Materials for Generation IV Nuclear Reactors
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Research and Development of Oxide Dispersion Strengthened Ferritic Steels for Sodium Cooled Fast Breeder Reactor Fuels

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10 R&D Sites

Tsuruga
Prototype fast reactor MONJU, Decommissioning of advanced thermal reactor FUGEN

Tono
High-level rad-waste research

Horonobe
High-level rad-waste research

Aomori
Decommissioning of nuclear ship, (ITER BA), etc.

Ningyotoge
Decommissioning of uranium enrichment plants

Tokai
Basic research, Safety studies, Neutron Science, Nuclear fuel cycle technologies, Rad-waste management and disposal, etc.

Kansai
Photon & synchrotron radiation science

Takasaki
Radiation science

Naka
Fusion R&D, ITER support

Oarai
Experimental reactors JOYO, HTTR and JMTR, Advanced reactor R&D including FBR cycle commercialization

Established on October 1, 2005 by integrating JAERI and JNC
Annual Budget: ～200BJPY, Employees: ～4,400

Outline

1. Fast Reactor Cycle Development in Japan

2. Introduction to ODS Steels

Oxide Dispersion Strengthened: ODS

3. Alloy Design

4. Microstructure Control
   (Nano-Size Oxide Particles)
   (Grain Morphology)

5. Mechanical Properties

6. Irradiation Tests

7. Future Mass Production

8. Conclusions

Chapter 1. FR Cycle Development in Japan

- “Feasibility Study” on Commercialized Fast Breeder Reactor Cycle Systems was conducted from JFY1999 to 2005.

- The final reports were compiled in March 2006, and reviewed by the Government (AEC, MEXT, METI).

- The Most Promising Concepts to Commercialize FR Cycle

Reactor:	**Sodium-cooled Fast Reactor with MOX fuel**
Fuel Cycle:	**Advanced Aqueous Reprocessing**
	Simplified Pelletizing Fuel Fabrication

- FR cycle technology development has advanced from “Feasibility Study” to “Project” since 2006.

AEC: The Japan Atomic Energy Commission
MEXT: The Ministry of Education, Culture, Sports, Science and Technology
METI: The Ministry of Economy, Trade and Industry

FR Cycle Development Program

2005 2010 2015 (JFY)

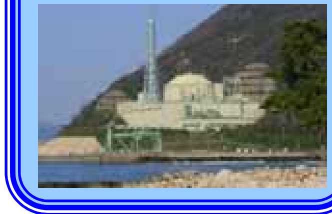
Feasibility Study
(JFY 1999-2005)

Identify The Most Promising Candidate Concept

C&R

Review & Basic Policy by MEXT &AEC

Experimental FR "JOYO"



R&D at Prototype FBR "MONJU"

Fast Reactor Cycle Technology Development Project (FaCT)

Decision of Innovative Tech. (2010)

Approved Confirmation (2015)

R&D of Innovative Technologies

Conceptual Design of Commercial & Demonstration FR Cycle Facilities

Commercialized FR Cycle

R&D at "MONJU"

- ◆ **Demonstrating its Reliability as a Operation Power Plant**
- ◆ **Establish Sodium Handling Tech.**

2015

Production of the Conceptual Designs of Commercial and Demonstration FR Cycle Facilities with R&D Programs

Basic Design & Construction

2025

Operation Start of Demonstration FR & its Fuel Cycle Facility

Validation of Economy & Reliability

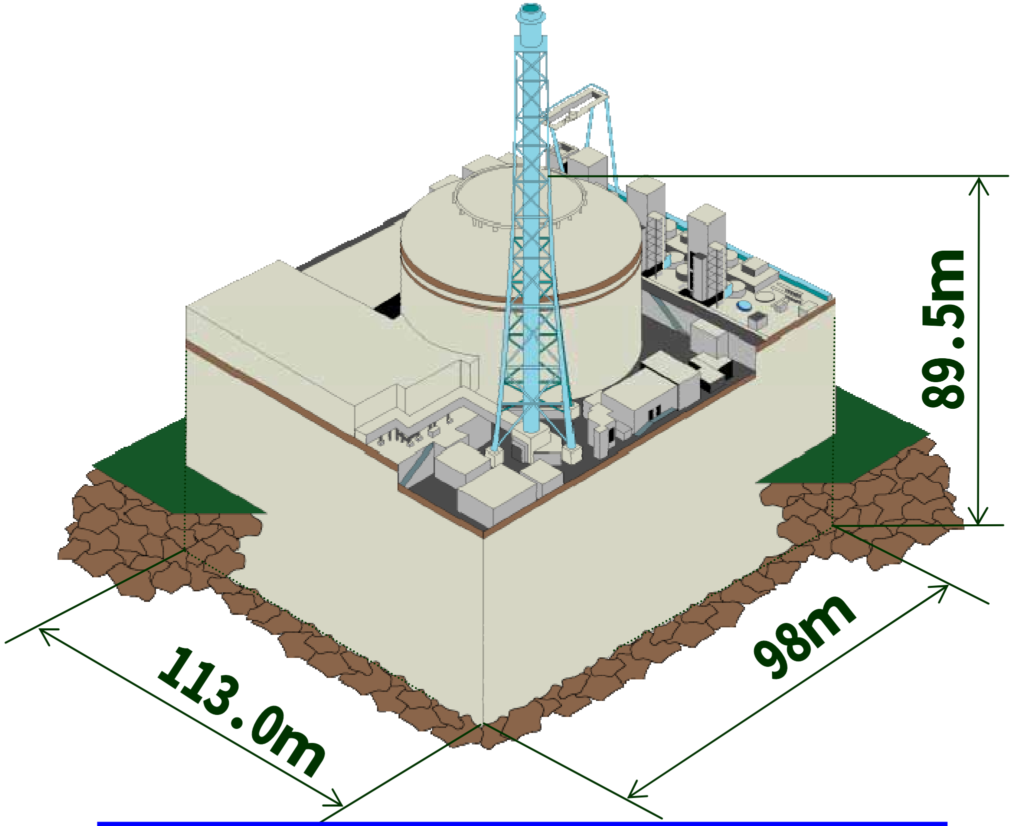
2050

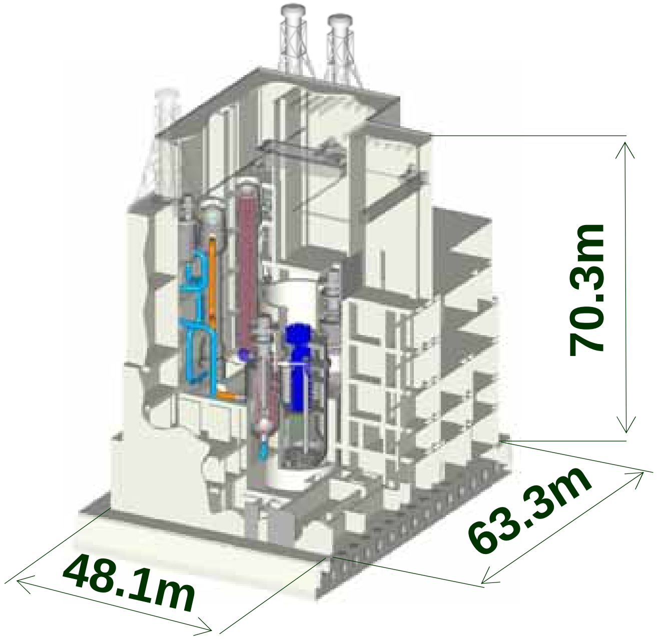
Commercially Introducing of FR Cycle Facilities

◆**Cooperation with related Organization** ◆**International Cooperation** (**GNEP, GEN-IV, INPRO etc.**)

Comparison of Reactor Size

Electricity output of JSFR is about 5 times larger while site area is about 1/4.





Prototype FBR MONJU
Thermal Output: 714 MWt
Electricity Output: 280 MWe

Japanese Sodium cooled Fast Reactor (JSFR)
Thermal Output: 3,570 MWt
Electricity Output: 1,500 MWe

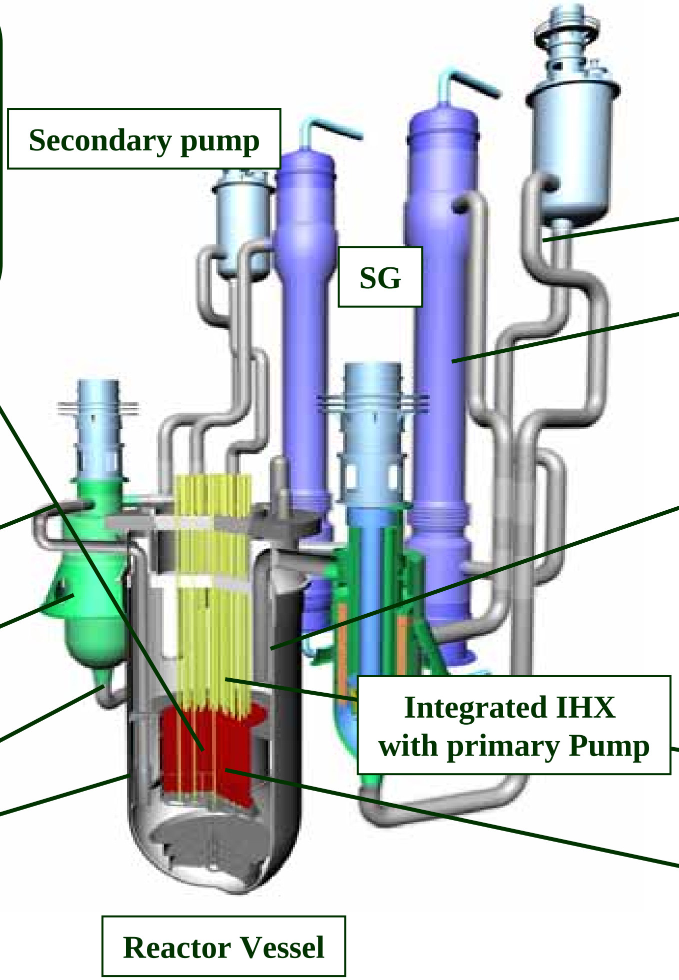
Japanese Sodium-cooled Fast Reactor (JSFR)

- 1,500 MWe large-scale SFR with MOX fuel,
- Innovative technologies for enhancement of reactor core safety, high economic competitiveness and countermeasures against specific issues of sodium

ODS Steels for cladding tube to achieve high burn-up at elevated temperatures

Innovative technologies to reduce plant materials and reactor building volume

- Two-loop cooling system
- Shortening of piping with high chromium steel
- Integrated Pump-IHX Component
- Compact reactor vessel



Prevention of sodium chemical reactions

- Double-wall piping
- High reliable SG with double-wall tube

Inspection and repair technology under sodium

Enhancement of reactor core safety

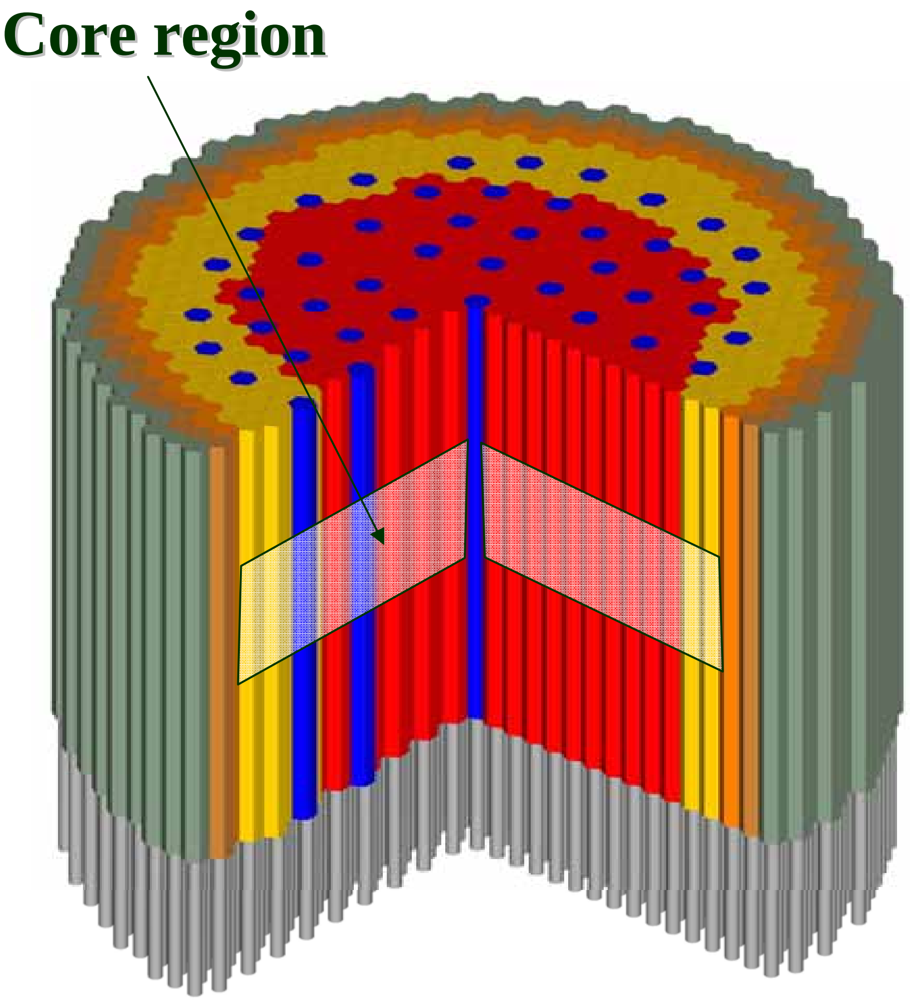
- Passive reactor shutdown system and decay heat removal by natural circulation
- Re-criticality free core

Reactor Core Configuration of JSFR

Neutronic Characteristics

Output power (MWt)	3570
Cycle length (months)	26
Refueling batch [core/RB]	4/4
Pu-enrichment (wt%) [Inner/outer]	18.3/20.9
Burnup reactivity (%dk/kk’)	2.3
Breeeding ratio	1.10
Discharge burnup (GWd/t) [core]	147
[core + blanket]	90
Pu fissile inventory (t/GWe)	5.7
Maximum neutron dose (dpa)	250
Sodium void reactivity ($)	5.3

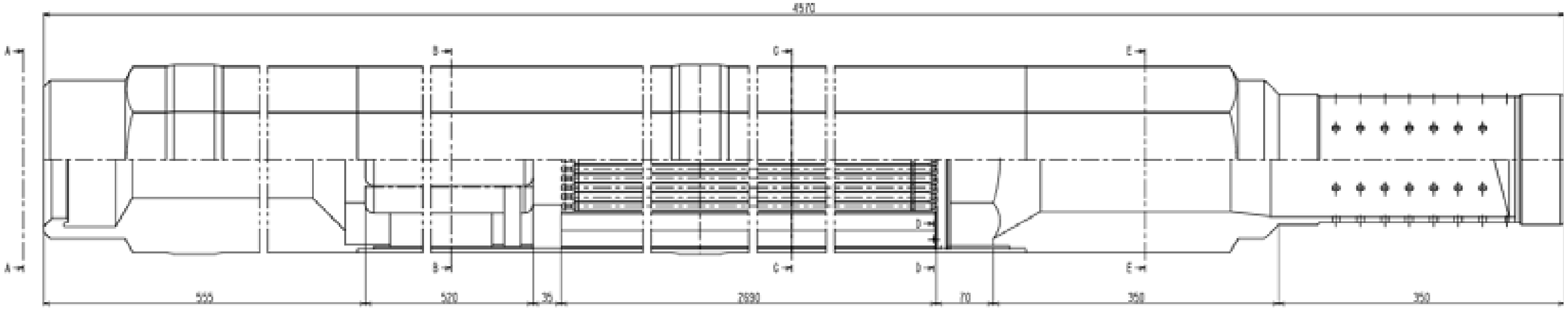
🟥 Inner core
🟨 Outer core
🟧 Radial blanket
🟫 SUS shield
🟩 Zirconium Hydride shield
🟦 Control rod



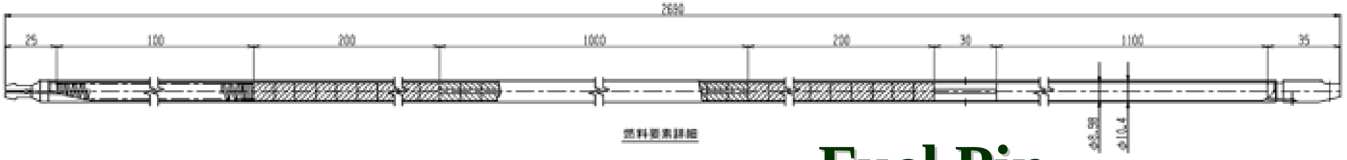
Cut view of reactor core

A Drawing of Core Fuel Pin and Subassembly

An example of design study for 1.5GWe SFR cores

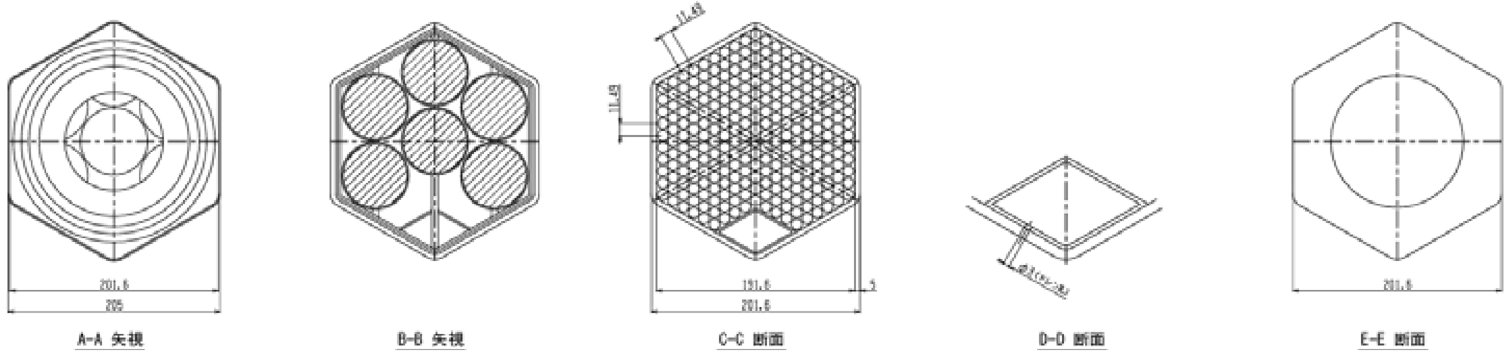


Subassembly



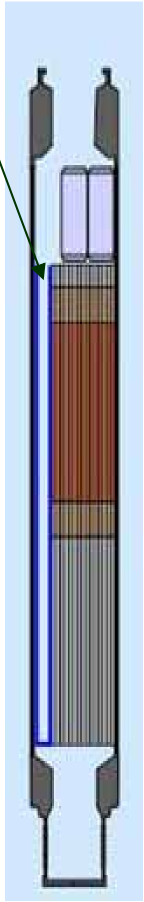
Fuel Pin

255 Pin Bundle



The original figure can be found in JAEA Research 2006-042 (2006), P.249

Molten fuel discharge duct at a corner of wrapper tube



Chapter 2. Introduction to ODS Steels

Crystallographic Consideration against Irradiation Damages

- Body Centered Cubic > Face Centered Cubic

Requirements and Target Performance for ODS Steels

- Reactor and Fuel Cycle
- Fuel Pin Mechanical Design

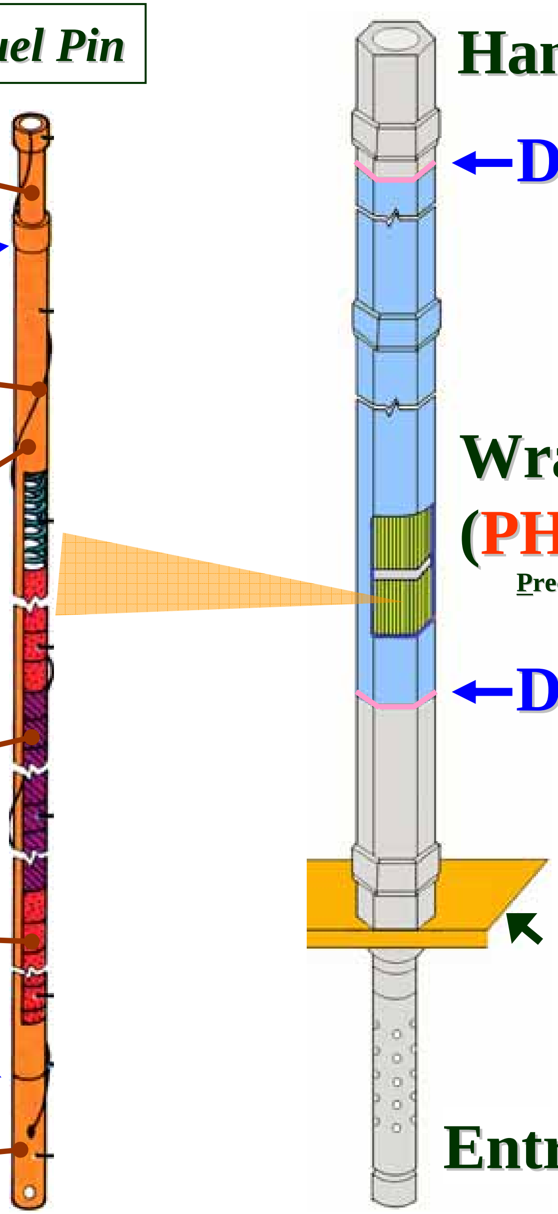
Manufacturing Process

- Powder Metallurgy
- Precision Seamless Tube Production

Break Through (Historical Review)

- Microstructure Control
- Pilger Cold Rolling Process

Materials for Core Fuel Pin and Subassembly



Fuel Pin

Upper End Plug
(ODS Ferritic Steel)

PRW Welded

Spacer Wire
(PH FMS)

Cladding Tube
(ODS Ferritic Steel)

Core Fuel Pellets (MOX)

Blanket Fuel Pellets (UO_2)

PRW Welded

Lower End Plug
(ODS Steel)

Subassembly

Handling Head(SUS316)

←Dissimilar Welded

Wrapper Tube
(PH FMS)

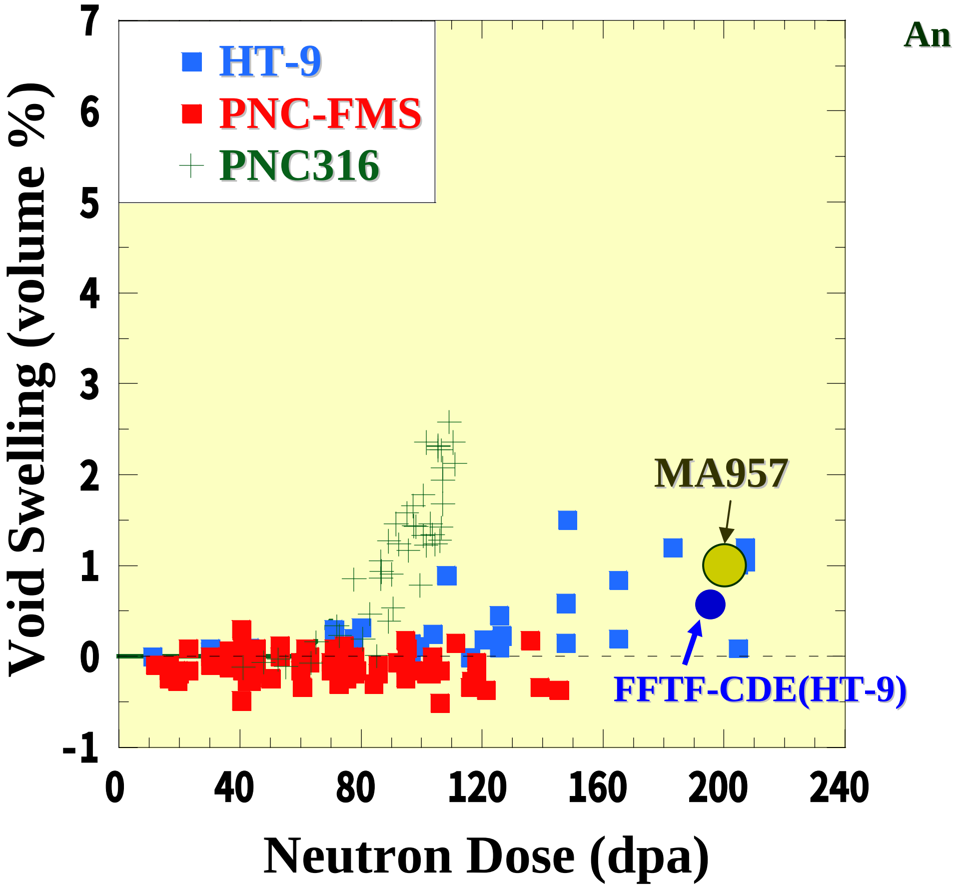
Precipitation **H**ardened **F**erritic **M**artensitic **S**teel

←Dissimilar Welded

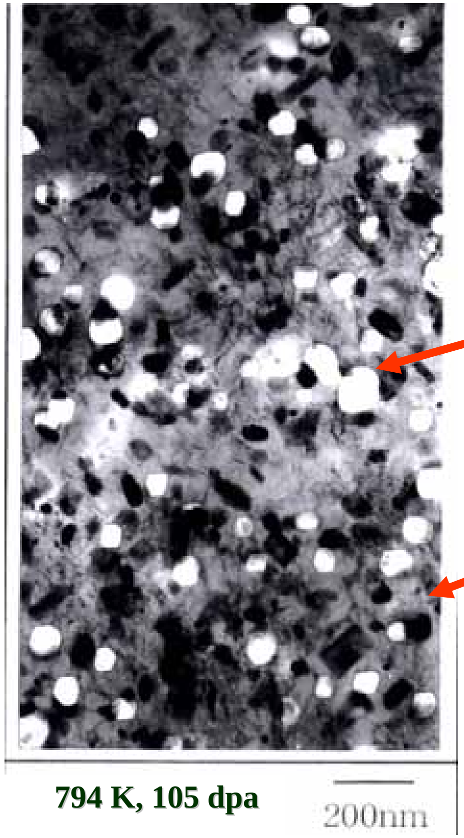
Core Support Structure
(SUS316)

Entrance Nozzle(SUS316)

Void Swelling in Various Steels



An observation by TEM for modified SUS316 irradiated in FFTF



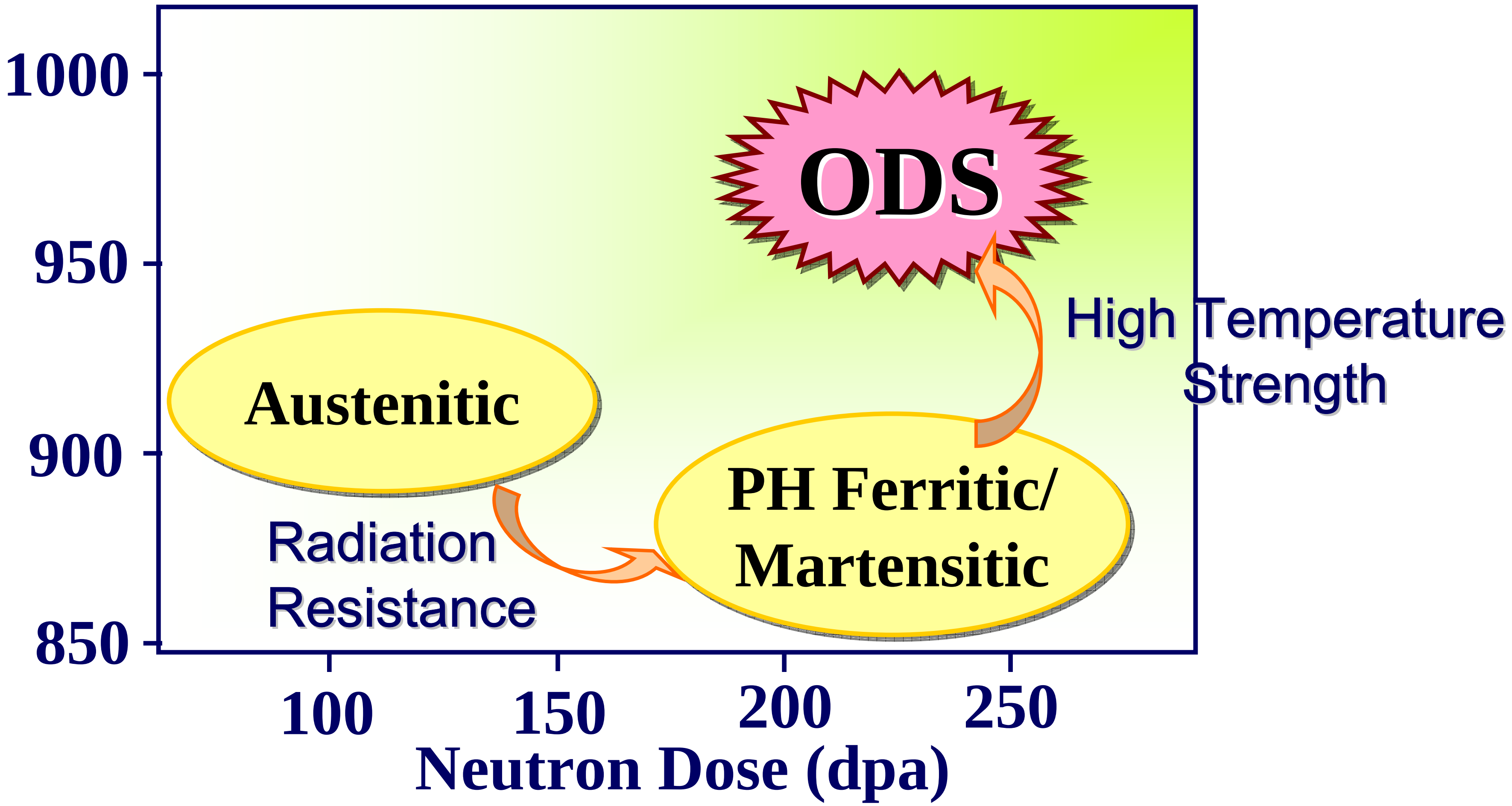
JNC TN9400 2000-075 (1999), P.171

Void swelling will be detrimental in austenitic stainless steels over 120 dpa.
BCC exhibits better swelling resistance than FCC.

→ Ferritic steels are promising for higher doses than 120 dpa.

HT-9: Fe-0.2C-12Cr-NiMnMoWVNb (Fully Martensitic: Precipitation Hardened), PNC-FMS: Fe-0.12C-11Cr-NiMnMoWVNb (Ferritic/Martensitic: Precipitation Hardened)
MA957: Fe-14Cr-0.3Mo-1Ti-0.25Y_2O_3 (Fully Ferritic: Oxide Dispersion Strengthened)
PNC316: Fe-16Cr-14Ni-1.7Mn-2.5Mo-0.25P-0.004B-0.1Ti-0.1Nb (Austenitic: Precipitation hardened and 20% Cold Work)

Why ODS?



Precipitation hardening will be lost in ferritic steels over 923 K.

↓

Oxide dispersion strengthening will be effective even over 973 K.

Requirements for Fuel Pin Cladding Tubes

1. Power Plant Performance: Higher Thermal Efficiency

○ Reactor Outlet Coolant Temperature: 823 K (550℃)

○ Reactor Inlet Coolant Temperature: 673 K (400℃)

↓

- Maximum (Hot Spot) Temperature: 973 K (700℃)

2. Fuel Cycle Performance: Higher Burnup

○ Discharge Average Burnup of Core Fuel: 150 GWd/t

○ Long Term Service: ~9 years (26 months × 4 Batches)

↓

- Peak Neutron Dose: ~250 dpa
- Peak Burnup: ~250 GWd/t
- Maximum Internal Pressure: ~12 MPa

Requirements for ODS Steels

High Temperature:~700℃
Long Term Service: ~9 y
→ **Creep & Tensile Properties**

Displacement Damage: ~250dpa
→ **Radiation Resistance (Dimensional Stability)**

Exposure to High Burnup Fuel & Flowing Na
→ **Chemical Compatibility**

Mechanical Properties are targeted to be

Ultimate Tensile Strength (UTS): >300 MPa @ 973 K
Internal Creep Rupture Strength: >120 MPa × 10^4@ 973 K hr
Uniform Elongation (UE): >1%

Manufacturing Process:1
Powder Metallurgy (PM) Process for Mother Tubes

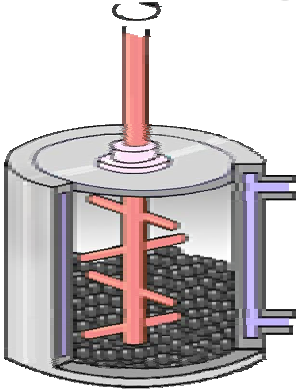
Dispersoid Size Control

Raw Material Powder

Pre-alloyed Powder
or
Elemental Powder

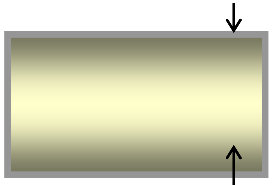
Y_2O_3 Powder

Mechanical Alloying (MA)
(Ball Milling)



Canning
Degassing@673K(400℃)

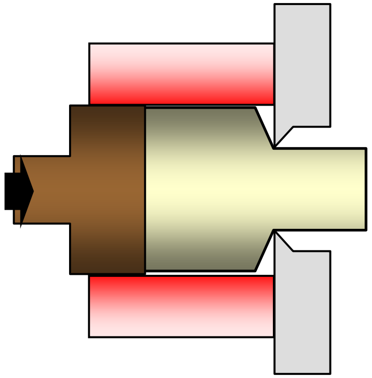
Mild steel capsule : Φ67mm



MA powder

Oxygen pick-up

Hot Extrusion@ 1,423 K(1,150℃)



Annealing
(Φ25mm)
Drilling

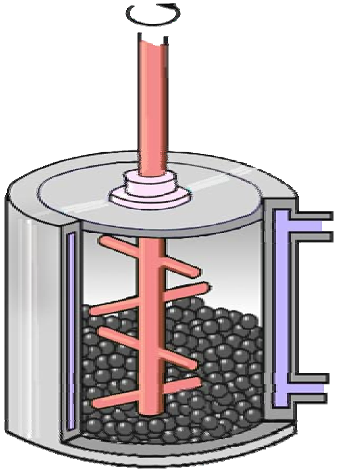
Mother Tube

OD18mm×ID12mm×L180mm:200g

High Energy Attrition Type Ball Mill for Mechanical Alloying

10D Attritor

Balls: 150 kg
Powders: 10kg/Batch
99.9999%Ar
220rpm×48hrs



Mechanically alloyed powders are exposed to air in recovery.

Photographs are supplied by Dr. Fujiwara of Kobelco Research Institute, Inc.

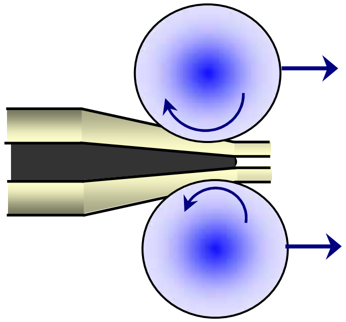
Manufacturing Process:2
Cold Rolling Process from Mother Tubes to Cladding Tubes

Grain Morphology Control

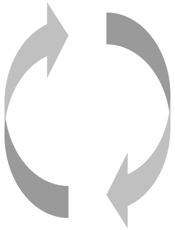
Mother Tube

OD18mm×ID12mm
×L180mm:200g

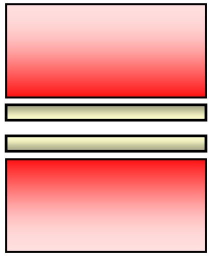
Cold-rolling: Pilger



Repeat

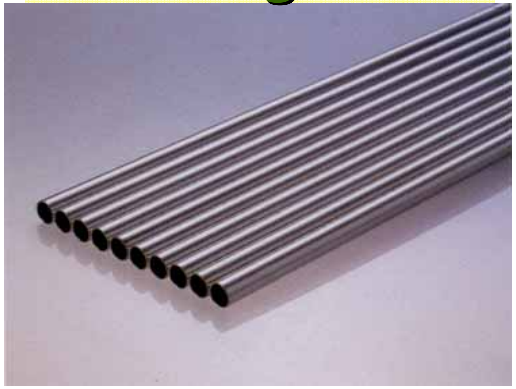


Intermediate Heat Treatment



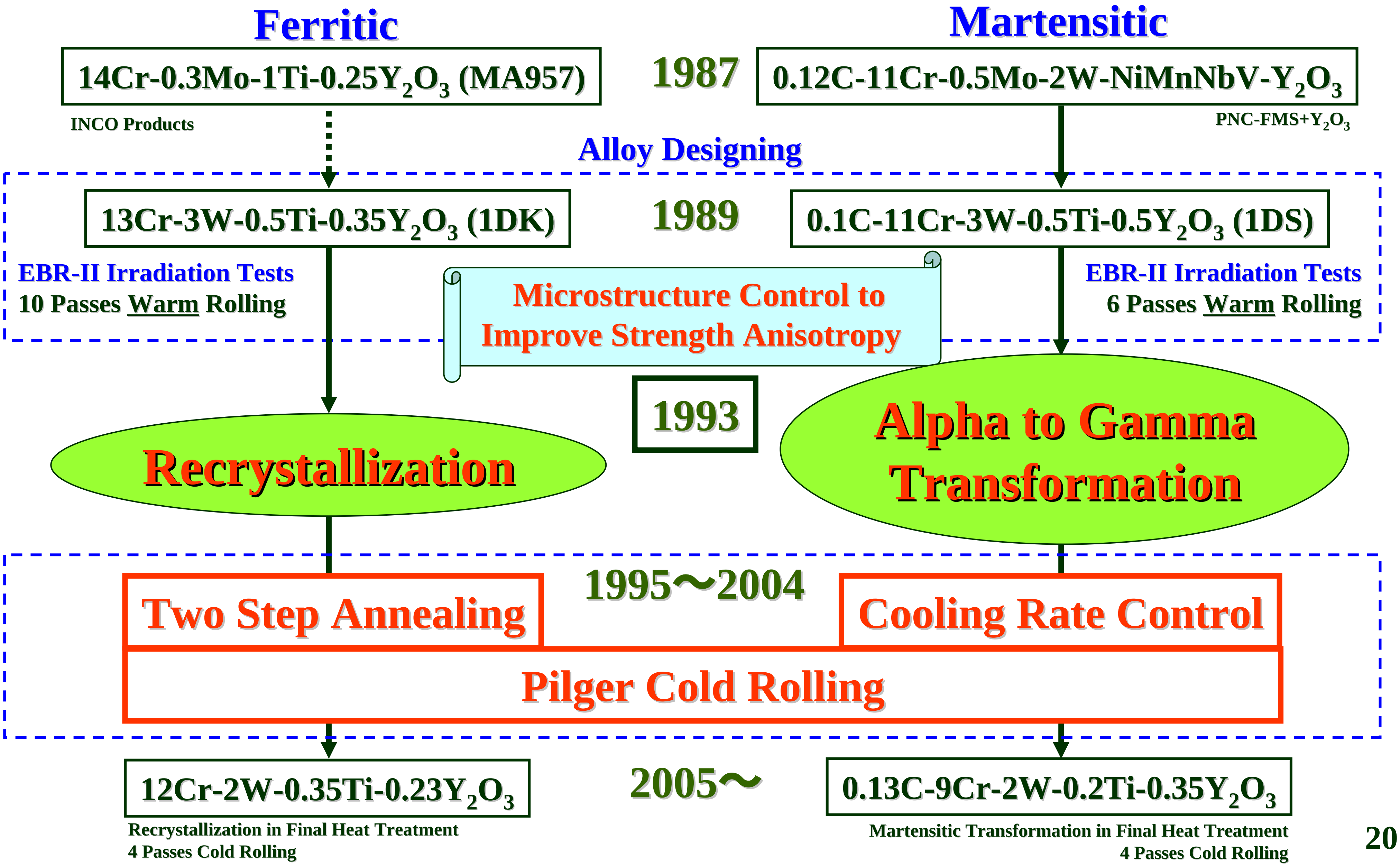
Final Heat Treatment

Cladding Tubes



OD8.5mm×ID7.5mm
×L1,850mm:180g

History and Major Break Through



Ferritic

Martensitic

1987

$14Cr-0.3Mo-1Ti-0.25Y_2O_3$ (MA957)

INCO Products

$0.12C-11Cr-0.5Mo-2W-NiMnNbV-Y_2O_3$

$PNC-FMS+Y_2O_3$

Alloy Designing

1989

$13Cr-3W-0.5Ti-0.35Y_2O_3$ (1DK)

EBR-II Irradiation Tests
10 Passes Warm Rolling

$0.1C-11Cr-3W-0.5Ti-0.5Y_2O_3$ (1DS)

EBR-II Irradiation Tests
6 Passes Warm Rolling

Microstructure Control to Improve Strength Anisotropy

1993

Recrystallization

Alpha to Gamma Transformation

1995～2004

Two Step Annealing

Cooling Rate Control

Pilger Cold Rolling

2005～

$12Cr-2W-0.35Ti-0.23Y_2O_3$

Recrystallization in Final Heat Treatment
4 Passes Cold Rolling

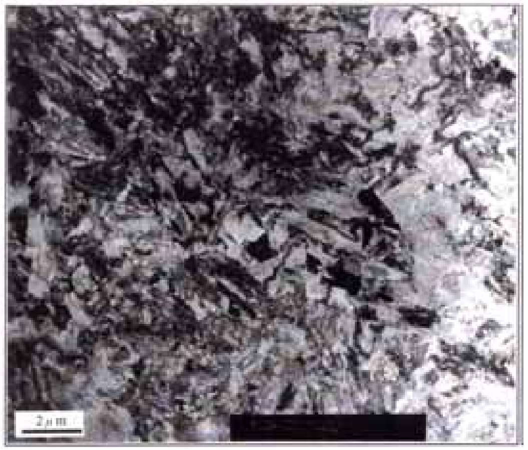
$0.13C-9Cr-2W-0.2Ti-0.35Y_2O_3$

Martensitic Transformation in Final Heat Treatment
4 Passes Cold Rolling

Two Candidates of ODS Steels for Further R&D in FaCT

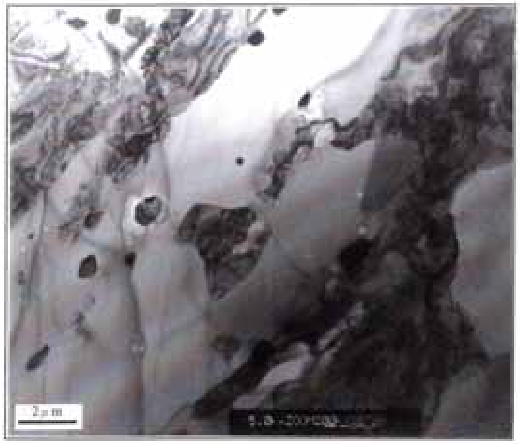
1. Primary Candidate: 9Cr-ODS

- $Fe-0.13C-9Cr-2W-0.2Ti-0.35Y_2O_3$
- Normalizing@1,323 K(1,050℃)×60min
→Tempering@1,053-1,073 K×60min
- Tempered Martensitic Matrix
- Alpha to Gamma Transformation
- Cooling Rate Control



2. Secondary Candidate: 12Cr-ODS

- $Fe-0.03C-12Cr-2W-0.26Ti-0.23Y_2O_3$
-Annealed@~1,423K(1,150℃)×~60min
- Recrystallization
- Two-step Annealing



Chapter 3. Alloy Design

Selecting alloying elements

- Understanding strengthening mechanisms
- Predicting phase diagrams

Optimizing chemical compositions

- Specimen preparations
- Tensile and creep tests
- Trade-off between strength, toughness and manufacturing

Unanticipated phenomena and empirical approach

- δ phase formation (not equilibrium phase) in 9Cr-ODS
- Recrystallization (highly empirical) in 12Cr-ODS

Alloying Concepts and Specifications

Element	C	Cr	W	Ti	Y_2O_3	Ex.O
Mechanism	M	M	S	D	D	D
9Cr-ODS	0.13	9.0	2.0	0.20	0.35	0.07
12Cr-ODS	0.03	12.0	2.0	0.26	0.23	0.07

in wt%

Impurity: Mn,Ni,N,Ar,Si,P,S

$$\text{Ex.O} = \text{TolalOxygen Content} - \frac{48}{178}\text{Yttrium Content}$$

Oxygen picked up during PM process is called as Excess Oxygen (Ex.O), but it is the most important alloying element !!

D: Dispersion Strengthening, S: Solution Hardening,
P: Precipitation Hardening, M: Phase Control

Martensitic/Ferritic & Precipitation Hardened: PNC-FMS

Element	C	Cr	Mn	Ni	Mo	W	V	Nb	N
Mechanism	M/P	M	M	M	S	S	P	P	P
PNC-FMS	0.12	11.0	0.60	0.40	0.50	2.0	0.20	0.05	0.05

Normalizing@1,373 K(1,100℃)×10min→Tempering@1,053 K(780℃)×60min

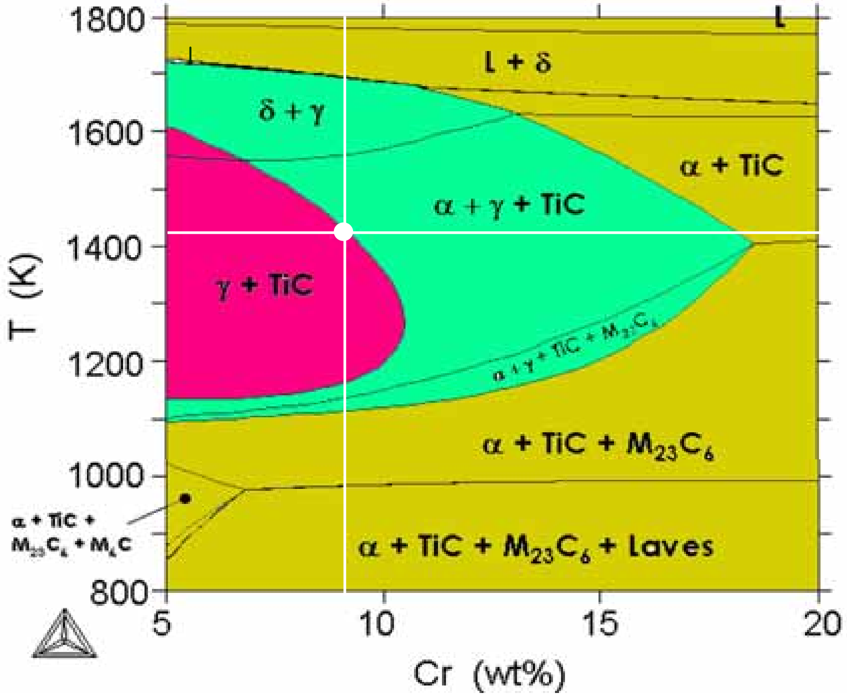
Cr Contents: Phase Control and Chemical Compatibility

●9Cr-ODS Steel: Martensitic

- Large capacity for trapping radiation-induced point defects.
→ Lower irradiation embrittlement

-Alpha to Gamma Transformation
→ Anisotropy-free mechanical properties

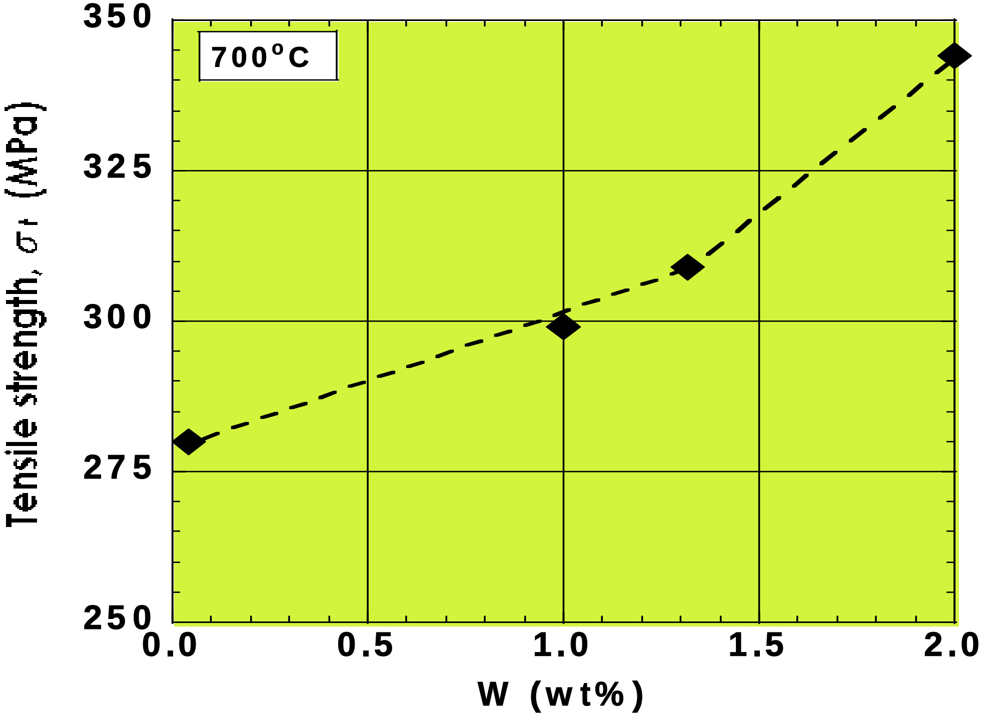
Fe-0.13C-2W-0.2Ti-xCr in mass%



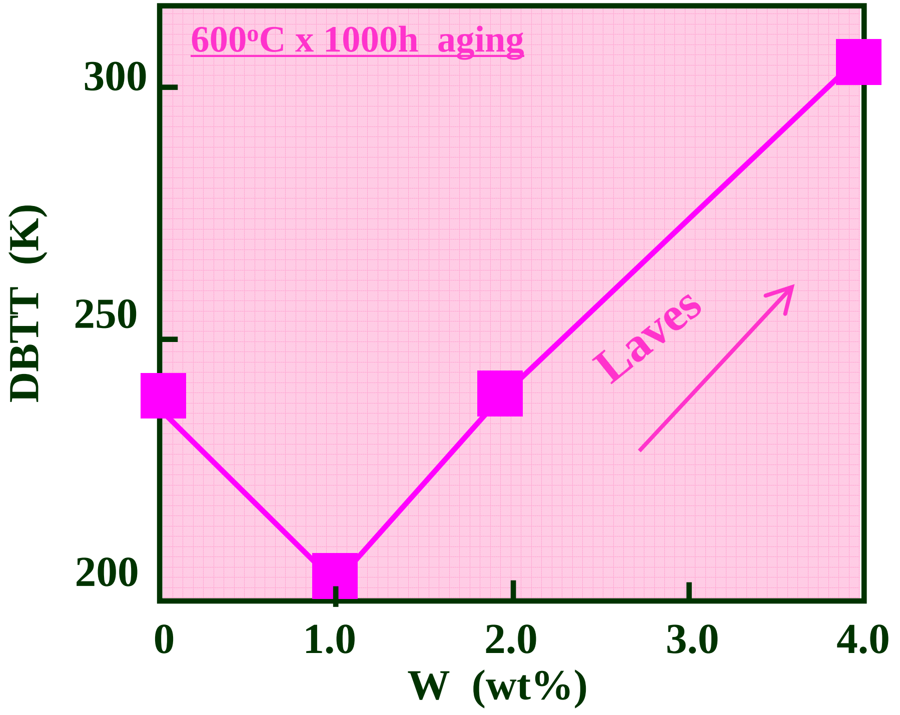
●12Cr-ODS: Fully Ferritic

- More stable oxide films on surface
Fuel-cladding chemical interaction (FCCI)
Nitric acid dissolution
→ Better corrosion resistance with minimal embrittlement

W Contents: Solution Hardening



9wt%Cr-0.13C-0.2Ti-0.35Y_2O_3-xW



9Cr-0.1C-xW

F.Abe et al, J. Nucl. Mater. 179-181 (1991) 663.

- Increasing to 2wt% improve considerablely high temperature strength.
- Excess addition over 2wt% will provide a concern with irradiation/thermal embrittlements due to laves precipitation.

⇨ **W 2wt%**

Oxide Dispersion Strengthening Theory

Smaller obstacles (dispersoids) interspacing provides
- higher threshold stress to dislocation motion and
- higher strength at elevated temperature.

↓

Finer dispersoids and higher number density is desirable.

Threshold stress to Dislocation Motion

$$\tau_{Or} = A_V \frac{Gb}{2\pi\bar{\lambda}}\left[\ln\left(\frac{\tilde{D}}{r_0}\right) + B_0\right]$$

- $\frac{1}{\tilde{D}} = \frac{1}{\bar{\lambda}} + \frac{1}{\bar{d}_s}$

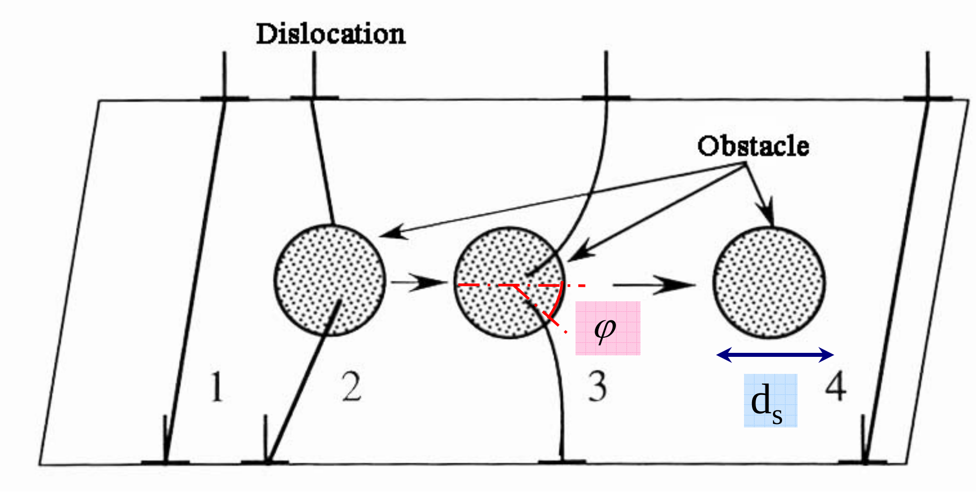
$\bar{\lambda}$ **: Average obstacle spacing**

$\bar{d}_s$ **: Average obstacle diameter**

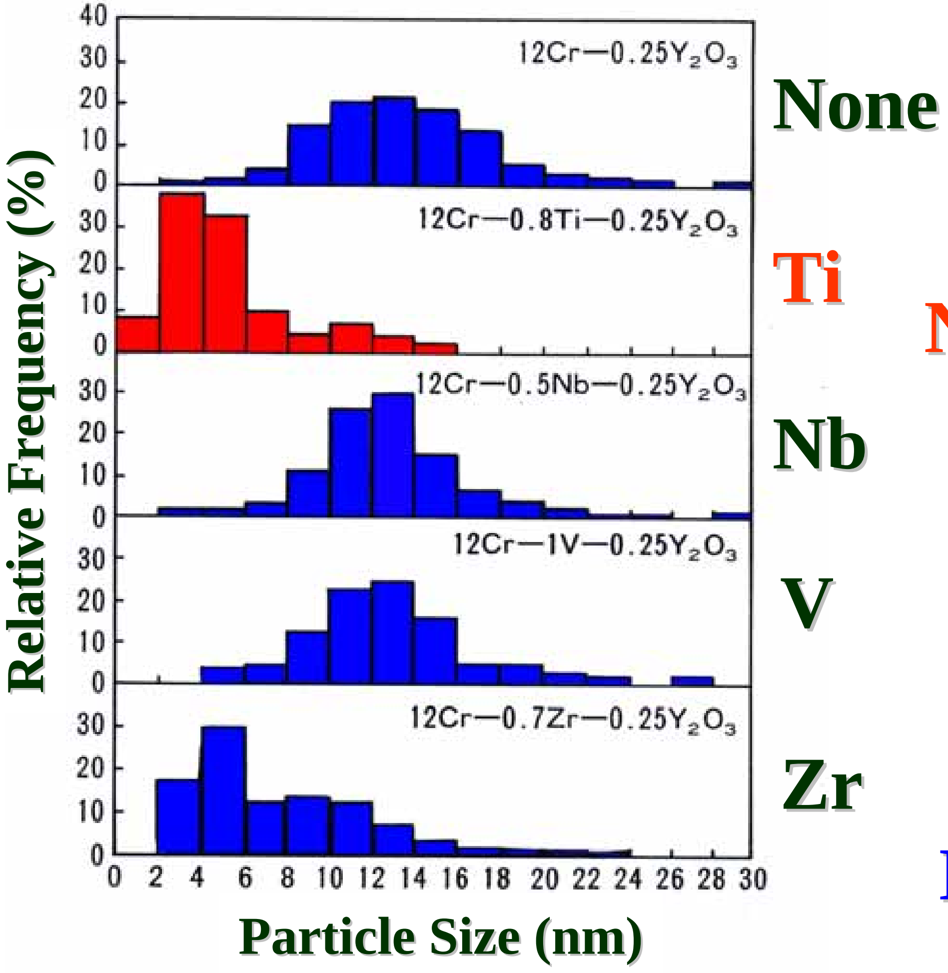
- **r_0 : dislocation core cut-off radius**

- $A_V = \begin{cases} \frac{1+\nu\sin^2\varphi}{1-\nu}\cos\varphi \quad (\varphi = 47°), B_0 = 0.6 & \textbf{: Screw dislocation} \\ \left(1-\frac{\nu}{1-\nu}\sin^2\varphi\right)\cos\varphi \quad (\varphi = 19°), B_0 = 0.7 & \textbf{: Edge dislocation} \end{cases}$

✓Srolovitz mechanism: Incoherent obstacle



Refinement of Dispersoid Size by Minor Alloying Elements



○Dispersoids will precipitate during hot-consolidation.

↓

Nucleation and Growth Kinetics in Co-Precipitation

○This refinement needs Ex.O.

↓

$Y_2O_3 + 2Ti + 4Ex.O = Y_2Ti_2O_7$

Detailed mechanism is not clear

Dispersoid sizes depend on minor alloying element(s) and amount of Ex.O.

↓

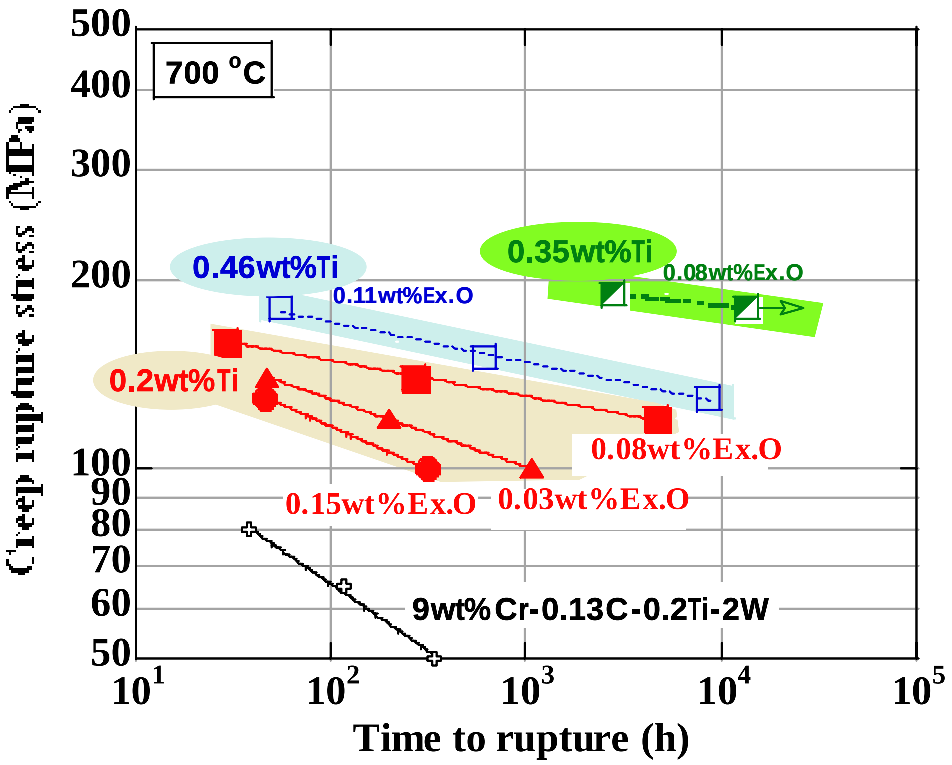
Titanium addition with Ex.O is very effective to enhance ODS effect.

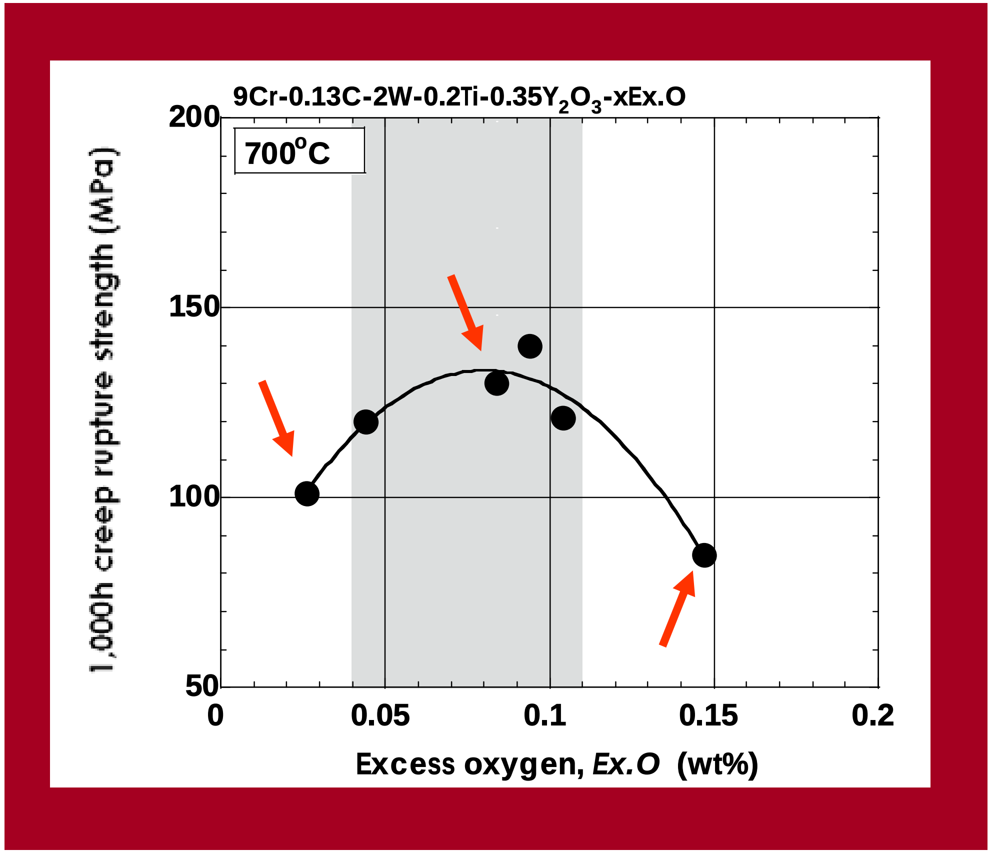
Y_2O_3, Ti & Excess Oxygen (Ex.O) Contents

○Increasing Y_2O_3 and Ti contents improve mechanical strength, while it leads to considerable degradation of tube manufacturability.

○Too much Ex.O degrades mechanical strength.

=> 0.2Ti-0.35Y_2O_3-0.08Ex.O

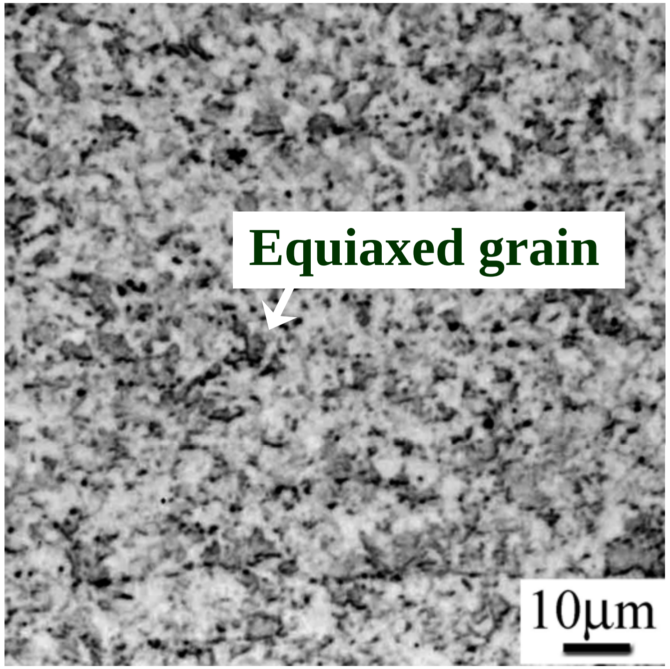




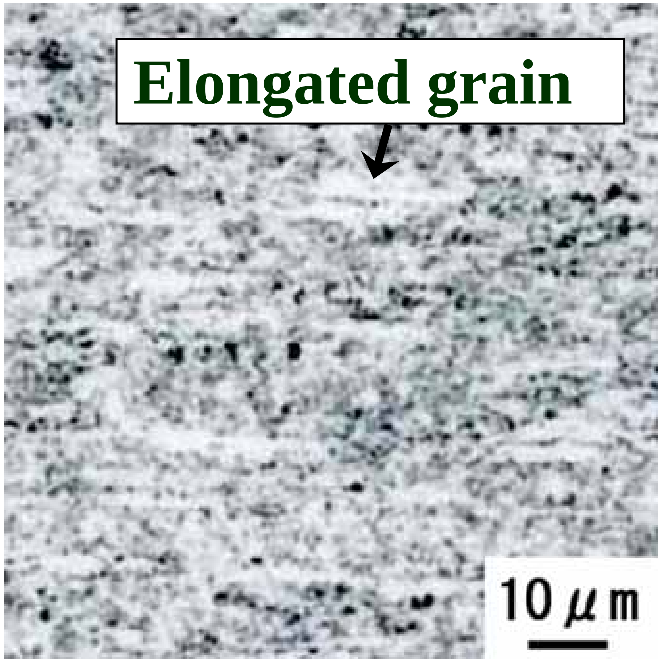
Phase Characterizations for the 9Cr-ODS Steel

δ phase disperses in martensitic with certain amount of Ex.O.

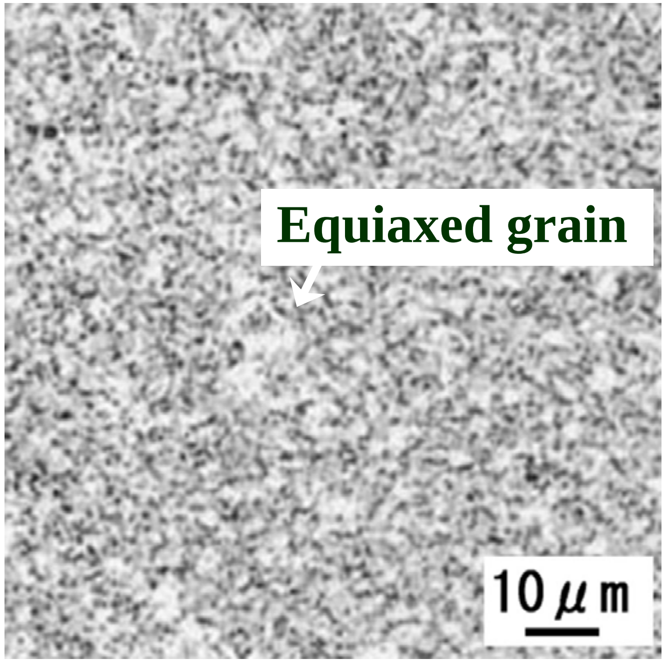
[Lo-O1] 0.2mass%Ti
0.03 mass%Ex.O



[ST-1] 0.2mass%Ti
0.08 mass%Ex.O

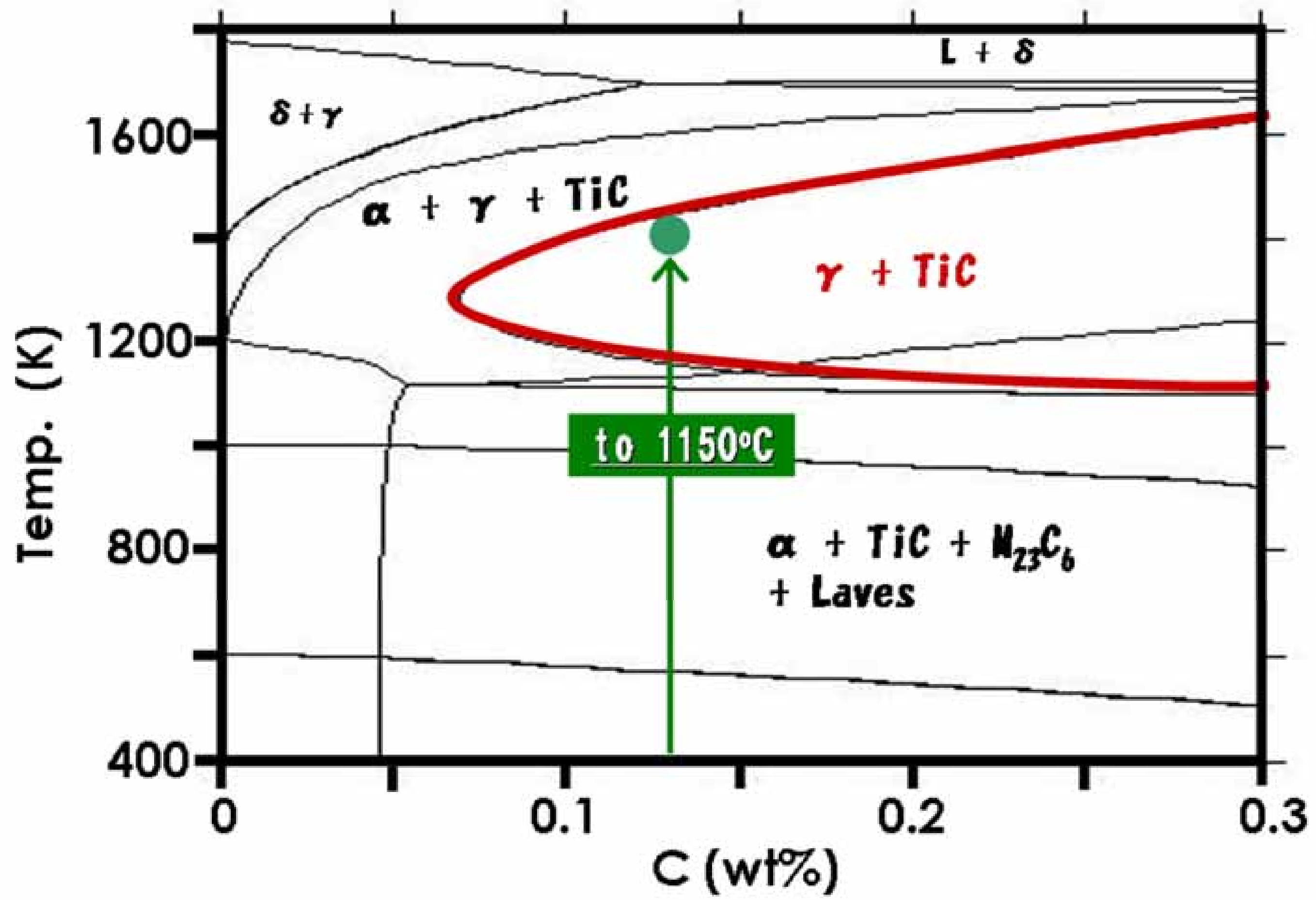


[Hi-O] 0.2mass%Ti
0.15 mass%Ex.O



Equiaxed Grain: Martensite
Elongated Grain: δ ferrite

Unanticipated Phenomena in the 9Cr-ODS Steel



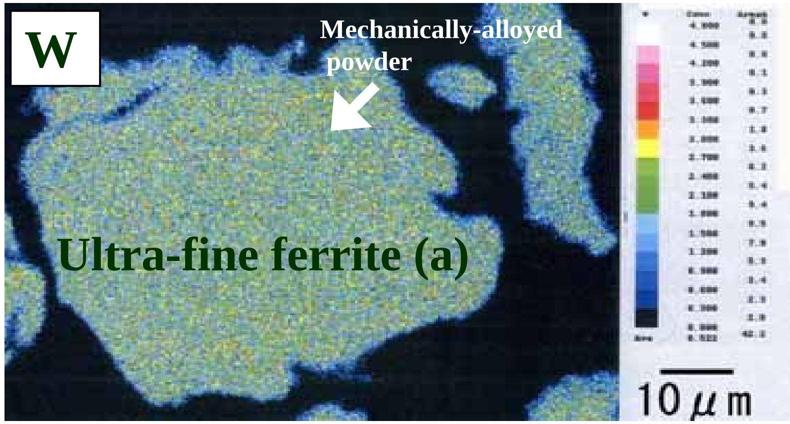
Phase diagram prediction does not indicate δ ferrite formation.

↓

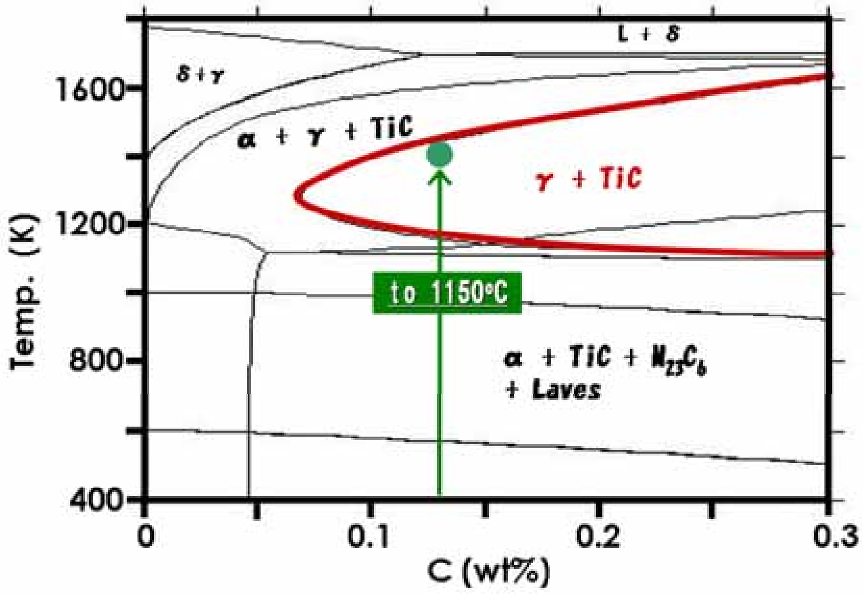
Is δ ferrite non-equibrium phase at 1150°C ?

δ Ferrite Formation during Hot Consolidation

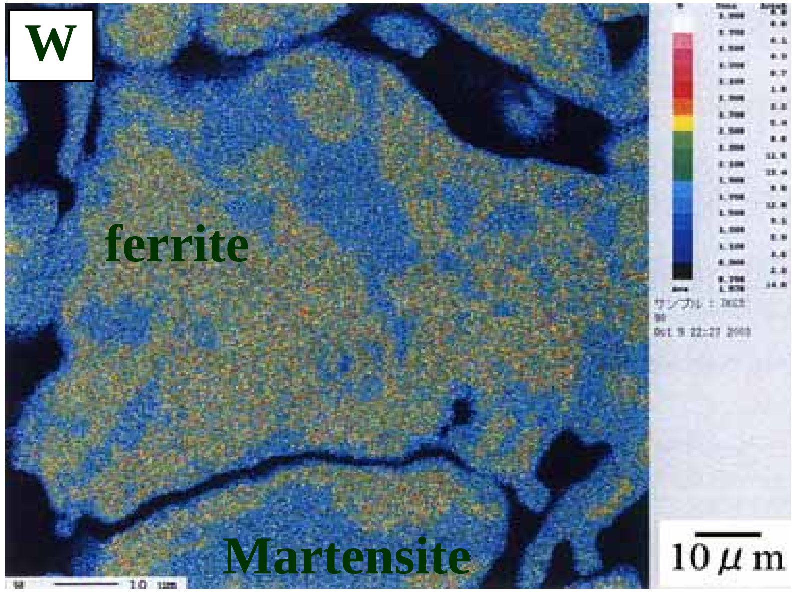
- **As mechanically alloyed**



Fine-grained structure produced by severe plastic deformation



- **1150°C annealed and quenched**

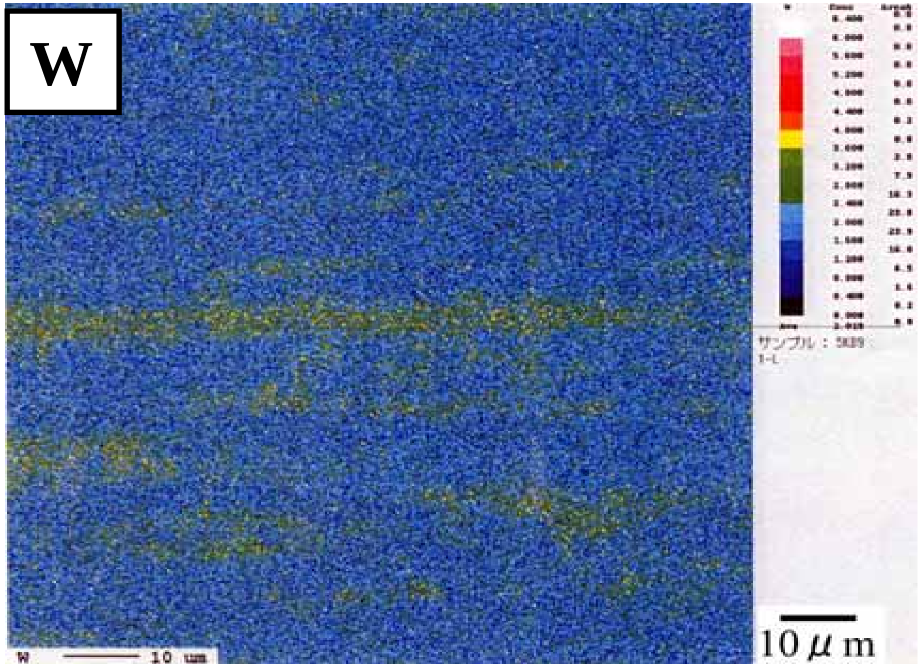


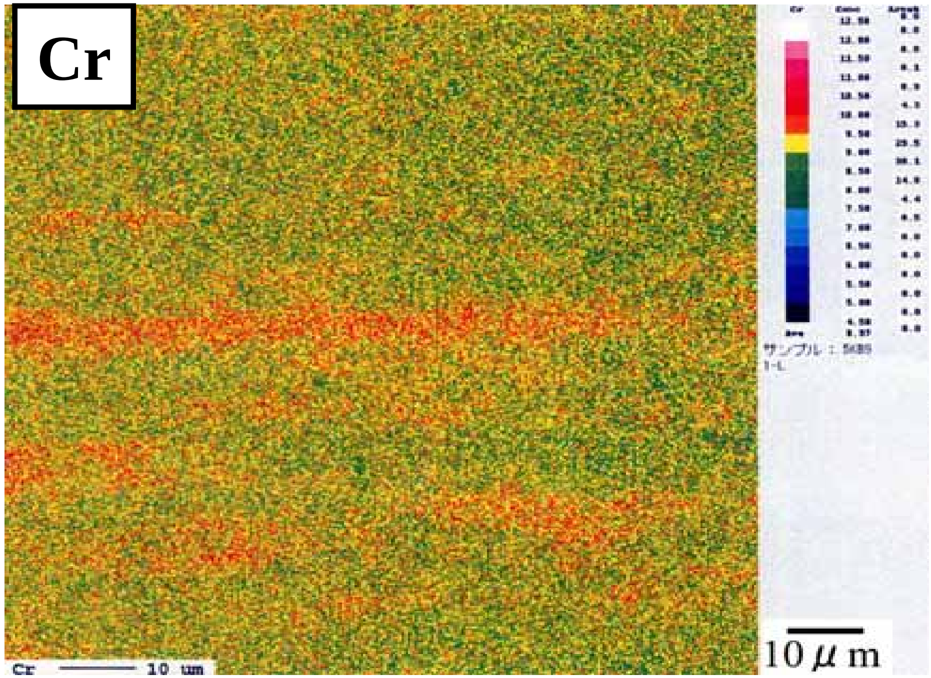
Ferrite(α)/austenite(γ) duplex

δ ferrite Formation
Non-equibrium phase at 1150°C.

EPMA Mapping of Longitudinal Section for the 9Cr-ODS Steel

- Ex.O=0.07wt%, Ti=0.2wt%



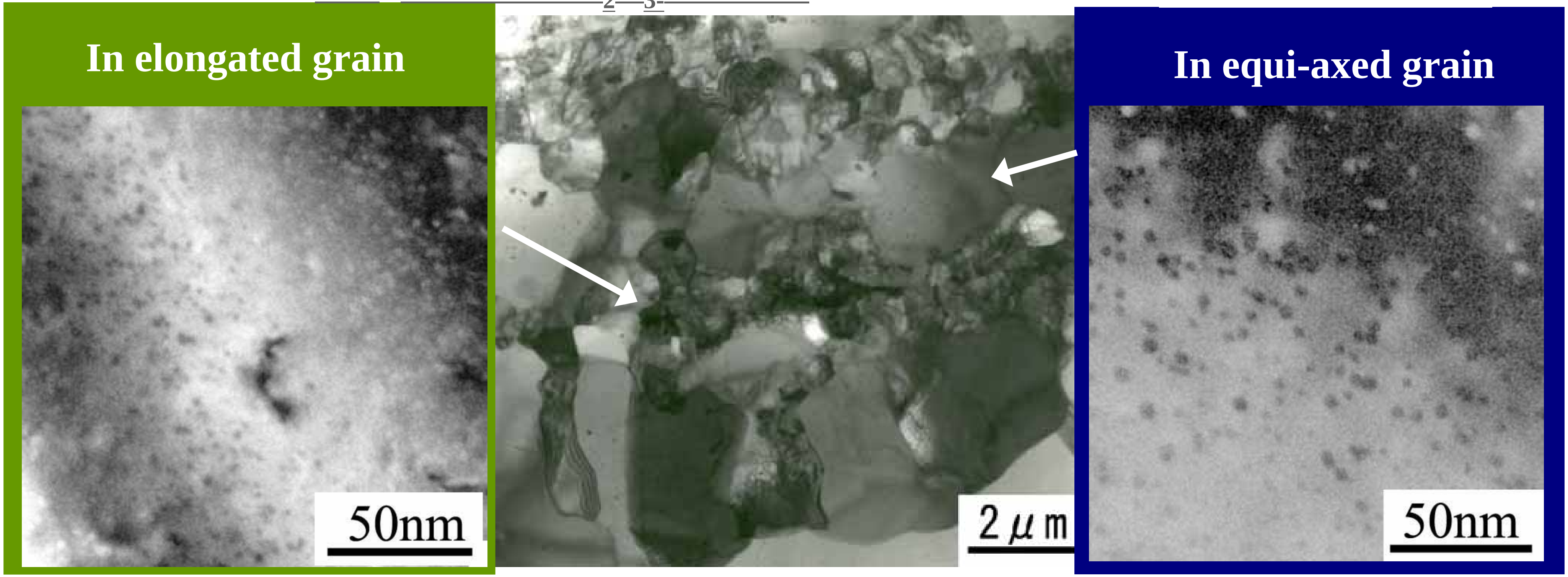


- Elongated grains (δ), where W and Cr are concentrated, exist in the matrix of high strength steel.
- Elongated grains can be also called as residual α-ferrite, which remained untransformed during 1150°C hot-consolidation.

TEM Observation for the 9Cr-ODS Steel

9wt%Cr - 0.13C - 2W - 0.2Ti - 0.35Y_2O_3 0.07Ex.O

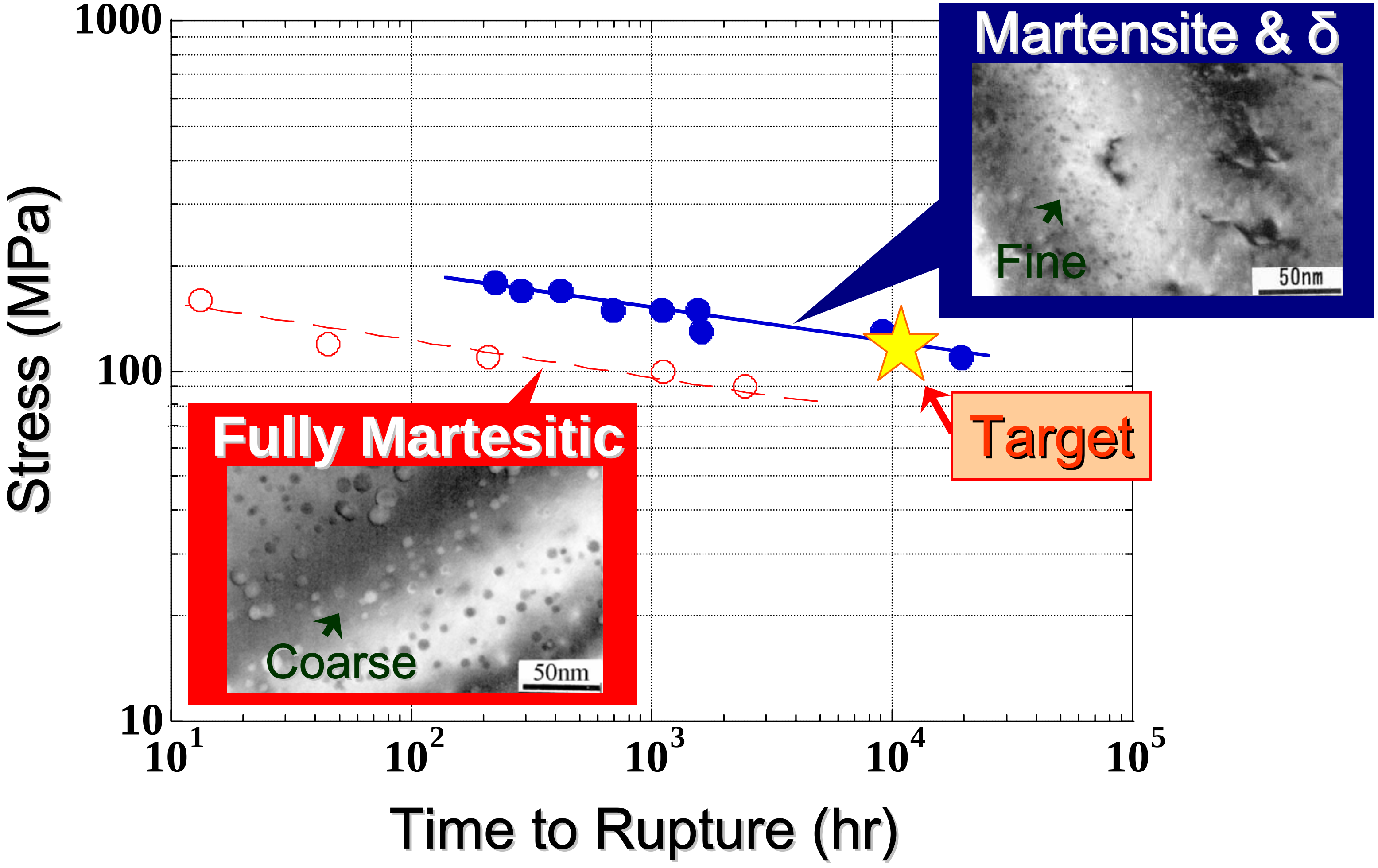
1050℃×1h, FC (30℃/h)



- Oxide particles is more finely dispersed in elongated grain (δ) than in equiaxed grain.
- The reason why the particle distribution between δ and martensite* are different is not clear.

*: Martensite should be gamma phase during hot-extrusion and heat treatments.

Composite Nature of the 9Cr-ODS Steel



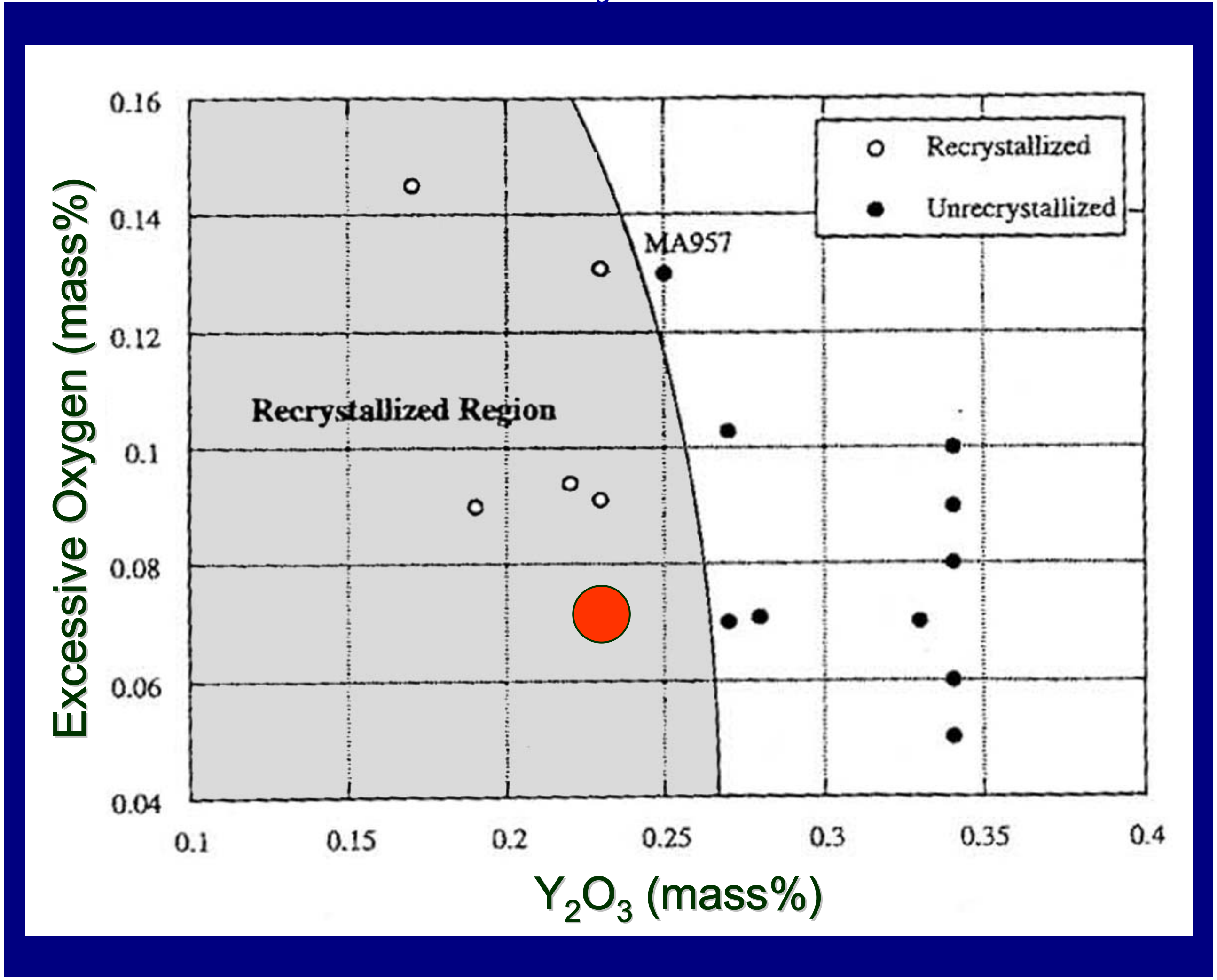
- Certain amount of δ ferrite is essential to attain the target.
- The 9Cr-ODS steel is re-enforced by δ ferrite.

Recrystallization Threshold in the 12Cr-ODS Steel

Too much Ex.O addition produces excessive, since Y-Ti-O particles are detrimental to recrystallization control.

=> 0.3Ti-0.23Y_2O_3

=> 0.07Ex.O



Summary of Alloy Effects

	Improvement	Degradation
Cr	✓Corrosion Resistance (≥ 12 mass%)	✓Irradiation/Thermal Embrittlement (Cr-rich phase: α’) ✓δ-ferrite in martensite
W	✓High-temperature Strength (Solution Hardening) (Finer Precipitation of $M_{23}C_6$)	✓Irradiation/Thermal Embrittlement (Laves, d-ferrite in Martensite)
Ti Y_2O_3 Ex.O	✓High-temperature Strength (ODS)(Dispersoid Morphology) ✓Non-equilibrium Phase Generation	✓Tube Manufacturability (Microstructure Controllability)

Excess Oxygen (Ex.O) is the most important alloying element, but its effect is not well clarified.

Concepts of Alloy Design

- 9Cr-ODS steel: 9Cr-0.13C-2W-0.2Ti-0.35Y_2O_3-0.07Ex.O

- C,Cr: Radiation-resistant martensitic matrix (microstructure control)
- W: Solution hardening, little laves precipitation
- Ti, Y_2O_3, Ex.O: Oxide dispersion strengthening
  δ ferrite formation

- 12Cr-ODS steel: 12Cr-0.13C-2W-0.26Ti-0.23Y_2O_3-0.07Ex.O

- Cr: Corrosion resistant fully ferritic
- W: Solution hardening, little laves precipitation
- Ti, Y_2O_3, Ex.O: Oxide dispersion strengthening
  Recrystallization

Chapter 4. Microstructure Control

Powder Metallurgy Process

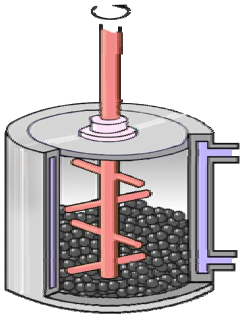
- Dispersoid Size/Distribution Control
- Uniform Solid Solution by Mechanical Alloying
- Precipitation of Nano Particles during Hot Consolidation

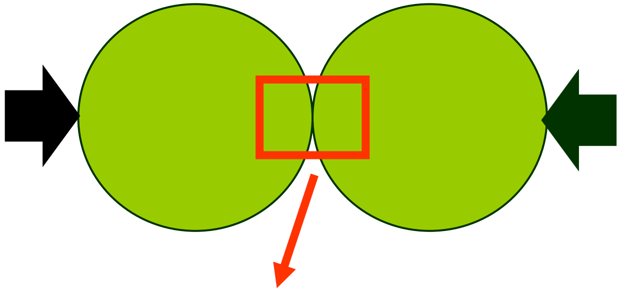
Precision Seamless Tube Production Process

- Grain Morphology Control
- Pilger Cold Rolling (Zircalloy Tubing)
- Intermediate and Final Heat Treatments
- Temperature History

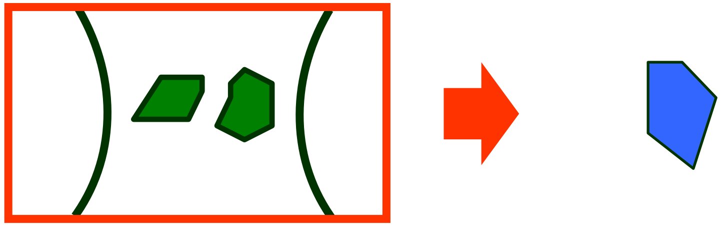
High Energy Attrition Type Ball Mill for Mechanical Alloying

Ball-to-Ball Collision → Uniform Solid Solution

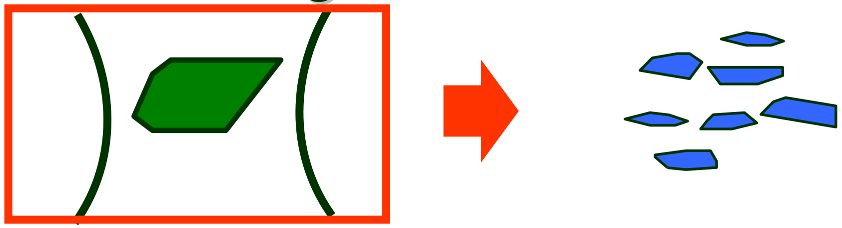




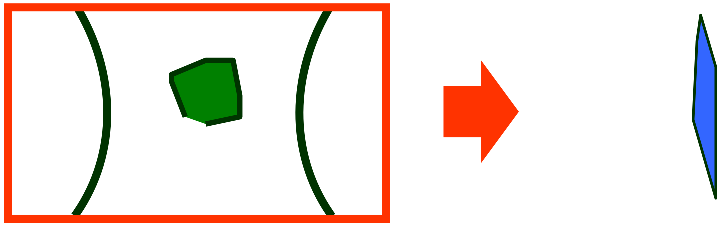
1.Consolidation



2.Fragmentation



3. Severe Plastic Deformation

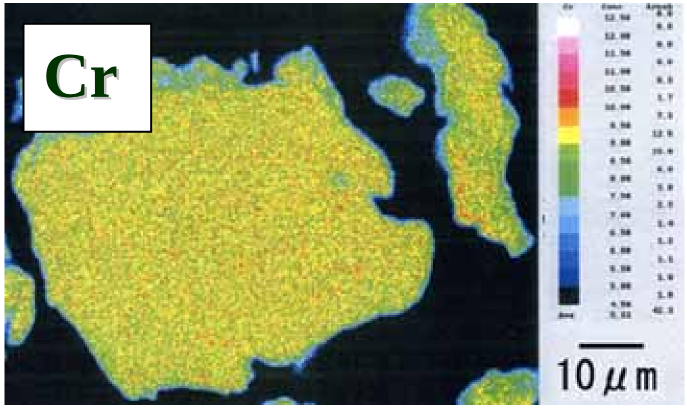


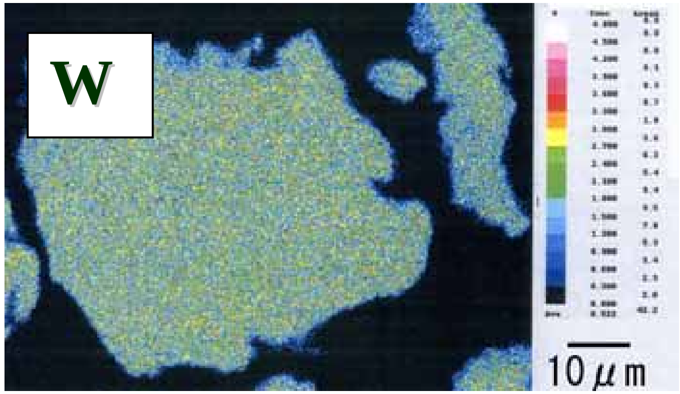
Instantaneous Deformation

+

Localized Heating!!

EPMA Mapping for As-mechanically Alloyed Powder





9Cr-0.2Ti-0.08Ex.O

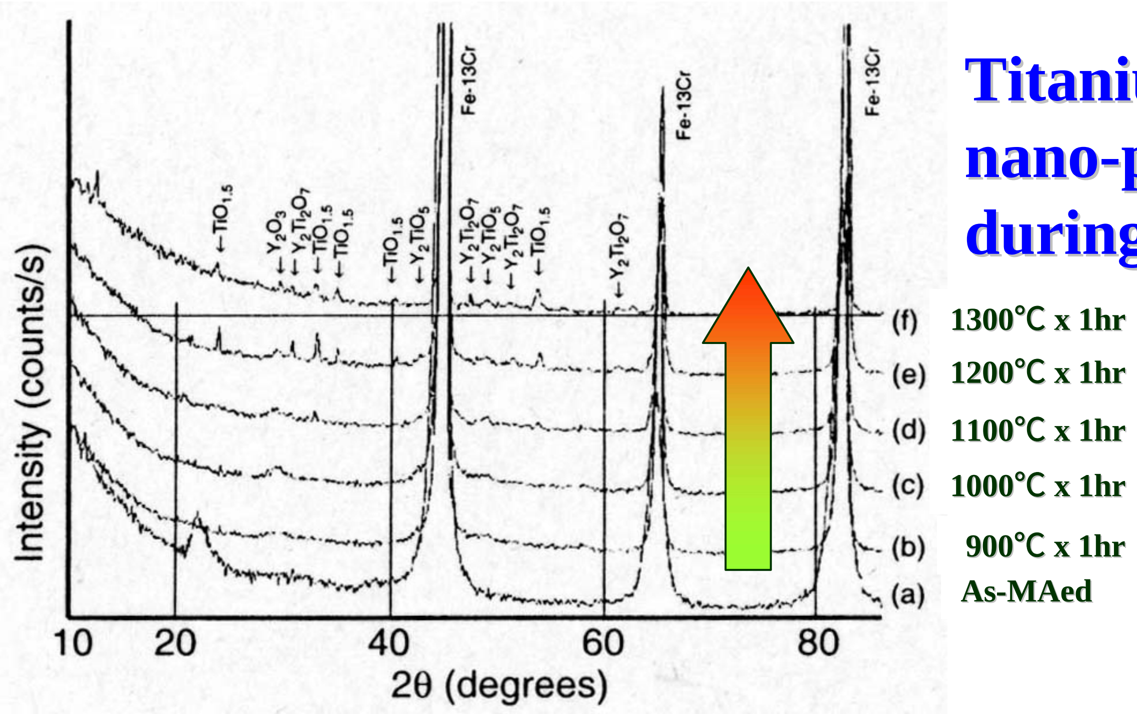
Precipitation of Nanometer Size Oxide Particles

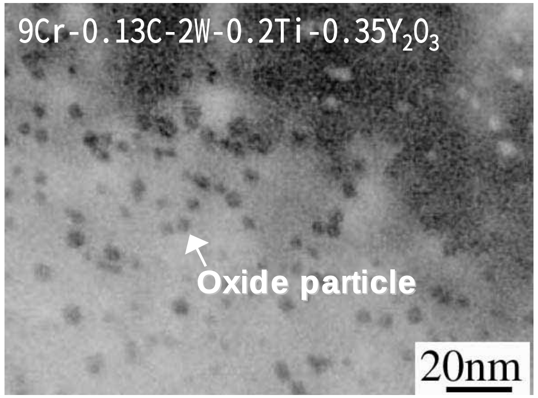
$$Y + Ti + O \rightarrow Y_2O_3 \cdot yTiO_x,$$

$$Ex.O = x \cdot y$$

Yttrium oxide dissociates and dissolves into matrix during mechanical alloying due to high energy ball milling.

Titanium and yttrium complex oxide nano-particles precipitate during hot consolidation process.





The original figure can be found in "T.Okuda and M. Fujiwara, J. Materials Science Letters, 14(1995)1600".

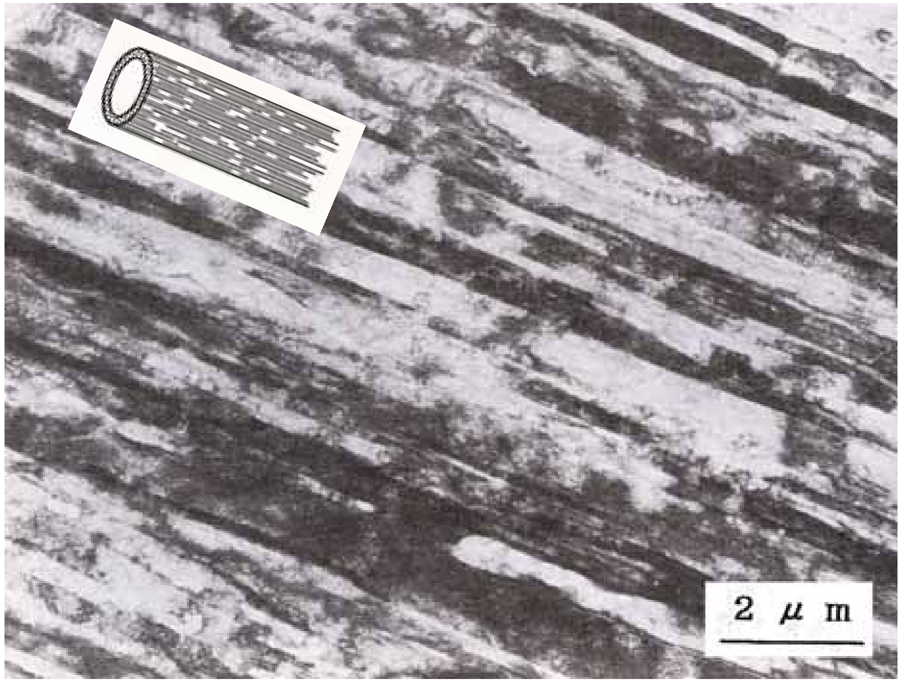
Mechanical Alloying and Hot Consolidation
→ Dispersoid Size Control

Grain Morphology Anisotropy Results in Poor Creep Rupture Strength under Internal Pressure

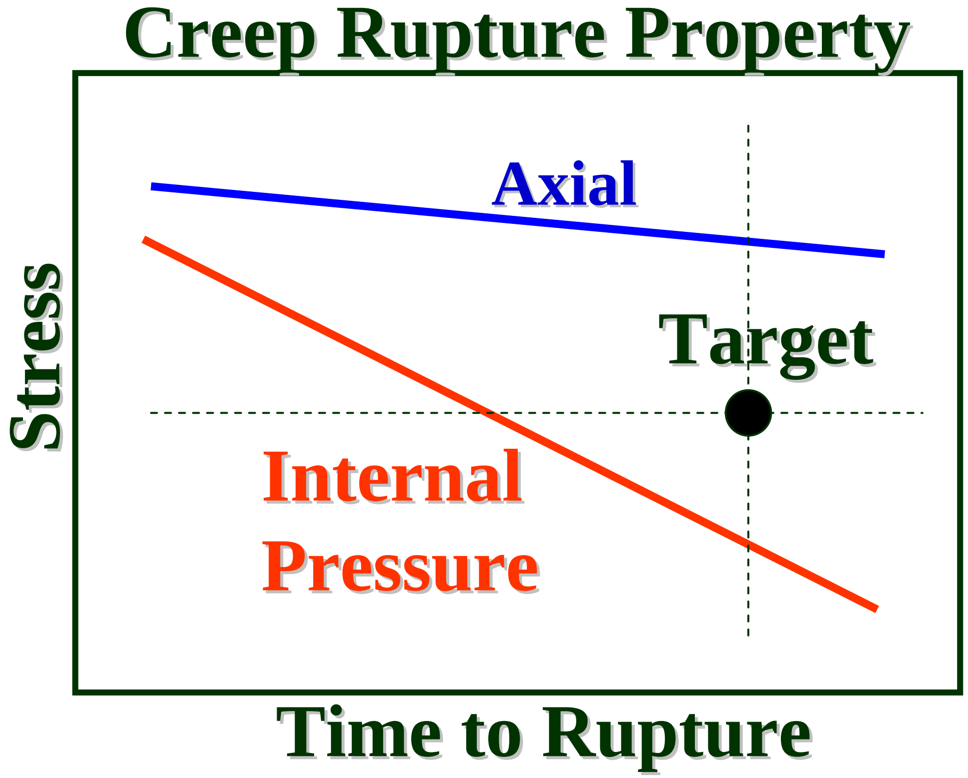
Finely elongated grain structure along rolling direction results in

- degradation of internal creep rupture strength and

- significant loss of ductility around 673 K

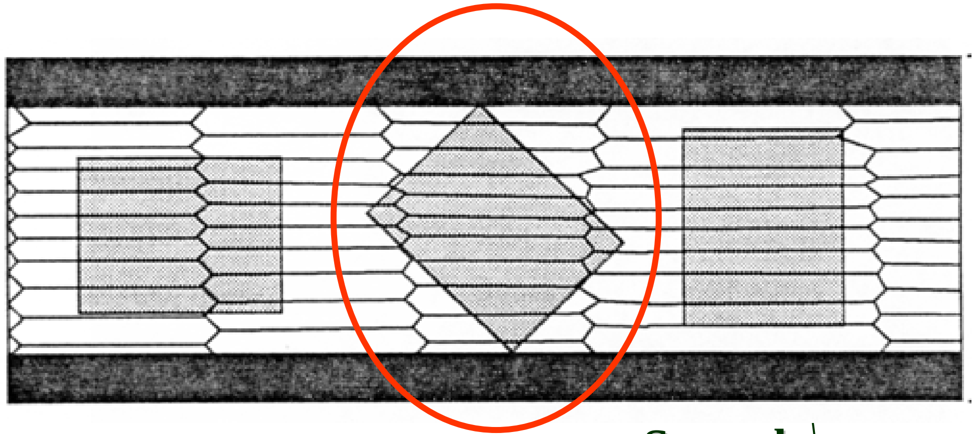


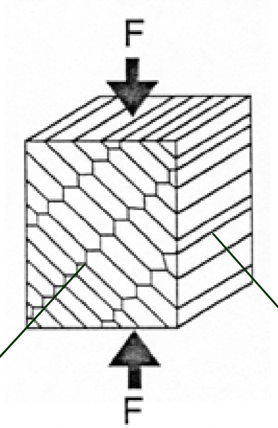
It looks like bamboo !!


Creep Rupture Property

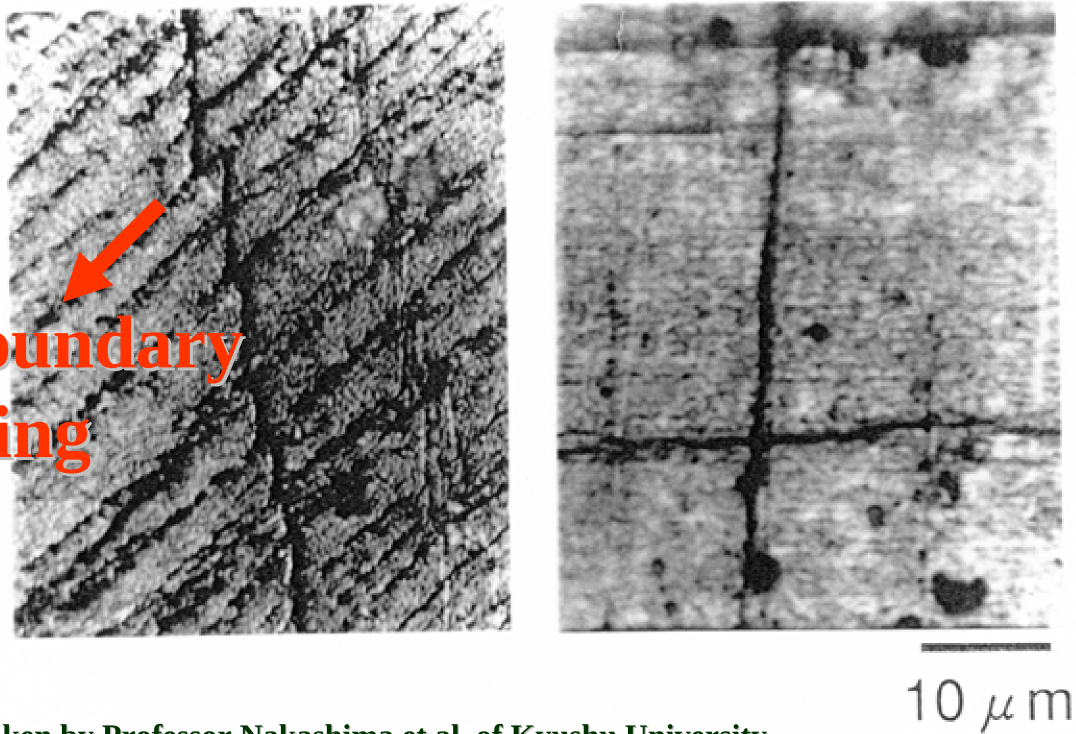
Grain Boundary Easily Slides at Elevated Temperature

Rolling Direction





Scratch



Grain Boundary Sliding

1. Specimen
- Cold-rolled plate
- Surface scratching

2. Mechanical test
- Compressive test
- 923 K

The original photographs were taken by Professor Nakashima et.al, of Kyushu University

What happens in cold rolling?

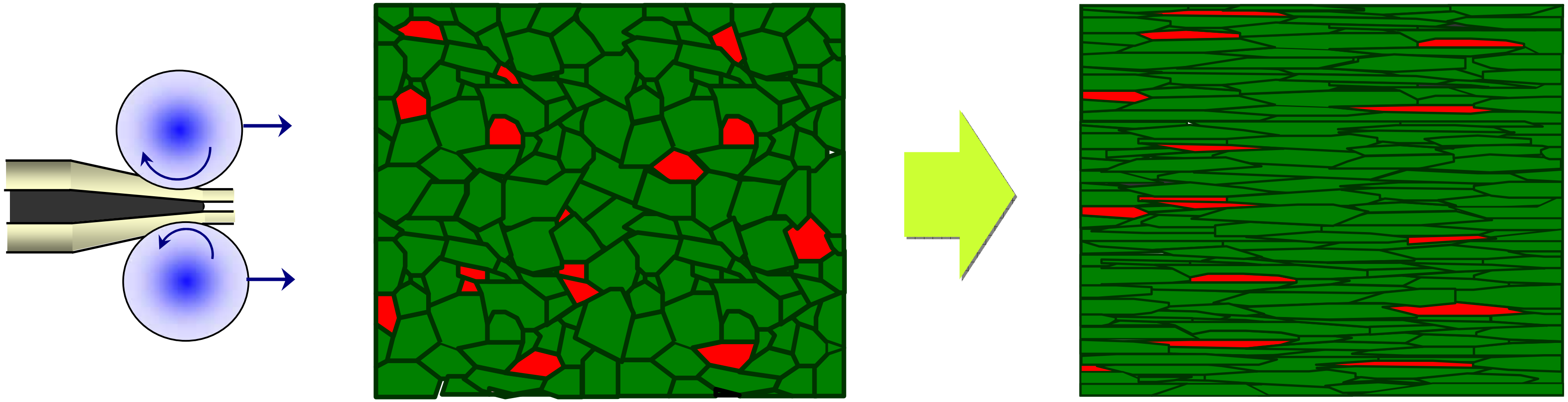
●Grains are elongated by uni-axial deformation

↓

Strength anisotropy

↓

Needs: Grain morphology modification !!



●Strain hardening due to cold work (area reduction: ~50%)

↓

Hardness increase (unacceptable further cold-rolling>Hv400)

↓

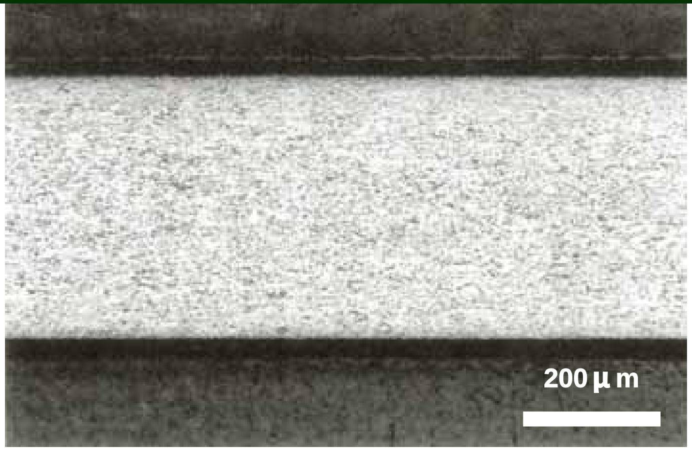
Needs: Intermediate heat treatment for softening !!

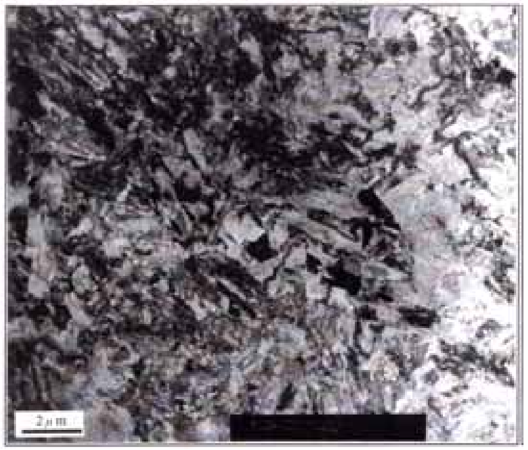
Grain Morphology in Final Products

9Cr-ODS Steel

$Fe-0.13C-9Cr-2W-0.20Ti-0.35Y_2O_3$

α/γ phase transformation

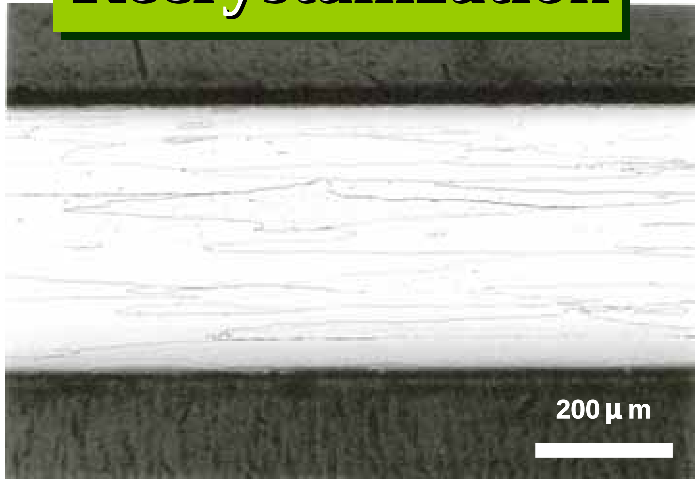


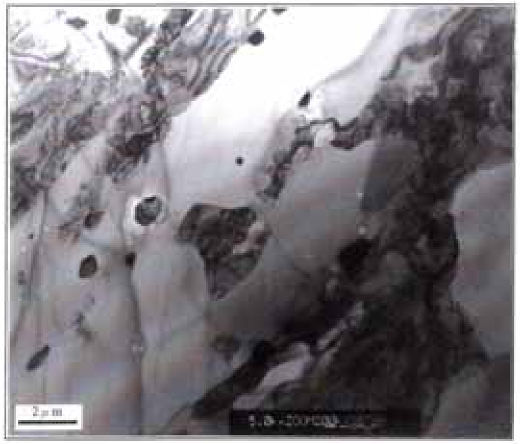


12Cr-ODS Steel

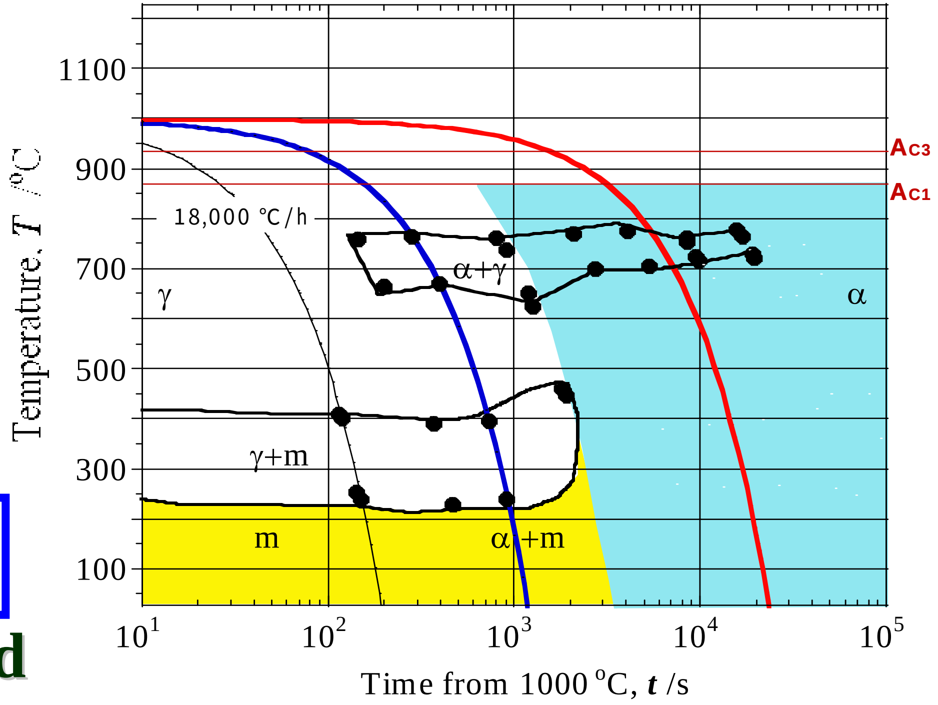
$Fe-0.03C-12Cr-2W-0.26Ti-0.23Y_2O_3$

Recrystallization





Cooling Rate and Subsequent Phases in the 9Cr-ODS Steel



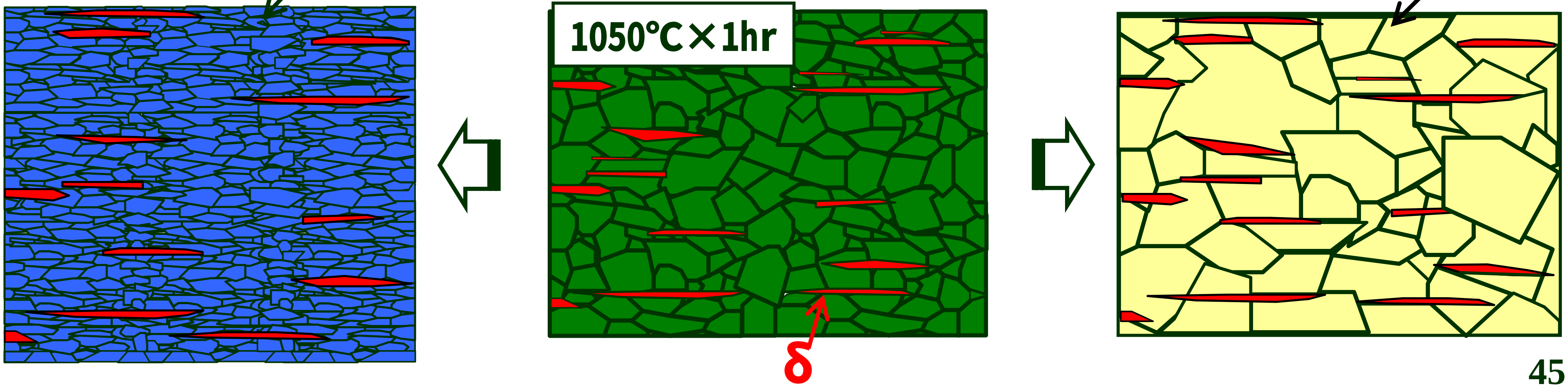
Continuous-Cooling-Transformation Diagram

Air Cooling

Martensite: Hard

Furnace Cooling

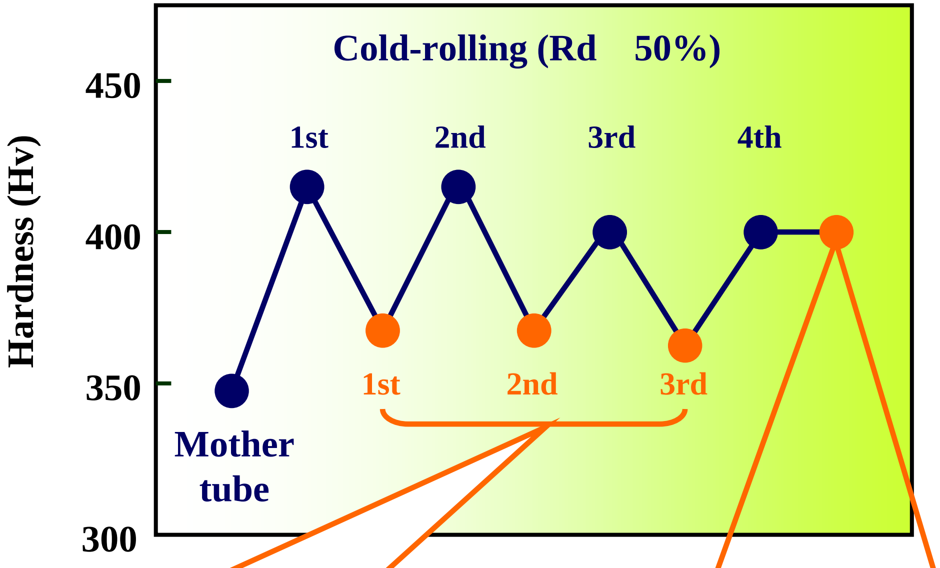
α : Soft



1050°C×1hr

δ

Simultaneous Control of Softening/Hardening Intention and Grain Morphology for the 9Cr-ODS Steel

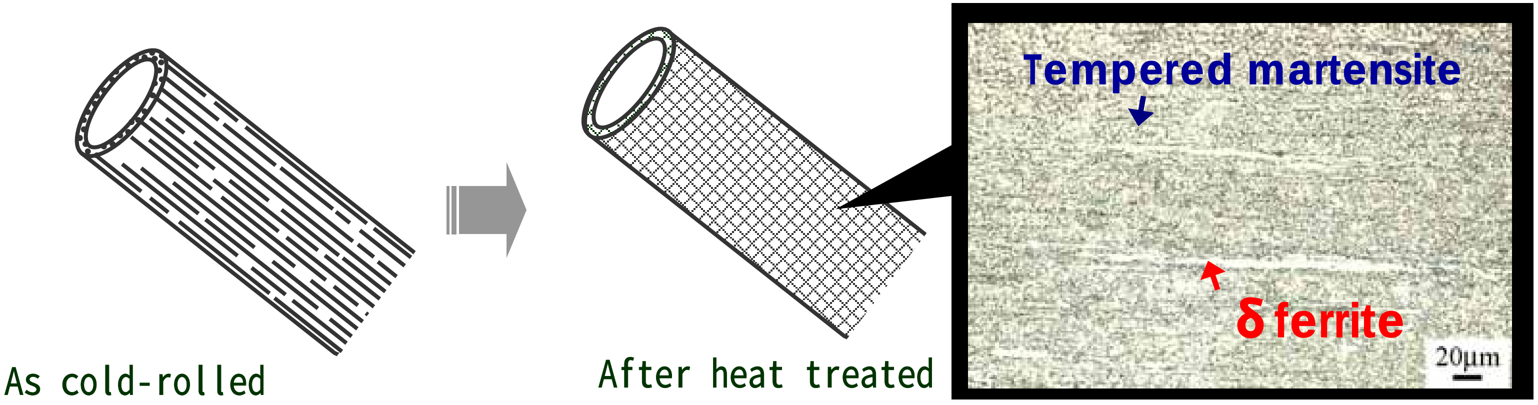


Intermediate Heat-treatment

1050°C×30 min×FC (150 °C/h)

Final Heat-treatment

1050°C×60 min×AC + 800°C×60 min×AC

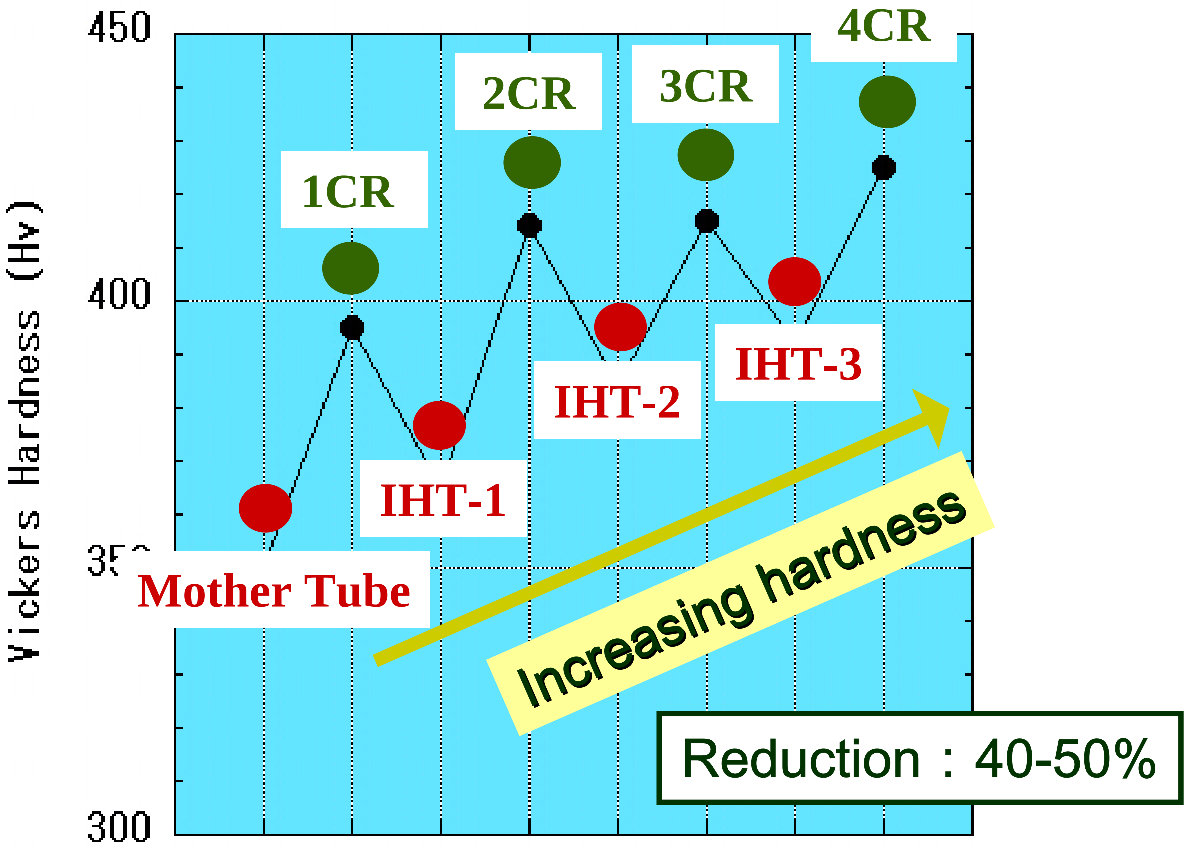


Technical Problems of the 12Cr-ODS Steel

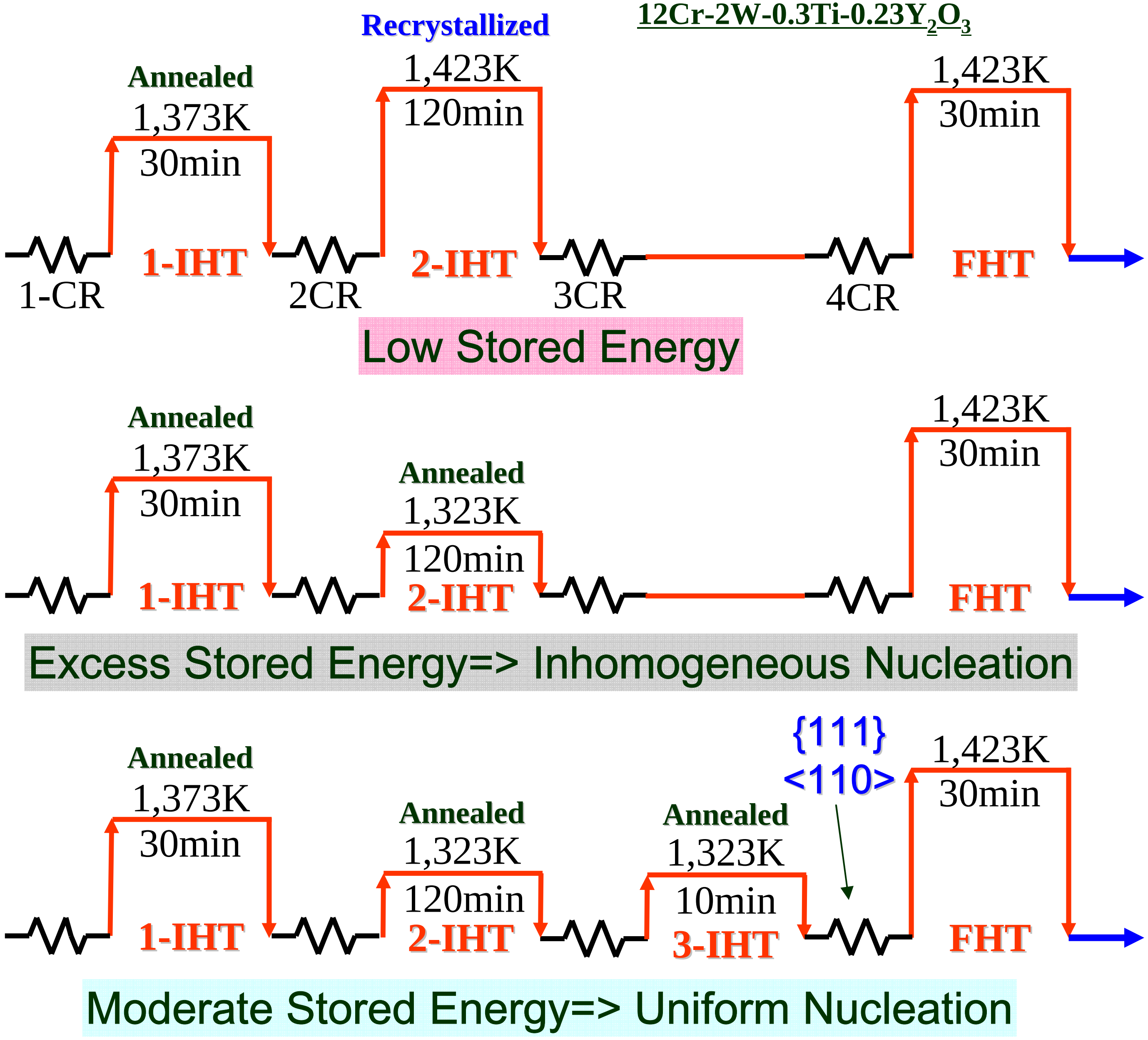
●Control of recrystalization is more difficult than that of alpha to gamma transformation.

●Recrystallized grains should be produced at the final heat-treatment to improve strength and ductility against internal pressure.

●Recrystallization phenomena depends empirically on texture evolution {111}<110>.



Effect of Stored Energy for Nucleation in the12Cr-ODS Steel

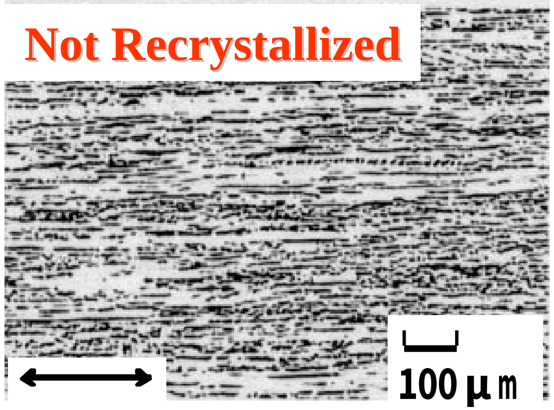


12Cr-2W-0.3Ti-0.23Y_2O_3

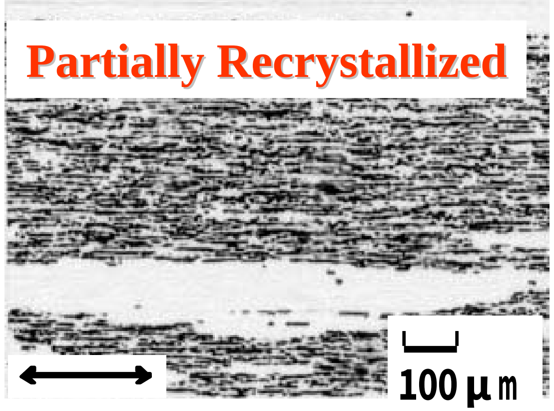
Low Stored Energy

Excess Stored Energy=> Inhomogeneous Nucleation

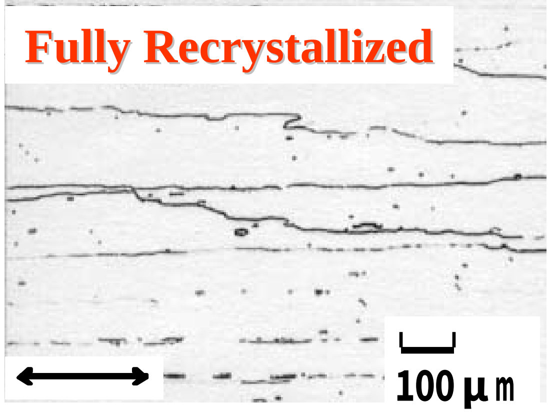
Moderate Stored Energy=> Uniform Nucleation



Not Recrystallized



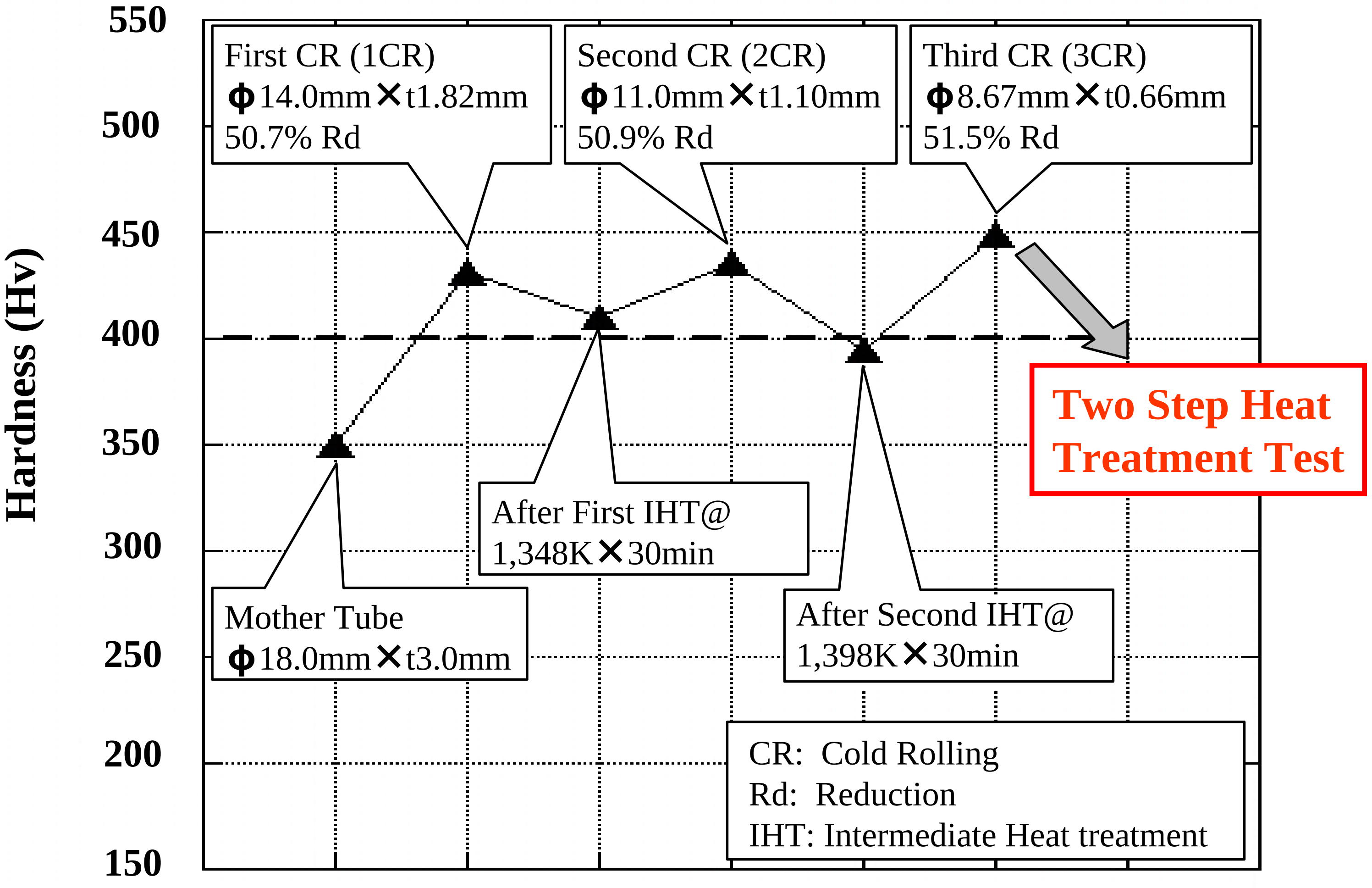
Partially Recrystallized



Fully Recrystallized

Specimen for Two-Step Heat Treatment Test

Preparation for 3rd Cold-Rolled Specimens without Recrystallizaation



First CR (1CR)
ϕ14.0mm × t1.82mm
50.7% Rd

Second CR (2CR)
ϕ11.0mm × t1.10mm
50.9% Rd

Third CR (3CR)
ϕ8.67mm × t0.66mm
51.5% Rd

Two Step Heat Treatment Test

After First IHT@
1,348K × 30min

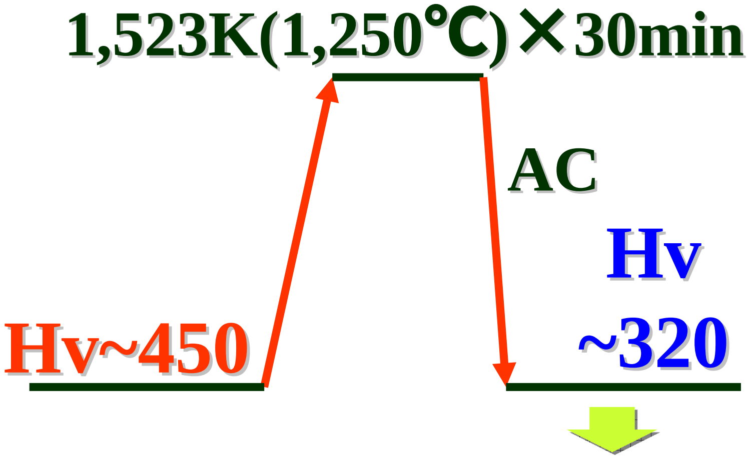
Mother Tube
ϕ18.0mm × t3.0mm

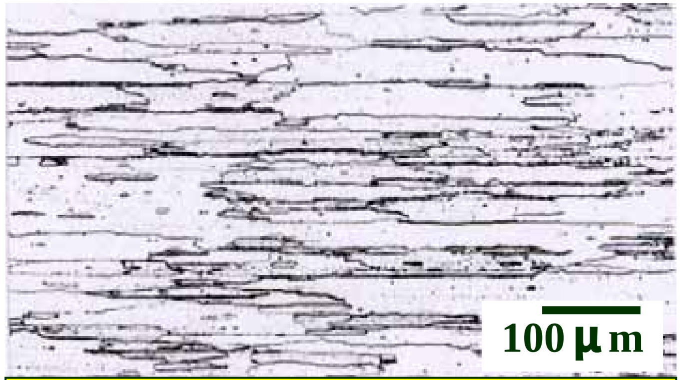
After Second IHT@
1,398K × 30min

CR: Cold Rolling
Rd: Reduction
IHT: Intermediate Heat treatment

One- and Two-Step Heat Treatment Tests after 3rd Cold Rolling

One Step Heat Treatment



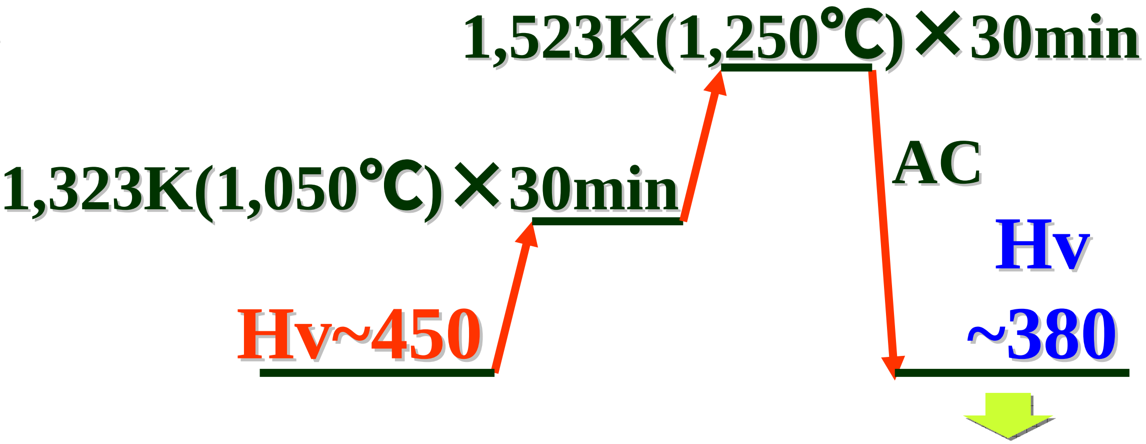


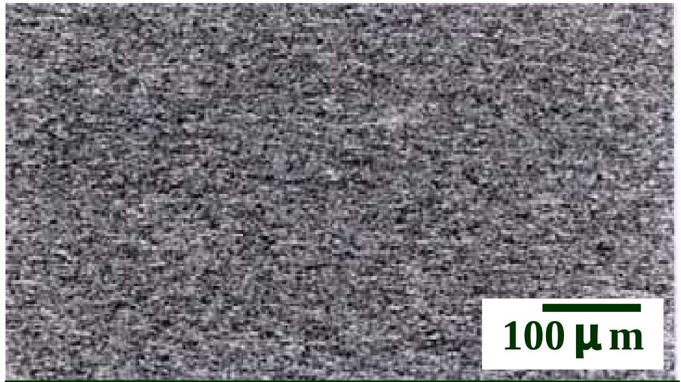
Recrystallized

Rolling Direction

↔

Two Step Heat Treatment

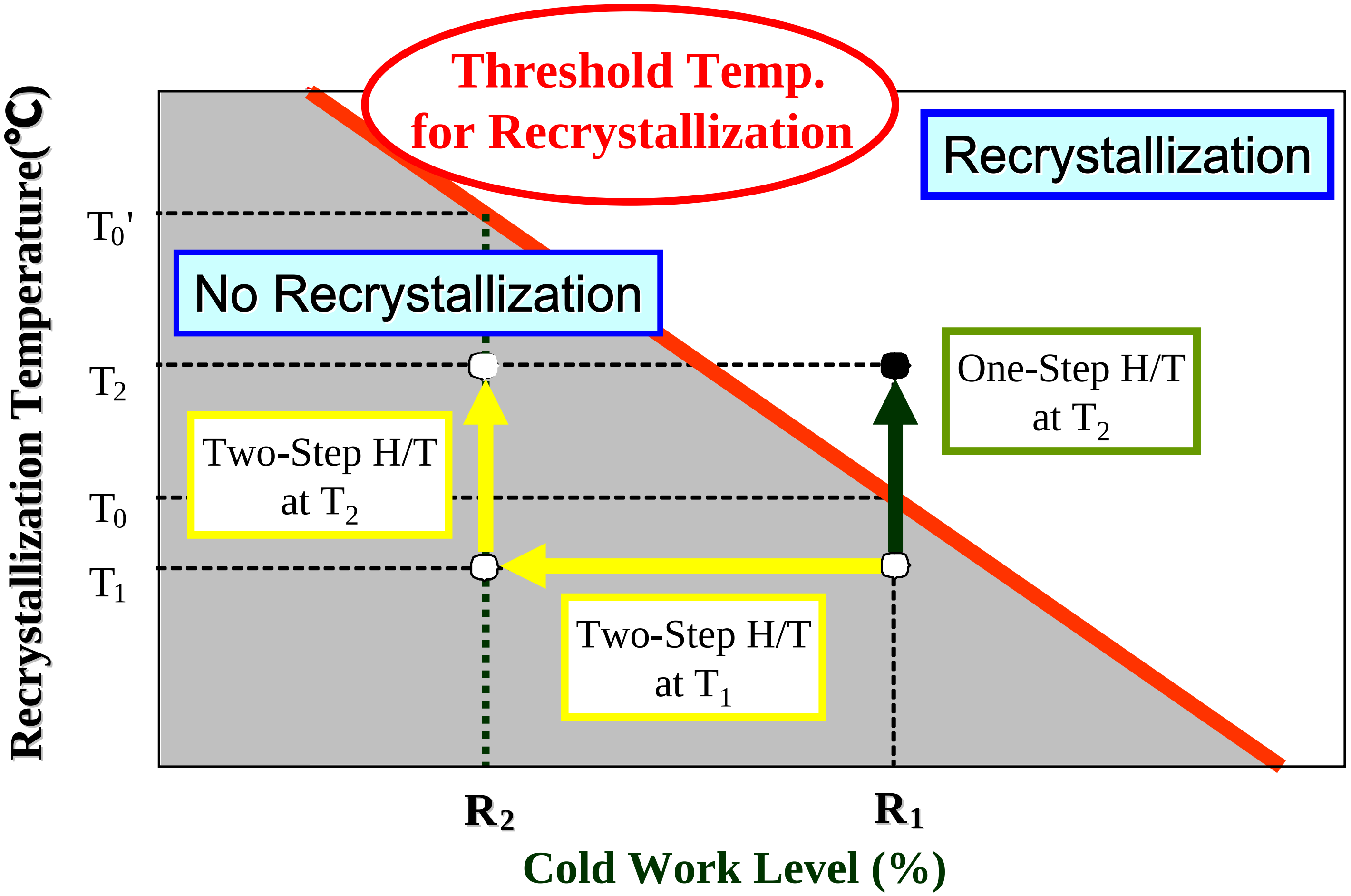




Not Recrystallized

- The aim of this test is to establish a heat treatment technique to consistenstly and reiliably produce the recovered structure (<400Hv) without recrystallization.

Basic idea of Two Step Heat Treatments for Recrystallization Control



- The first step H/T provides the recovery of strain energy which elevates the threshold temperature for recrystallization.
- The second step H/T at higher temperature can sufficiently soften cladding tube without recrystallization.

Grain Morphology Control

- 9Cr-ODS Steel: Alpha to Gamma Transformation.

  - Intermediated heat treatment with slower cooling rate softens and allows to cold roll without cracking.

  - Final heat treatment (normalizing + tempering) produces equiaxed tempered martensite (with δ phase) with little strength anisotropy.

- 12Cr-ODS: Recrystallization after Two-step Annealing

  - Once recrystallization took place in the course of cold-rolling process, recrystallized microstructure can not be obtained at final heat treatment.

  - “Two-step annealing” is useful to soften the cold-rolled tubes without premature recrystallization in intermediate heat treatments.

Chapter 5. Mechanical Properties

Deformation Mode

Steady State Operation

Noble Fission Gas Accumulation

↓

Internal Gas Pressure Increase

↓

Creep Rupture Property
(Long Term:~9 years)
(Max. ~12 MPa)

Off-Normal Operation

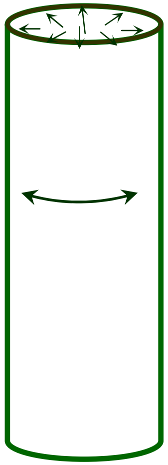
Over Power Transient

↓

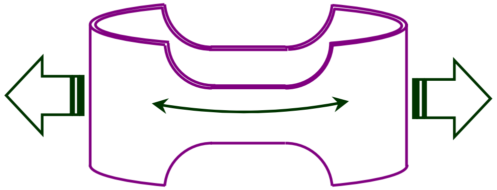
Fuel Pellet Expansion
(Fuel-to-Cladding Mechanical Interaction)

↓

Tensile Property
(Short Term)



Pressurized Tube
Specimen



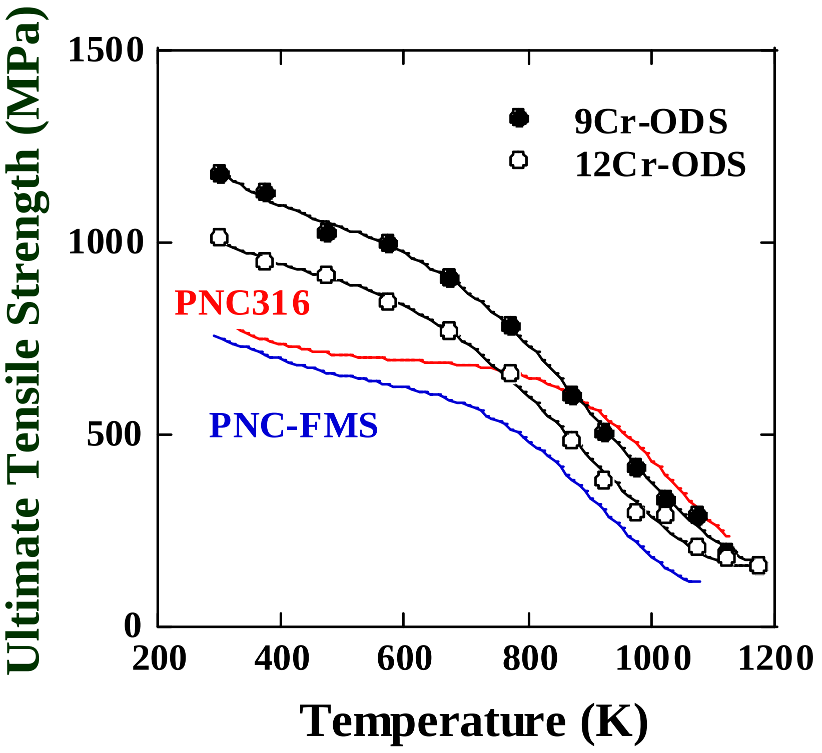
Ring Tensile
Specimen

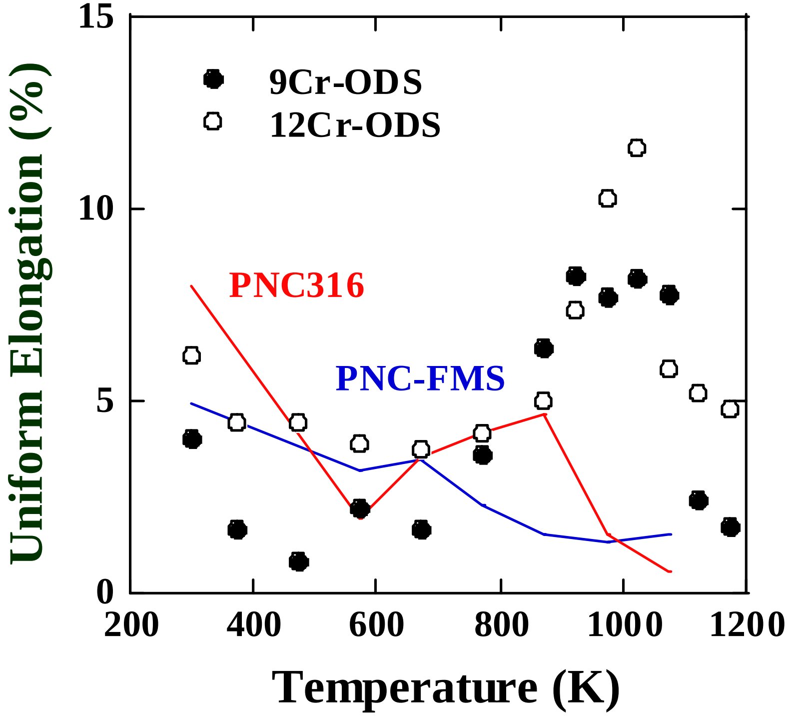
Material Strength Standard for Fuel Pin Mechanical Design

Material strength	1. Short-term tensile strength (σ_y, σ_u)
	2. Creep rupture strength (σ_R)
	3. Fatigue strength
	4. Transient burst rupture strength
Weld material strength	1. Short-term strength (σ_y, σ_u)
	2. Creep rupture strength
	3. High-cycle fatigue property
Stress-strain correlation	1. Short-term stress-strain
	2. Thermal creep strain
	3. Irradiation creep strain
Environmental effects	1. Environmental effect adjusting factor
	2. Sodium corrosion
	3. Fuel-to-cladding chemical interaction: FCCI
	4. Void swelling
Physical properties	1. Young modulus, 2. Poisson ratio, 3. Thermal expansion, 4. Thermal conductivity, 5. Heat capacity, 6. Density, 7. Transformation point
Specifications	1. Manufacturing process, 2. Chemical composition, 3. Final heat treatments

JNC TN9400 2005-015 (2005)

Tensile Property

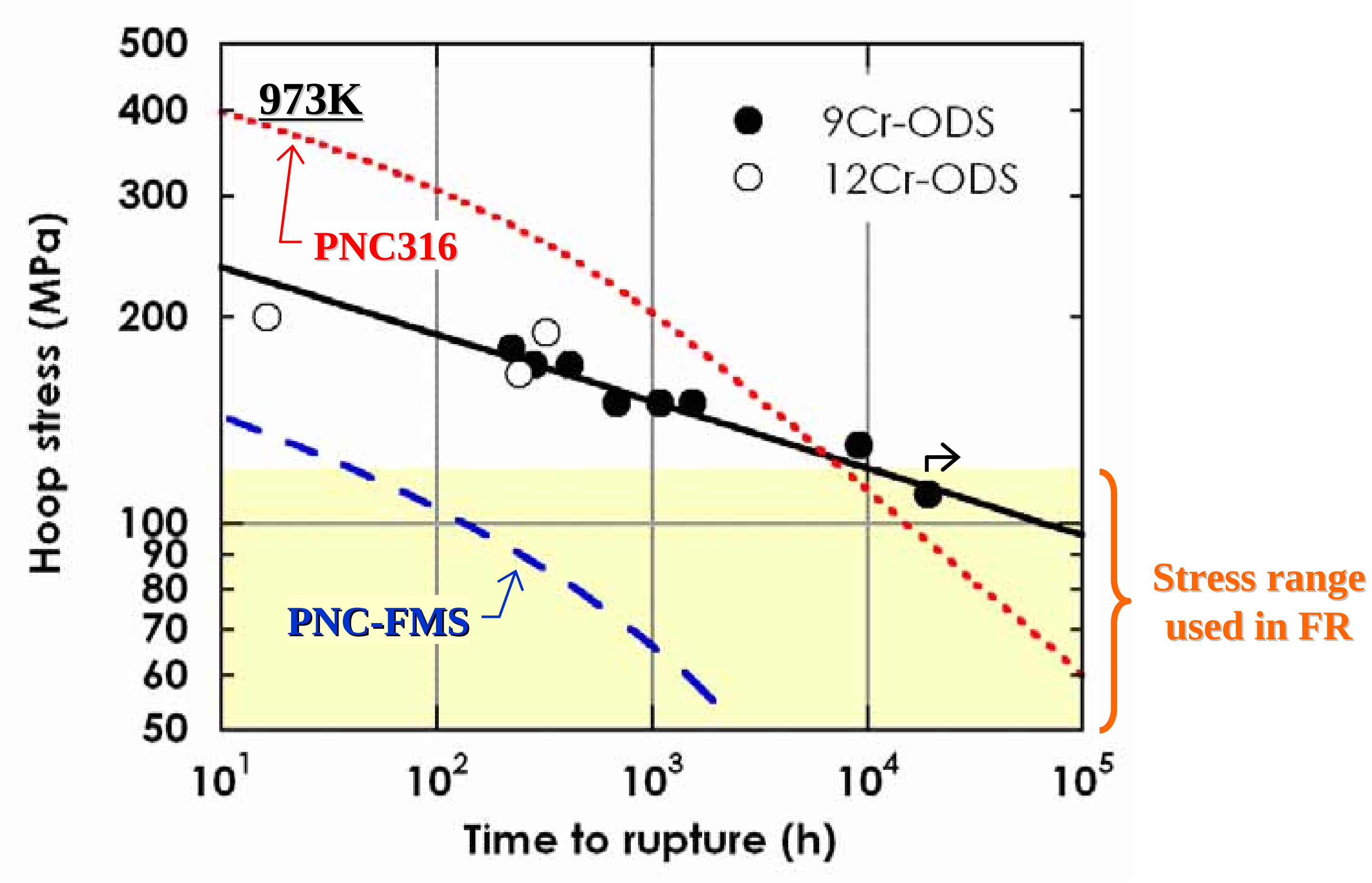




Ultimate tensile strength of 9Cr-ODS steel is much higher than precipitation hardened PNC-FMS.

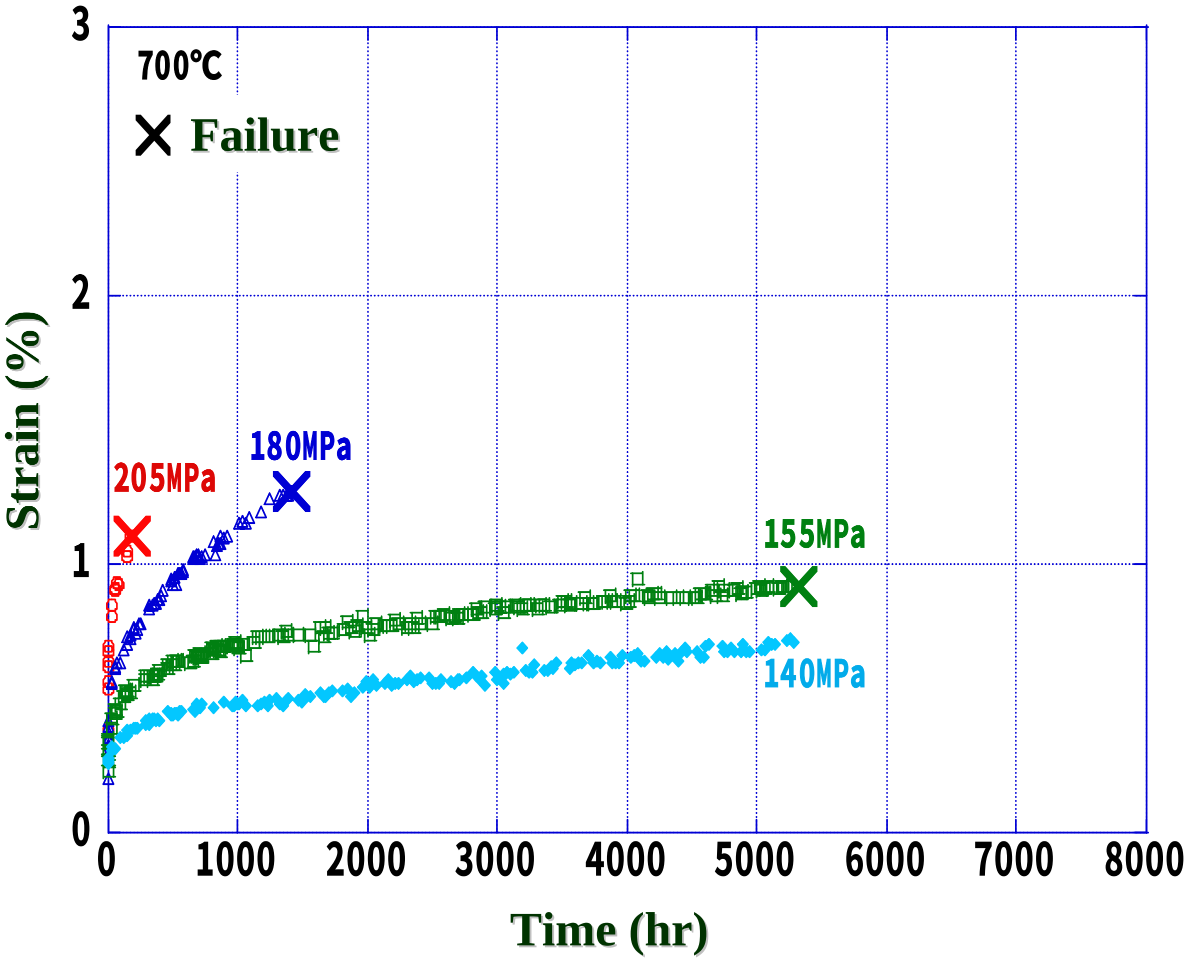
Uniform elongation of both 9Cr- and 12Cr-ODS steels are comparable with PNC316 and PNC-FMS.

Creep Rupture Properties



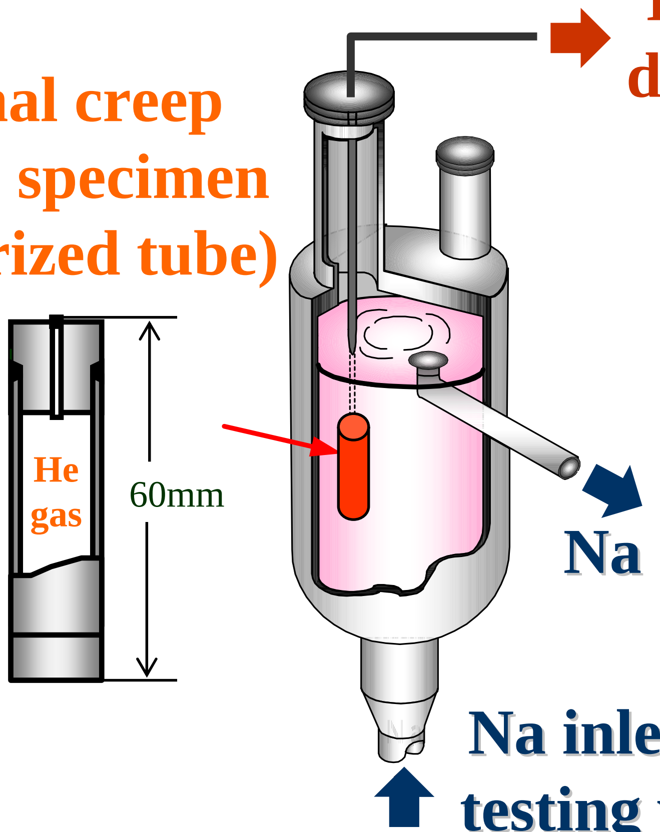
Creep rupture strength of both 9Cr- and 12Cr-ODS steels under internal pressures meet the target: 120 MPa for 10,000 hr at 973 K.

Creep Curves of 9Cr-ODS Steel



Creep curves under axial stress show little ternary stage in both 9Cr- and 12Cr-ODS steels

Sodium Immersion Effect on Creep Rupture Property

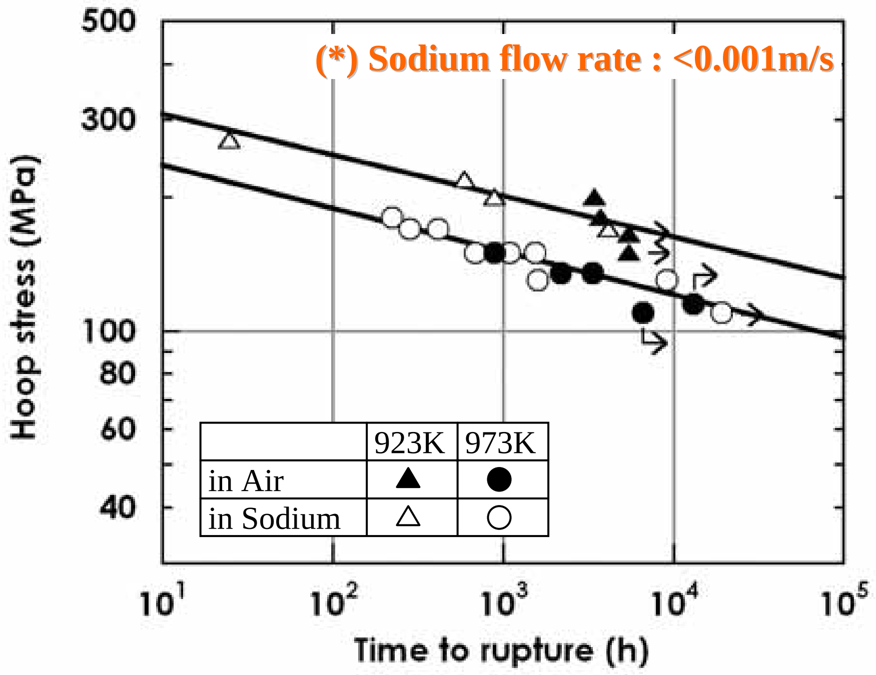


Internal creep rupture specimen (Pressurized tube)

He gas detector

Na outlet

Na inlet into testing vessel


(*) Sodium flow rate : <0.001m/s

-The 9Cr-ODS steel has superior sodium compatibility up to 973 K in stagnant sodium condition.

-Decarburization and Ni intrusion will be more influential For the 9Cr-ODS steel in flowing sodium and in-pile conditions.

Chapter 6. Irradiation Tests

Step1. Material Specimen Irradiations

- Screening of candidate materials
- Fundamental investigation of irradiation behavior
- Material Strength Standards

↓

Step2. Fuel Pin Irradiations

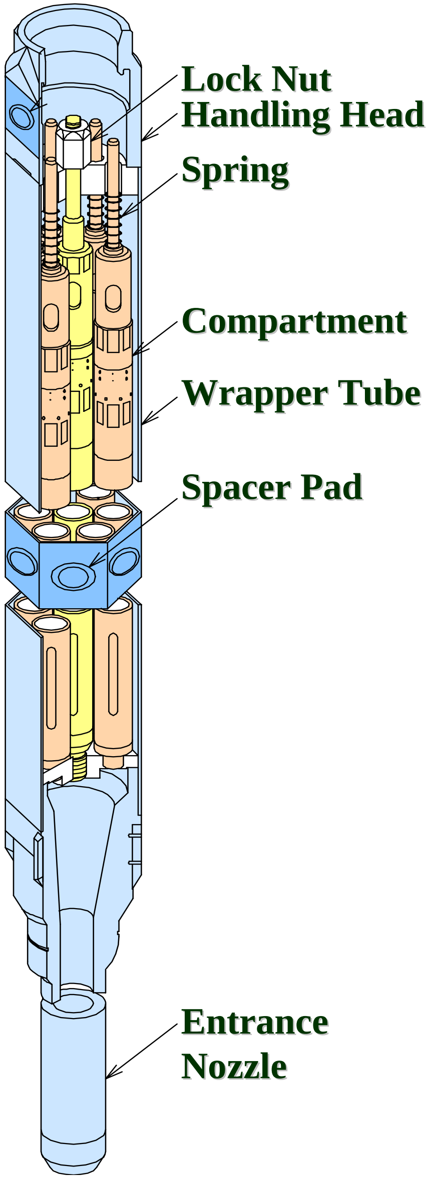
- Step 1 +
- Fuel to cladding mechanical/chemical interaction

↓

Step3. Leading/Dedicated Subassembly Irradiations

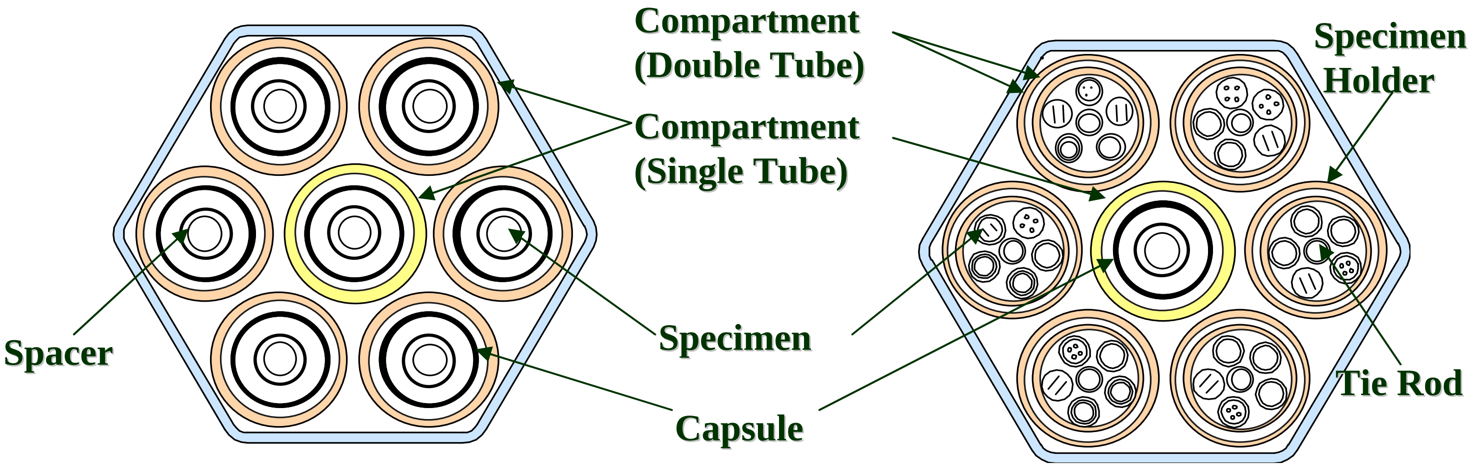
- Demonstration

Material Irradiation Rig: Off-line Type
- CMIR & SMIR -



Cross section of Structure Materials Irradiation Rig (SMIR)

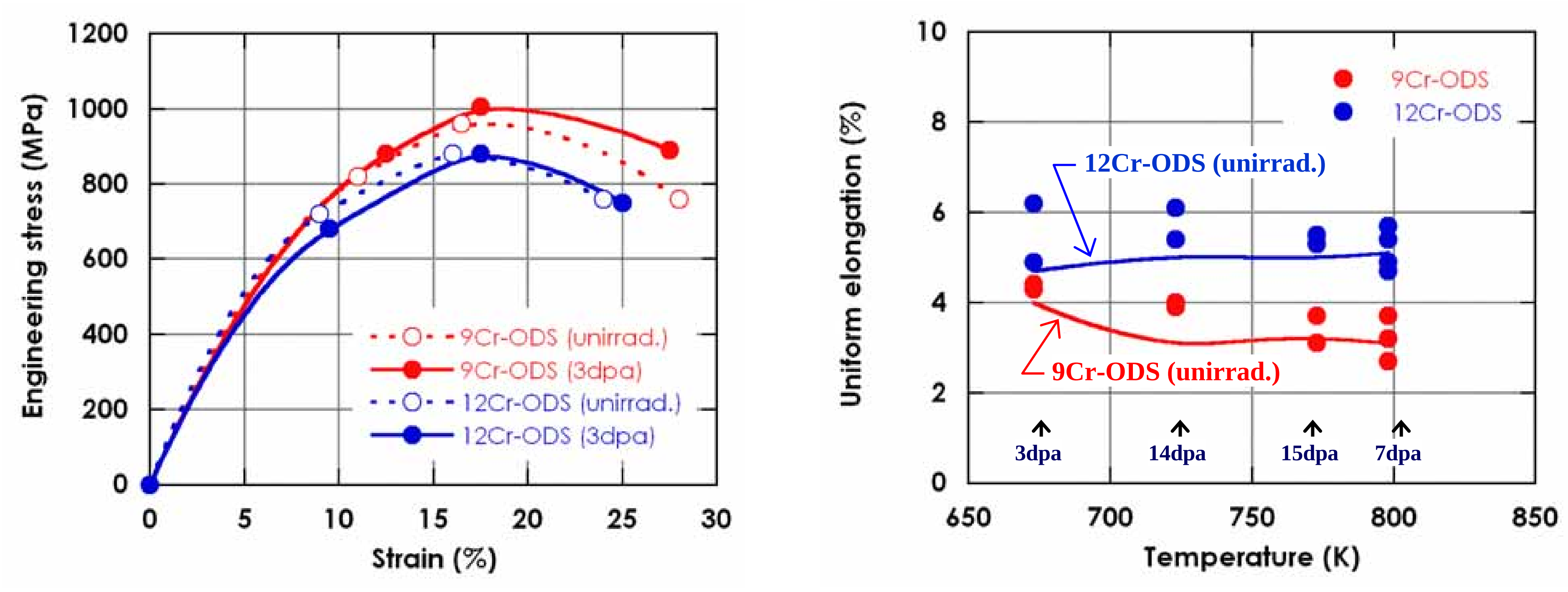
Cross section of Core Materials Irradiation Rig (CMIR)



Example of Specimen and Capsule

Example of Miniature Specimen and Holder

Irradiation Effect on Tensile Property



(a) Stress-strain curve

(b) Uniform elongation

Strength and ductility levels of irradiated ODS steel cladding tubes are adequately maintained.

Fuel Pin Irradiations in EBR-II

JAEA(PNC)-DOE Collaboration

12 Fuel Pins

- 1DK: 13Cr-3W-0.5Ti-0.35Y2O3-0.07Ex.O
- 1DS: 0.1C-11Cr-3W-0.5Ti-0.5Y2O3-0.1Ex.O
- Higher Smear Density MOX Fuel Pellets
- PRW

Irradiation: Nov.26, 1992- Sep.15, 1994

- Two subassembly: SPA-1B, C2
- LHR: ~48 kW/m, Tcm:~640℃
- Burnup:~6.3 at%, Fluence:~24 dpa

Post Irradiation Examinations

- Profilometry, Ceramography

Fuel Pin Irradiations in BOR-60 (1)

- JAEA-RIAR Collaborative Work -

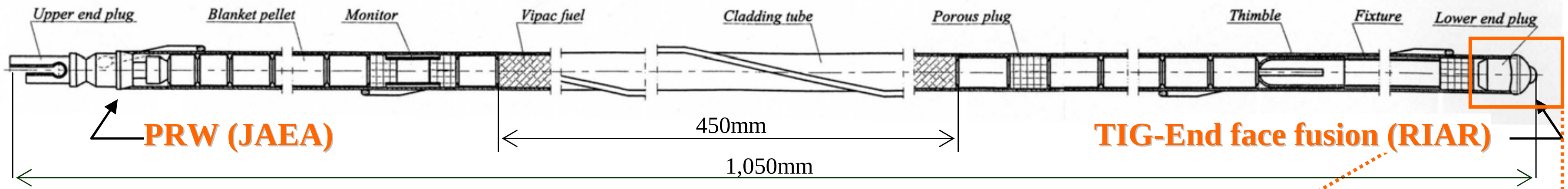
Objectives

- To investigate irradiation performance of ODS clad fuel pins
- To demonstrate and confirm the integrity up to 150 GWd/t

Specification of ODS Clad Fuel Pins

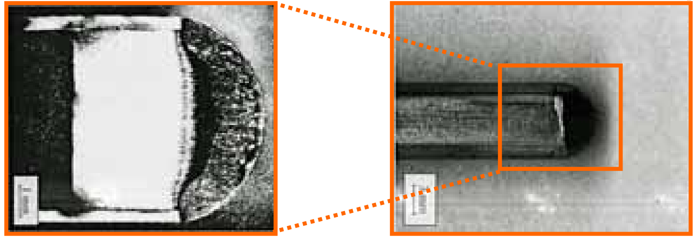
Fuel pin		Cladding material	Fuel	Pu/(Pu+U)	Smear density
Outer diameter	Length				
6.9 mm	1,050 mm	9Cr-ODS 12Cr-ODS	Vibro-packed MOX fuel	15 wt%	9.0 g/cm^3

Fuel Pin Structure and Manufacturing



PRW (JAEA)

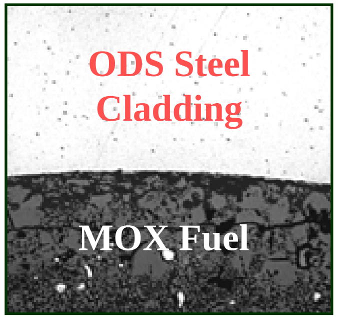
TIG-End face fusion (RIAR)

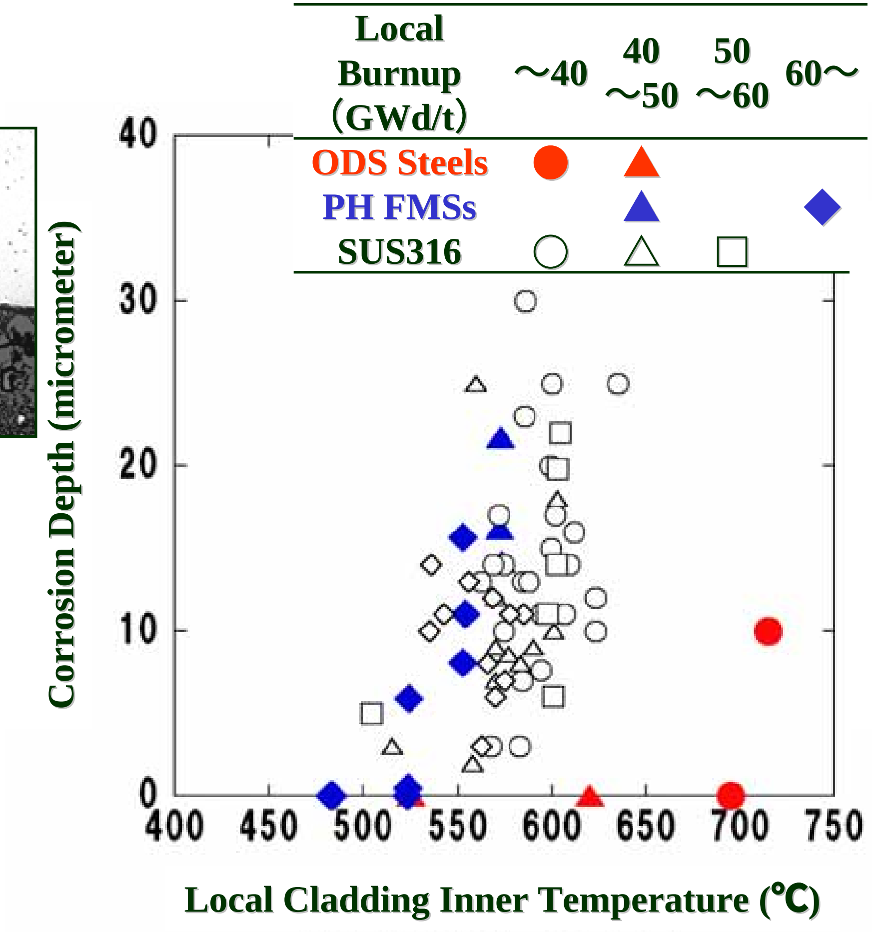


RIAR: Research Institute of Atomic Reactors

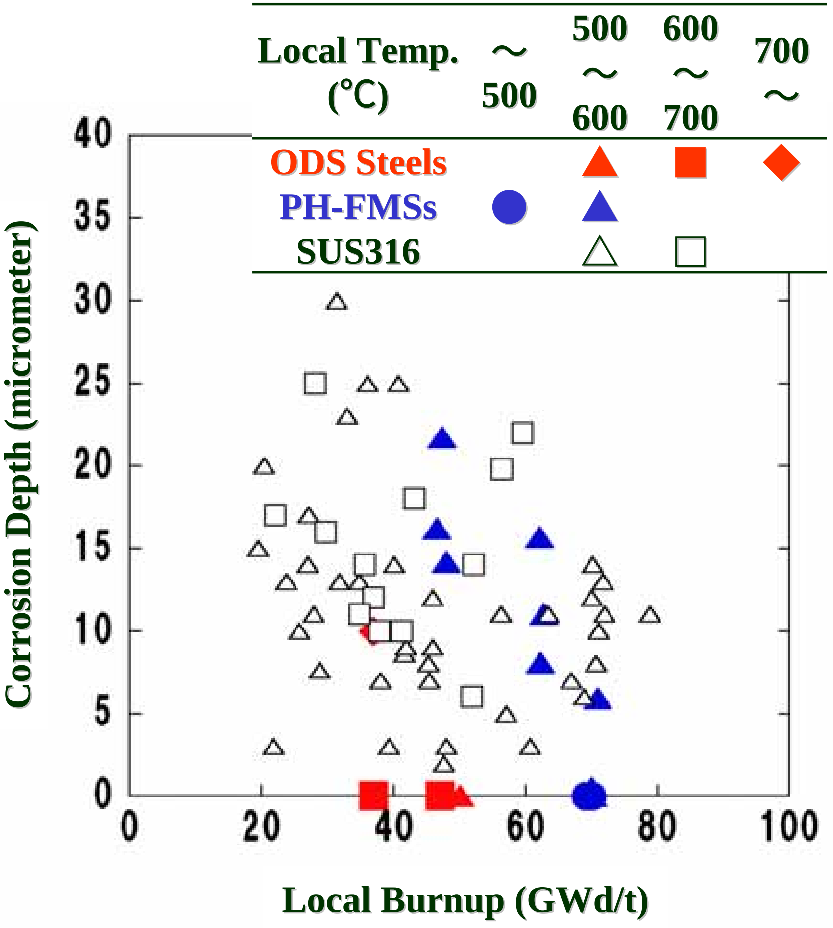
Fuel Pin Irradiations in BOR-60 (2)
- JAEA-RIAR Collaborative Work -

Fuel-to-Cladding Chemical Interaction





Local Burnup (GWd/t)	～40	40～50	50～60	60～
ODS Steels	●	▲		
PH FMSs		▲		◆
SUS316	○	△	□	



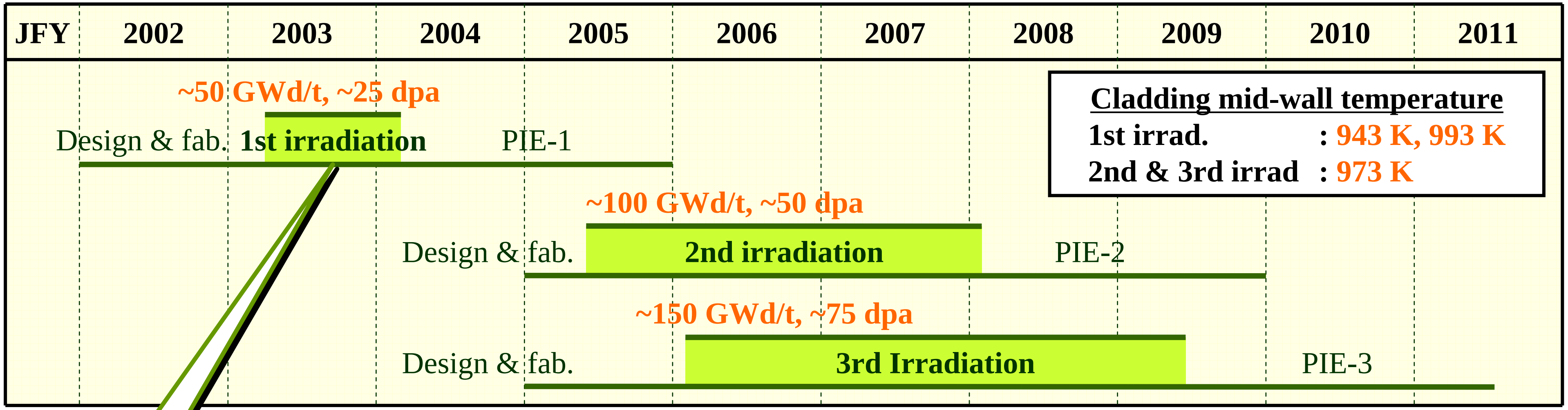
Local Temp. (℃)	～500	500～600	600～700	700～
ODS Steels		▲	■	◆
PH-FMSs	●	▲		
SUS316		△	□	

In case of BOR-60 irradiations, vibro-packed fuel contains uranium getter to control stoichiometry. This low stoichiometric condition (oxygen-to-metal ratio: ~1.93) will strongly influence (suppress) internal corrosion behavior

Fuel Pin Irradiations in BOR-60 (3)

- JAEA-RIAR Collaborative Work -

■Schedule and Irradiation Conditions



JFY 2002 2003 2004 2005 2006 2007 2008 2009 2010 2011

~50 GWd/t, ~25 dpa

Design & fab. **1st irradiation** PIE-1

~100 GWd/t, ~50 dpa

Design & fab. **2nd irradiation** PIE-2

~150 GWd/t, ~75 dpa

Design & fab. **3rd Irradiation** PIE-3

Cladding mid-wall temperature

1st irrad. : 943 K, 993 K

2nd & 3rd irrad : 973 K

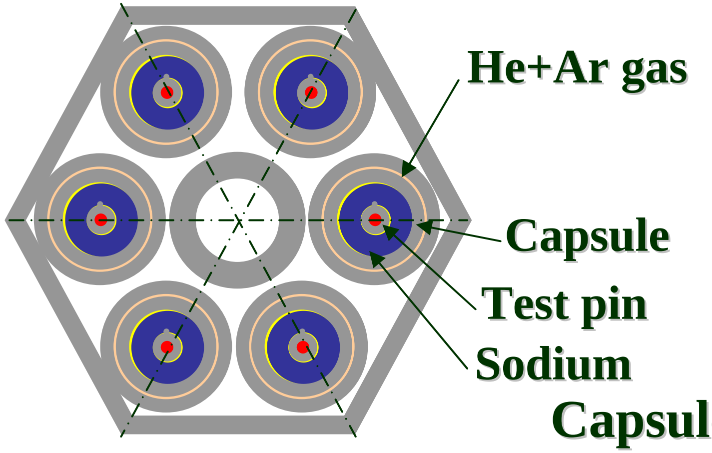
◆Results

- Irradiation results of **CDF up to 0.3** were achieved without fuel pin failure.

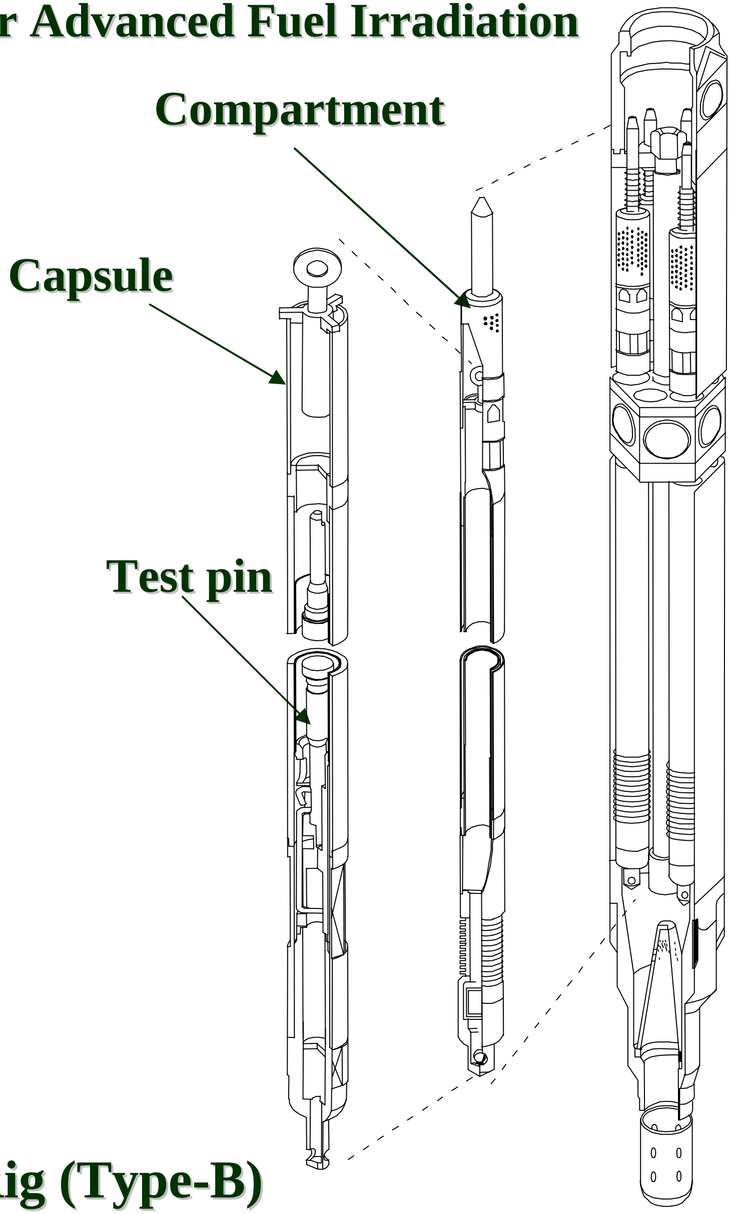
- Maximum corrosion depth observed was 10 micrometer which is comparable with PNC-FMS.

Fuel Pin Irradiations in JOYO
- Irradiation Rig -

1. Cladding tubes
   - Material: 9Cr-ODS
   - OD8.5mm x ID7.5mm
2. Fuel pellets
   - $(U, Pu)O_{2-x}$ (O/M = high & low)
   - OD7.3mm x ID2.2mm
   - Fuel column length : 400mm
3. Irradiation conditions
   - Target LHR: 450W/cm
   - Target Clad temp.: 700℃
   - Target burnup: ~220GWd/t



Capsule Rig for Advanced Fuel Irradiation



Capsule-type Irradiation Rig (Type-B)

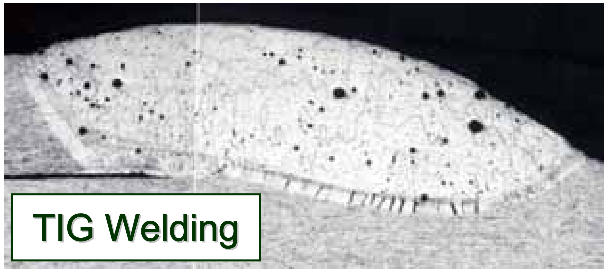
PRW Technology (1)

When ODS steels are heated far beyond 1,473 K (1,200℃), nano-meter size dispersoids will coarsen and lose dispersion strengthening effects and argon gas bubble will precipitate at grain boundary.

↓

Pressurized Resistance Welding (PRW) Process*

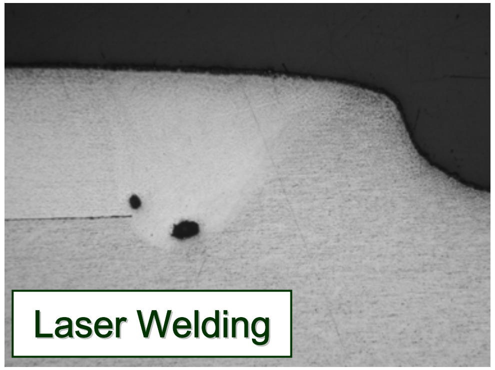
*: Electrical resistance heating of the components while maintaining a continuous force sufficient to forge weld without melting.



TIG Welding

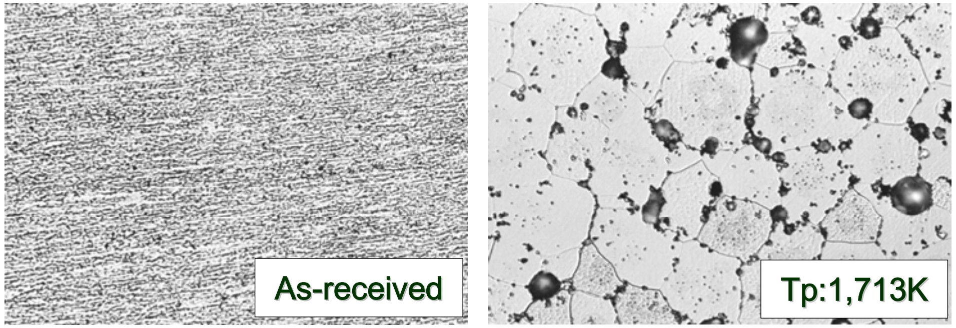
Loss of Original Microstructure

Blow Holes



Laser Welding

Argon Gas Bubble Precipitation by Over Heating



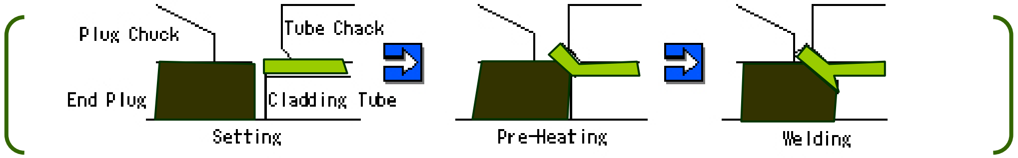
As-received

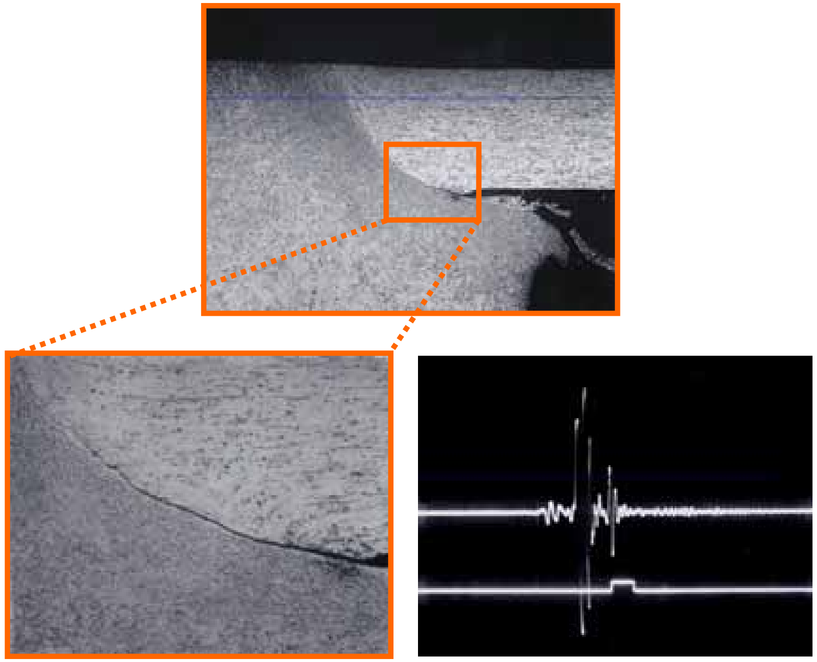
Tp:1,713K

Photographs are taken by Fuel Technology Research and Development Section, Fuel Technology Department, Plutonium Fuel Development Center, Tokai Research and Development Center

PRW Technology (2)

■Welding Process



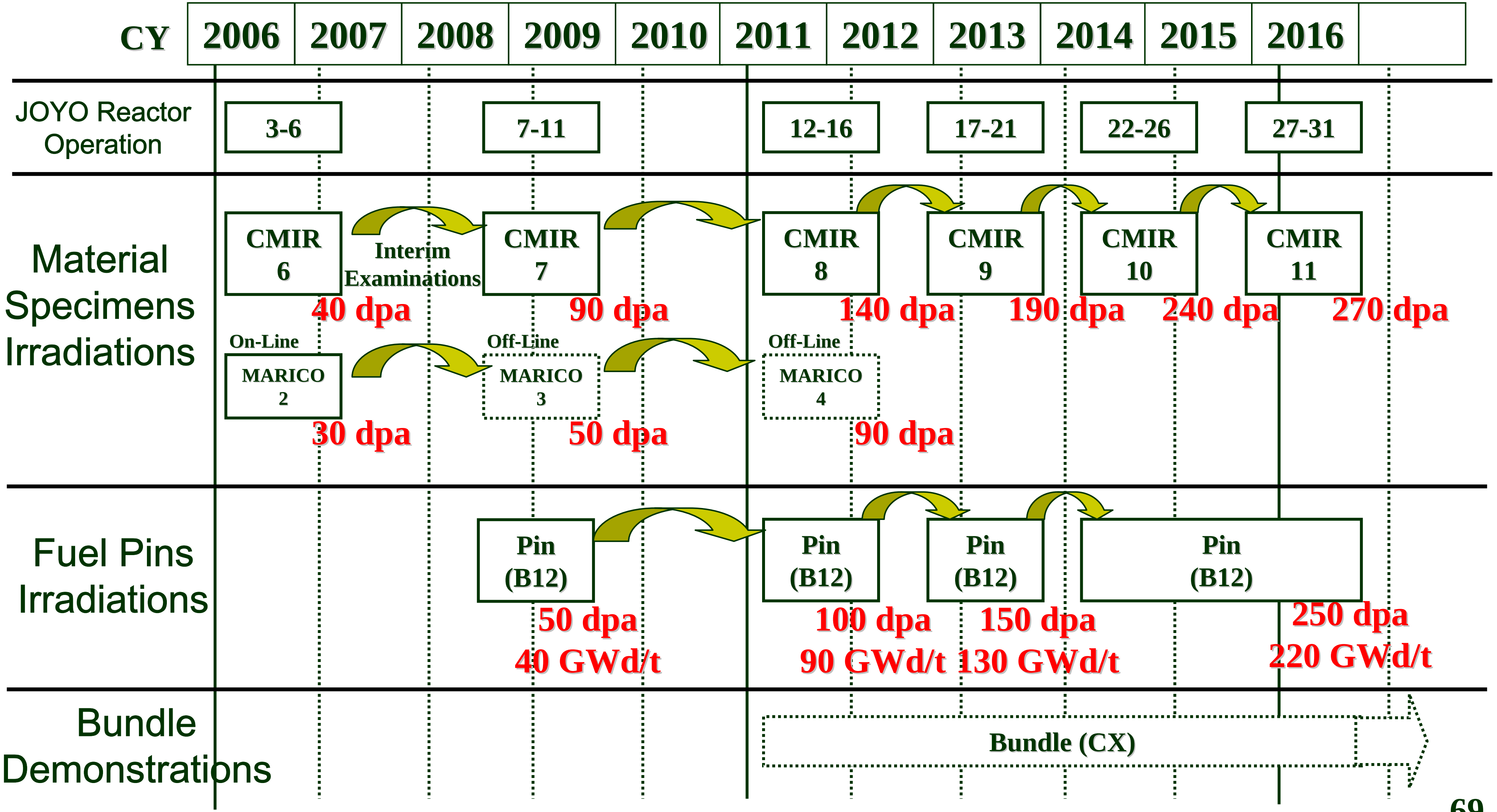


Ultrasonic Inspection

Available for fuel pin end-plugs and pressurized tube specimens

Schedule of JOYO Irradiation Tests

<Scheduled at beginning of FaCT project>



CY 2006 2007 2008 2009 2010 2011 2012 2013 2014 2015 2016

JOYO Reactor Operation: 3-6, 7-11, 12-16, 17-21, 22-26, 27-31

Material Specimens Irradiations:

CMIR 6 — 40 dpa

Interim Examinations

CMIR 7 — 90 dpa

CMIR 8 — 140 dpa

CMIR 9 — 190 dpa

CMIR 10 — 240 dpa

CMIR 11 — 270 dpa

On-Line MARICO 2 — 30 dpa

Off-Line MARICO 3 — 50 dpa

Off-Line MARICO 4 — 90 dpa

Fuel Pins Irradiations:

Pin (B12) — 50 dpa, 40 GWd/t

Pin (B12) — 100 dpa, 90 GWd/t

Pin (B12) — 150 dpa, 130 GWd/t

Pin (B12) — 250 dpa, 220 GWd/t

Bundle Demonstrations: Bundle (CX)

Chapter 7. Future Mass Production

Optimistic Estimation of Market Scale for ODS Steel Cladding Tubes

	CFBR
Core Fuel Pin Geometry	OD8.5mm×L3,000mm
Core Fuel Pins/Subassembly	255
Fuel Subassembly	~800
Operation Cycle Length	~26 month
Number of the Tubes	~24,000 /year*
Weight of the Tubes	~7,000 kg/year**

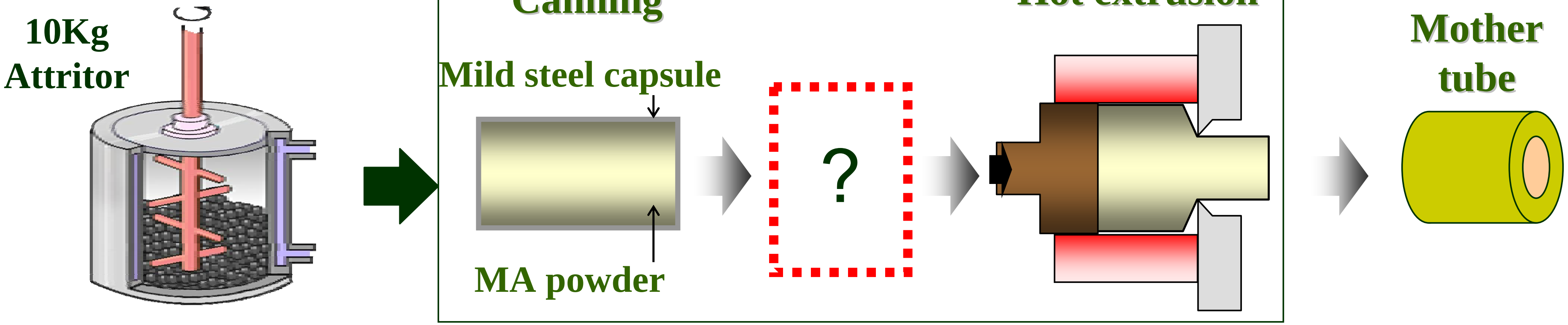
*: does not include loss in manufacturing and fuel pin fabrication processes.
**: The weight of raw steel powders may be three times larger than that of products.

JOYO Irradiation Tests: 24 tubes/year (4.4kg)

↓

Scale-up of factor 1,000 is necessary for commercialization!!

Key Process for Large Scale Production



Hot Consolidation

Mechanical alloying

10Kg
Attritor

Canning

Mild steel capsule

MA powder

?

Hot extrusion

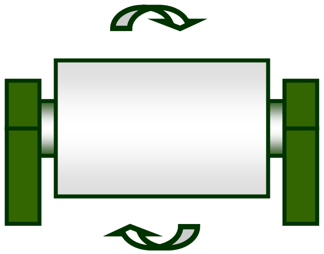
Mother tube

● **Present process**

● **Large scale process**

Large & Multiple Attritor
(30Kg x 4units)

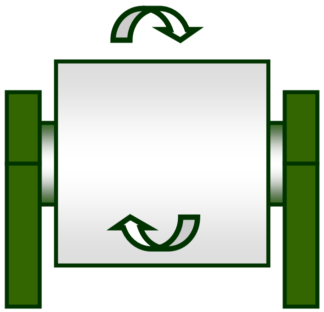
Large
Ball Mill
(>250Kg)



Additional Consolidation before Hot Extrusion
for Large Size Hollow Billet

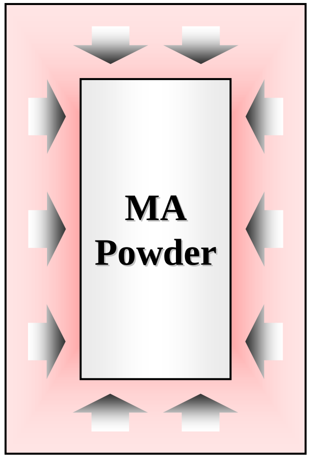
① **Pre-consolidation by HIP**
② **Hollow Capsule Direct Hot Extrusion**

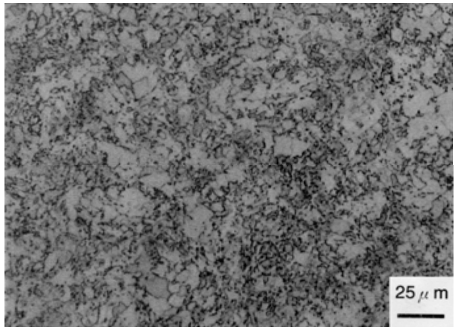
Pre-Consolidation by HIP and Hot Extrusion



Mechanical Alloying
(1ton Ball Mill)

Hot Iso-static Pressing



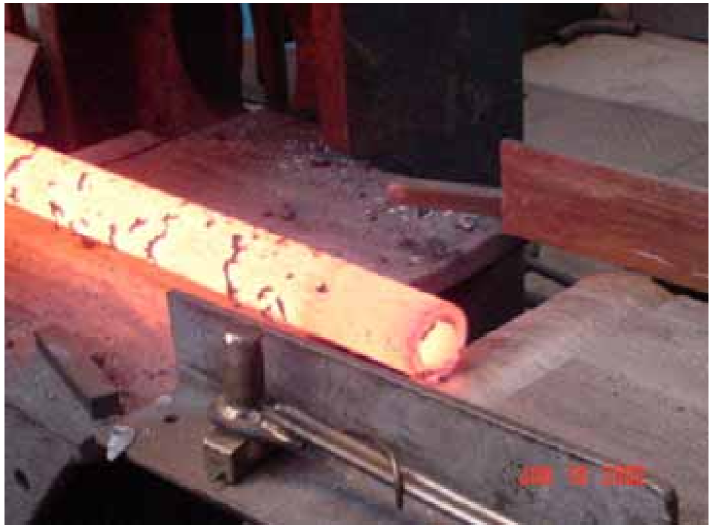


Optical micrograph of HIPed product

HIPed Billet: OD225mm×ID60mm ×L500mm: 150kg

Hot Extrusion Test of HIPed Billets@1,453K

Machining
Drilling



Annular Billets: OD65mm×ID48mm ×L10,000mm:120kg

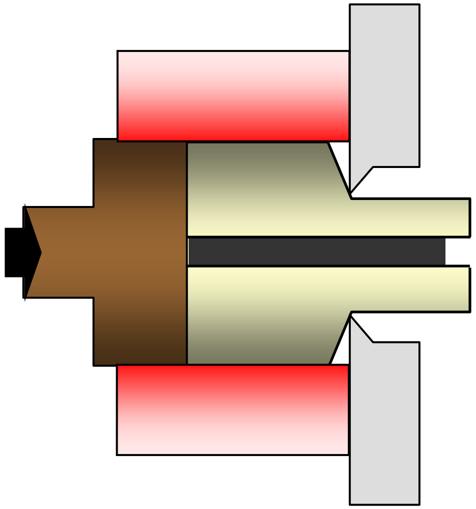
Direct Hot Extrusion by Hollow Capsule

Powder Filling

Assembling

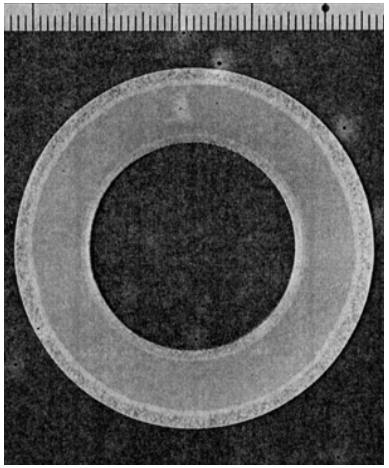
OD147mm×ID32mm
×L590mm:30kg

Hot-Extrusion 2,000ton Press
@1,423K, Extrusion Ratio 13



Annular Billets:OD32mm×ID21mm
×L2,000mm:7.3kg

High Chromium Steel Liner Cross Section



Conclusions: 1

Candidate

Martensitic 9Cr-ODS steel: Primary

Fully ferritic 12Cr-ODS steel: Secondary

Alloy Design: Strength, Ductility, Microstructure Control

- **Phase and Grain Morphology Control: C,Cr, (Ex.O)**
- **Solution & Dispersion Hardening : W, Ti, Y_2O_3, Ex.O**

Microstructure Control:

- **Nano-size oxide particles precipitate during PM process**
- **Grain morphology must be controlled during tubing process**
- **9Cr-ODS: Alpha to Gamma Transformation**
  → Cooling Rate Control
- **12Cr-ODS: Recrystallization**
  → Two Step Heat Treatment

Conclusions: 2

Mechanical Properties

- Tensile and Creep Rupture Strength met the Initial Target
- Environmental Effects

Manufacturing Process for Future Mass Production

- Screening Feasible Technologies

  Larger Size Hot-Extruded Billets

  → ○Pre-consolidation by HIP

  → △Direct Hollow Capsule Extrusion

Irradiation Tests

- Material Specimen Irradiations in JOYO in progress
- Fuel Pin Irradiation Tests in BOR-60 since 2003
- PRW technology

Activity in FaCT Project by 2015

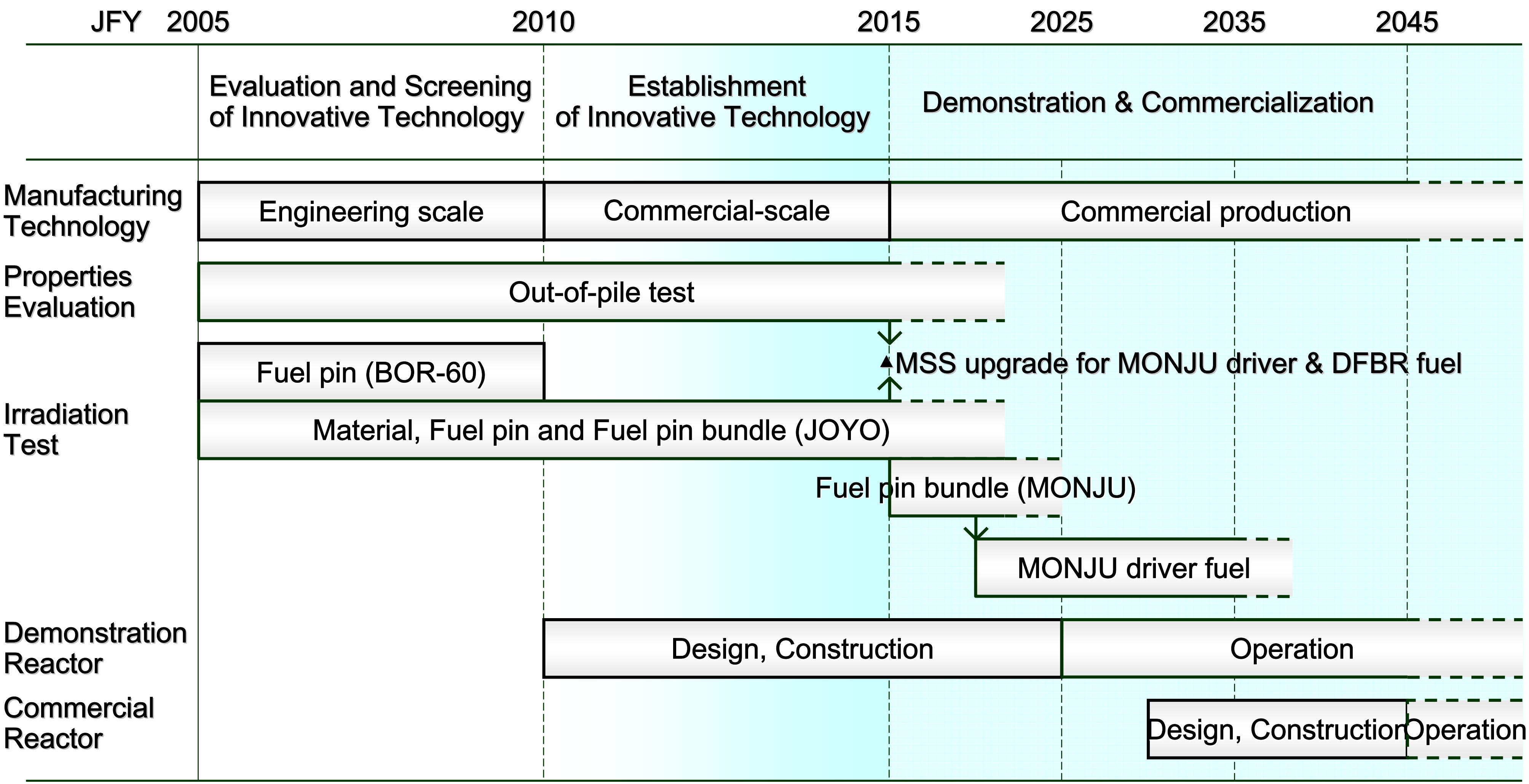
Manufacturing Technology Development for Mass Production

- Applicable for the annual production size:~10,000 tubes
- Larger Mother Tube Volume
- Longer Final Product
- Quality Assurance

Demonstration of High Burnup Capability

- Material Specimen Irradiations in JOYO up to 250 dpa
- Fuel Pin Irradiations in BOR-60 to 2009; 150GWd/t/75dpa
- Fuel Pin Irradiations in JOYO; ~180GWd/t/~210dpa
- Upgrading Material Strength Standard

Project Schedule



JFY 2005 2010 2015 2025 2035 2045

Evaluation and Screening of Innovative Technology

Establishment of Innovative Technology

Demonstration & Commercialization

Manufacturing Technology: Engineering scale → Commercial-scale → Commercial production

Properties Evaluation: Out-of-pile test

Irradiation Test: Fuel pin (BOR-60); Material, Fuel pin and Fuel pin bundle (JOYO); MSS upgrade for MONJU driver & DFBR fuel; Fuel pin bundle (MONJU); MONJU driver fuel

Demonstration Reactor: Design, Construction → Operation

Commercial Reactor: Design, Construction → Operation

Evaluate and Select Innovative Technologies by 2010: ODS clad fuel pin data at 150GWd/t and evaluation of high burnup capability

Establish Innovative Technologies by 2015: ODS clad fuel pin data at 250GWd/t and fuel pin bundle irradiation at medium burnup

Demonstrate Innovative Technologies by 2025: Fuel pin bundle/subassembly demonstration in MONJU

Representative Publications

[1] S. Ukai, S. Mizuta, M. Fujiwara, T. Okuda, T. Kobayashi, J. Nucl. Sci. Technol., Vol.39, No.7, pp.778-788 (2002).
[2] J. Bottcher, S. Ukai, M. Inoue, Nucl. Technol.,Vol.138, pp.238-245 (2002).
[3] S. Ukai, H. Hatakeyama, S. Mizuta, M. Fujiwara, T. Okuda, J. Nucl. Mater.,Vol.307-311, pp.758-762 (2002).

[4] S. Ukai, T. Kaito, S. Ohtsuka, T. Narita, M. Fujiwara, T. Kobayashi,
ISIJ International, Vol.43, No.12, pp.2038-2045 (2003).

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[12] T. Narita, S. Ukai, T. Kaito, S. Ohtsuka, M. Fujiwara, J. Nucl. Sci. Technol., Vol.42, No.9, pp.825-832 (2005).
[13] T. Kaito, S. Ukai, S. Ohtsuka, T. Narita, GLOBAL2005, Paper No.169, October 9-13, 2005, Tsukuba, Japan (2005).
[14] S. Ohtsuka, S. Ukai, H. Sakasegawa, M. Fujiwara, T. Kaito, T. Narita, Mater. Trans., Vol.46, No.3, pp.487-492 (2005)
[15] S. Ohtsuka, S. Ukai, M. Fujiwara, T. Kaito, T. Narita, J. Phys. Chem. Solids., Vol.66, pp.571-575 (2005).

[16] S. Ukai, T. Kaito, S. Ohtsuka, T. Narita, H. Sakasegawa,
Transactions of the 2006 ANS Annual Meeting, June 4-8, 2006, Reno, USA (2006).

[17] S. Ohtsuka, S. Ukai, H. Sakasegawa, M. Fujiwara, T. Kaito, T. Narita, J. Nucl. Mater., Vol.367-370, pp.160-165 (2007)
[18] T. Kaito, S. Ohtsuka, M. Inoue, GLOBAL2007, Paper No.005_175716, September 9-13, 2007, Idaho, USA (2007).