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International Centre for Theoretical Physics**



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**Joint ICTP/IAEA School on Physics and Technology of Fast Reactors  
Systems**

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**Principles of Design of Radiation Resistant Materials for Fast Reactor Fuel  
Assembly: SCOPE**

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Kalpakkam  
India*

# Principles of Design of Radiation Resistant Materials for Fast Reactor Fuel Assembly: SCOPE

## INTRODUCTION TO STEELS;

STRENGTHENING MECHANISMS IN AUSTENITIC STEELS;

20 % CW 316 AUSTENITIC STAINLESS STEELS TO D9: DESIGN PRINCIPLES;

AUSTENITICS TO FERRITIC STEELS &

OXIDE DISPERSION STRENGTHENED STEELS.

**RECAP:** Reactor circuits: core, out-of-core & balance of plant;

Severe service conditions for core component materials;

Major problems: void swelling, irradiation growth, hardening, creep and embrittlement;

Main cause is point defects, defect clusters & their interaction with matrix defects;

Void swelling: condensation of excess vacancies into voids, CW, dose, dose rate, temperature type of sinks influence the growth rate

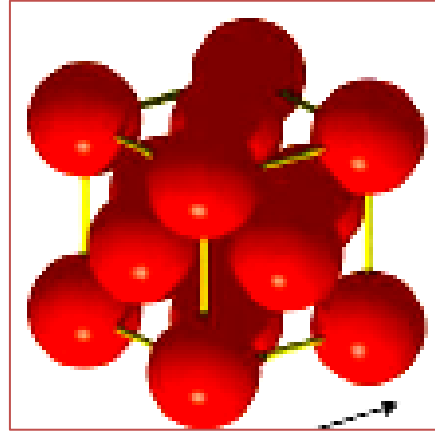
Irrad. Hard.: increase in strength due to defect clusters

Irrad. Creep : diametral strain increase due to SIPA, SIPN & void swelling

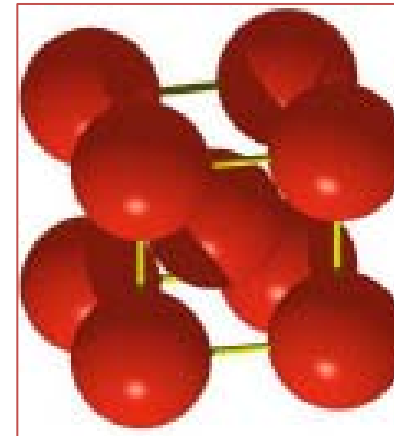
Irrad. Embrittlement : DBTT reduces with reduction in upper shelf energy, due to increase in yield strength due to irradiation .

# PHASES IN STEELS

PHASE TRANSFORMATIONS IN Fe ALLOYS	
Temperature	
1550 °C	Liquid
1500 °C	$\delta$ + Liquid
1400 °C	$\delta$
1200 °C	$\gamma$ + $\delta$
920°C	$\gamma$
800°C	$\alpha$ + $\gamma$
RT	$\alpha$

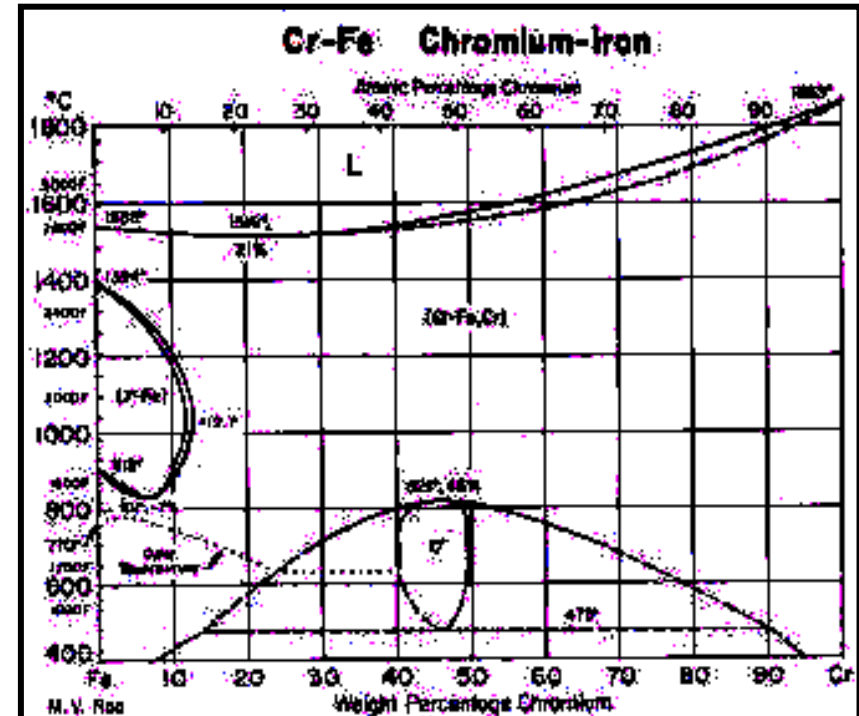
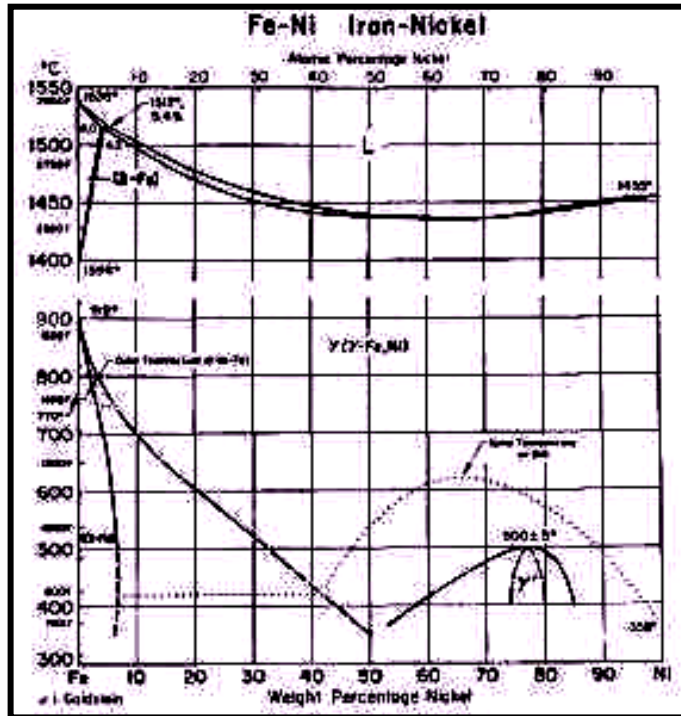
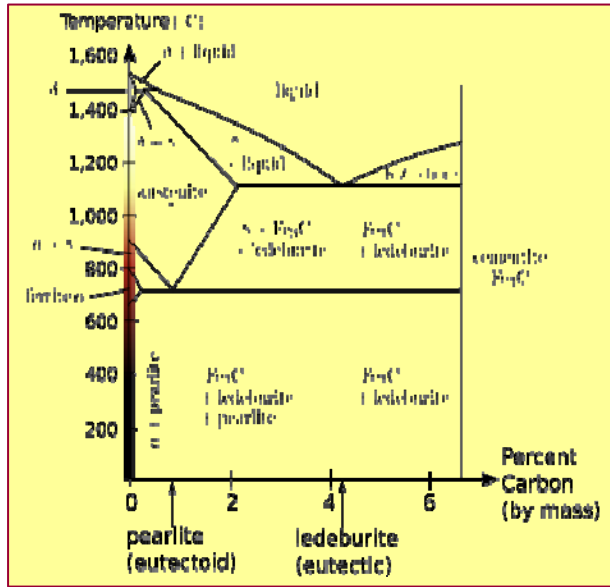


**AUSTENITE -  $\gamma$**



**FERRITE -  $\alpha$**

# Relevant phase diagrams



# TARGETS FOR FAST REACTOR MATERIALS SCIENTISTS -

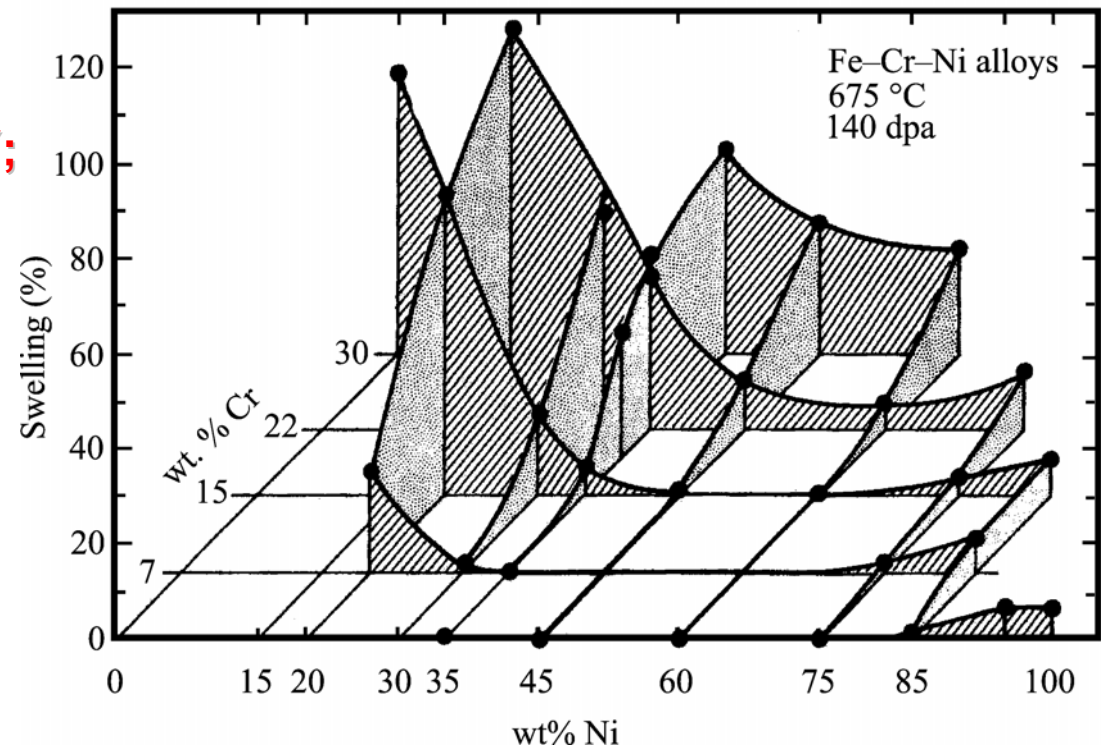
**HIGH BURN-UP;**

**HIGH TEMPERATURE CAPABILITY;**

**LONG LIFE.**

Ni → BENEFICIAL FOR  
SWELLING RESISTANCE;

Cr → BAD BEYOND 15 %.



Ni -- ■ --- 0.26 eV

Cr -- ■ --- 0.06 eV

Can you explain why binding energy with vacancies influences void swelling ?

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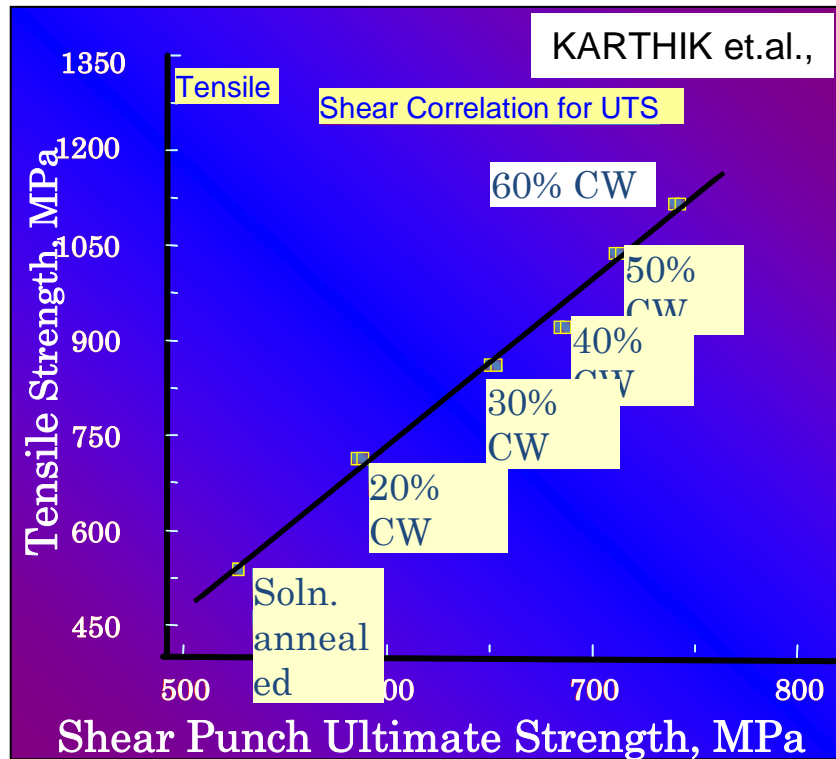
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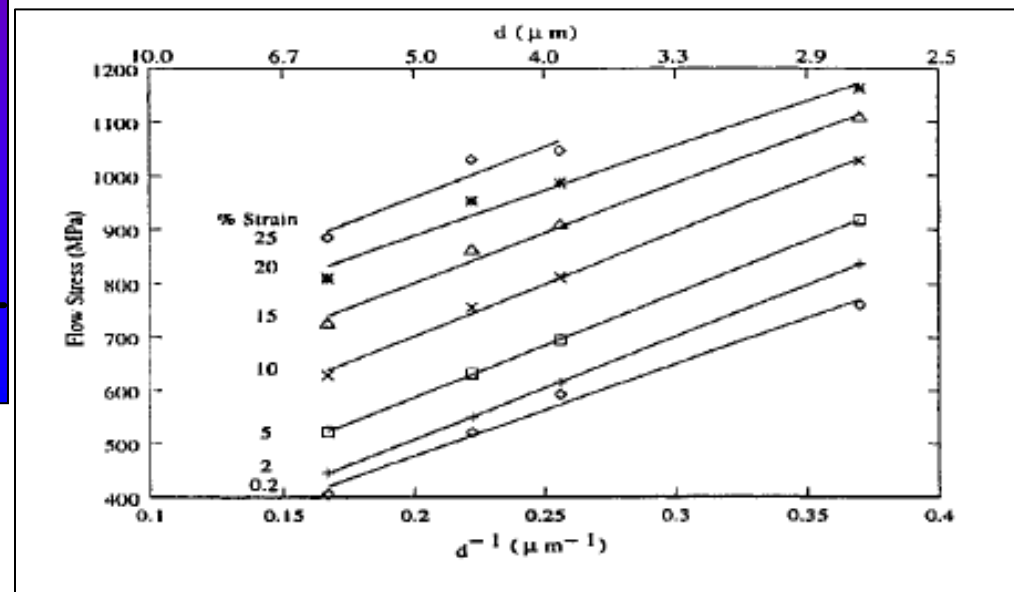
AUSTENITICS TO FERRITIC STEELS &

OXIDE DISPERSION STRENGTHENED STEELS.

# IMPROVING SWELLING RESISTANCE & MECHANICAL PROPERTIES



## STRENGTHENING BY CARBON & MOLYBDENUM ADDITION



**20 % COLD WORKED 316 STAINLESS STEEL ---→ FOR FAST REACTOR CORE**

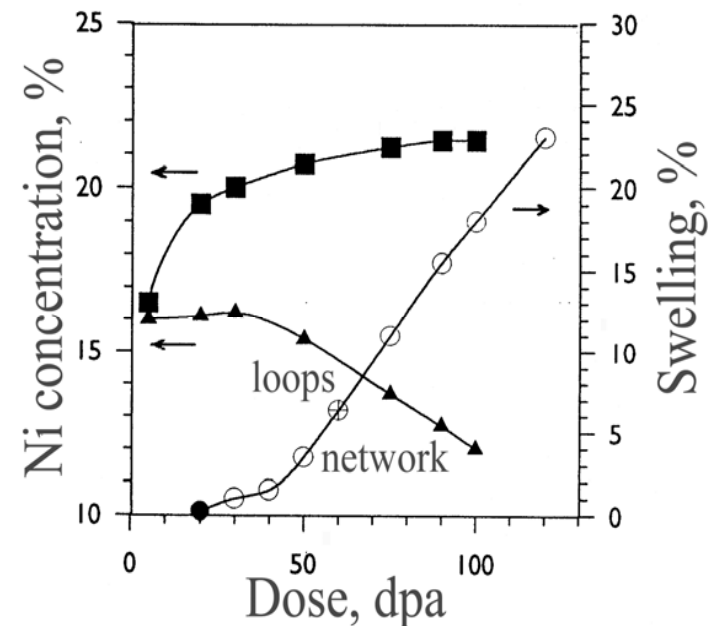
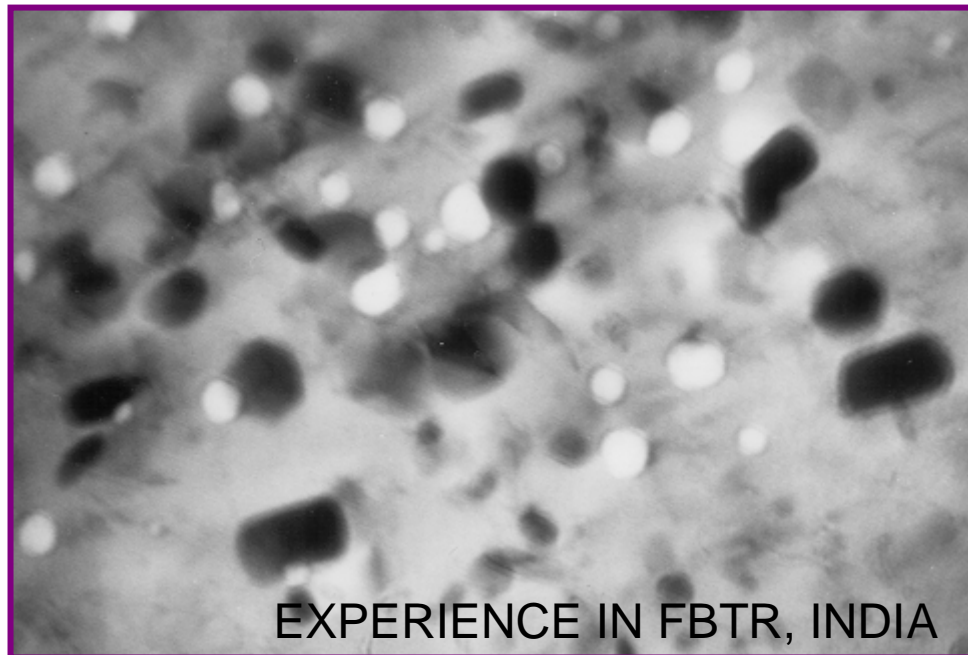


# WAS 20 % cw SS OK?

**NO !!! ACHIEVABLE BURN-UP IN FRENCH REACTOR WAS ONLY ~ 40 dpa !!!**

**IN-REACTOR EXPERIENCE NOT SATISFACTORY :**

**Ni<sub>3</sub>Si DUE TO RIS & VOIDS AROUND G-PHASE ;  $\gamma'$  PHASE – SOLID SOLUTION DECAY**



**WHAT NEXT ?**

**ADD Ti TO HAVE TWO ADVANTAGES: BIND THE VACANCIES (0.3 eV),  
FORM TiC- COHERENT PRECIPITATES**

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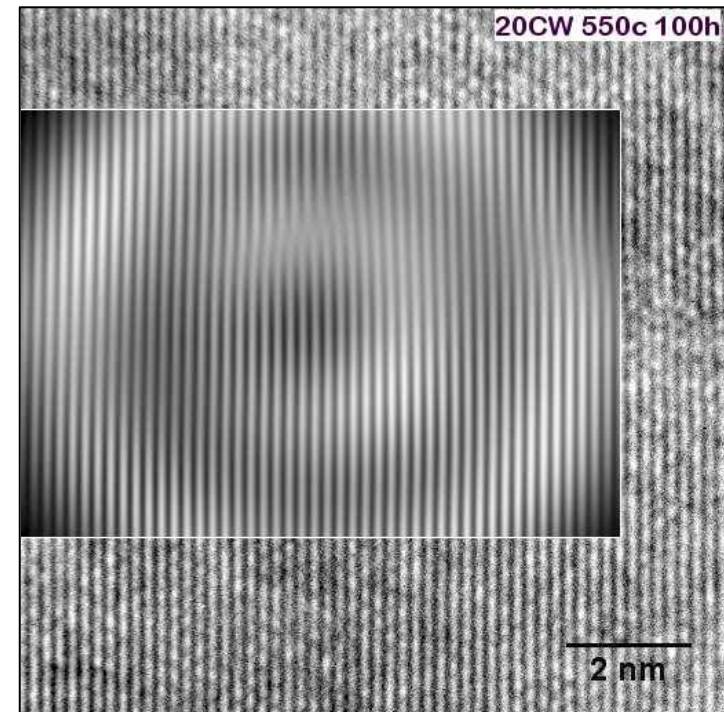
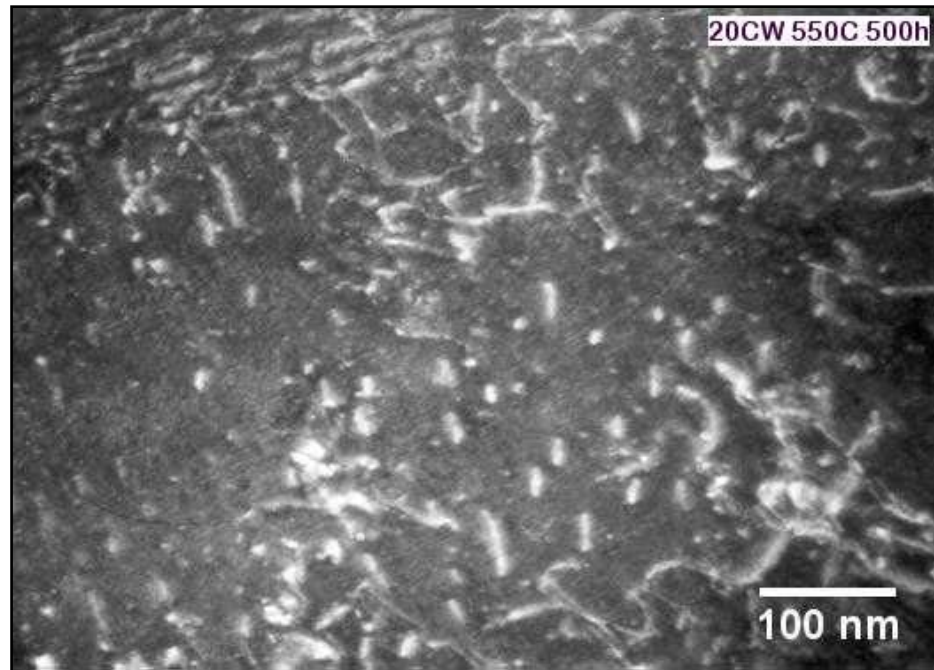
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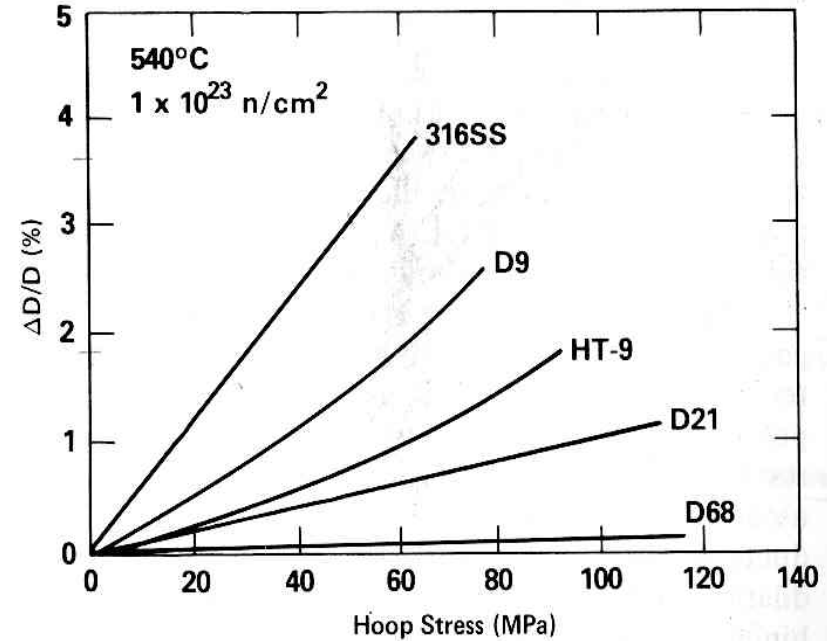
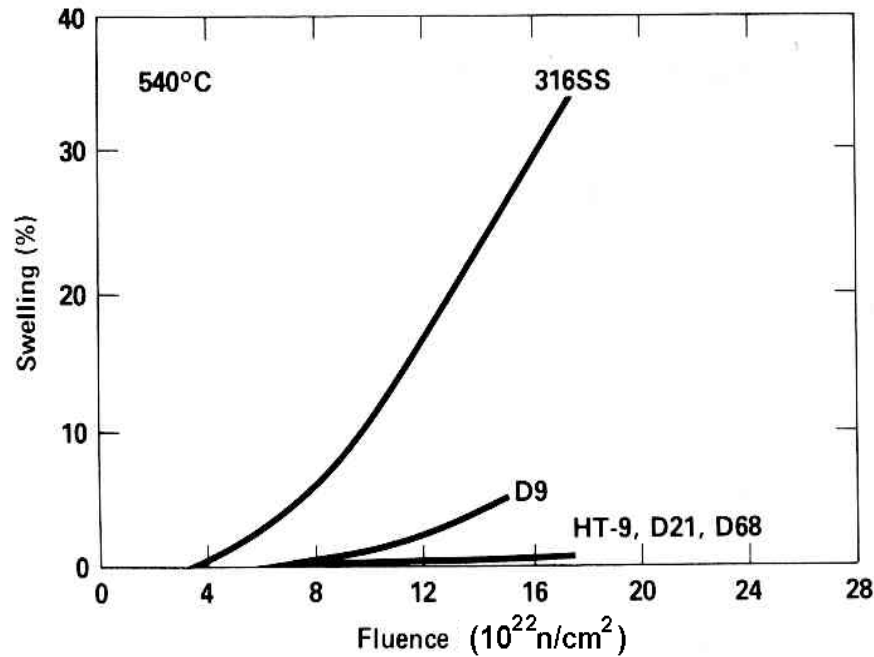
AUSTENITICS TO FERRITIC STEELS &

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20 % cw 316 STAINLESS STEEL --→  
15 % Ni-15 %Cr-Ti-SS(D9)



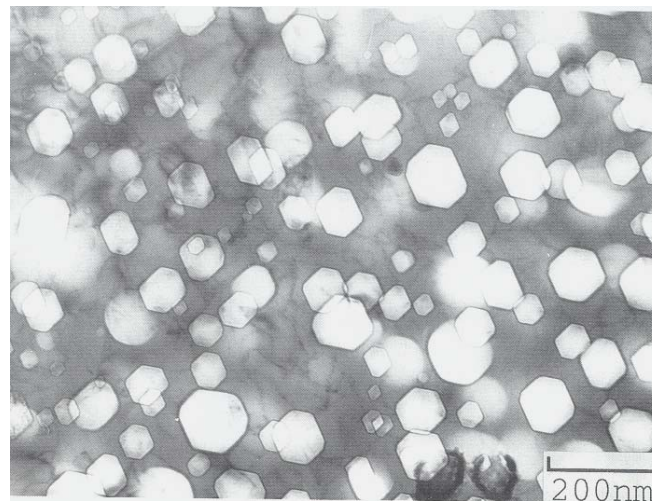
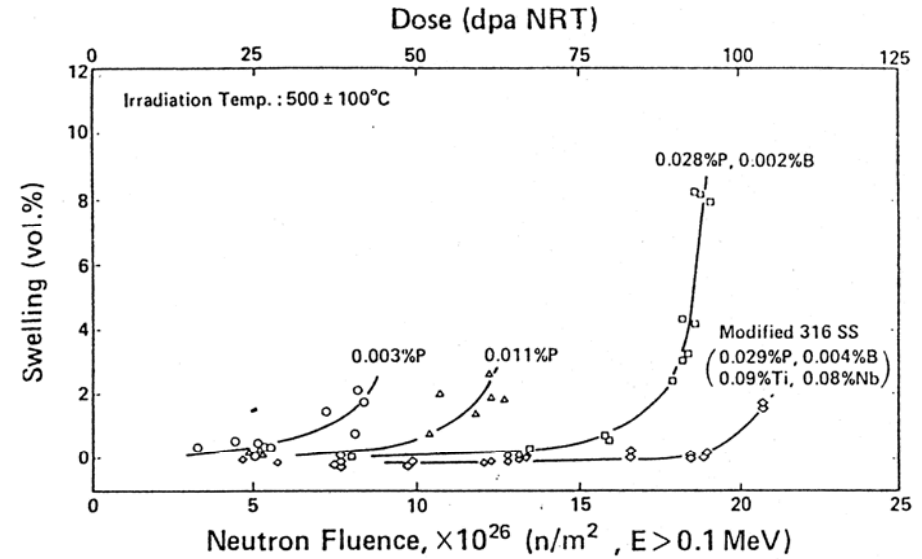
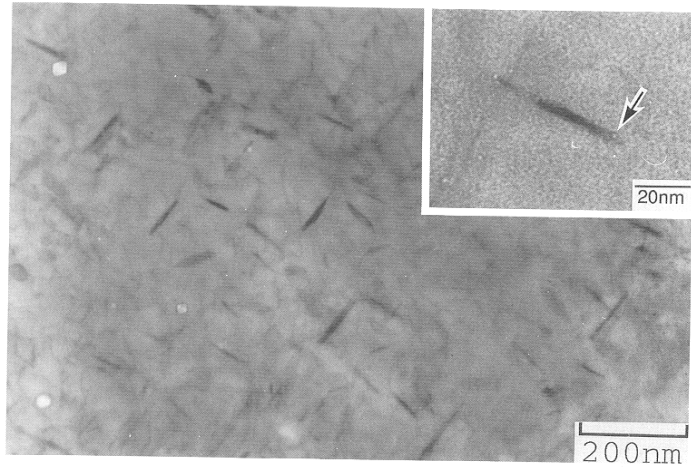
# Void swelling & irradiation creep



**MAXIMUM BURN-UP RAISED TO ~ 80 dpa,  
AFTER OPTIMISING Ti/C RATIO**

**CAN THE BURN-UP LIMIT BE INCREASED FURTHER ????**

# 100-120 dpa with D9i



# Role of alloying elements-every element in steels composition

**B** plays positive role;

increases radiation resistance IF IT IS IN SS of ASS;

Boron reduces diffusion mobility of carbon and nitrogen ;

restricts formation of carbides and intermetallics.

concentration of Ni, Mo, Si, C, Nb in  $\gamma$ -solid solution is same as original.

**Silicon** has positive role;

diffusion mobility on some orders higher in comparison with main components of austenitic steels.

silicon, reduces vacancies super saturation and, accordingly, depress rate of their nucleation.

# Role of alloying elements-every element in steels composition

**Ti – positive role;**

Ti-Vacancy BE 0.3 eV; Cv are absorbed by Ti;

Supersaturation of vacancies reduced and swelling reduced;

Ti successfully suppress swelling only together with silicon and phosphorus or with both of them.

**Phosphorous – positive role in small amounts;**

Diffusion of P-V complex very high with high BE;

phosphorus affects the microstructure via phosphorus-defect interaction at

lower temperatures and via phosphides formation at higher temperatures.

# Precipitates classification

- Precipitates EVOLVE IN AUSTENITE DURING LONG TERM SERVICE.
- TWO MAJOR CLASSIFICATION :
- **MC (mainly TiC, NbC, VC) Fe<sub>2</sub>P or Ni<sub>3</sub>Ti** (in a few cases) SUPPRESS SWELLING (enhance point defect recombination at particles-matrix interface) - DESIRABLE
- **M<sub>6</sub>C** and **G-phases** : DRASTICALLY INCREASE SWELLING - solution decay (remove Ni & Si from austenite) - UNDESIRABLE



# DESIGN PRINCIPLES OF D9i

**ADD ELEMENTS WITH HIGH BINDING ENERGY WITH VACANCIES, LIKE P, Si**

**-----> ADJUSTMENT OF MINOR ELEMENTS ;**

**PLAY WITH COMBINATION OF UNDERSIZED AND OVERSIZED ATOMS  
TO CAPTURE BOTH VACANCIES & INTERSTITIALS;**

**COMBINE THE COHERENT PRECIPITATES WITH + AND – MISFIT VOLUME  
TO ATTRACT OPPOSITE TYPE OF POINT DEFECTS.**

**SOMEHOW REDUCE OVERALL POINT DEFECT CONCENTRATION  
&  
VACANCY SUPERSATURATION, IN PARTICULAR.**

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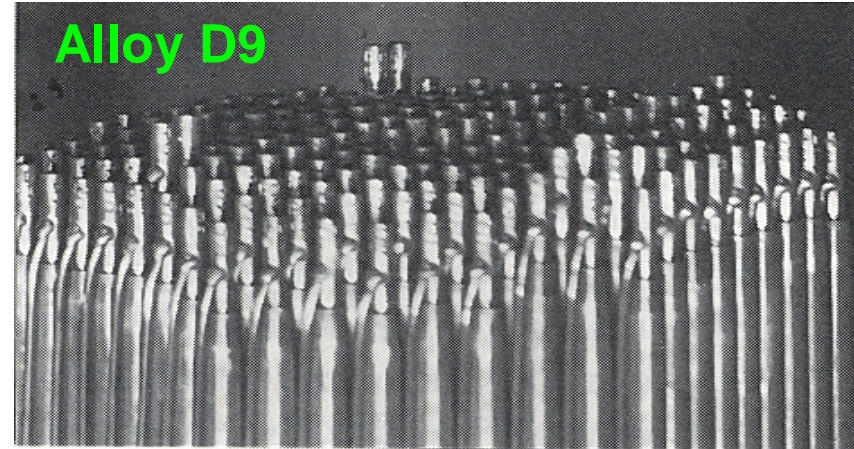
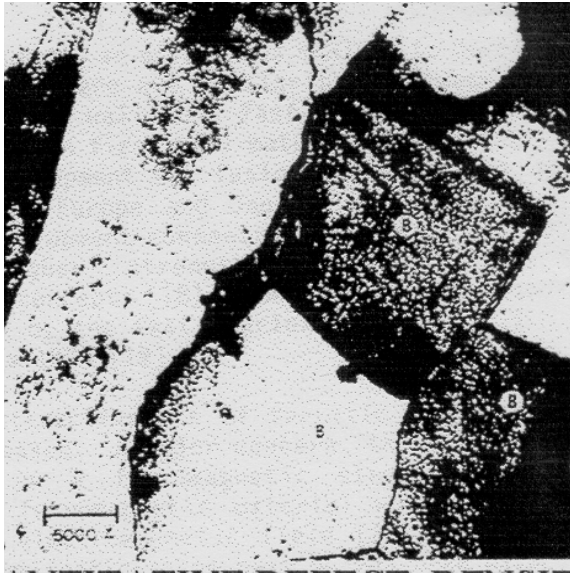
# CAN BURN-UP OF FUEL BE INCREASED FURTHER ?

200 – 250 dpa ???

is there a max. limit for burn-up ?

What limits it?

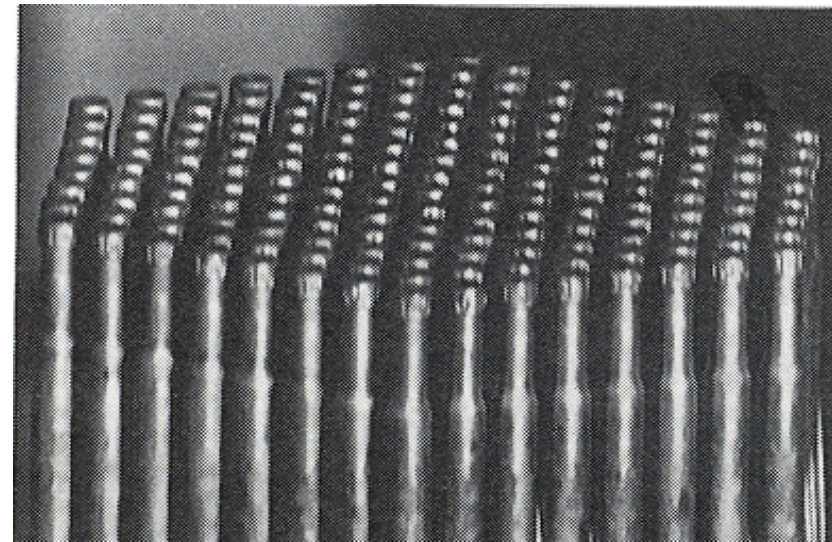
## SERENDIPITY IN DEV. OF SWELLING RESISTANT MATERIALS



- ✓  $E_m^V = 0.5\text{eV}$  ( $< \gamma$ )
- ✓ B.E.-C/-  $0.8\text{eV}$ ; ( $\gg \gamma$ )
- ✓ Strong  $\gamma$ -C Attraction;
- ✓ low Relaxation volume  
reduced bias

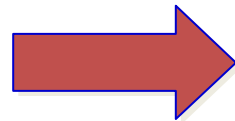
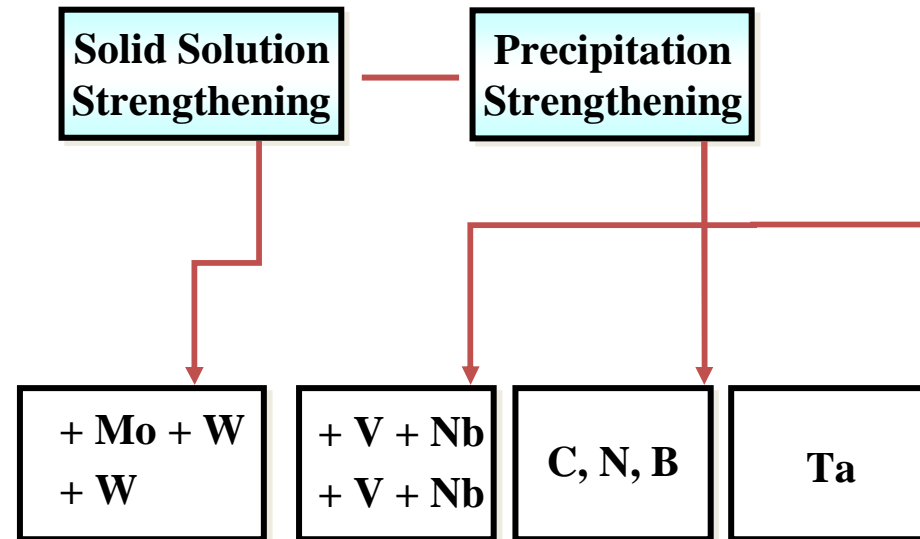
Burn-up upto 180 – 200 dpa

high temperature capability REDUCED



# Strengthening Mechanism of FMS

## Strengthening mechanisms of FMS Steels



- Effect of **B** addition
- Optimization of **C, N**
- Optimization of **Nb**
- Effect of **Ta** addition

# Evaluation of Minor Element Effect

Group	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
	I	II											III	IV	V	VI	VII	VIII
Period	hydrogen 1 H 1.0079			1) Cr 2) Mo, W, Re 3) V, Nb, Ta, Ti 4) C, N			5) B 6) Si, Mn 7) Ni, Cu, Co 8) Al, P, S											helium 2 He 4.0026
2	lithium 3 Li 6.94	beryllium 4 Be 9.01218											boron 5 B 10.81	carbon 6 C 12.011	nitrogen 7 N 14.0067	oxygen 8 O 15.999	fluorine 9 F 18.998403	neon 10 Ne 20.18
3	sodium 11 Na 22.98977	magnesium 12 Mg 24.305											aluminum 13 Al 26.98154	silicon 14 Si 28.086	phosphorus 15 P 30.97376	sulfur 16 S 32.07	chlorine 17 Cl 35.453	argon 18 Ar 39.948
4	potassium 19 K 39.0983	calcium 20 Ca 40.08	scandium 21 Sc 44.95591	titanium 22 Ti 47.867	vanadium 23 V 50.9415	chromium 24 Cr 51.996	manganese 25 Mn 54.93805	iron 26 Fe 55.84	cobalt 27 Co 58.9332	nickel 28 Ni 58.693	copper 29 Cu 63.55	zinc 30 Zn 65.4	gallium 31 Ga 69.723	germanium 32 Ge 72.6	arsenic 33 As 74.9216	selenium 34 Se 79	bromine 35 Br 79.904	krypton 36 Kr 83.8
5	rubidium 37 Rb 85.468	strontium 38 Sr 87.62	yttrium 39 Y 88.9058	zirconium 40 Zr 91.22	niobium 41 Nb 92.9064	molybdenum 42 Mo 95.94	technetium 43 Tc [97.9072]	ruthenium 44 Ru 101.1	rhodium 45 Rh 102.9055	palladium 46 Pd 90	silver 47 Ag 107.868	cadmium 48 Cd 112.41	indium 49 In 114.82	tin 50 Sn 118.71	antimony 51 Sb 121.76	tellurium 52 Te 127.6	iodine 53 I 126.9045	xenon 54 Xe 131.3
6	caesium 55 Cs 132.9054	barium 56 Ba 137.33	57-71 *	hafnium 72 Hf 178.5	tantalum 73 Ta 180.9479	tungsten 74 W 183.84	rhenium 75 Re 186.207	osmium 76 Os 190.2	iridium 77 Ir 192.22	platinum 78 Pt 195.08	gold 79 Au 196.9666	mercury 80 Hg 200.6	thallium 81 Tl 204.383	lead 82 Pb 207.2	bismuth 83 Bi 208.9804	polonium 84 Po [208.9824]	astatine 85 At [209.9871]	radon 86 Rn [222.0176]
7	francium 87 Fr [223.0197]	radium 88 Ra [226.0254]	89-103 **	rutherfordium 104 Rf [263.1125]	dubnium 105 Db [262.1144]	seaborgium 106 Sg [266.1219]	bohrium 107 Bh [264.1247]	hassium 108 Hs [269.1341]	meitnerium 109 Mt [268.1388]	darmstadtium 110 Ds [272.1463]	roentgenium 111 Rg [272.1535]	ununbium 112 Uub [277]	ununtrium 113 Uut [284]	ununquadium 114 Uuq [289]	ununpentium 115 Uup [288]	ununhexium 116 Uuh [292]	ununseptium 117 Uus [291]**	ununoctium 118 Uuo [294]**

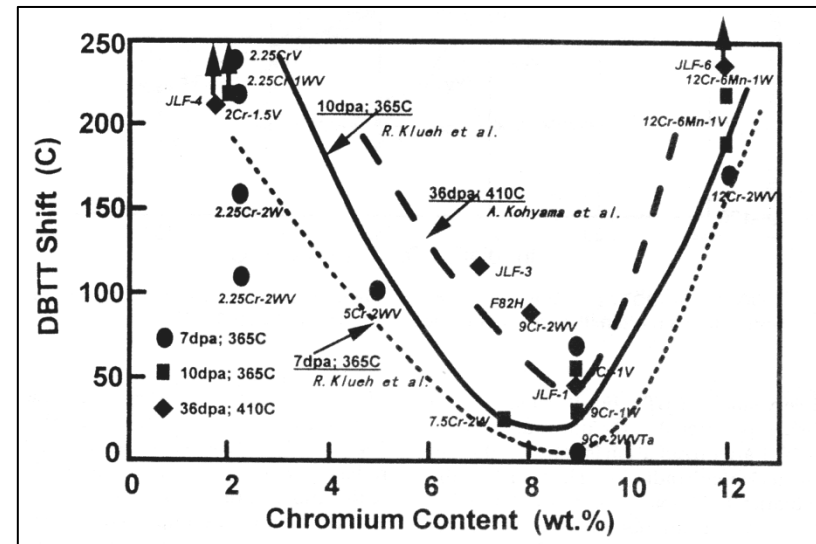
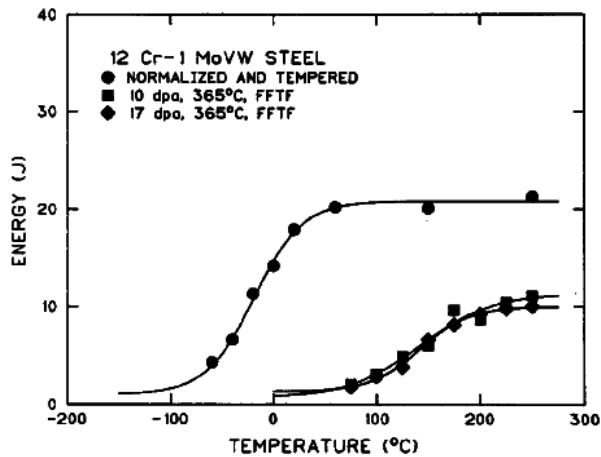
- 1) Cr: Precipitation hardening
- 2) Mo, W, Re: Solid solution hardening
- 3) V, Nb, Ta, Ti: Precipitation hardening
- 4) C, N: Precipitation hardening
- 5) B: Stabilization of precipitates
- 6) Si, Mn: Stabilization of precipitates
- 7) Ni, Cu, Co: Stabilization of microstructure
- 8) Al, P, S: Stabilization of microstructure

Evaluation  
of base data

- Crystal structure/atomic radius
- valence/Electronegativity/MP
- nucleus embrittlement
- Formation of  $\delta$ -ferrite
- Phase transformation temp. ( $M_s$ ,  $A_1$ )

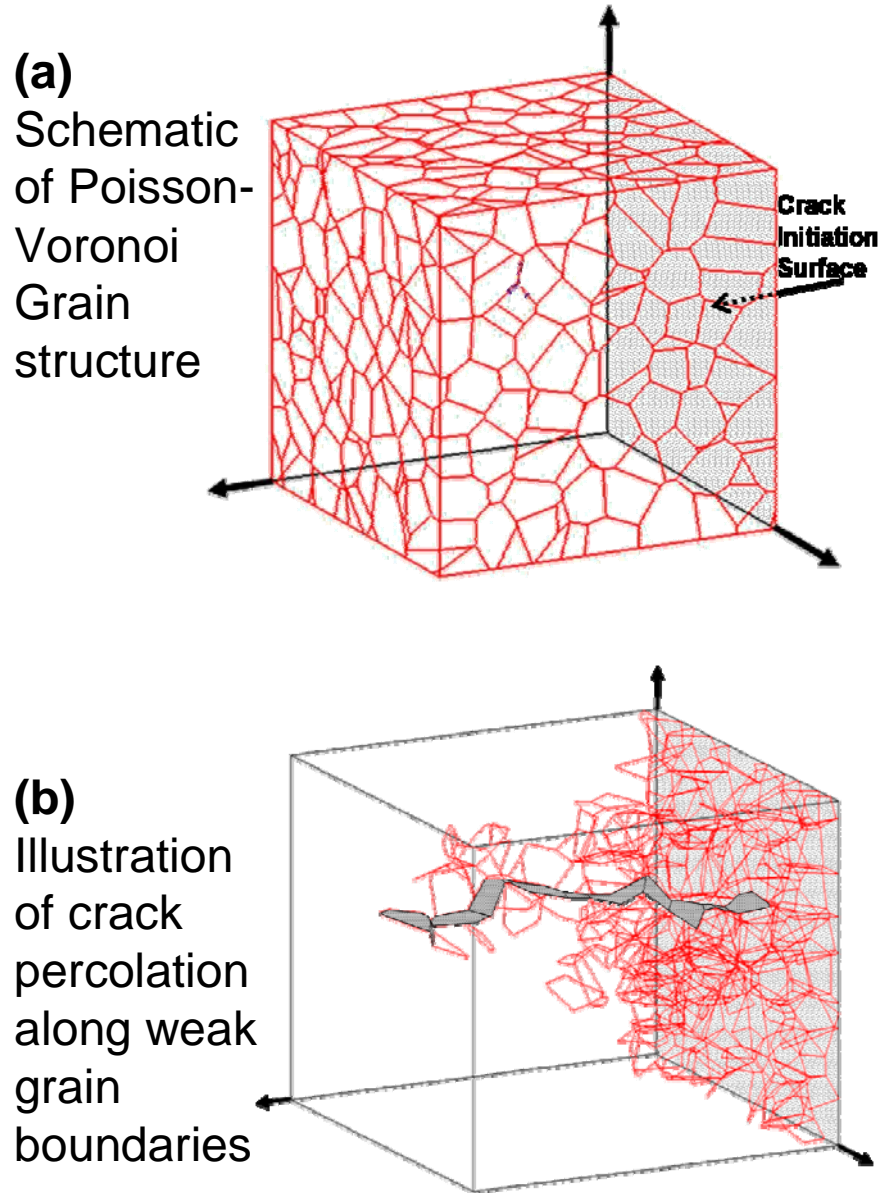
# MAIN PROBLEMS

- HIGH TEMPERATURE LIMIT
- EMBRITTLEMENT

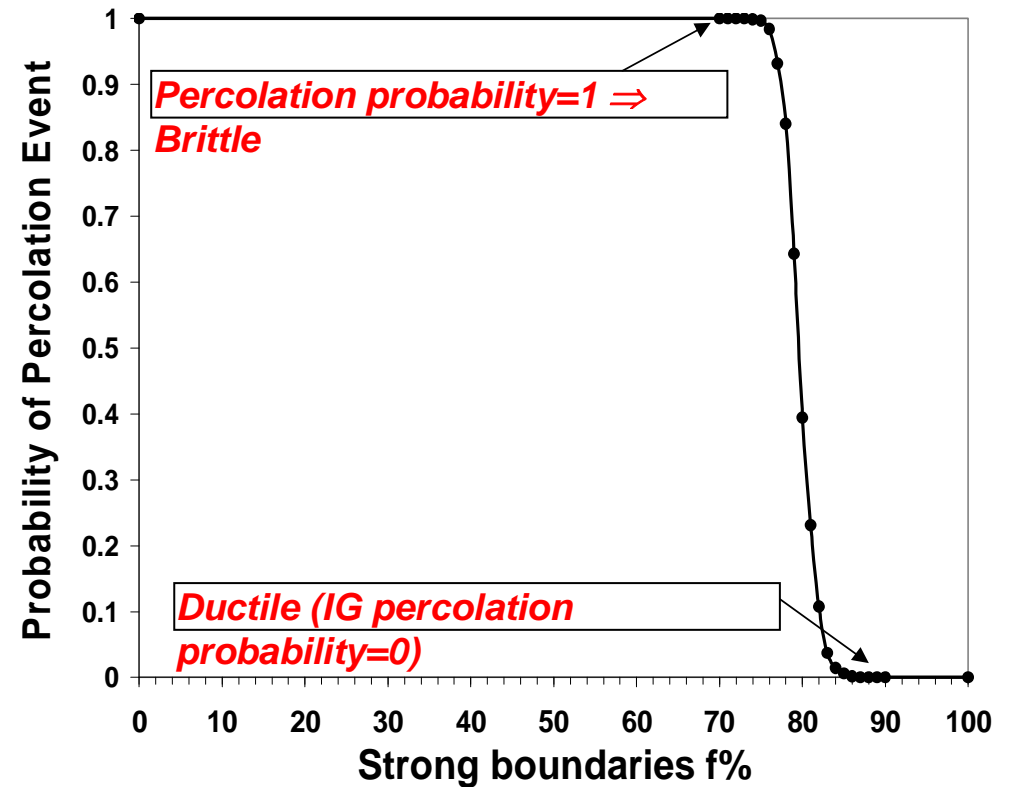


Charpy impact curves of HT9 (12Cr-1MoVW) in the unirradiated condition and after irradiation to 10 and 17 dpa at 365 °C in FFTF.

**Figure 1: Intergranular crack connectivity in a simulated Grain structure**

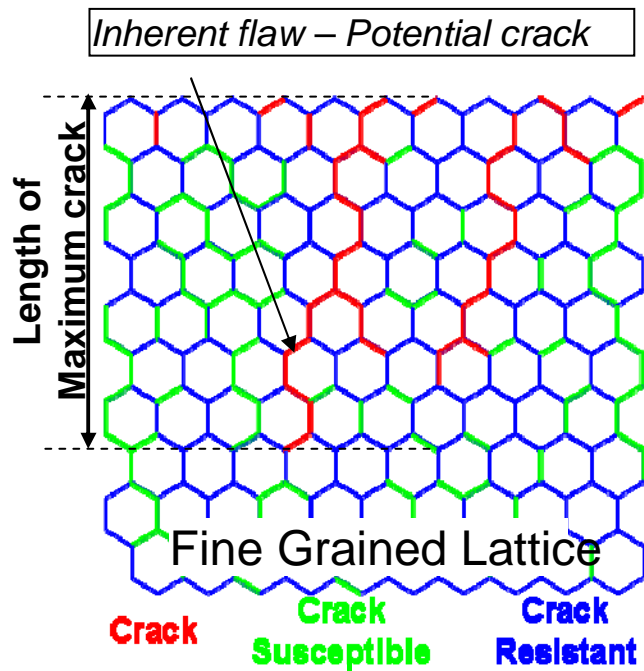


**(c)** Percolation probability vs % of crack resistant/strong grain boundaries





# Illustration of grain size effect on length of potential crack



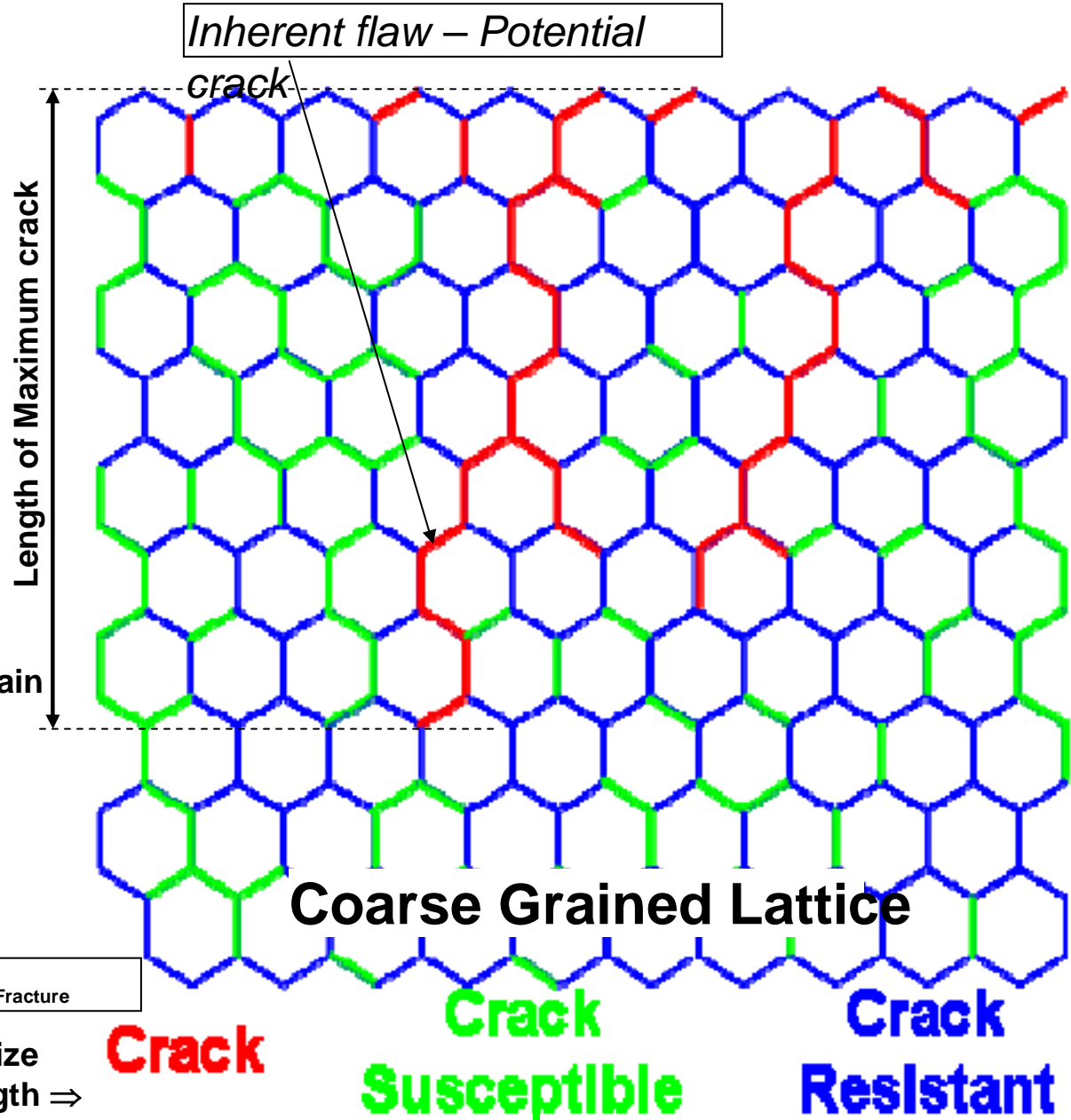
For the give percentage of crack resistant boundaries, crack size in grain units is same, but in length units, is scaled by the factor of grain size  $\Rightarrow$  Length of potential inherent flaw is more in coarse grained structure

Condition for Brittle failure:

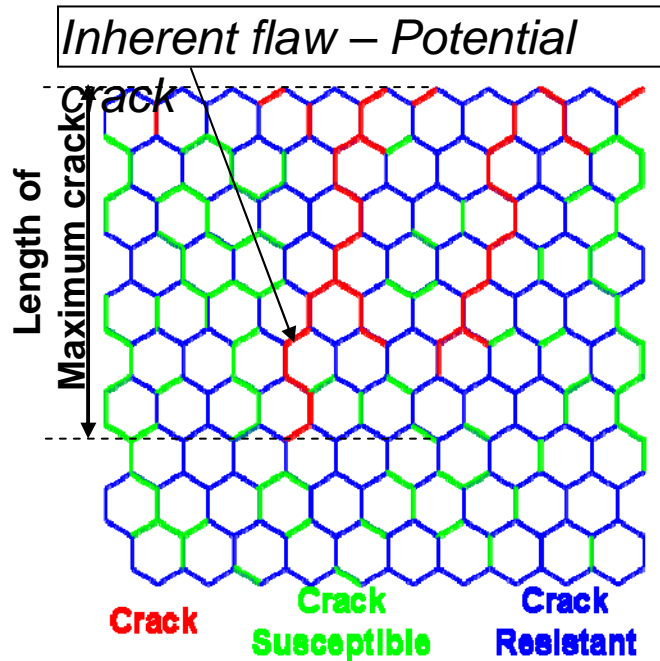
$$\text{Crack length}_{\text{Potential}} > \text{Critical crack length}_{\text{Fracture}}$$

Mechanics

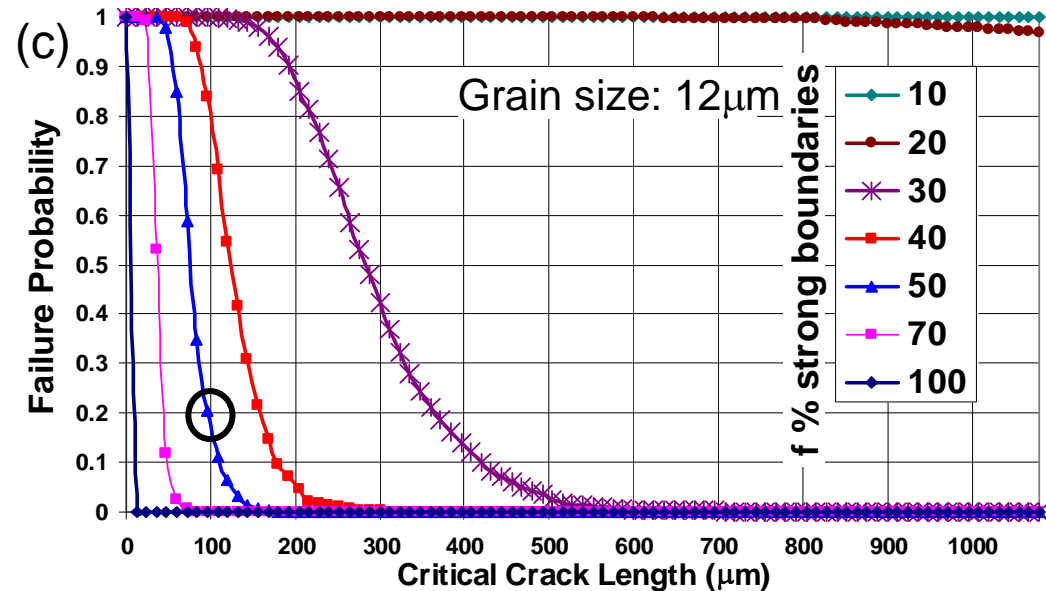
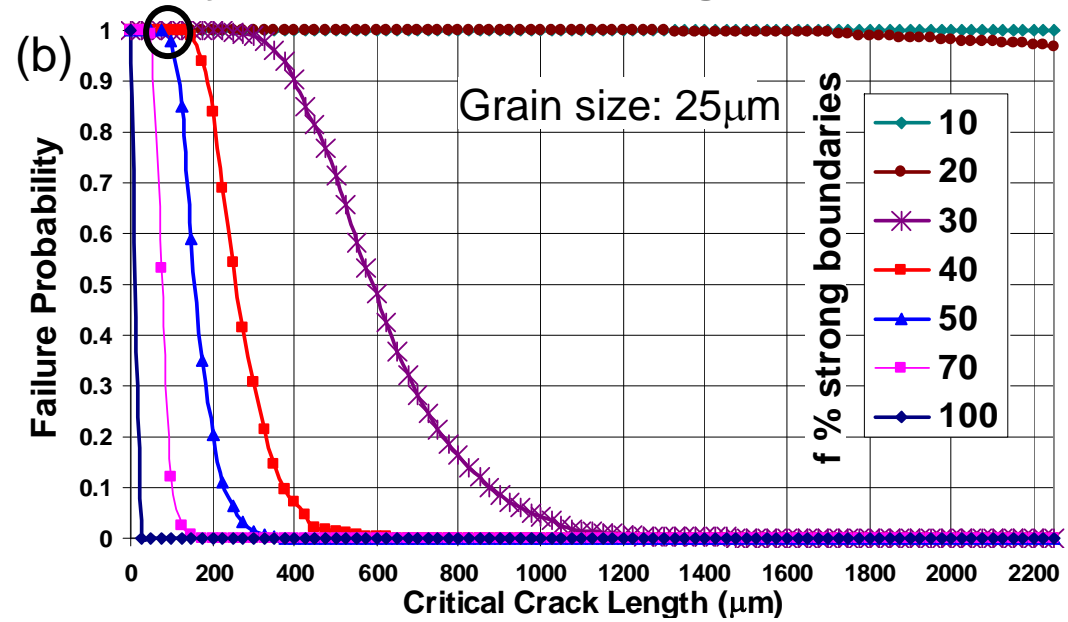
Fore all values of  $f$ , smaller grain size gives a smaller potential crack length  $\Rightarrow$  small grain size is beneficial



**Figure 2: Intergranular crack connectivity in a simulated Hexagonal Grain lattice**



(a) Methodology for crack connectivity along susceptible (weak) grain boundaries



\* For critical crack length of 100 $\mu\text{m}$  and 50% strong boundaries, the failure probability for coarse grained (25 $\mu\text{m}$ ) structure is  $\sim 0.99$  compared to 0.2 for a fine grained (12 $\mu\text{m}$ ) structure

250 dpa with temp. capability up to  
600 & more ?????

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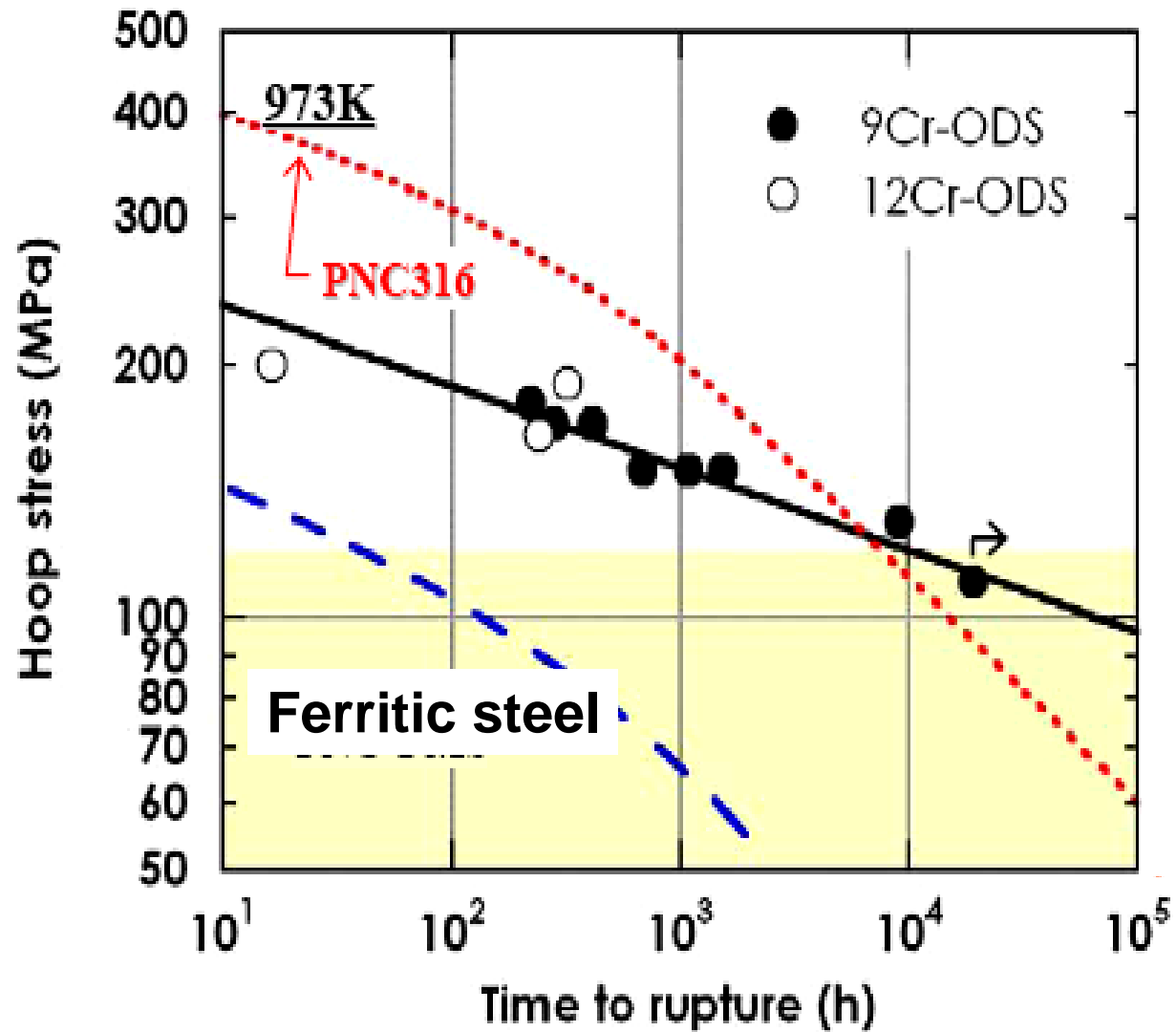
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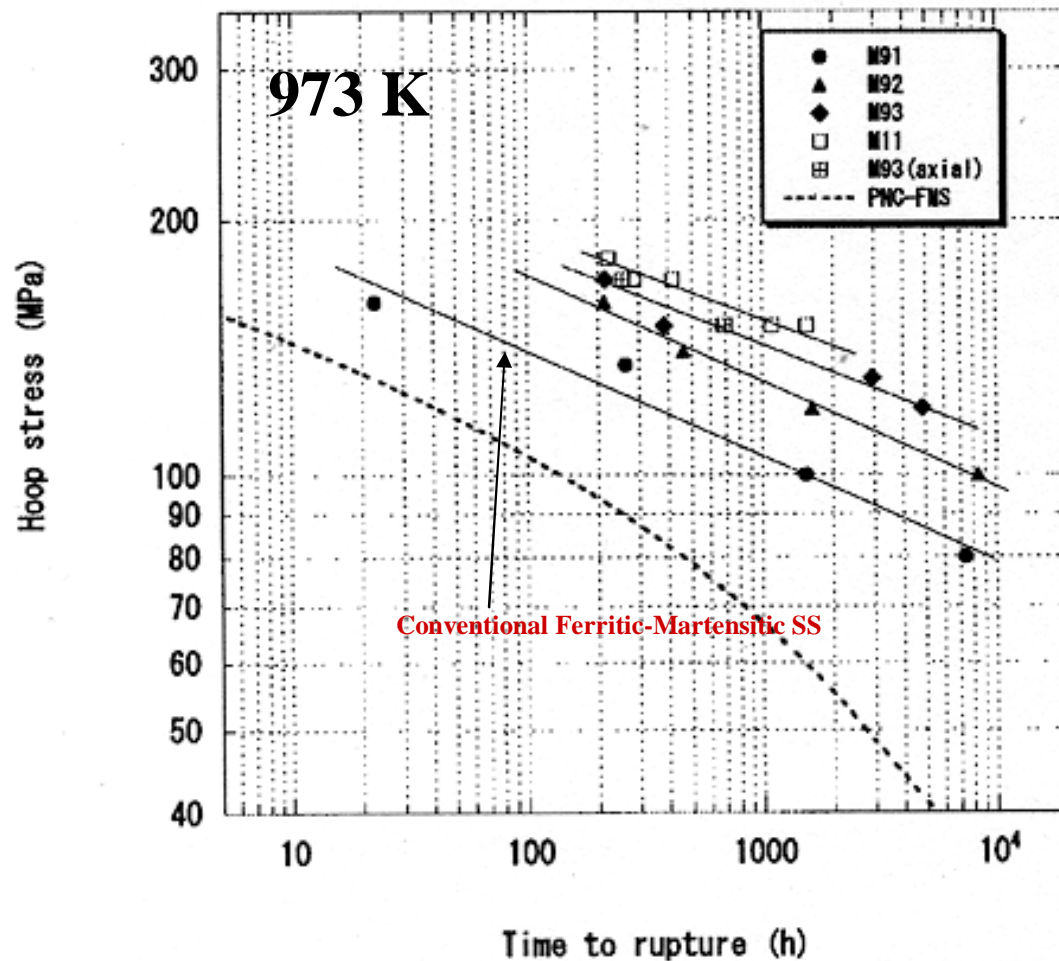
**OXIDE DISPERSION STRENGTHENED STEELS.**

# 9Cr-ODS steel

9Cr-0.24 %  $Y_2O_3$



## 9Cr ODS Martensitic Steel Claddings

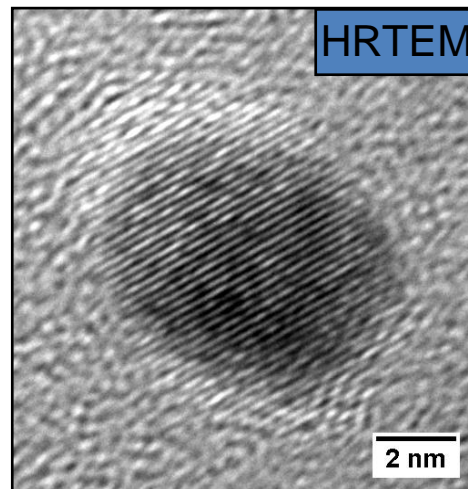
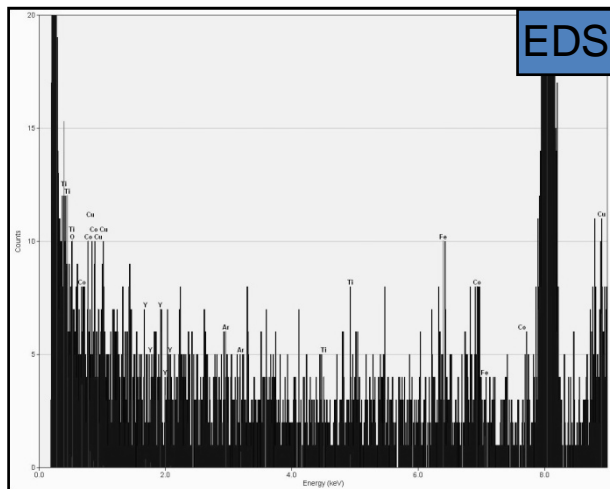
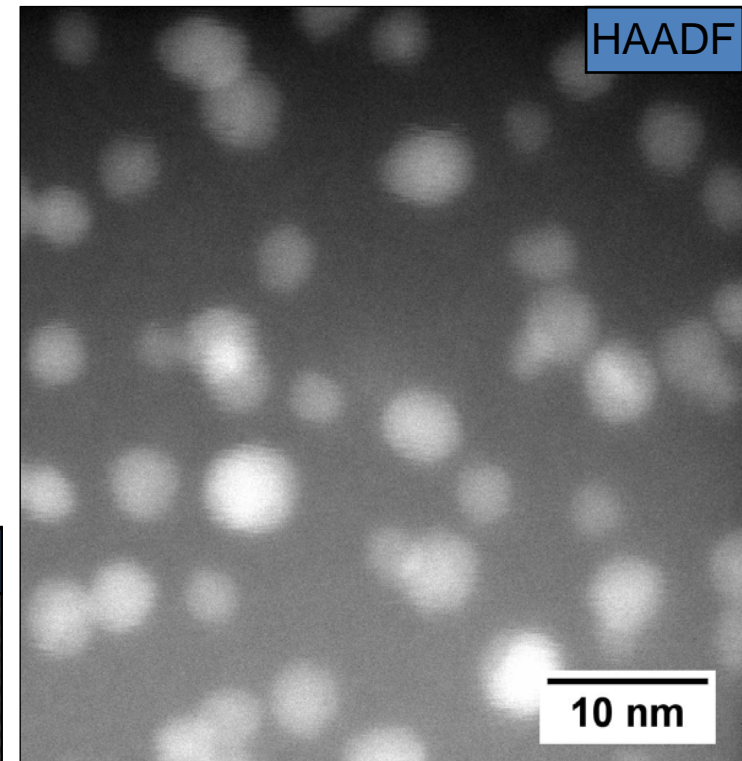
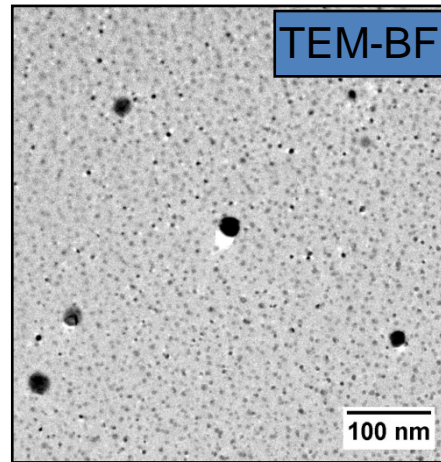
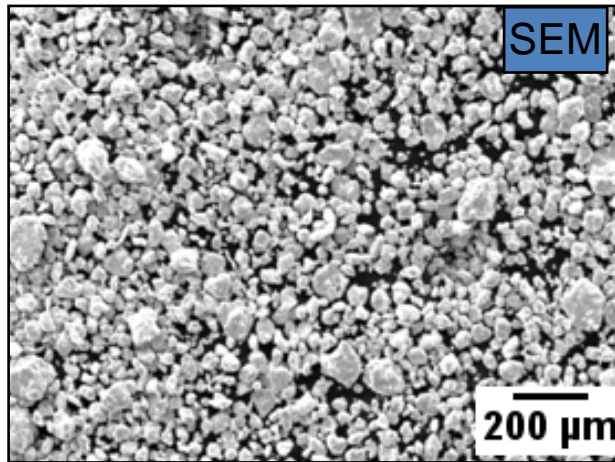


- Better creep strength than ASS
- DBTT is close to room temperature
- No carbon leaching in sodium environment
- Ferritic structure provides better resistance to neutron damage and has better void swelling resistance. (Compared to austenitics)

Creep strength increases with increasing Ti content from 0.1(M91) to 0.2 (M93) wt% and  $Y_2O_3$  from 0.30 (M92) to 0.37 (M11) wt%

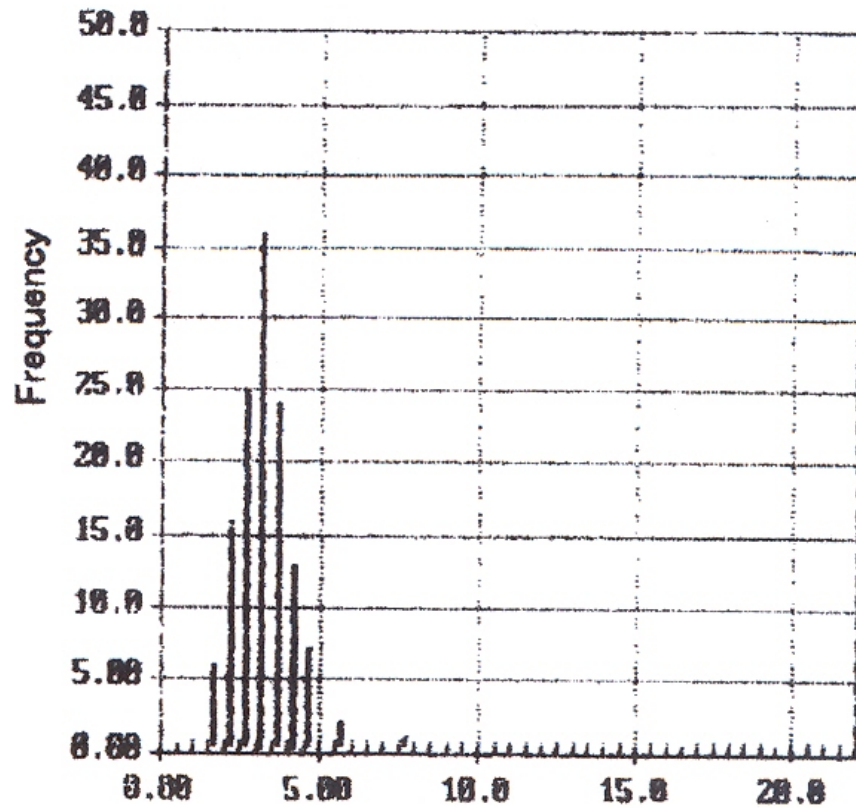
S. Ukai, S. Mizuta, M. Fujiwara, T. Okuda and T. Kobayashi, J. Nuclear Science and Technology, 2002, Vol. 39, No. 7, pp. 778-788.

# ODS Steels for future FBR Applications

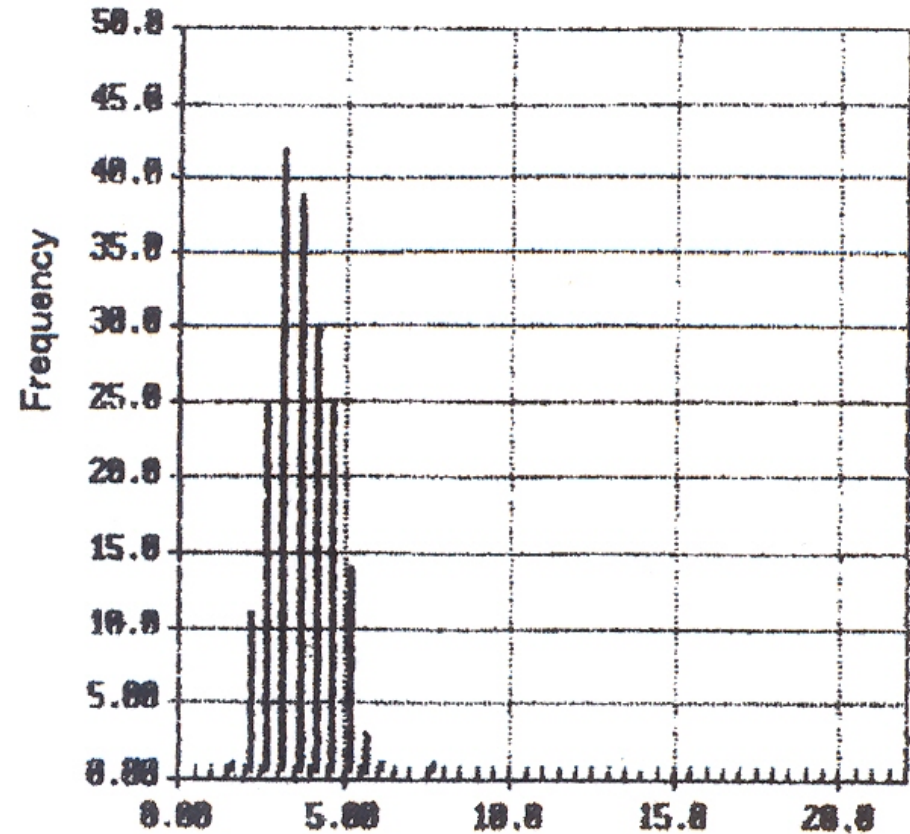


Qualitative Z-contrast for  
detection of microchemistry  
variations

# Effect of Ti and $Y_2O_3$ on dispersive particles distribution



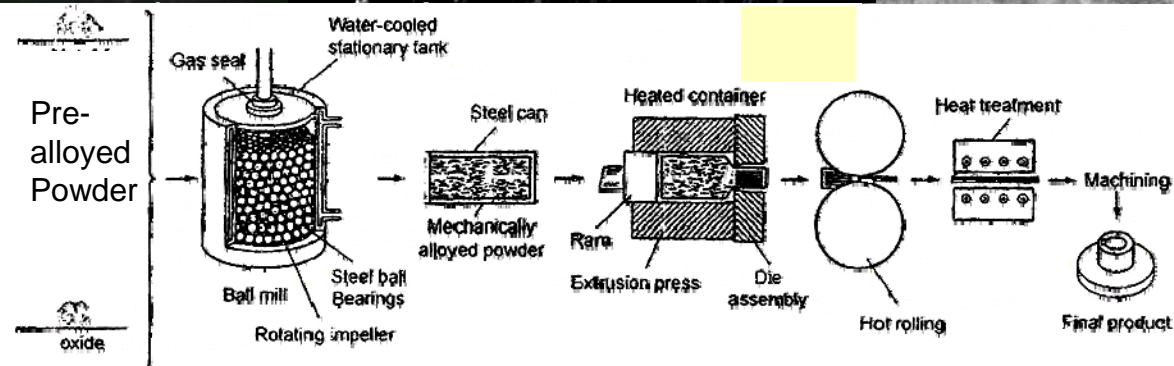
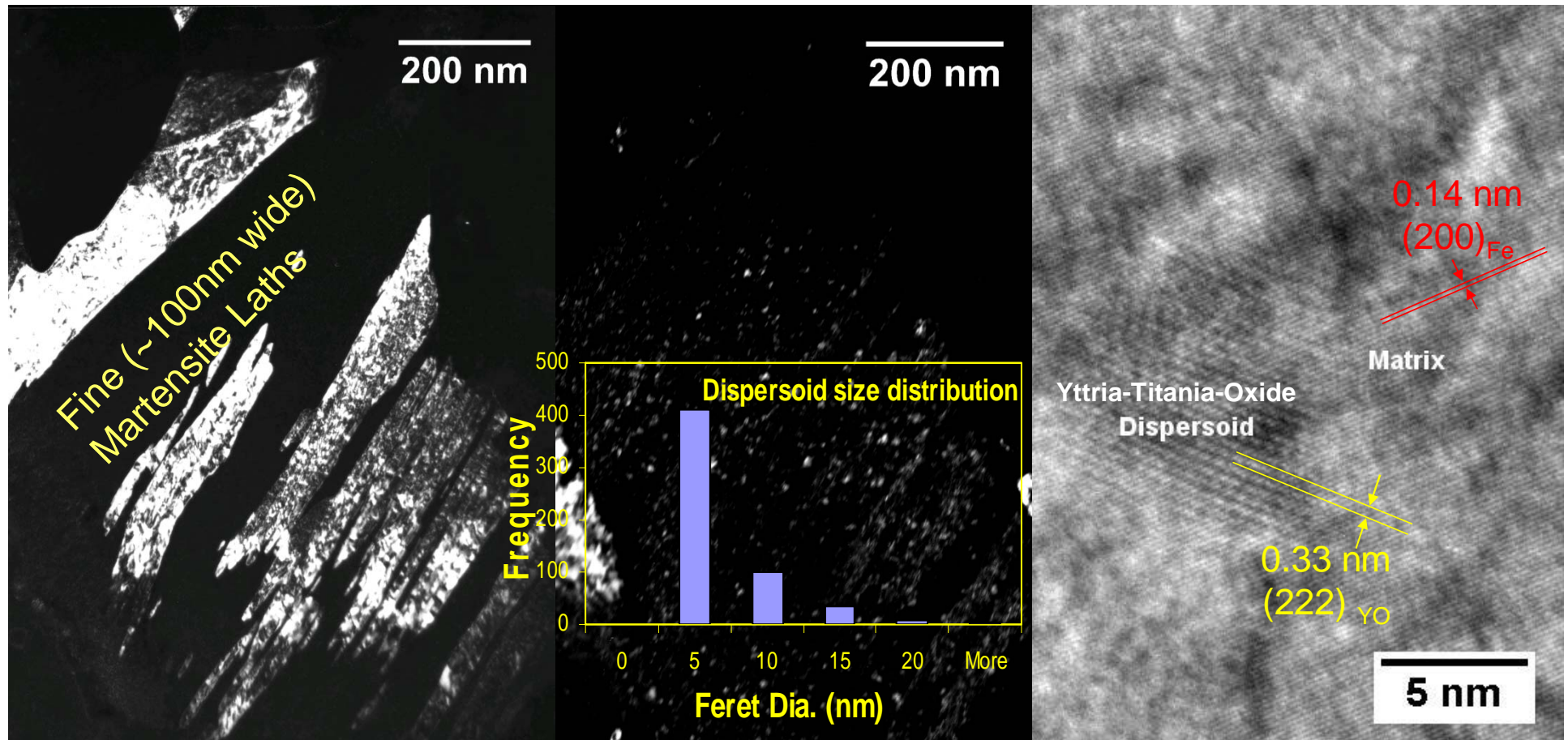
Ti – 0.20,  $Y_2O_3$  – 0.30 (wt %)



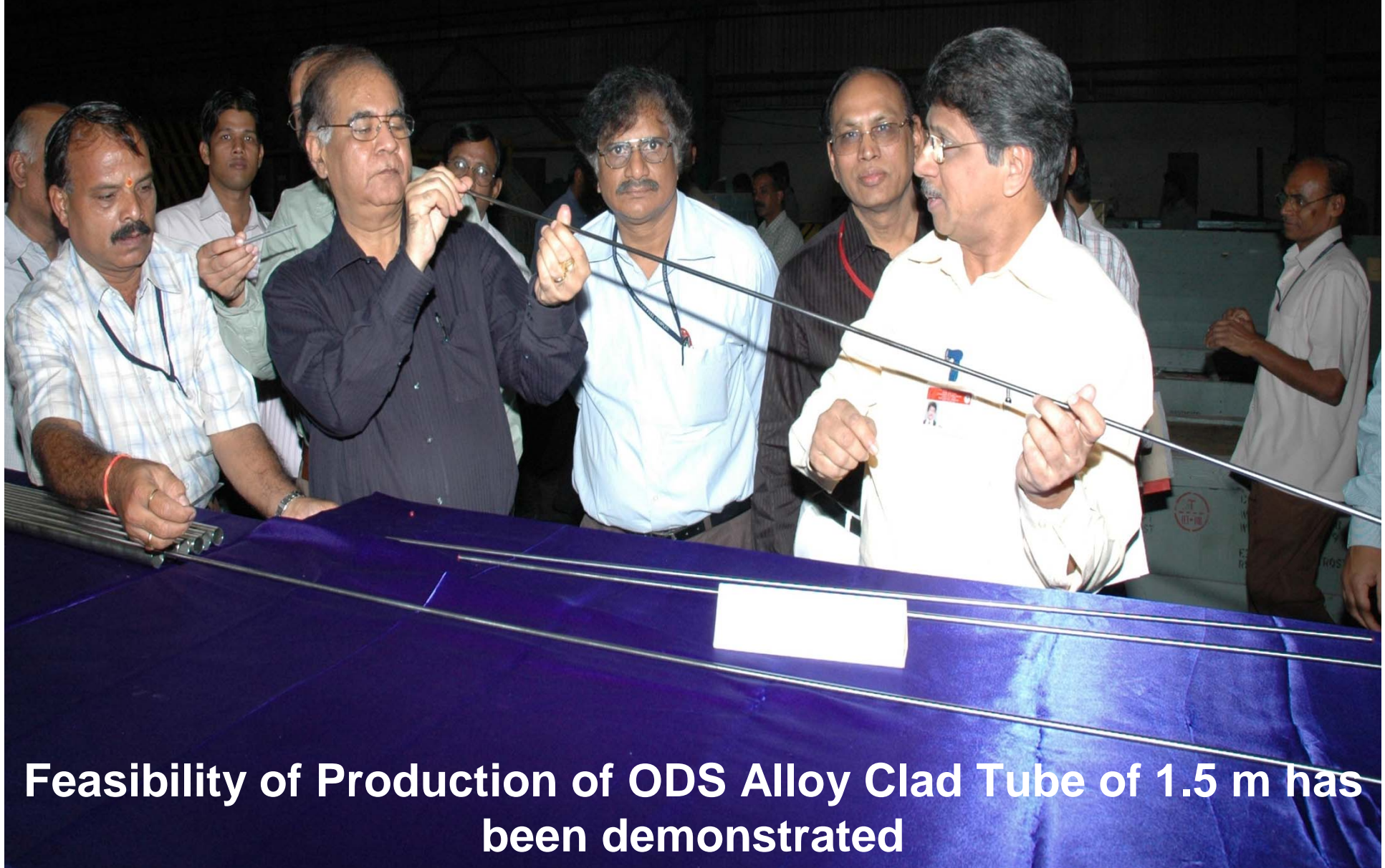
Ti – 0.20,  $Y_2O_3$  – 0.35 (wt %)



# Indian ODS: Fe-9Cr-2W-0.2Ti-0.35Y<sub>2</sub>O<sub>3</sub>-0.16C (MA / Extruded at 1050°C)

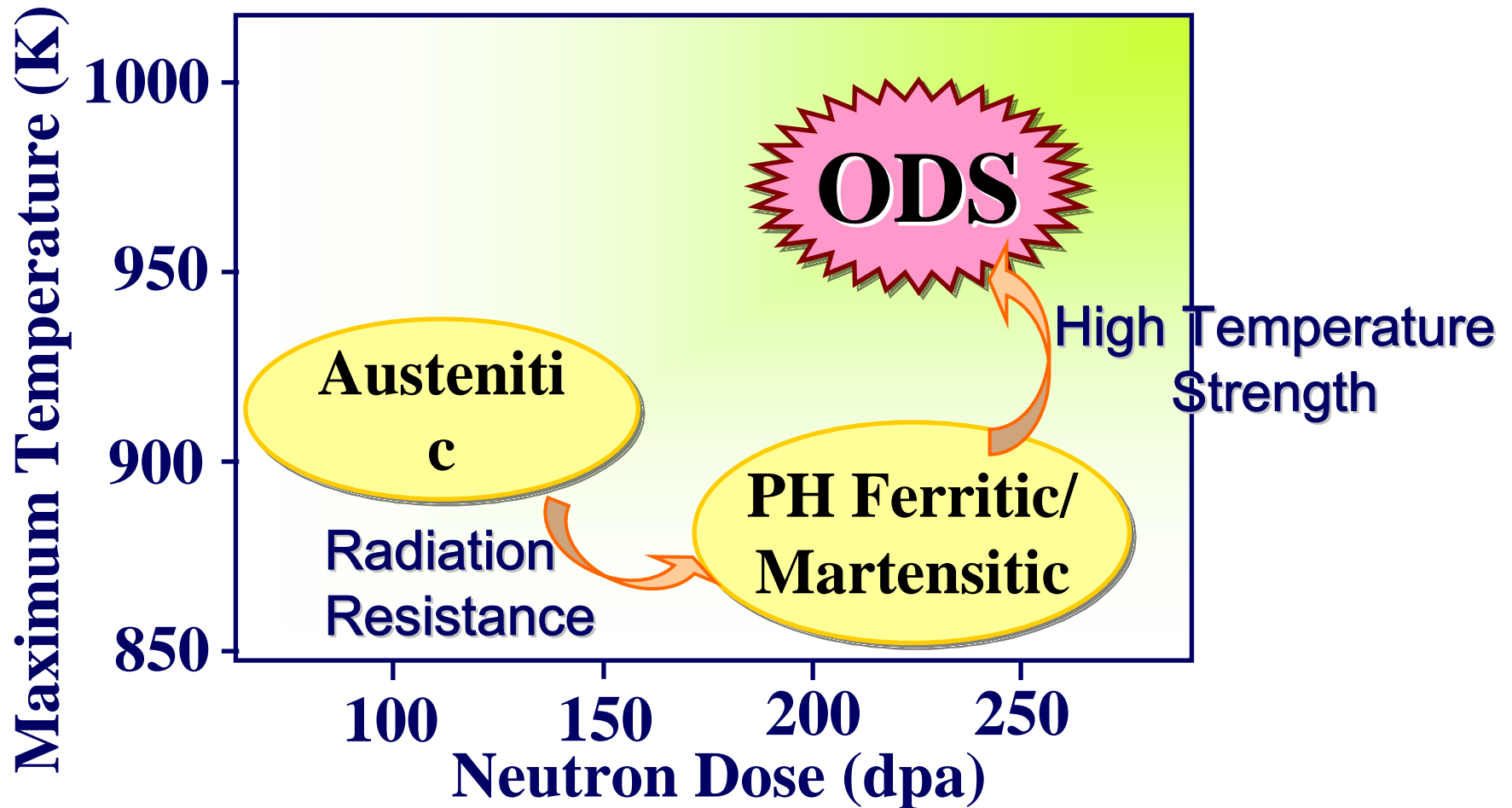


# VISUAL INSPECTION OF ODS CLAD TUBES AT NFC, HYDERABAD



**Feasibility of Production of ODS Alloy Clad Tube of 1.5 m has  
been demonstrated**

## $\gamma$ Alloys to $\alpha$ alloys to ODS

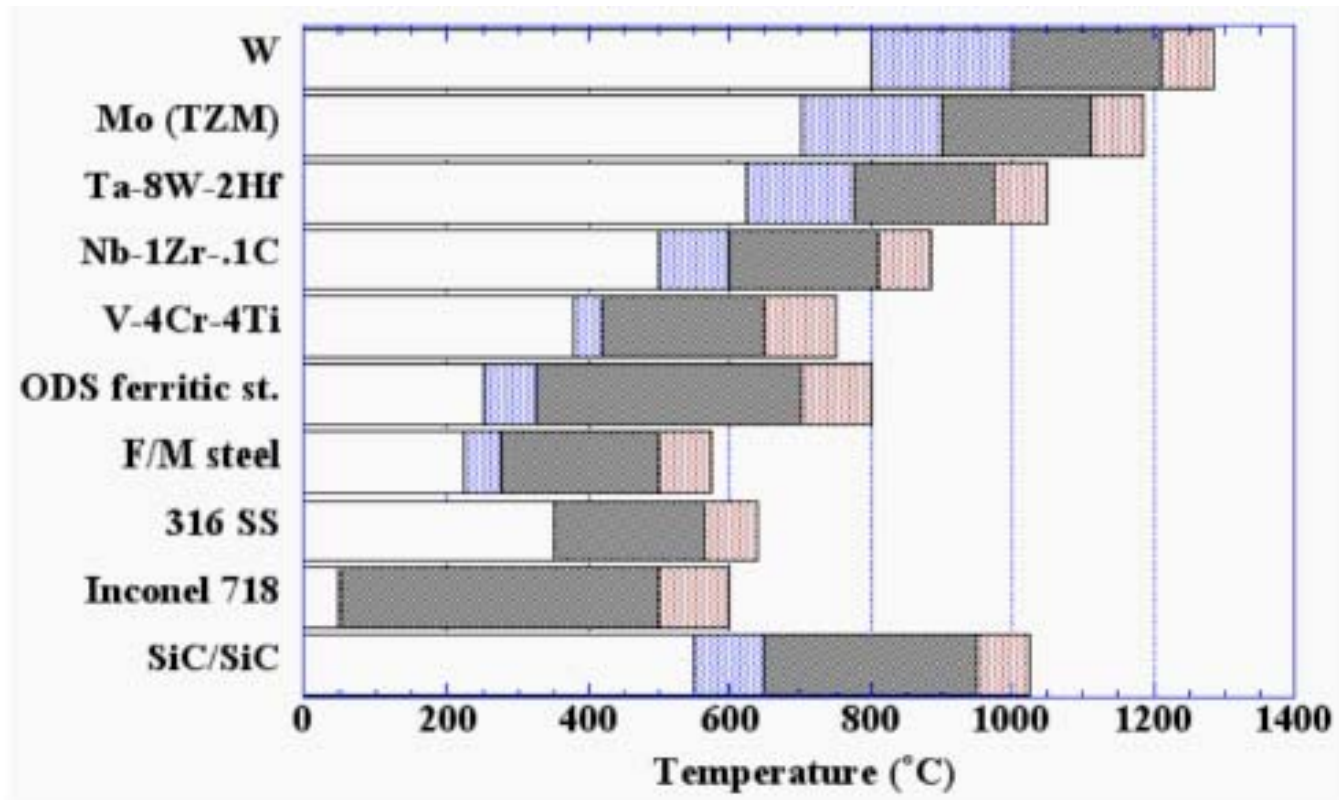


**Precipitation hardening will be lost in ferritic steels over 923 K.**



**Oxide dispersion strengthening will be effective even over 973 K.**

# HIGH TEMPERATURE MATERIALS



**THANK YOU VERY MUCH FOR YOUR PATIENT LISTENING;**

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