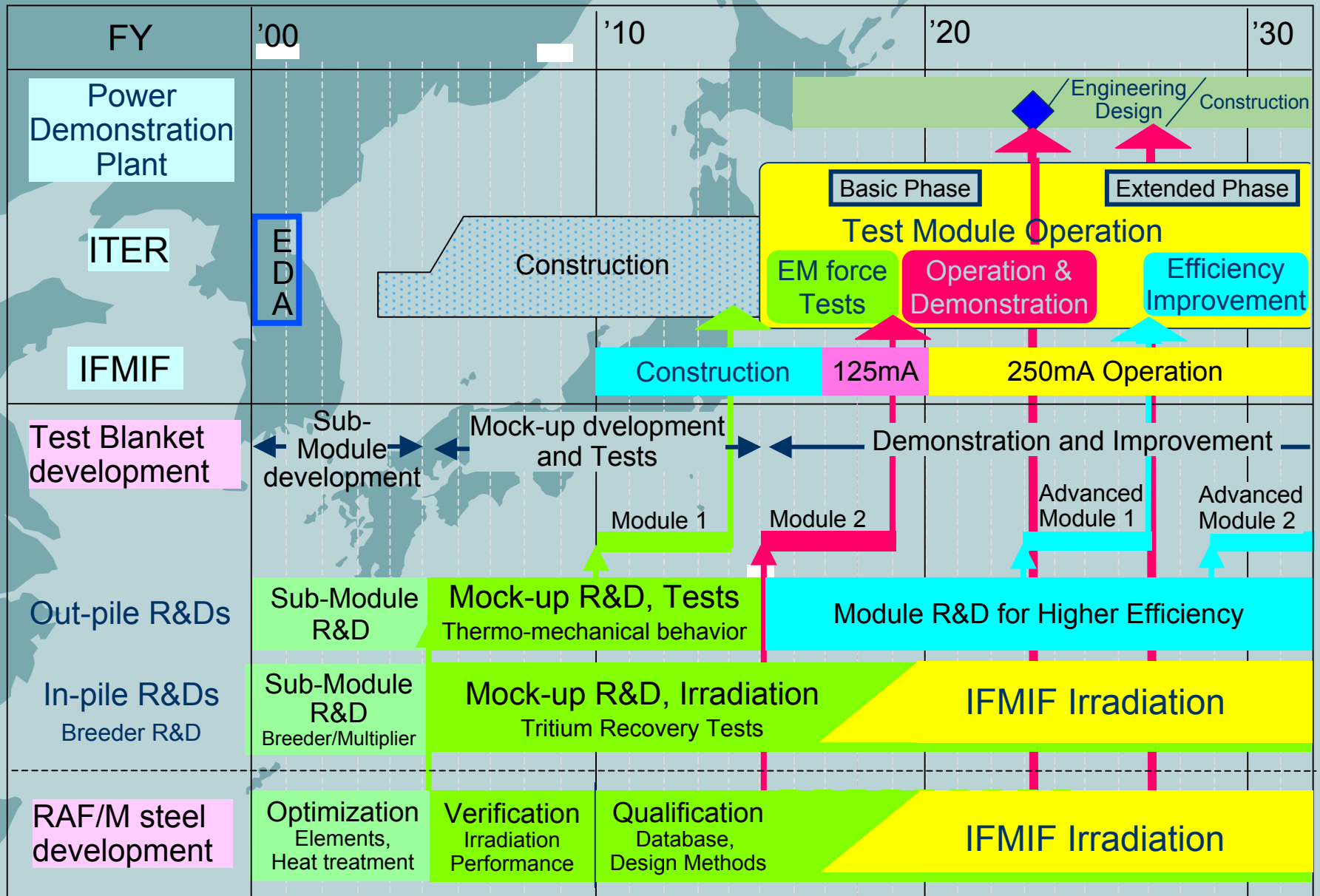




***Research Emphasis and Status (1)***

**Reduced Activation Ferritic/Martensitic Steels**

# Blanket and RAF/M steel Development

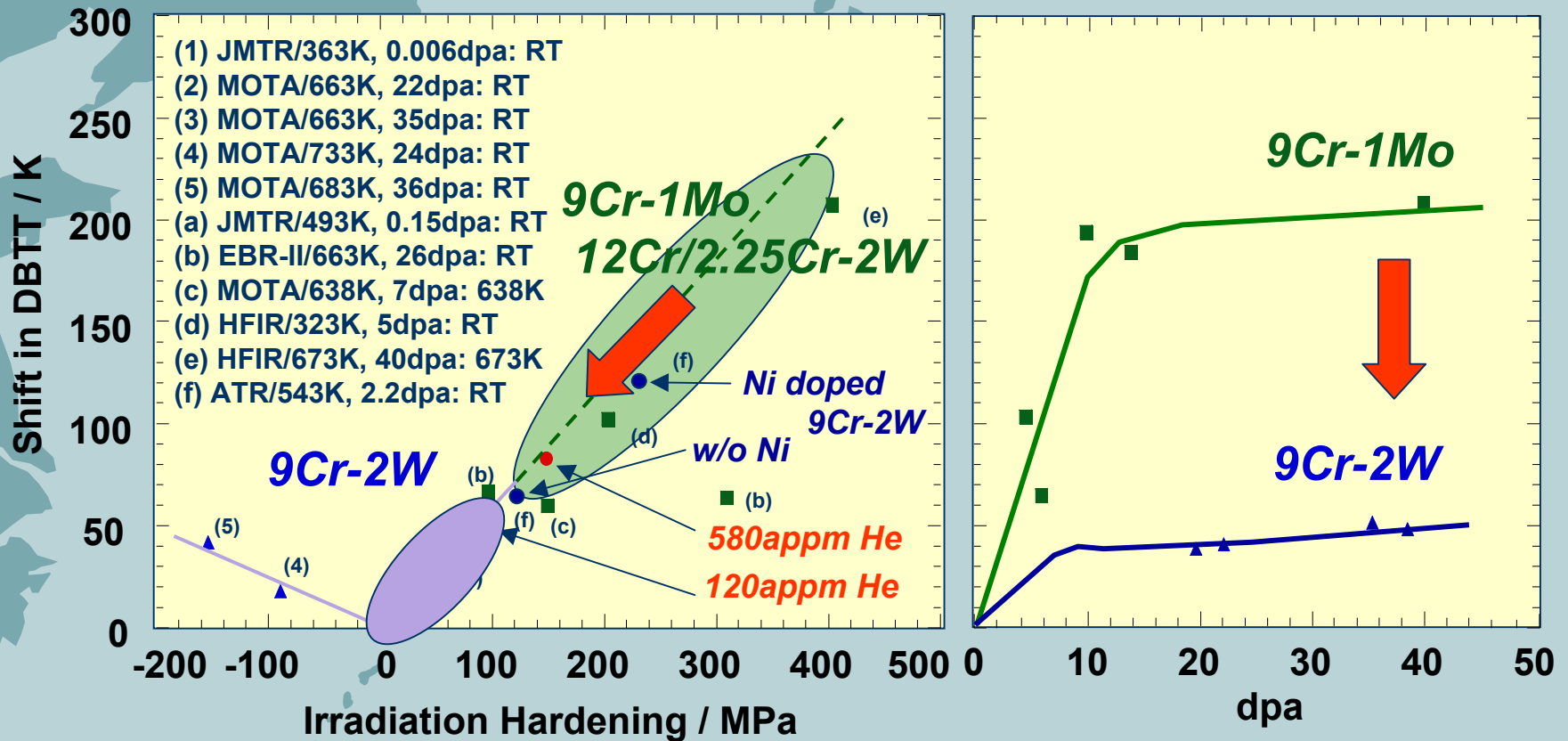


# Research Status and Emphasis - **RAFM**

- Large heats of F82H (8Cr-2W) and JLF-1 (9Cr-2W) were produced in Japan as reference heats and characterized by IEA test program
  - Data obtained on various physical and mechanical properties
  - Well characterized and recommended heat treatment and welding procedures have been established
  - Irradiation data are being obtained for matrix and weld joints
- The resulting data are compiled in relational database
  - With emphasis on the traceability of the results
- Large heat (9 ton) of JLF-1 was melted and thick plate (38mm, 25mm) processing is on-going
  - Production of 20 ton heat of JPCS (F82H/JLF-1) is under negotiation for the early production in FY2005

# Optimization of Total Performance for Fusion

## ● Suppression of irradiation embrittlement

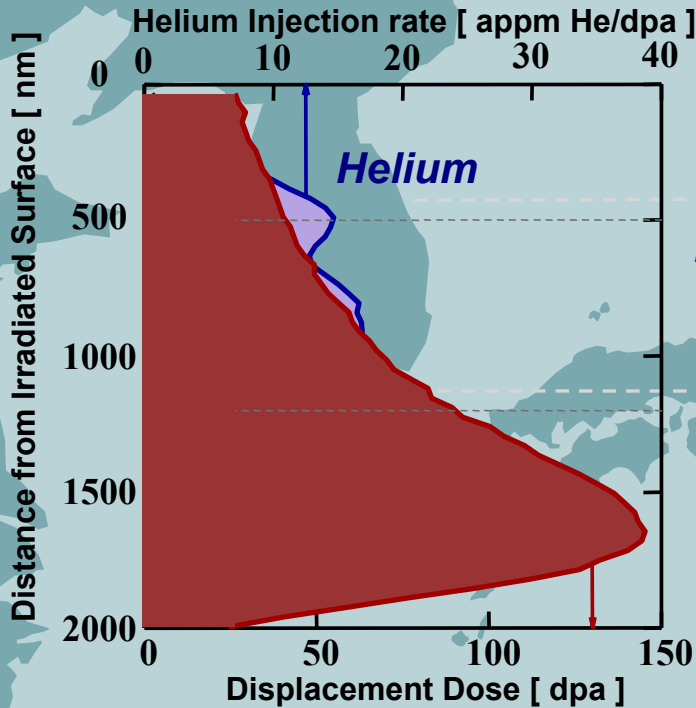


- # Successful substitution of steel elements
- # Optimization of Cr content (8–9%Cr) with 2W
- # Optimization of whole processing

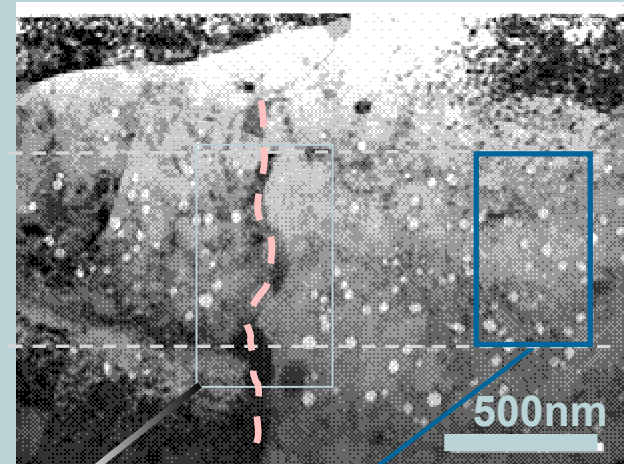
# Mechanistic Studies of Microstructure Evolution Under Fusion Environment

## Simulation Radiation Damage Study

**Dual-Ion Irradiation : at 743 K with 6.4 MeV Fe<sup>3+</sup> + Energy-Degraded 1.0 MeV He<sup>+</sup>**

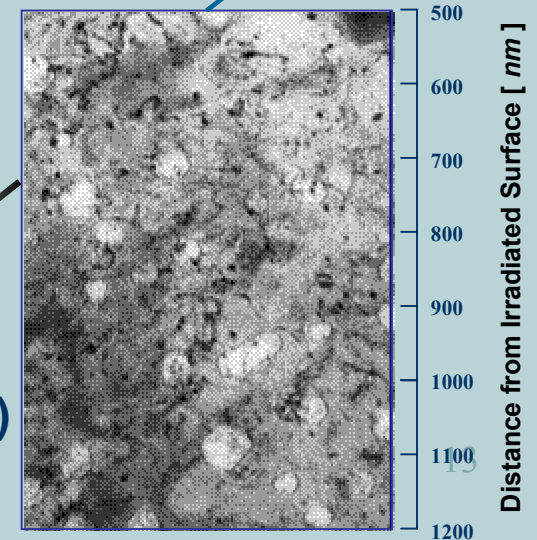


Depth Profile  
6.4MeV Fe<sup>3+</sup>, Degraded 1.0MeV He<sup>+</sup>



Void-depleted zones along lath boundaries

Cavity and dislocation image  
Swelling : 3.2 % (60 dpa @850 nm)



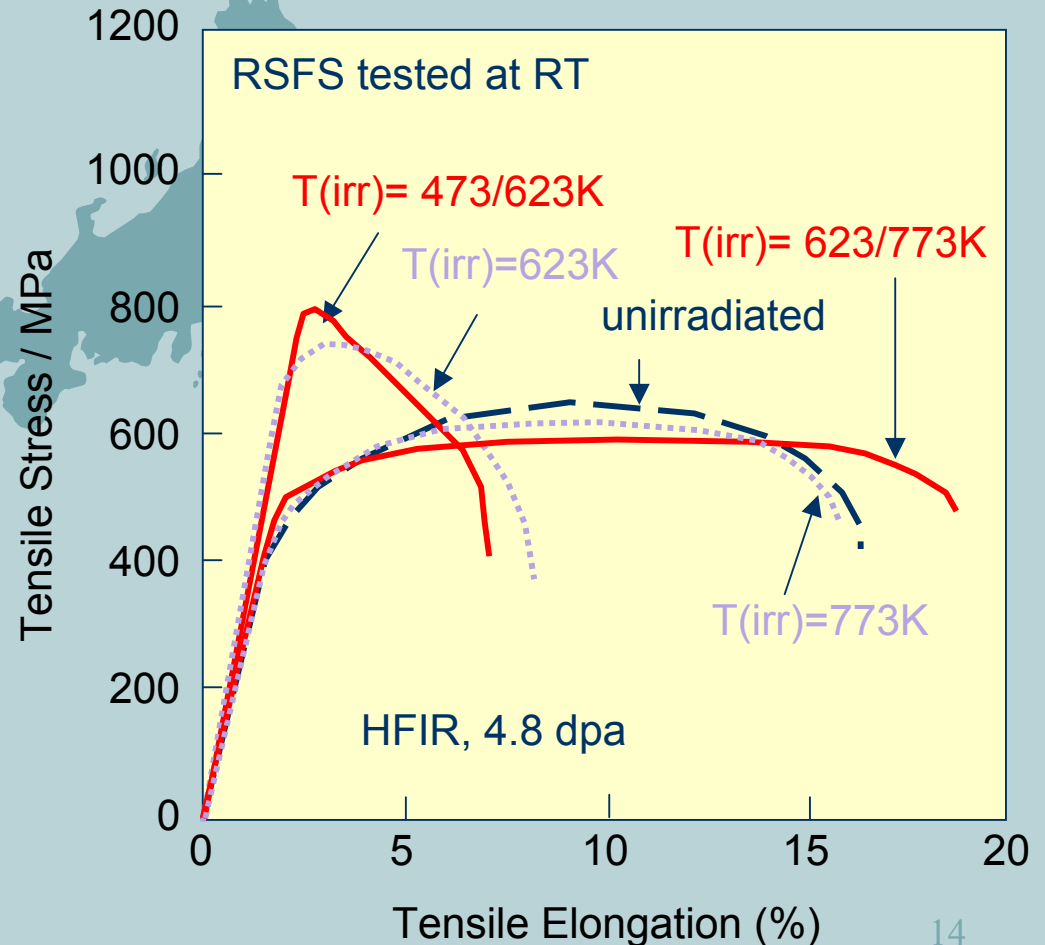
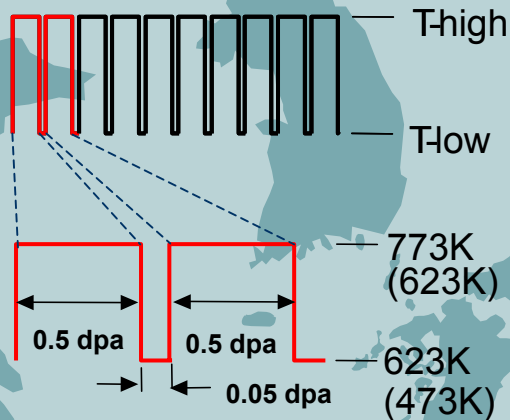
$g = [110]$

$Z \approx [\bar{1}11]$

# Mechanistic Studies of Microstructure Evolution Under Fusion Environment

## ● Insensitive to varying temperature irradiation

Varying Temperature Irradiation  
(HFIR: 0.5 x 10 cycles)



Tensile properties are similar between constant and varying temperature.

# RAF. Relational Database

- On going under international collaboration -

The screenshot displays the Microsoft Access interface for the RAF Relational Database. The main window, titled "IRRADIATION CAPSULE INFORMATION for Browse, Edit and Add", shows details for capsule JP-10. A search window, "TENSILE SPECIMEN SEARCH (Expert Mode)", is open, allowing users to filter specimens based on material (F82H) and irradiation parameters (dpa, IrrTemp, TestTemp). The search criteria are defined as follows:

```

Criteria
((MatName) Like 'F82H' And ((TestTemp) >= 0 And
[TestTemp] <= 1000) And ((IrrTemp) >= 0 And [IrrTemp] <=
1000) And ((dpa) >= 0 And [dpa] <= 100)) Or ((MatName)
Like 'F82H' And ((TestTemp) >= 0 And [TestTemp] <=
1000) And [IrrTemp] = NULL)
    
```

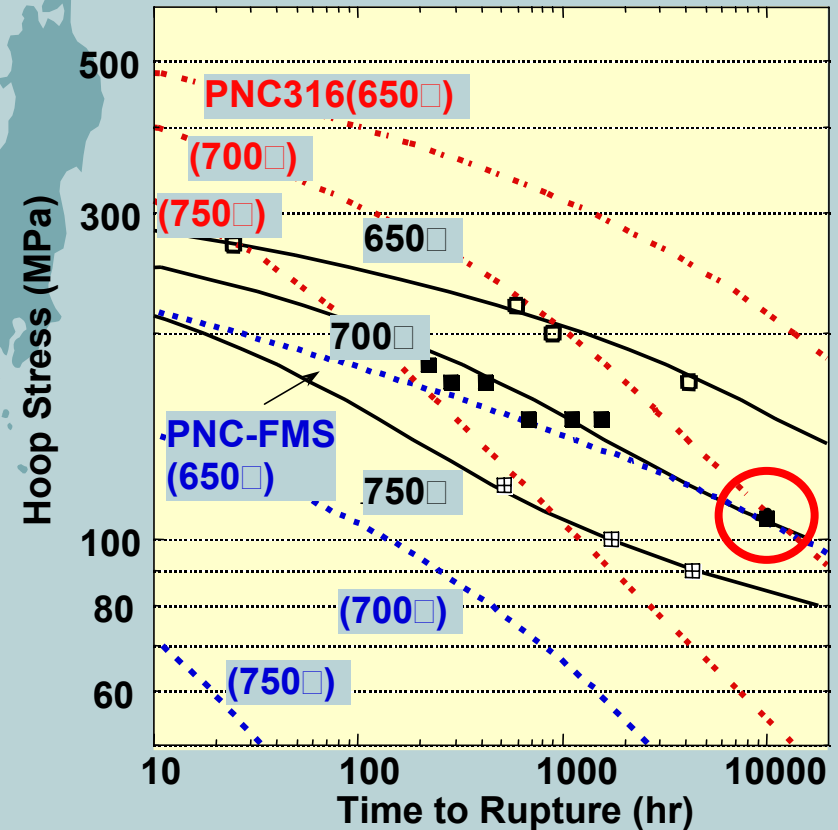
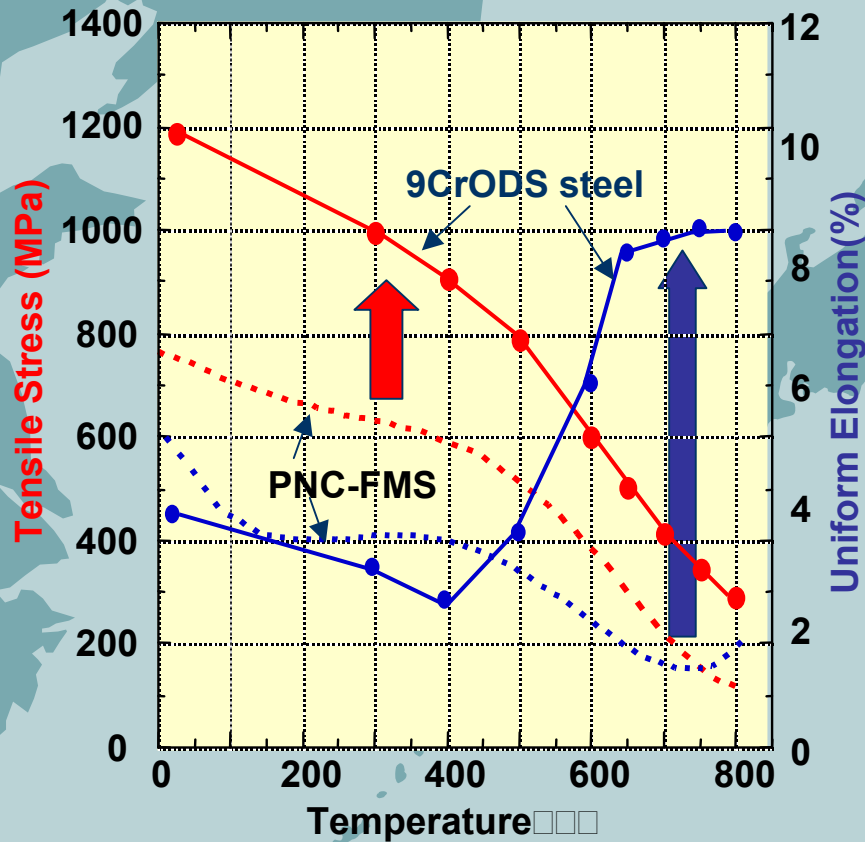
The capsule information table includes the following data:

Name	JP-10
Type	HFIR-PTP
Power	20161 Mwd
Start Date	90/07/20
End Date	91/09/17
Neutron Fluence	3.12E+22 n/cm2 (E<0.5eV)
Total Neutron Fluence	2.64E+22 n/cm2 (0.5eV<E<0.1MeV)
Neutron Fluence 1	2.12E+22 n/cm2 (E>0.1MeV)
Neutron Fluence 2	1.07E+22 n/cm2 (E>1MeV)
(316SS)	17.3 dpa
@CL (316SS)	903 appmHe
	-3.53E-18
	-9.59E-04

The search window also features a "Criteria" field with a complex logical expression and buttons for "Search Specimens" and "CLOSE". The background window displays the database title "US/JAERI HFIR/ORR IRRADIATION EXPERIMENT DATABASE" and a "Capsule Information" button.

# ODS RAFS R & D for Fusion

- Big improvement of high temp. properties



Time to rupture: 100 times longer than RAFS



***Research Emphasis and Status (2)***

# **Vanadium and Its Alloys**

# Roadmap for Vanadium Alloy Development

Calendar year

2005

2010

2015

2020

2030

2040

Advanced Powerplant

Design

(Full blanket)

Power Generation Plant

Design

Construction

Operation

(Sector blanket)

ITER

Blanket Module Test

(Small unit  
test module)

(Full size  
test module)

(Design data)

(Miniaturized  
specimen test)

(Engineering  
database)

(Design data)

## V Alloy Development

### Alloy optimization

(composition, processing)

### Qualification by fission neutrons

(creep, fracture, He effect)

## V/Li Blanket Technology

### MHD coating

(static and dynamic corrosion test)

### Tritium recovery

### Blanket module design/R&D

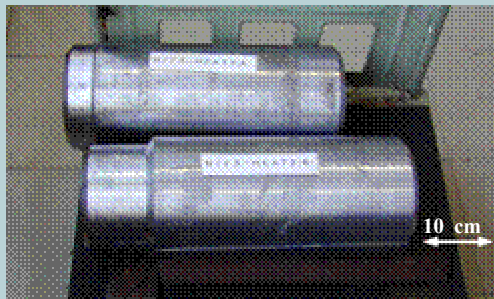
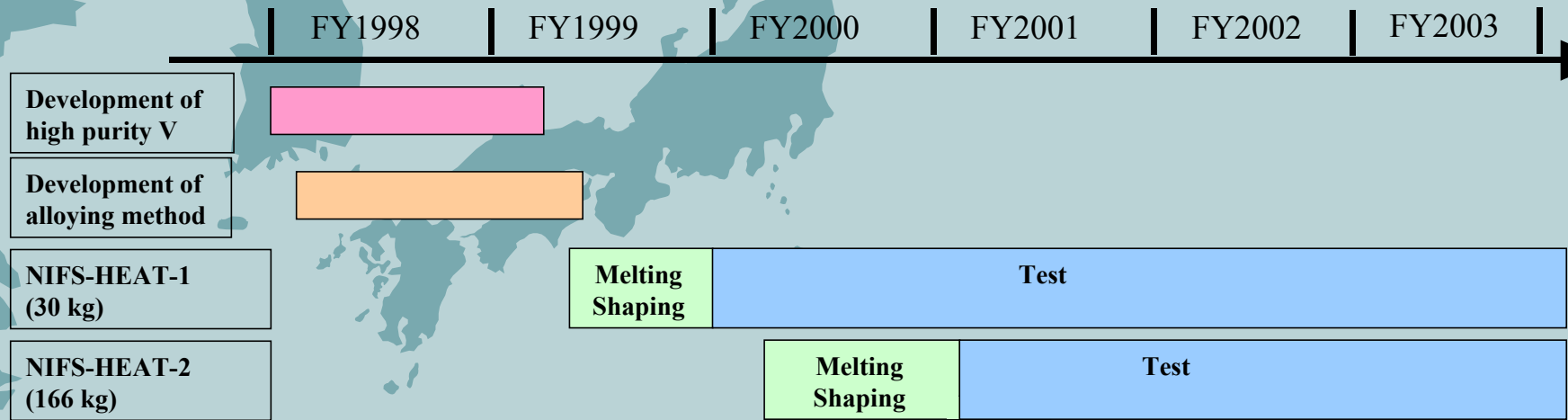
IFMIF

Irradiation Test, Materials Qualification

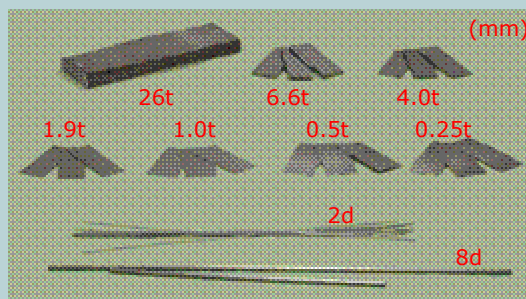
# Production and Characterization of High Purity V-4Cr-4Ti (NIFS-HEATs)

- NIFS-HEAT-1 has been tested by Japanese Universities
- NIFS-HEAT-2 was distributed for international collaboration

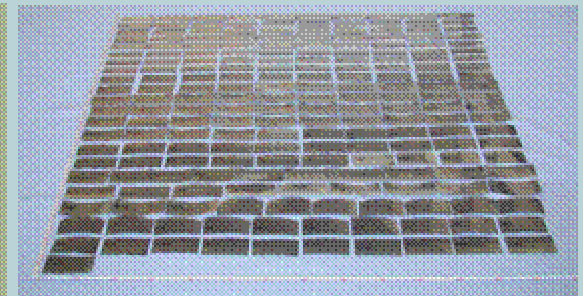
April 98



NIFS-HEAT-2 ingots



Products

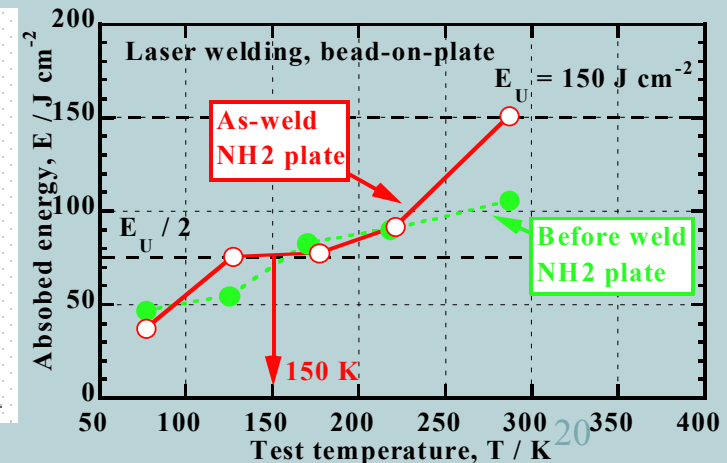
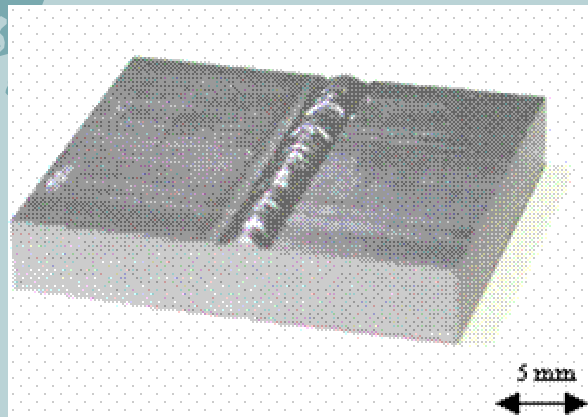
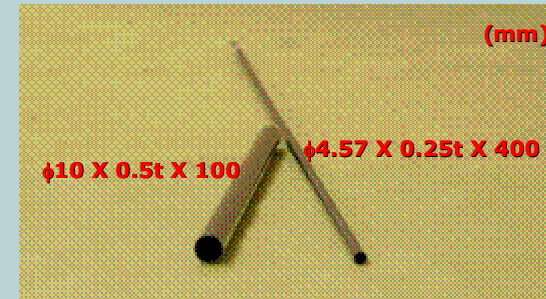
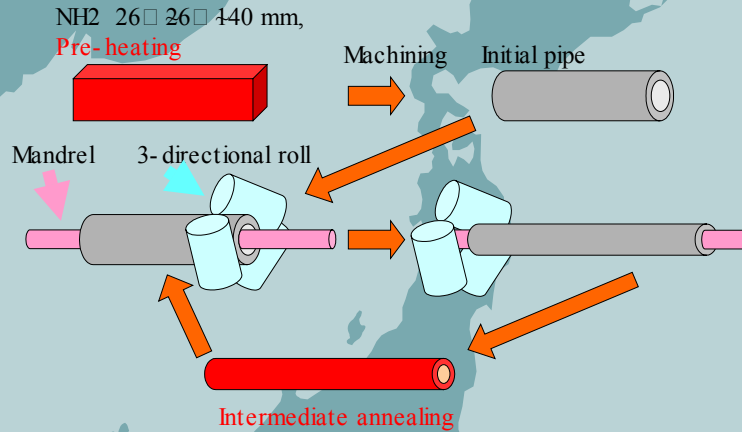


Specimens distributed

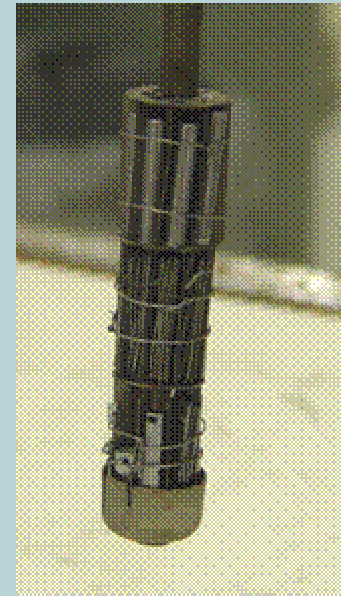
# Fabrication Technology Tubing, welding.

⌘ Based on the control of impurities and precipitates, plates, sheets, wires and tubes were successfully fabricated

⌘ Good properties of laser-weldment by atmospheric control and PWHT



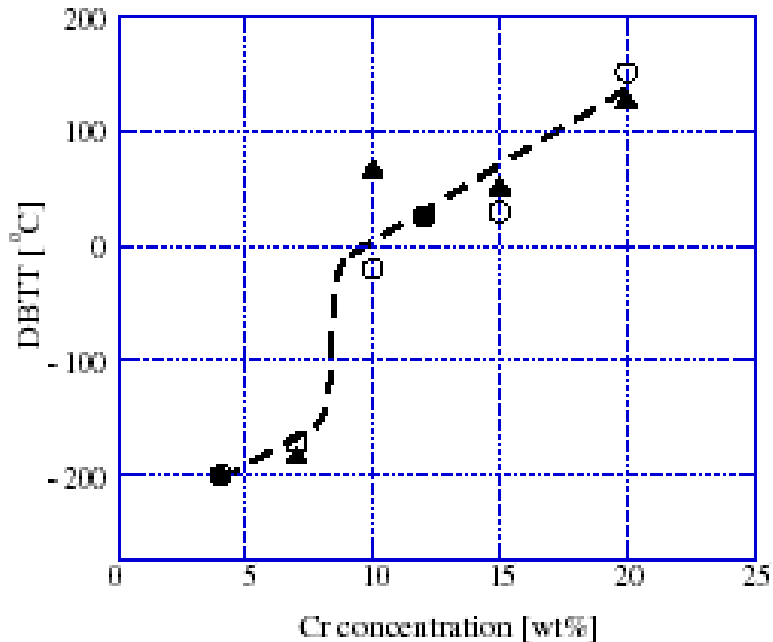
# Thermal and Irradiation Creep Tests



Specimen holder for thermal creep tests

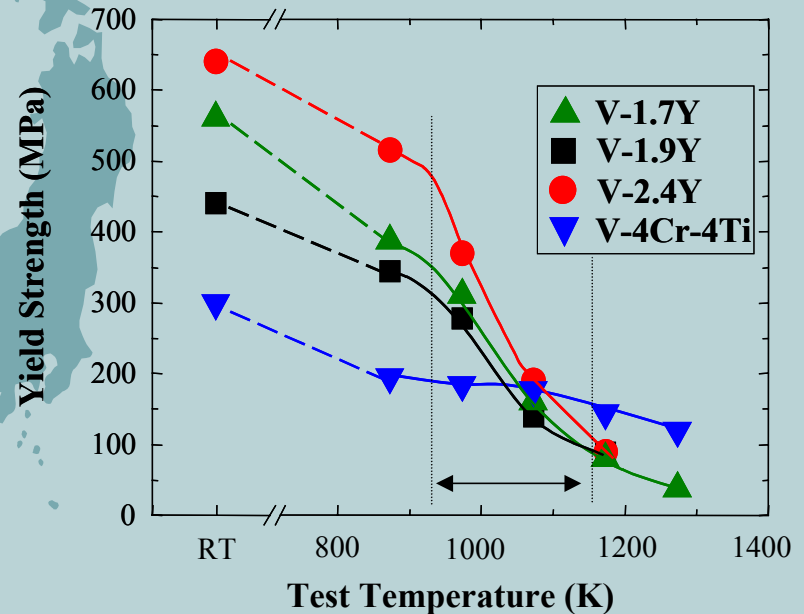
- ⌘ Thermal creep tests in Li is in progress
- ⌘ Irradiation tests in Li is going on in HFIR

# Improvement of Vanadium Alloys



V-xCr-4Ti alloy

- Cr level may be increased to ~7 wt% without degrading fracture properties



Mechanically Alloyed V-xY

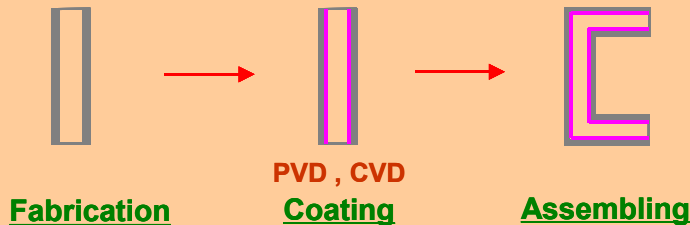
- Higher strength at <800C relative to V-4Cr-4Ti

# In-situ $\text{Er}_2\text{O}_3$ Coating on V alloy

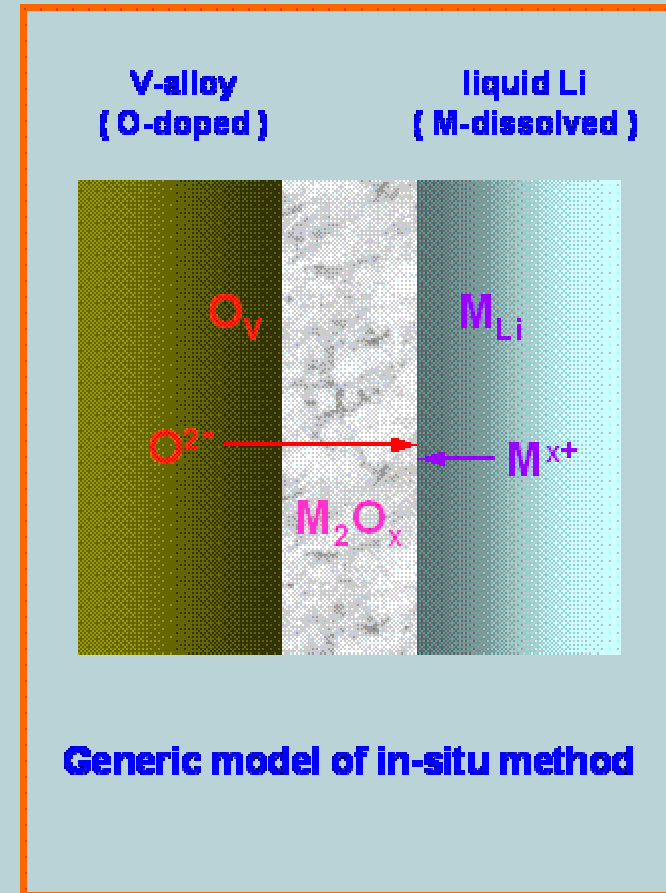
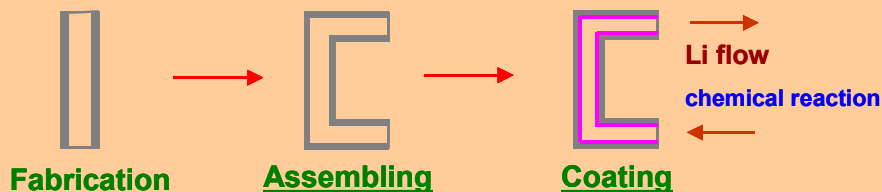
- $\text{Er}_2\text{O}_3$  was identified as promising candidate MHD insulator coating material (JUPITER-II)
- In addition to PVD coating, in-situ method has been developed.
- Advantages of in-situ coating
  - Coating on complex component
  - Healing without disassembling coating

## Type of coating

### a) Ex-situ coating



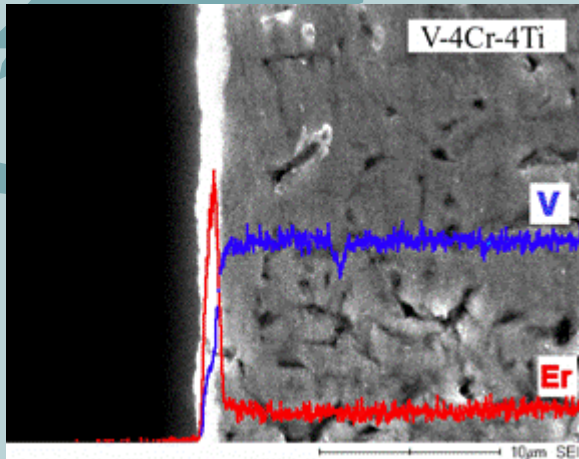
### b) In-situ coating



# In-situ Formation of $\text{Er}_2\text{O}_3$ Layer

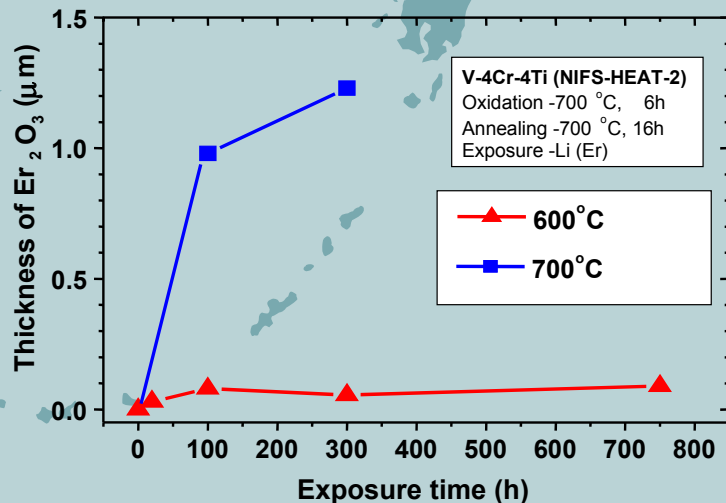
$\text{Er}_2\text{O}_3$  coating formed successfully on V-4Cr-4Ti

SEM and EDS of cross section



oxidized	700°C, 6h
annealed	700°C, 16h
exposed in Li (Er)	600°C, 300h

XPS and ERD confirmed the  $\text{Er}_2\text{O}_3$  phase



$\text{Er}_2\text{O}_3$  layer grow and saturate

Stable to 750h at 600C

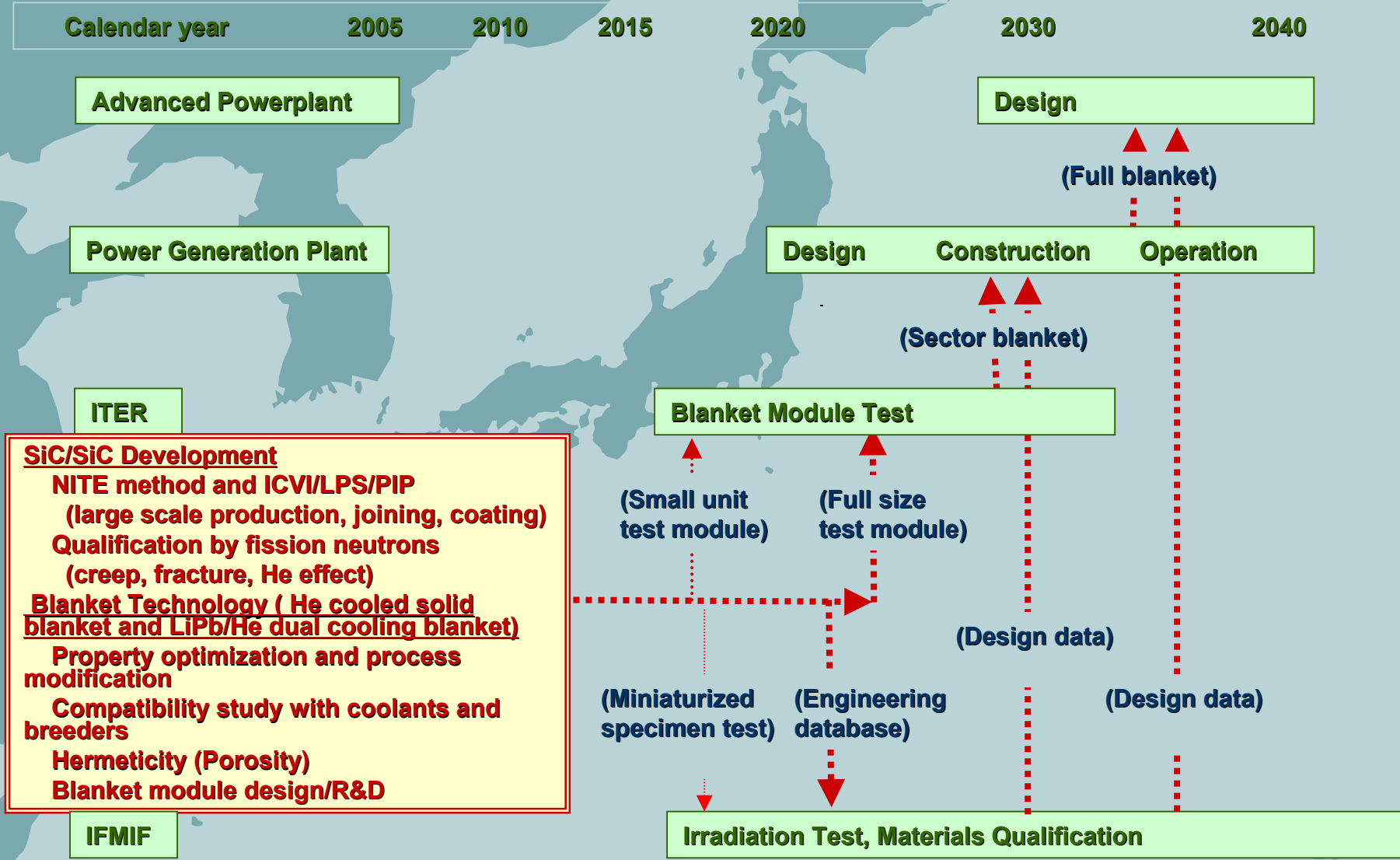




***Research Emphasis and Status (3)***

**SiC/SiC Composite Materials**

# Roadmap for SiC/SiC Composite Development



**SiC/SiC Development**  
 NITE method and ICVI/LPS/PIP  
 (large scale production, joining, coating)  
 Qualification by fission neutrons  
 (creep, fracture, He effect)  
**Blanket Technology ( He cooled solid  
 blanket and LiPb/He dual cooling blanket)**  
 Property optimization and process  
 modification  
 Compatibility study with coolants and  
 breeders  
 Hermeticity (Porosity)  
 Blanket module design/R&D

# What Types of Blanket System with SiC/SiC

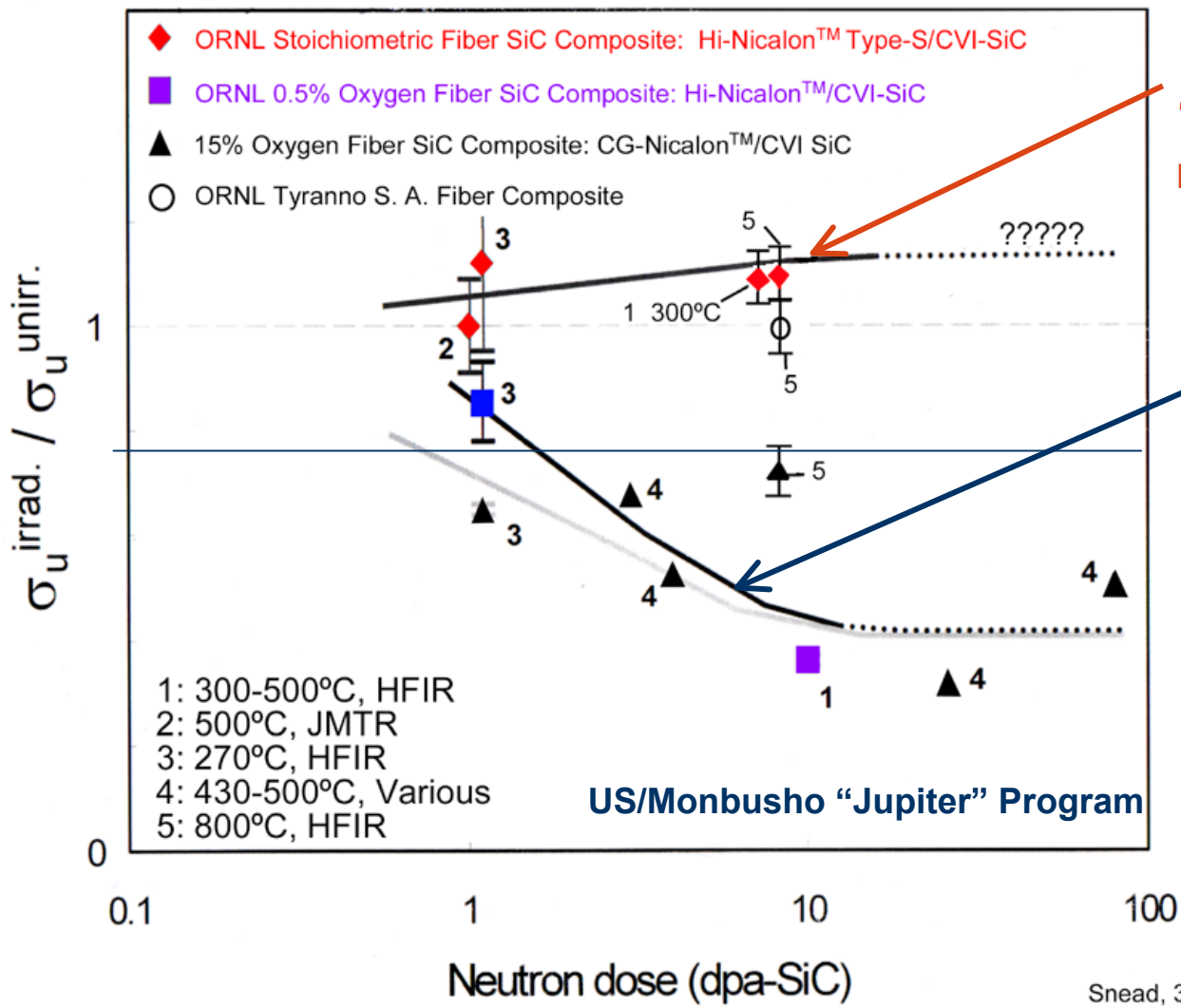
## 1: He Cooled Solid Blanket

- a) The SiC/SiC has to be compatible with He, Solid breeder and Be at high temperature
- b) SiC/SiC should keep **high thermal conductivity** and low tritium retention. “sealing layers” is an option
  - a) the degradation of thermal conductivity under neutron irradiation is critical from heat flow point of view
- c) The degradation of mechanical properties under high fluence neutron irradiation at elevated temperature is an issue.

## 2: He/Li-Pb Dual Cooled Liquid Blanket

- a) The SiC/SiC inserts have to be compatible with Pb-17Li and He
- b) SiC/SiC should keep **low electrical conductivity** and low tritium retention. “sealing layers” is an option
  - a) the change in conductivity is critical from pressure drop point of view and flow balance
- c) The degradation of mechanical properties under high fluence neutron irradiation at elevated temperature is an issue

# We Now Have First Radiation-Resistant SiC Composite



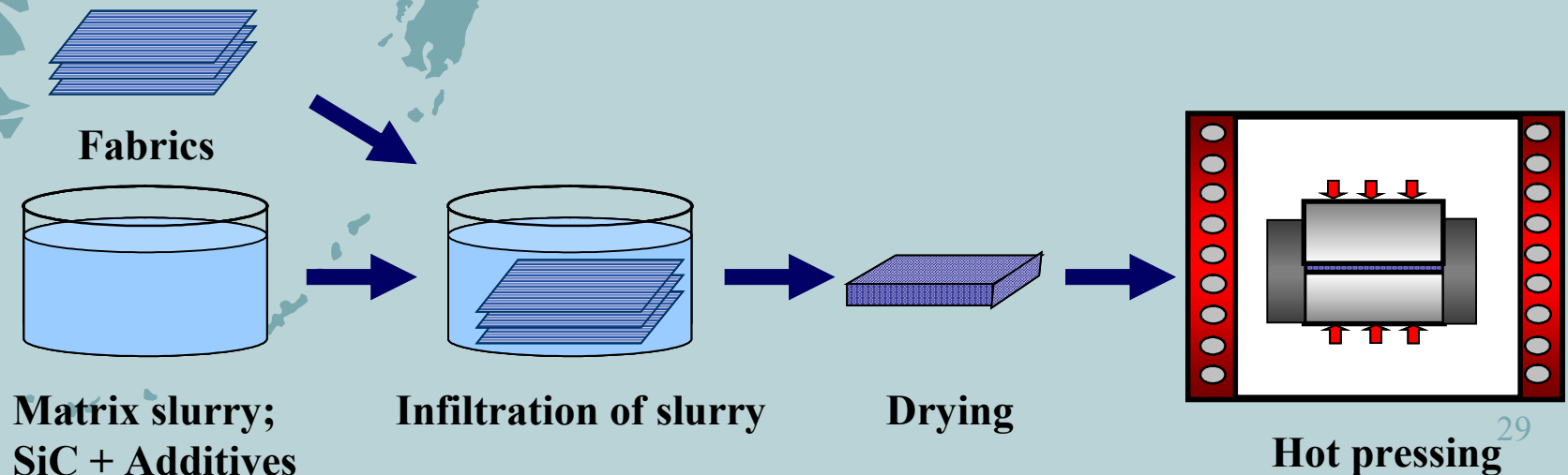
Bend strength of irradiated "advanced" composites show no degradation up to 10 dpa

1st- and 2nd generation irradiated SiC/SiC composites show large strength loss after doses >1 dpa

# A Solution to the severe requirements for Nuclear Application is NITE Process

## NITE: Nano-Infiltration Transient Eutectic Phase Process

- High Density and Excellent Hermeticity
- High thermal conductivity
- Chemical stability
- Flexibility in size and shape
  - Applicability of existing near net-shaping techniques
- Low production cost



# Why NITE Process is the solution ?

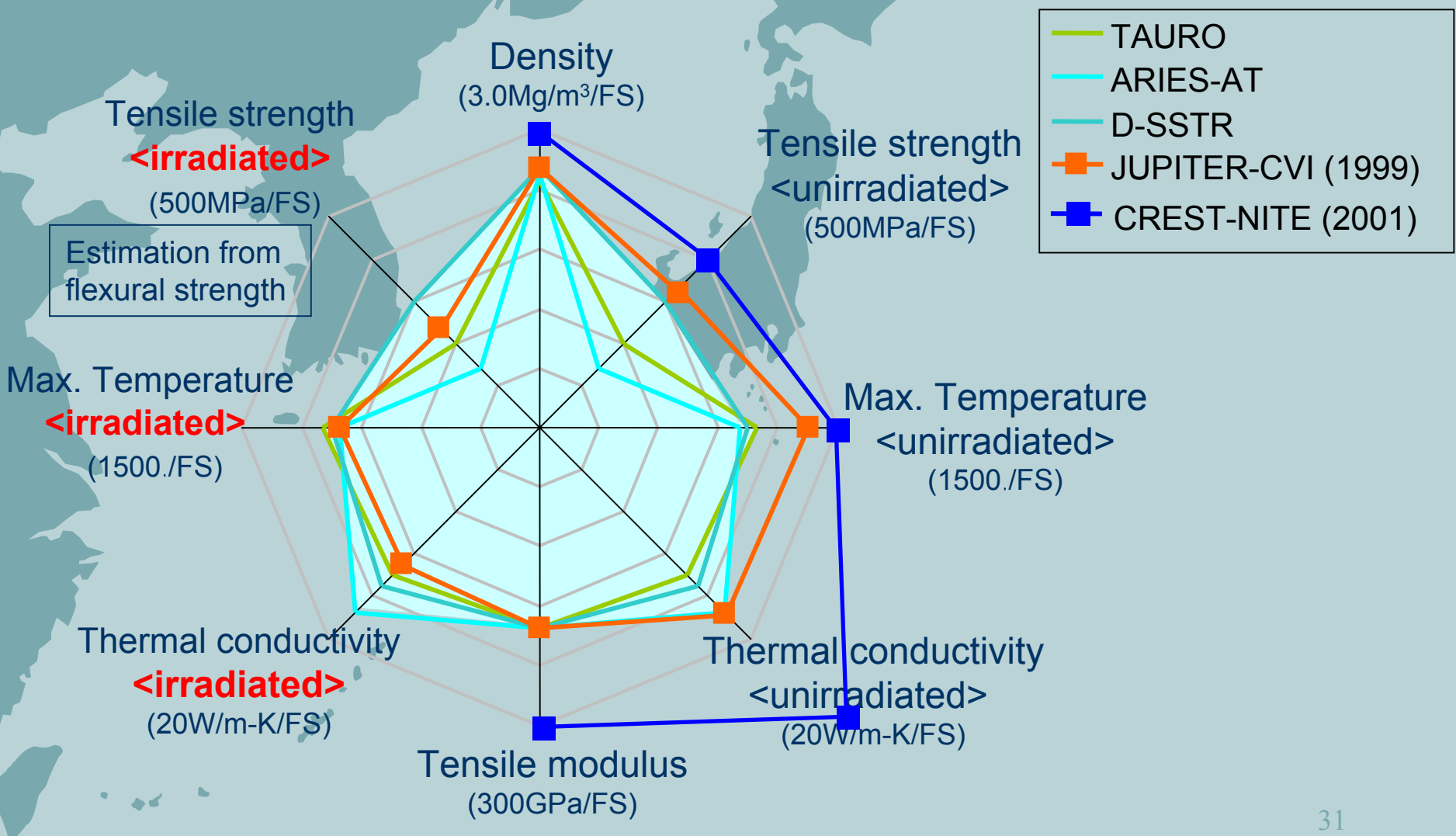
- **The Combinations of :**
  - **Near stoichiometry SiC fiber:**  
Heat resistance in inert environment  $\sim 1800^{\circ}\text{C}$
  - **Nano Powder:**  
Large specific surface area  
Flexibility in chemistry adjustment and surface control
  - **Protective interphase (fiber coating):**  
CVD-carbon, Phenolic resin-derived carbon, CVD-BN
  - **Nano powder-based matrix slurry:**  
Infiltration to intra-fiber bundle openings  
Minimize binding agents to reduce detrimental impurities



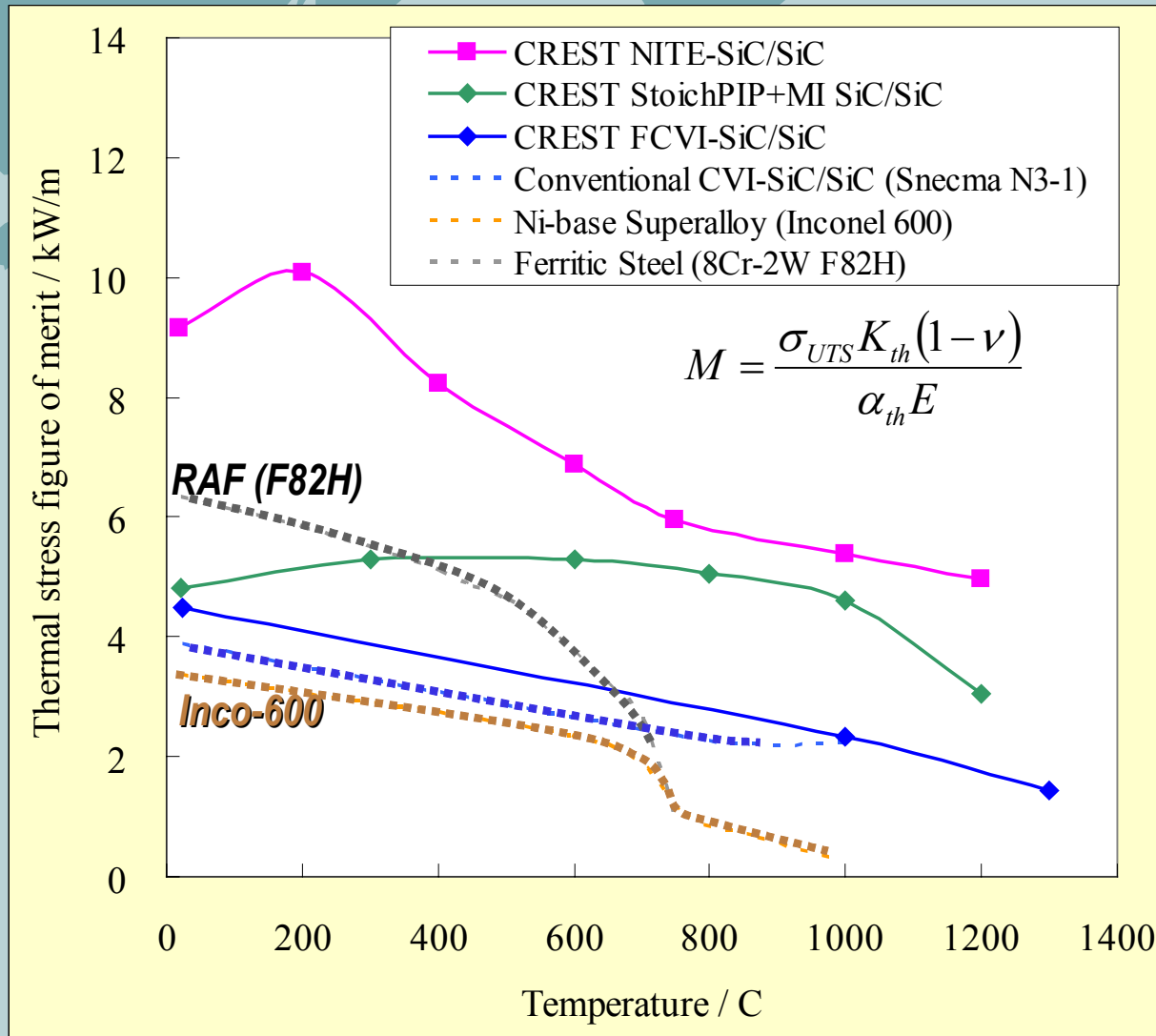
*Make it possible to produce SiC/SiC*

**High purity and highly crystallized fiber and matrix  
with sound interface and optimized microstructure**

# Current Status of NITE Composites



# Thermal/Mechanical Properties of NITE Composites



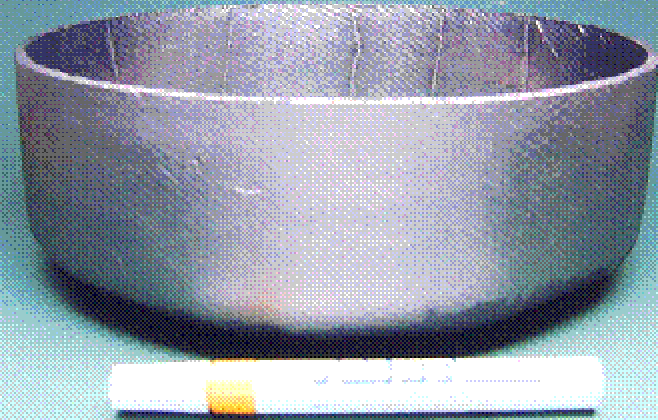


# Shape Flexibility of NITE Composites

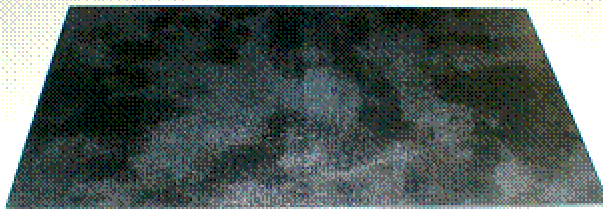
**Thick Block: 97 x 97 x 70mm**



**Combustor Liner**



**Thin Plate: 195 x 195 x 2.0mm**

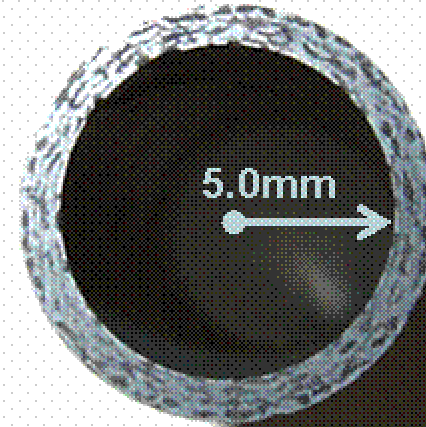
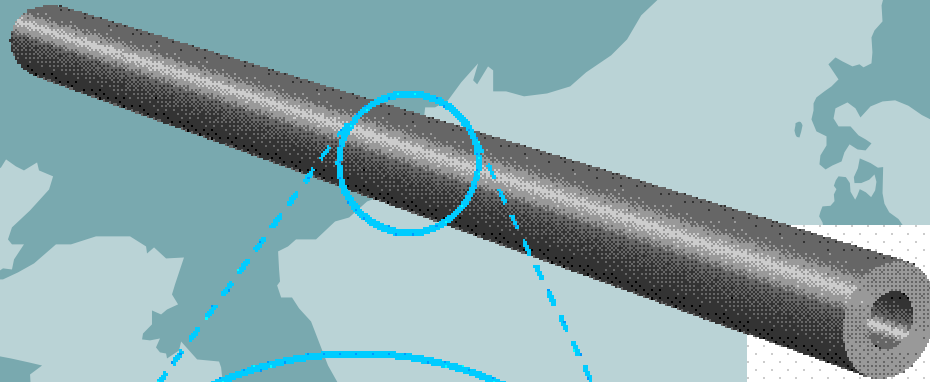


**Tube:  $\Phi$ 31-33 x 60.0mm**



# 2D SiC/SiC Fuel Pin for GFR by NITE Process

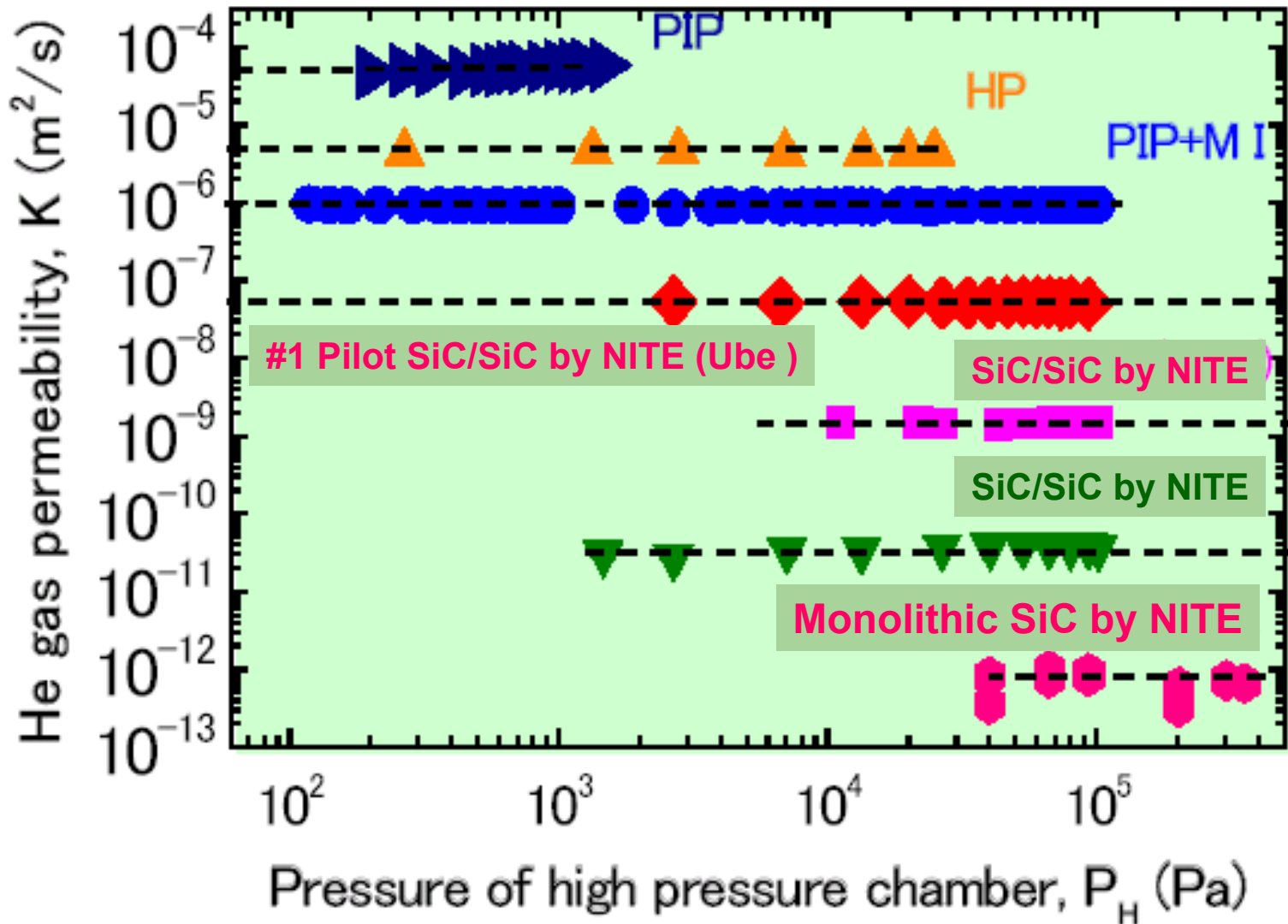
- The first trial at Ube Co. Ltd., 9/12/03 -



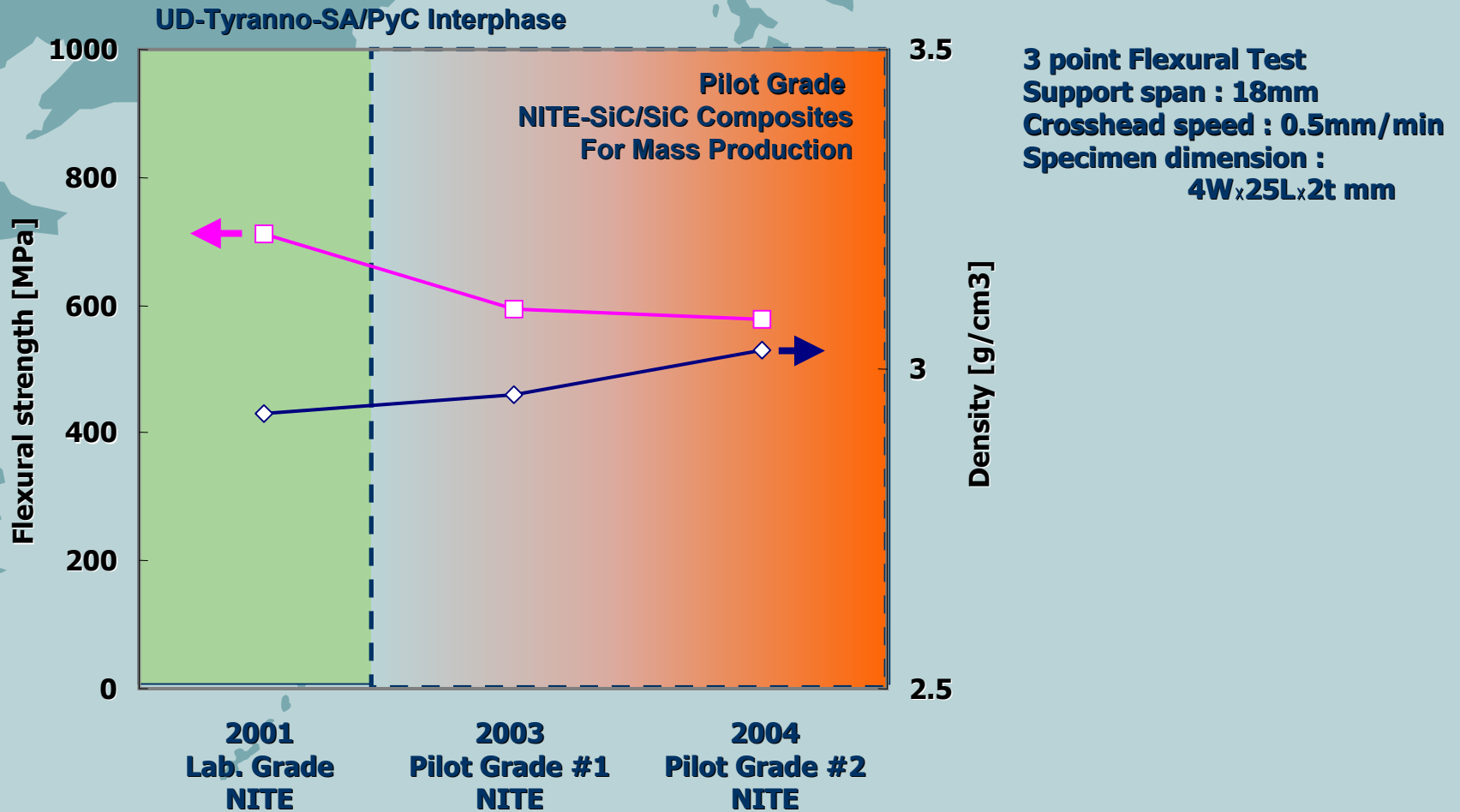
Wall thickness: 1.0mm

Goal: 3m(length) x 10mm(inner diameter) x 1mm(wall thickness)

# Permeability of NITE Composites



# Progress in Large Scale Fabrication of NITE Composites at Ube Industries Co.

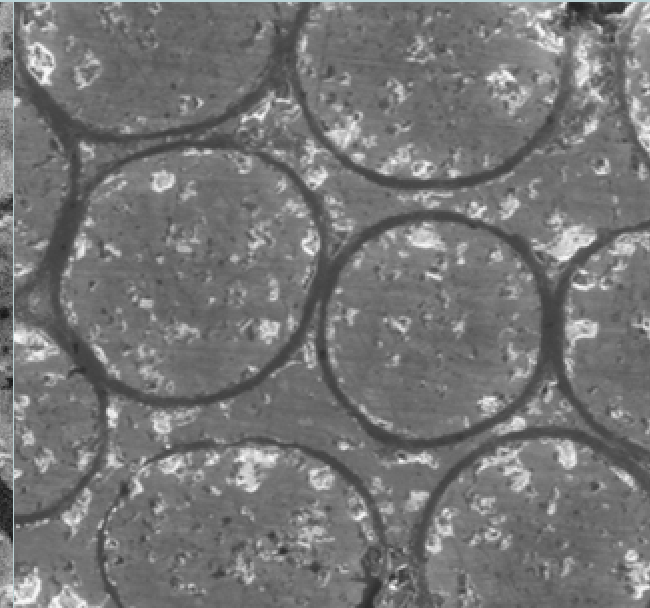
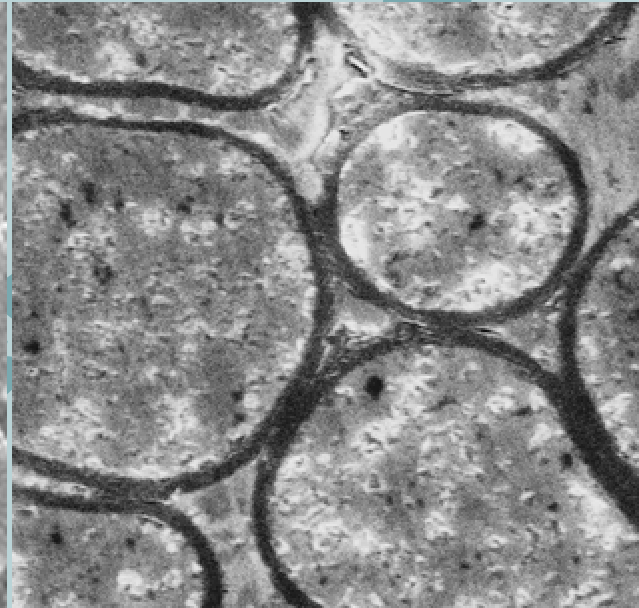
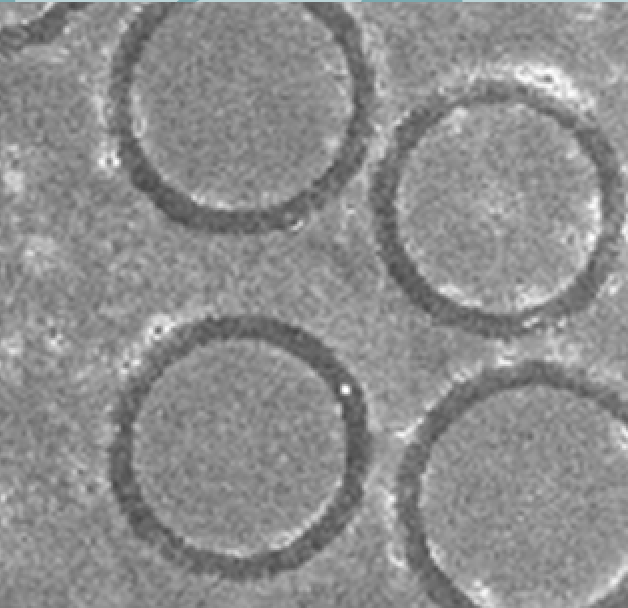


# Progress in Large Scale Fabrication of NITE Composites at Ube Industries Co.

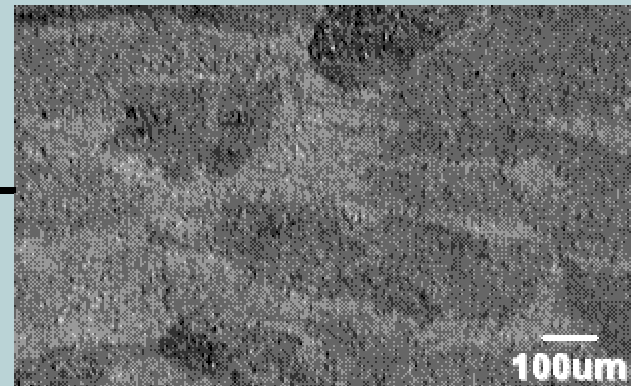
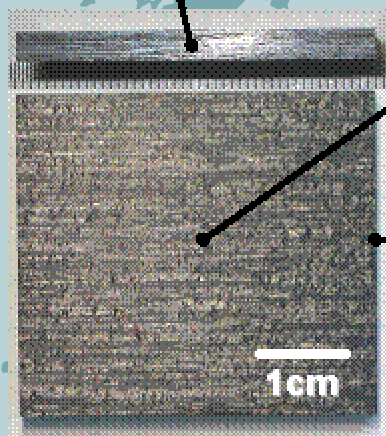
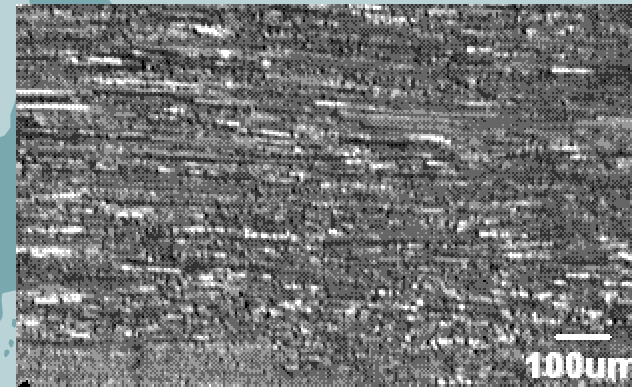
2001 Lab. Grade  
NITE-SiC/SiC Composites

2003 Pilot Grade #1  
NITE-SiC/SiC Composites

2004 Pilot Grade #2  
NITE-SiC/SiC Composites



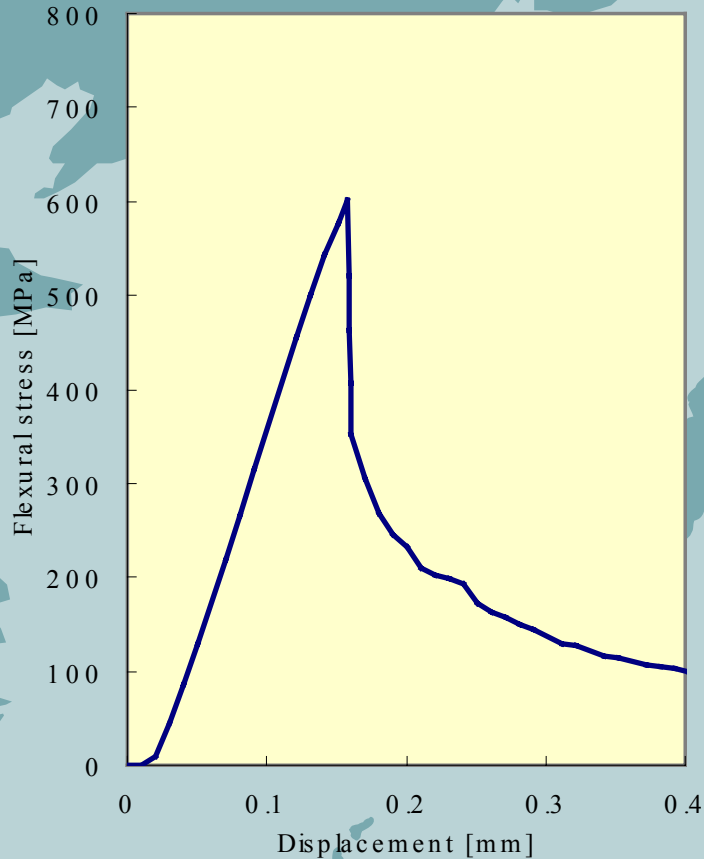
- ❑ Specimen Size: 40W×45L×4Tmm (27 UD-prepreg sheets stacking)
- ❑ Fiber: Tyrano SAK (7.5μm.800fiber/bundle)
- ❑ Fiber Coating: Pyro-Graphite.0.5μmt
- ❑ Vf: 45vol%
- ❑ Fabrication Process: NITE method (1800., 1hr, 200kg/cm<sup>2</sup>., in Ar )



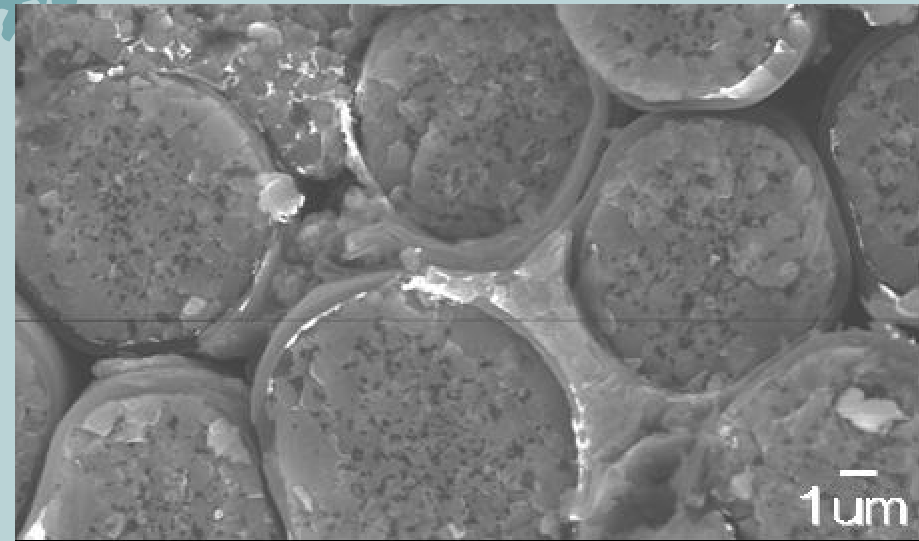
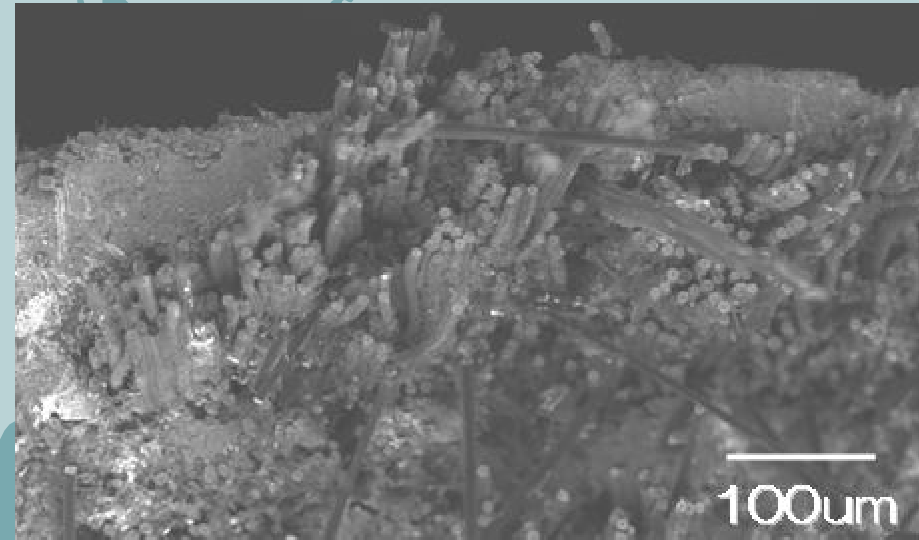
**Dimension : 36.48W×40.5L×3.34T mm**  
**Weight : 14.98 g / Bulk Density : 3.03 g/cm<sup>3</sup>**

# Three Point Bending Test Results (Pilot Grade #2-1)

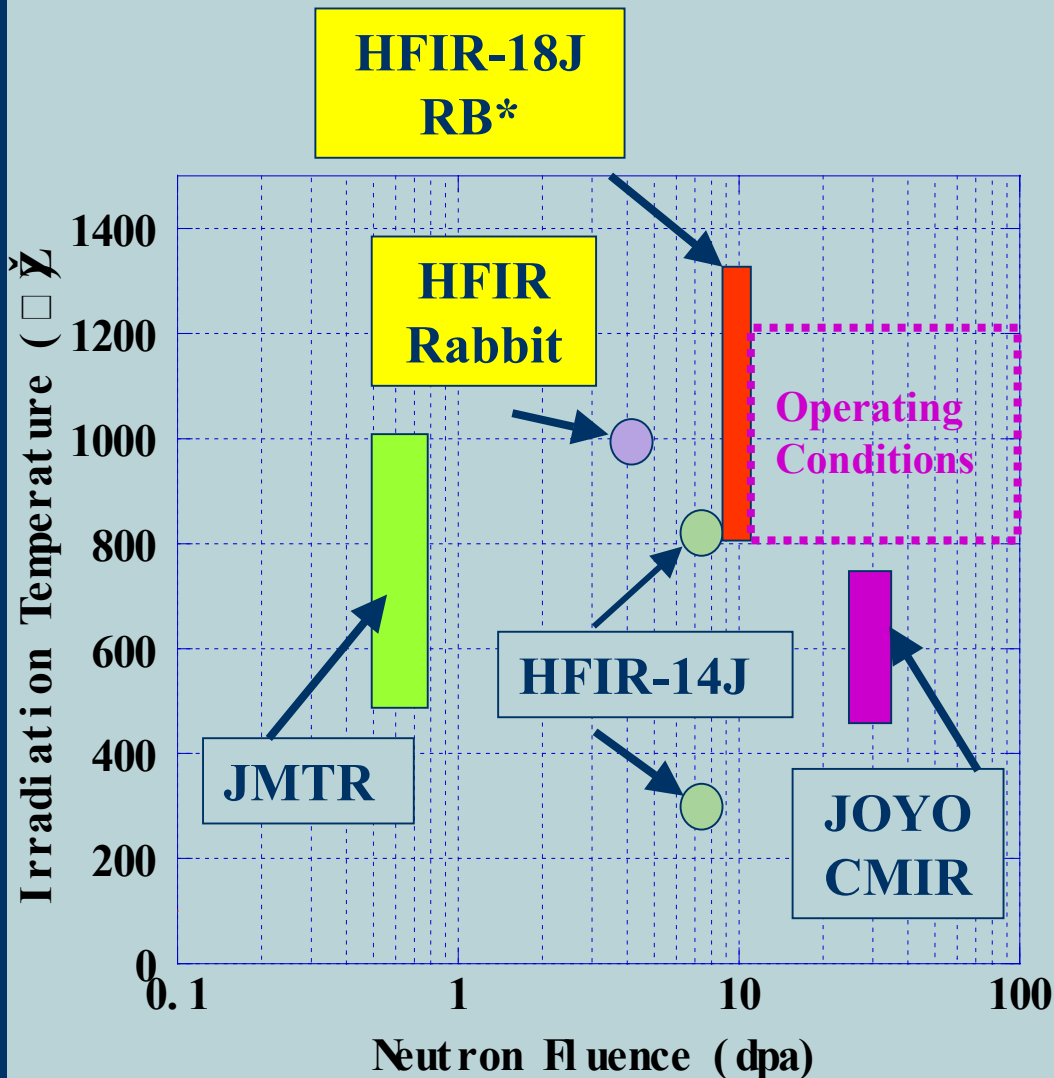
- Specimen Size: 4.2Wx25.2Lx1.7t mm
- Span.18 mm
- Cross Head Speed: 0.5 mm/min



- Three Point Bend Strength: 601 MPa
- Elastic Modulus.150 GPa
- Fracture Mode.  
Pseudo Elasticity with Fiber Pull Outs



# Neutron Irradiation Research Status



## Issues to be studied

.Max. Temperature Limit under Irradiation

.Compatibility of solid breeding materials and SiC or SiC/SiC

.Irradiation behavior of joining or coating



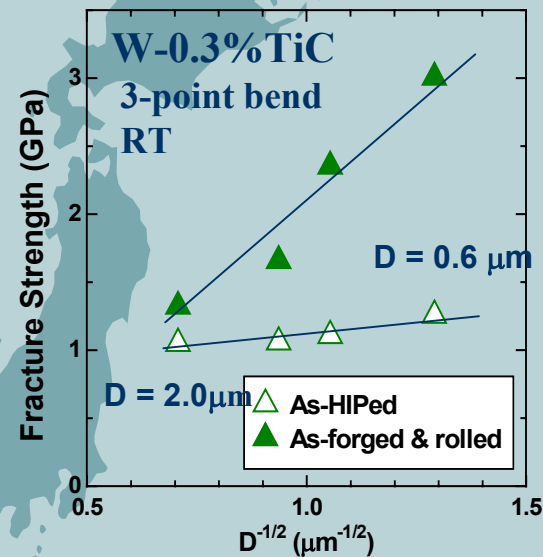
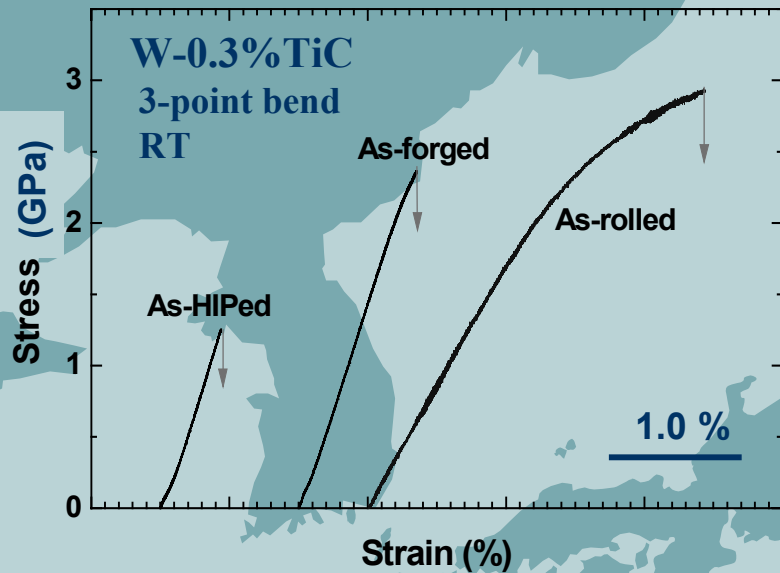
Feasibility studies and design activities have been started since 2003.



***Research Emphasis and Status (4)***

**Refractory Alloys**

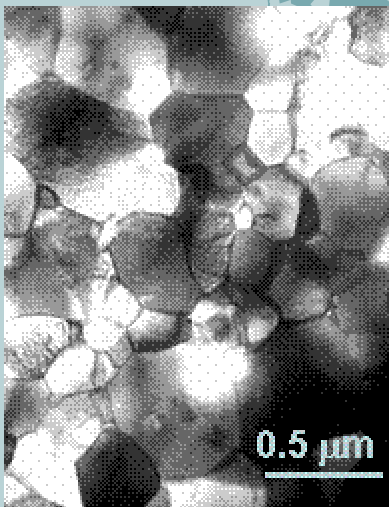
# Development of Ultra-Fine Grained, Nano-Particle Dispersed W-TiC Alloys with Improved Ductility



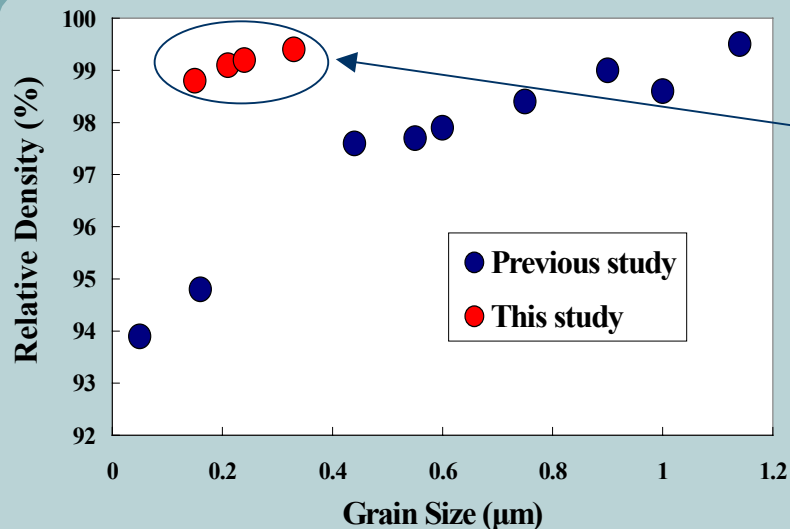
The beneficial effect of hot working becomes prominent with decreasing grain size of HIPed compacts.



Need of consolidated body with very small grain size less than  $0.5 \mu\text{m}$

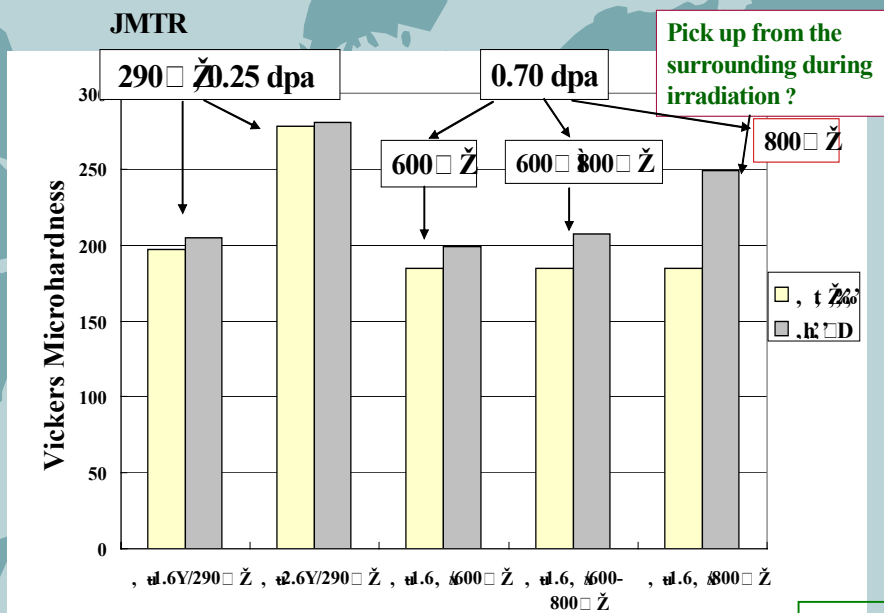
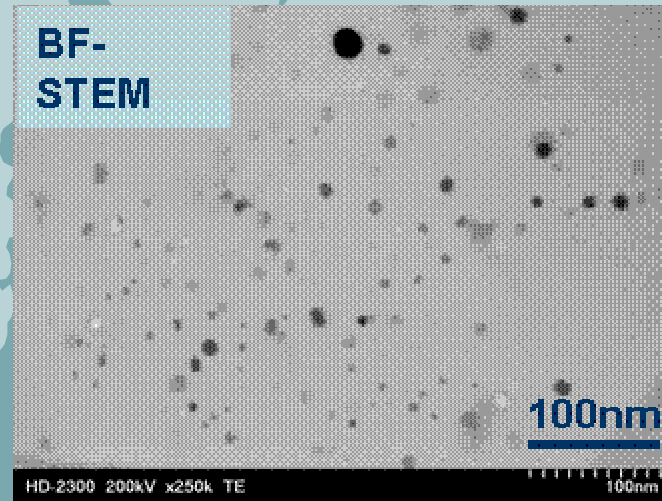
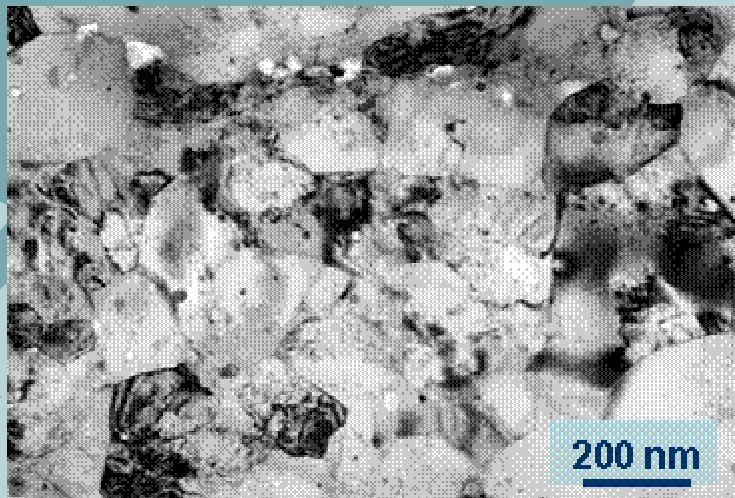


Relative Density vs Grain Size

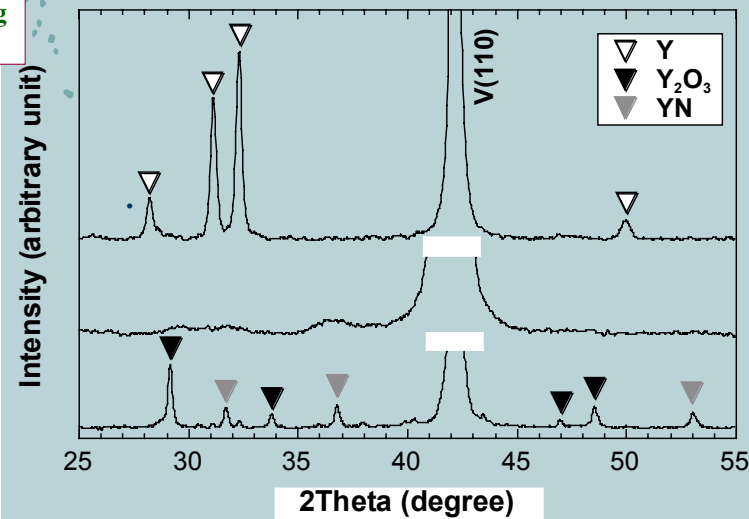


Success in fabrication of ultra-fine grained W-TiC with a high relative density of  $\sim 99\%$

# Development of Ultra-Fine Grained, Nano-Particle Dispersed V-Y Alloys with Good Radiation Resistance



## V-1.7Y



Less hardening by JMTR irradiation

MA causes the added Y particles to dissolve into V.  $Y_2O_3$  and YN form by reaction of Y with solute O and N during HIP.

***Research Emphasis and Status (5)***

**Modeling and Mechanistic Studies**

# Emphasis in Modeling and Mechanistic Studies

## ■ Issues of simulation-fusion correlation

- Recoil spectrum
- Transmutation
- Damage rate
- Temperature variation

## ■ Materials performance mechanisms

- Fracture and deformation of irradiated materials

## ■ Test technology

- Qualification of small scale test technology

## ■ System oriented issues

- Life time evaluation

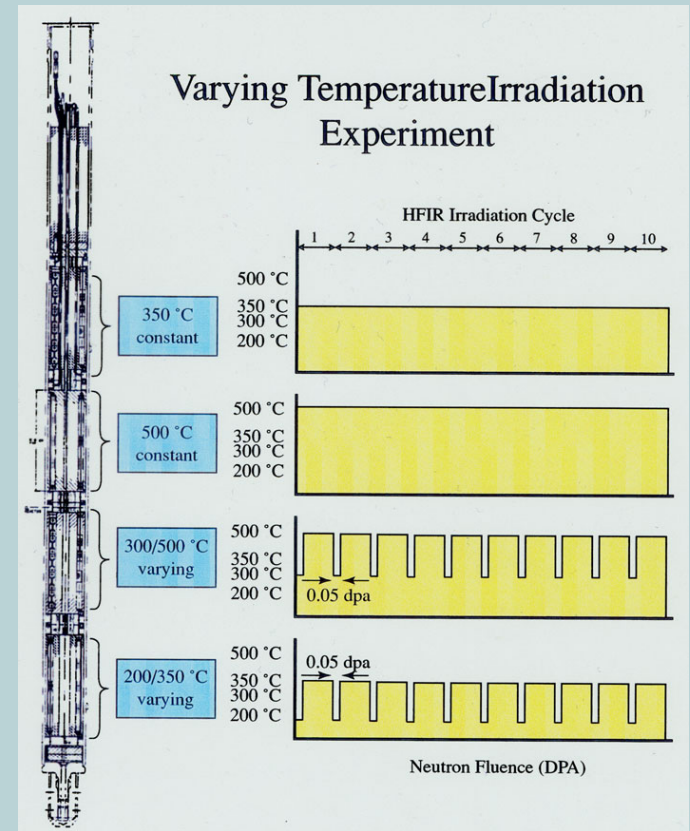
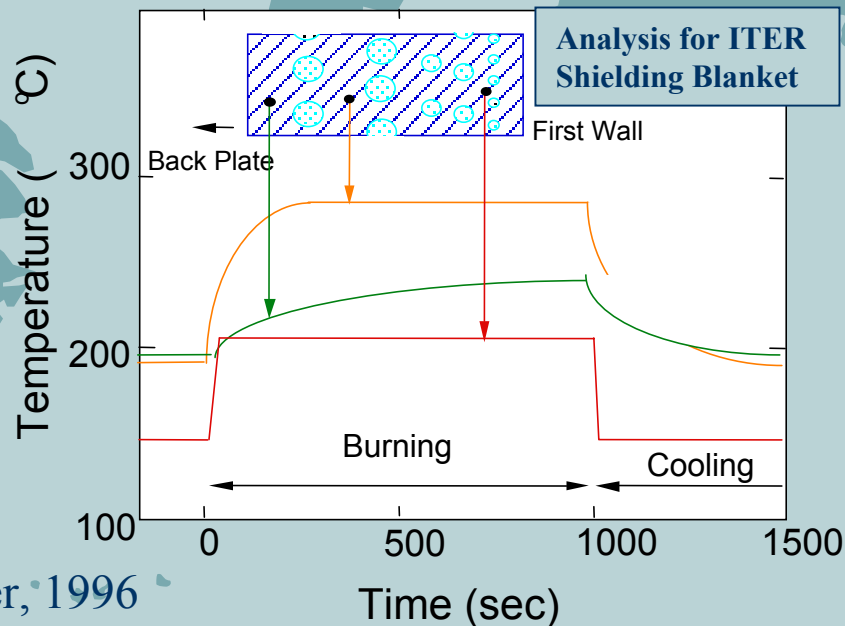
# Multi Scale Modeling

- For understanding materials performance under irradiation, analyses of physical phenomena in quite wide range of time and scale are required
  - psec to decades
  - sub nm to m
- Various modeling with quite different length and time scales are developed and interrelated with each other
  - First principle atomistic modeling of point defect properties
  - Molecular Dynamics simulation of primary defect production
  - Kinetic Monte Carlo simulation of defect diffusion
  - Rate Theory of defect accumulation, solute segregation and precipitation, void swelling and creep
  - 3-D Dislocation Dynamics simulation for mechanical property change
- Multi Scale Testing and Evaluation Research (MUSTER) facility is introduced in IAE Kyoto University including JEOL F-2200 FE TEM with omega filter

# Varying Temperature Irradiation Experiment

- Fusion reactor materials will be subject to temperature transient during irradiation
- Symmetrical isothermal and temperature-variant irradiation was carried out in HFIR by JUPITER project and have been conducted in JMTR

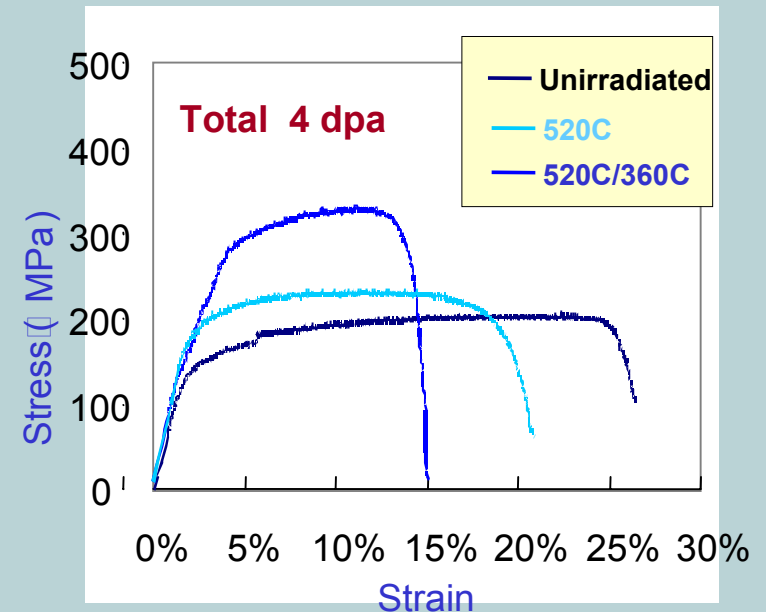
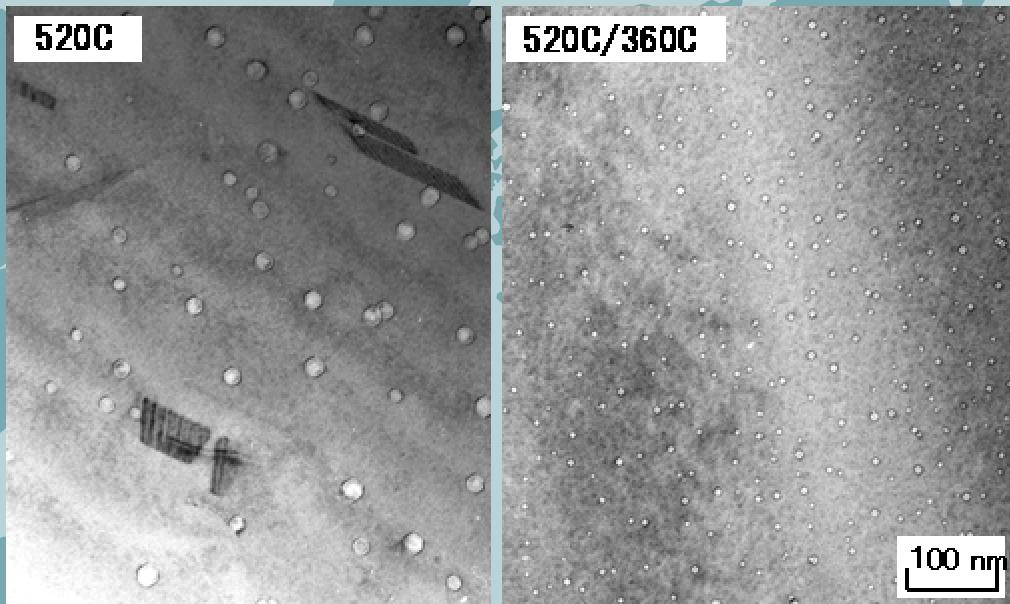
- 10% low temperature excursion



Muroga, 2001

# Examples of Varying Temperature Effects

- Lower temperature transient resulted in increase in void density and decrease in void size in unalloyed vanadium
  - Mechanical property is consistent with the microstructures
  - The difference explained by rate theory analysis
  - However, the temperature transient effects are generally more moderate for radiation-resistant materials (e.g. V-4Cr-4Ti)



Watanabe, Fukumoto unpublished



# Summary (1)

- The realization of fusion power system with advantages in safety, environment and economy depends on the development of high-performance and low activation materials
- Efforts are focused on developing RAFM, vanadium alloys and SiC/SiC composites
  - Compositional and microstructural optimization to yield the necessary properties
  - Technology development specific to the design concepts
- Irradiation effects with helium is the common critical issue
  - Strongly motivates intense 14MeV neutron irradiation facility

# Summary (2)

- **Continuous use of the present irradiation facilities in elaborate manners will enhance the efficiency and soundness of the materials development**
- **Modeling and fundamental studies of radiation effects play a very important part in enhancing the predictability of materials performance in fusion conditions based on the limited irradiation data.**



## 核融合炉用低放射化フェライト JLF-1 鋼

大同 9 トン 炉溶解スラブ

〔インゴット〕

表1 溶解材の化学組成 mass%

C	Si	Mn	P	S	Cr	V	Al	Ta	W	Ni	Co	Mo	B	N
0.10	0.10	0.46	0.003	0.003	8.94	0.20	0.002	0.08	1.99	0.06	0.001	0.006	0.0003	0.019

〔スラブサイズ〕

- ・ 厚さ 130×幅 1200×長さ 2000 →40mm 厚に圧延
- ・ 厚さ 110×幅 1200×長さ 2000 →18mm または 25mm 厚に圧延

〔製造条件(予定)〕

20 ≤ t ≤ 40mm: 1150°C加熱-860°C CR-CLC	760°C Temper
10 ≤ t < 20mm: 1150°C加熱-900°C CR-AC	760°C Temper