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Microstructural effect of solute addition to Fe-15Cr-20Ni steels irradiated in Joyo

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Background

The second-generation advanced austenitic steel for near-term application for FBR



PNC1520 by JNC as Monju(FBR) core materials

Effects of solute atom addition

Cr, Ni	•••	well-known
C, Si, Ti, P, Nb, Mo, etc.	•••	not well-known

C, Si, P, Ti, Nb, Mo

Radiation -induced, enhanced, modified –Precipitation
affect the formation process of cavity → swelling behavior

Objectives

To classify the effect of minor solute addition for the microstructural evolution in Fe-15Cr-20Ni model alloys

Experimental procedure

<Specimens>

Fe-15Cr-20Ni austenitic stainless steel

1. **Fe-15Cr-20Ni**
2. -0.06C
3. -0.025P
4. -0.004B
5. -0.25Ti-0.06C
6. -0.1Nb-0.06C
7. -0.5Si
8. -2.5Mo
9. -0.24Ti-0.1Nb-0.06C

10. **PNC1520**

(Fe-15Cr-20Ni-1.5Mn-2.5Mo
-0.03P-0.8Si-0.25Ti-0.1Nb-0.06C)

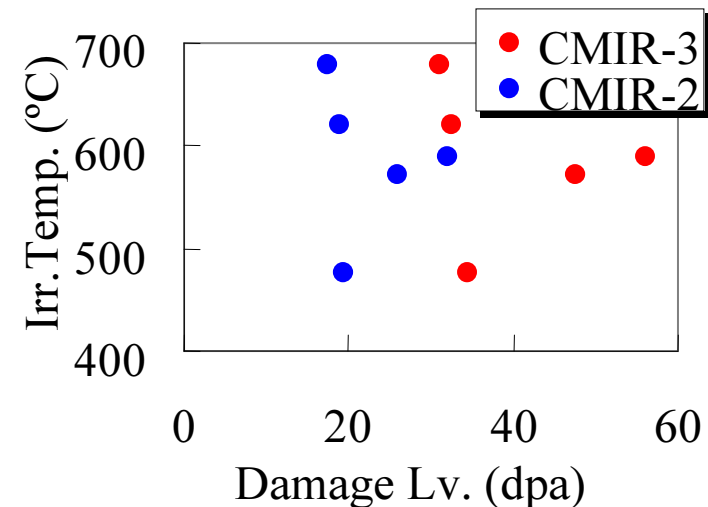
Heat treatment : SA

<Irradiation condition>

Reactor : JOYO

Irr. Temp. : 476°C - 679°C

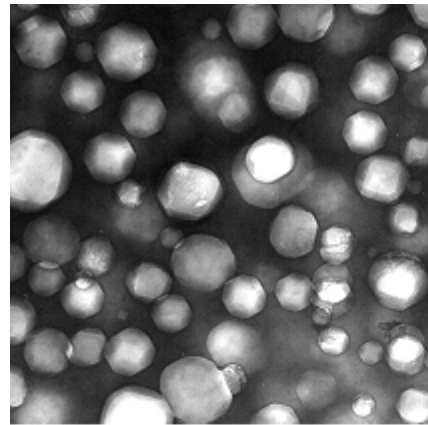
Damage Lv. : 20 – 56 dpa



<PIE>

TEM observation (EDS-2Dmapping + Electron diffraction analysis)

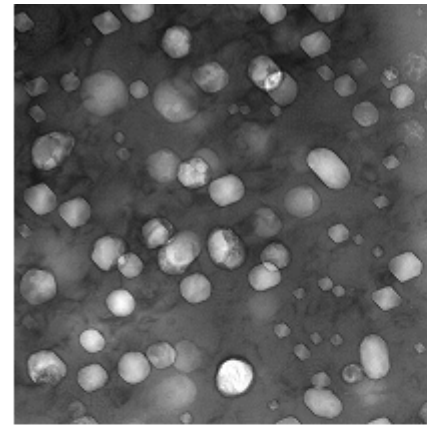
476° C 35dpa - Cavity image



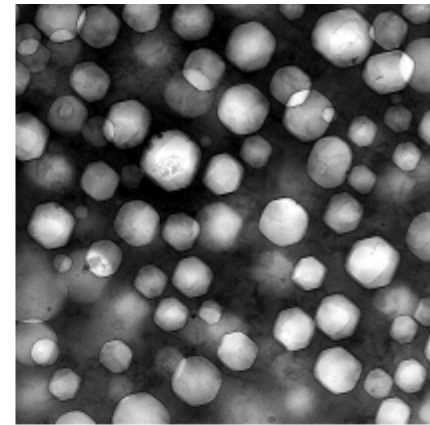
Fe-15Cr-20Ni



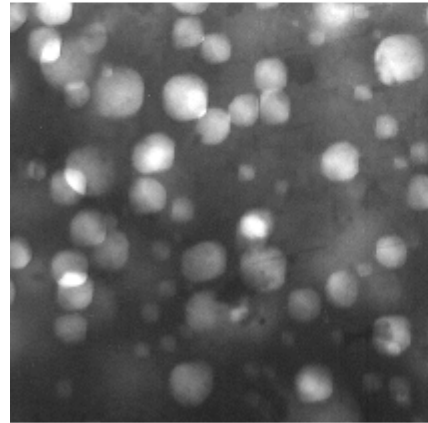
0.06C



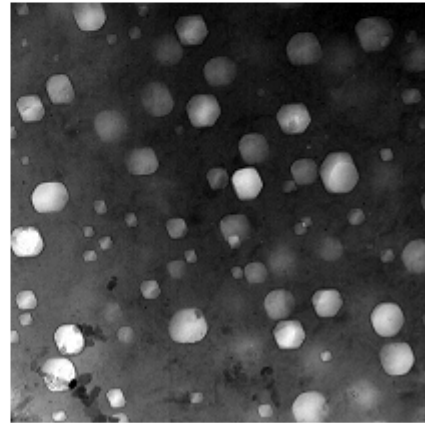
0.025P



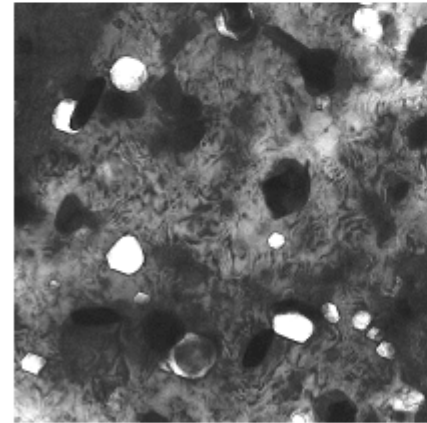
0.004B



0.25Ti



0.1Nb



PNC1520

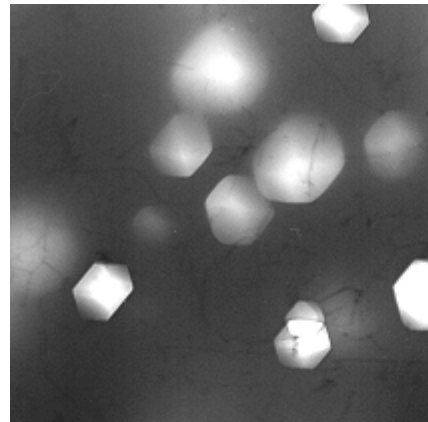
0.5μm

476°C

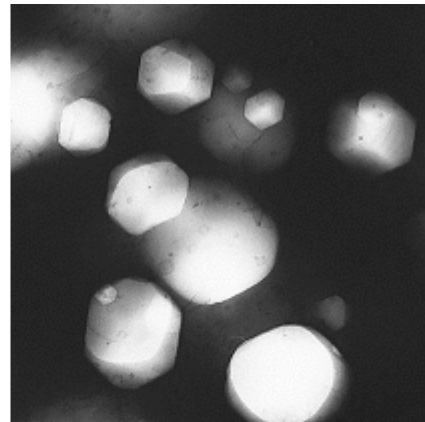
6.9×10^{26} n/m²

34.5 dpa

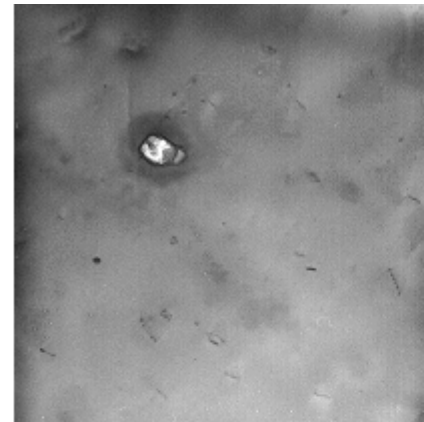
621° C 33dpa - Cavity image



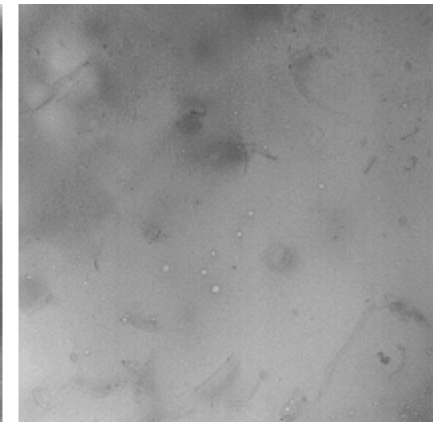
Fe-15Cr-20Ni



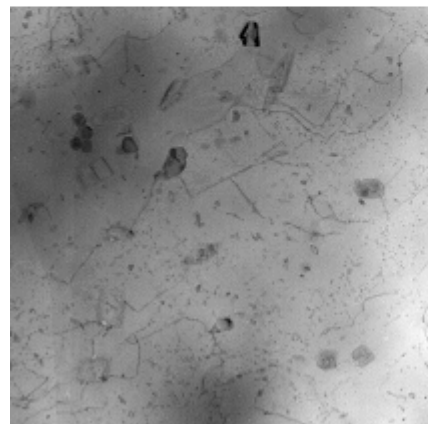
0.06C



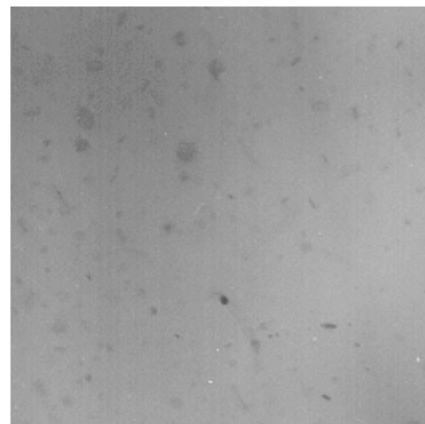
0.025P



0.004B



0.25Ti



0.1Nb

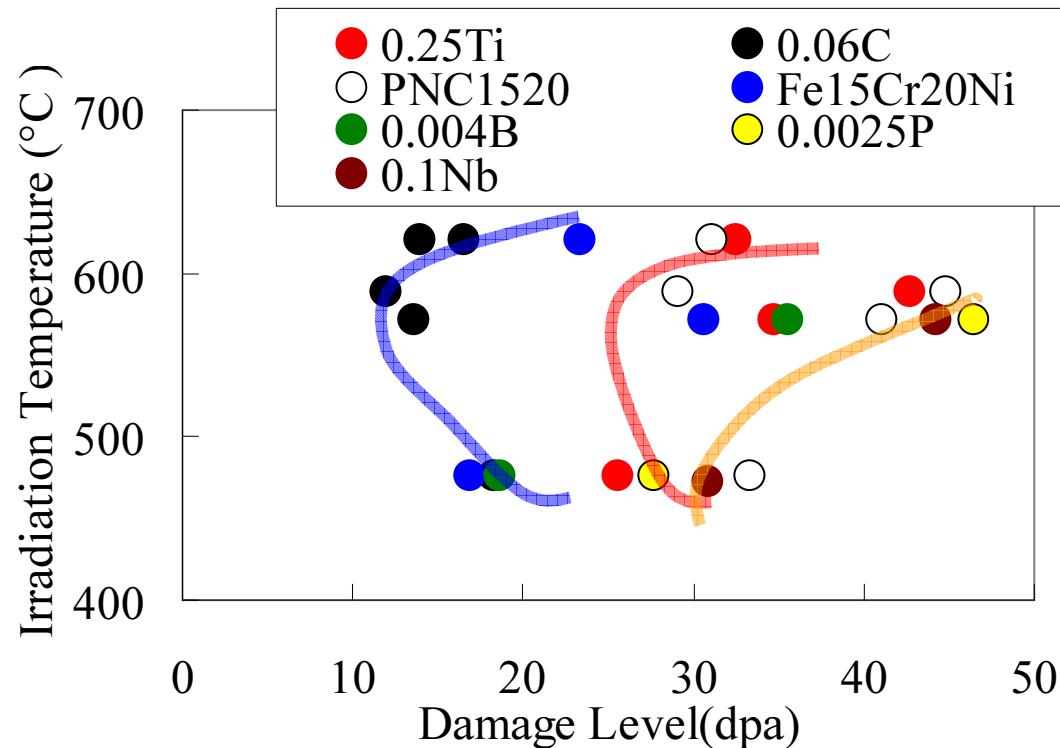


PNC1520

0.5μm

621°C
 6.5×10^{26} n/m²
32.5 dpa

Incubation dose – damage level

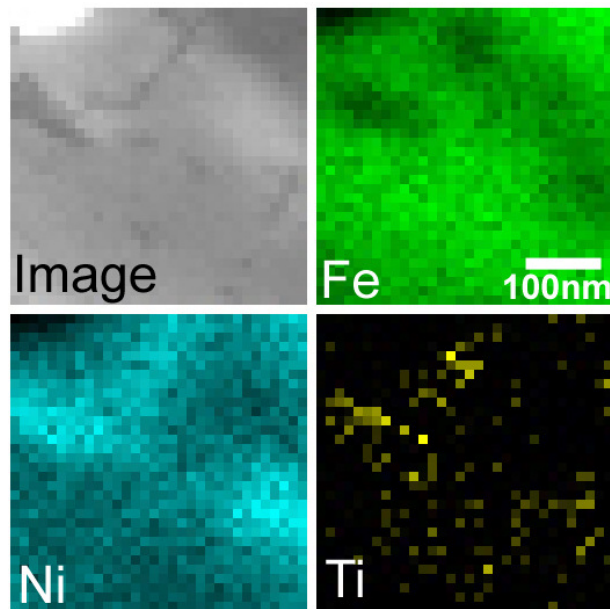


Incubation damage level
Estimation extrapolating
with a 1%/dpa of swelling
rate from swelling data

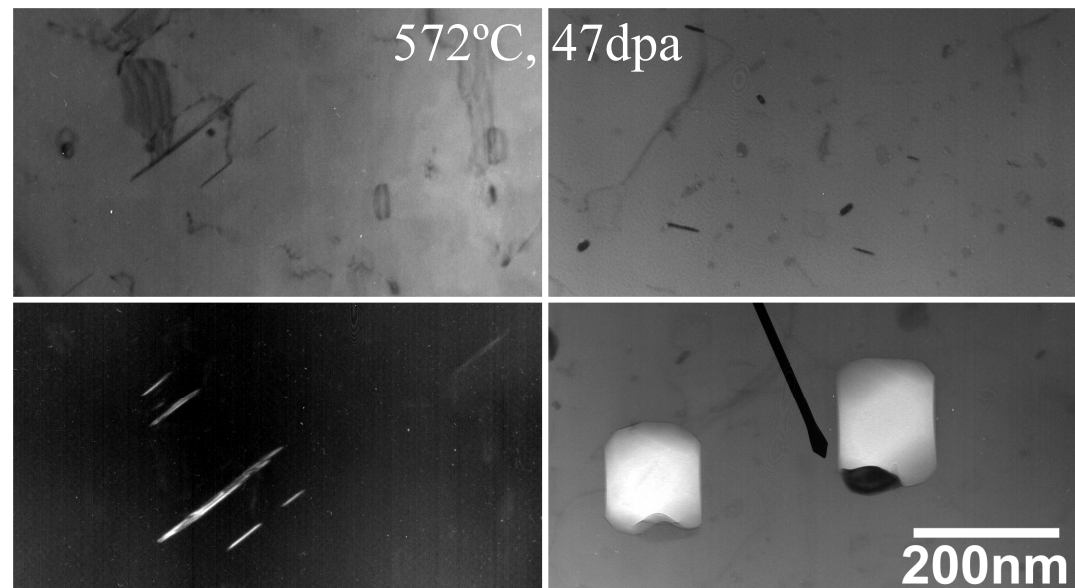
Three types of swelling behavior

- Fe-15Cr-20Ni, 0.06C : incubation damage level $\approx <20$ dpa
- PNC1520, 0.25Ti : incubation damage level ≈ 30 dpa
- 0.04B, 0.0025P, 0.1Nb : strong suppression of swelling at higher T

Typical feature of precipitates in Fe-Cr-Ti modified alloys (Ti & Nb)



0.25Ti-0.06C



0.25Ti-0.06C

0.1Nb-0.06C

Only 0.25Ti addition alloys show the frank loop formation for the model alloys.

Ti atoms aggregate on the extra half plane of the Frank-loop.

✧ Ti addition makes the stacking fault energy lower during irradiation.

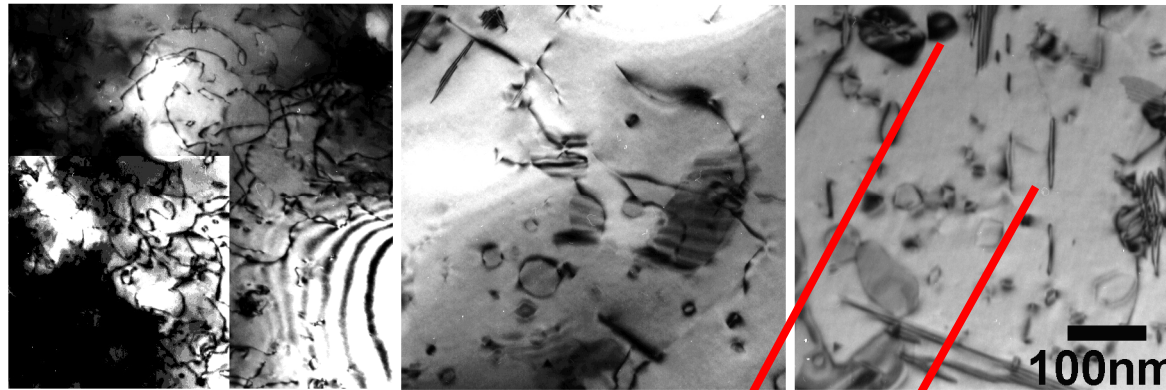
Comparison between Nb addition and Ti addition

Ti → inside of Frank-loops, Nb → incoherent MC precipitates from low T

A Nb addition easily makes MC precipitates

✧ The contribution for swelling suppression : Nb addition > Ti addition

Synergistic effect of Ti and Nb addition

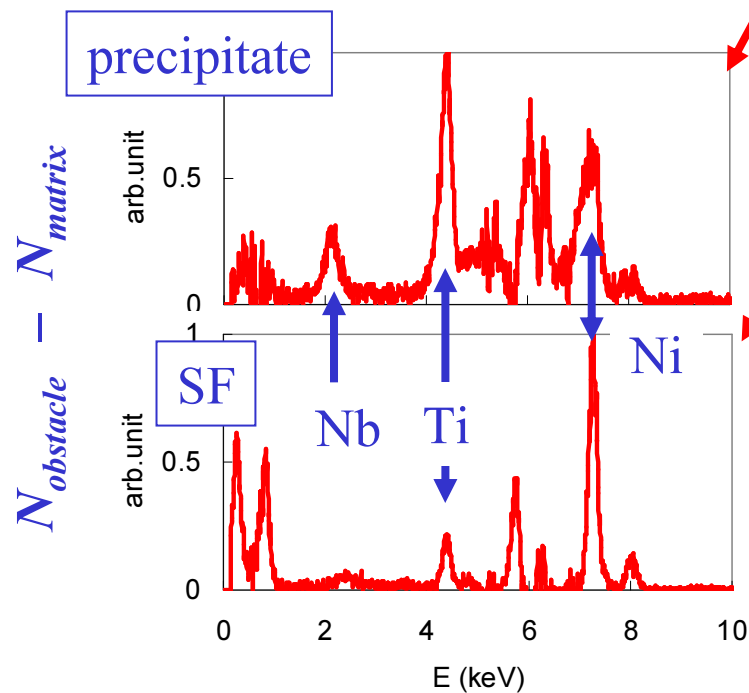


480°C, 35dpa

570°C, 48dpa

620°C, 33dpa

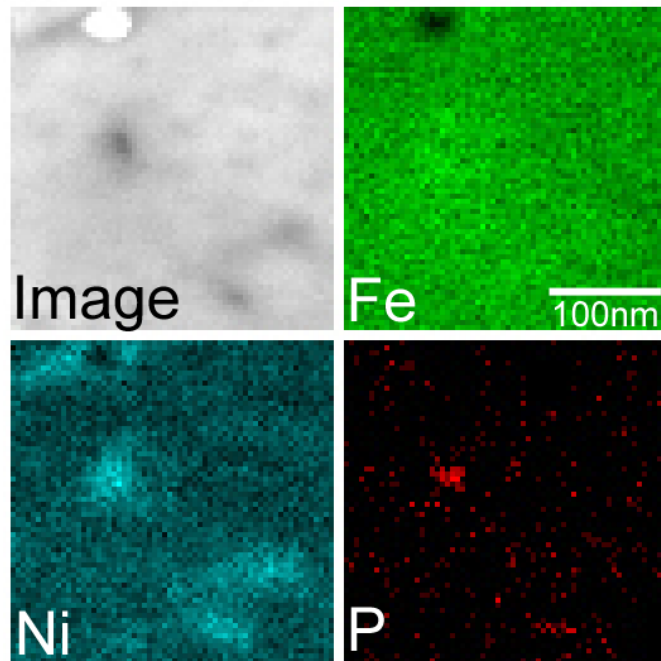
$M_{23}C_6$ on GB.
In 480-620°C,
(Nb,Ti)C presipitate and
Ti segregation on
stacking fault



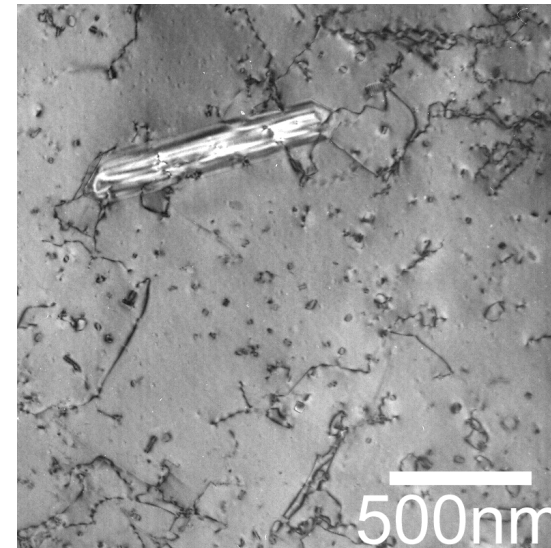
- ◆ No segregation of Nb on stacking fault.
- ◆ segregation element on stacking fault is only Ti.
- ◆ Stable formation of stacking fault at high temperature (620°C).

No synergistic effect of Ti and Nb addition
The contribution for swelling suppression :
Nb addition > Ti addition

Typical feature of precipitates in Fe-Cr-Ti modified alloys (P & B)



0.025P



0.004B

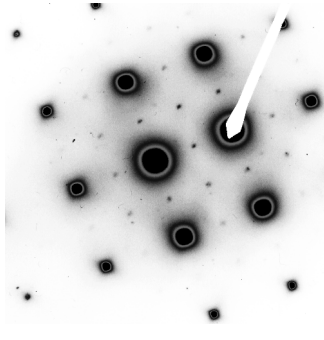
P addition :

Low density of MP at high temperature (Ni concentrate around MP precipitates)
Phosphor in solution is important for the suppression of the swelling

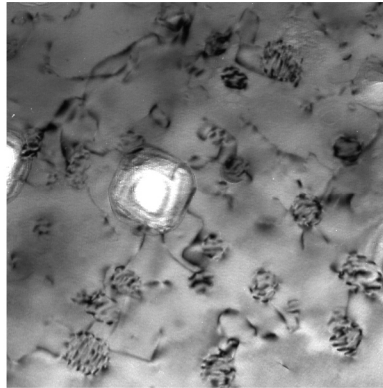
B addition :

No precipitates in the matrix, and some traces of boron precipitates were observed.
Most of boron precipitates are burn up during irradiation → He release
No bubble were observed → **it is unclear problem to explain the suppression of swelling**

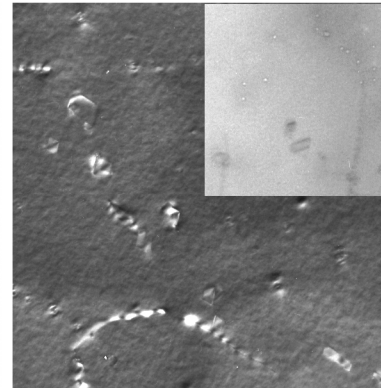
Typical feature of precipitates in Fe-Cr-Ti modified alloys (Si)



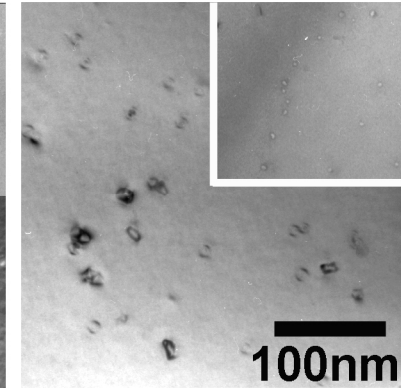
$\text{Ni}_3\text{Si} - \gamma'$ phase



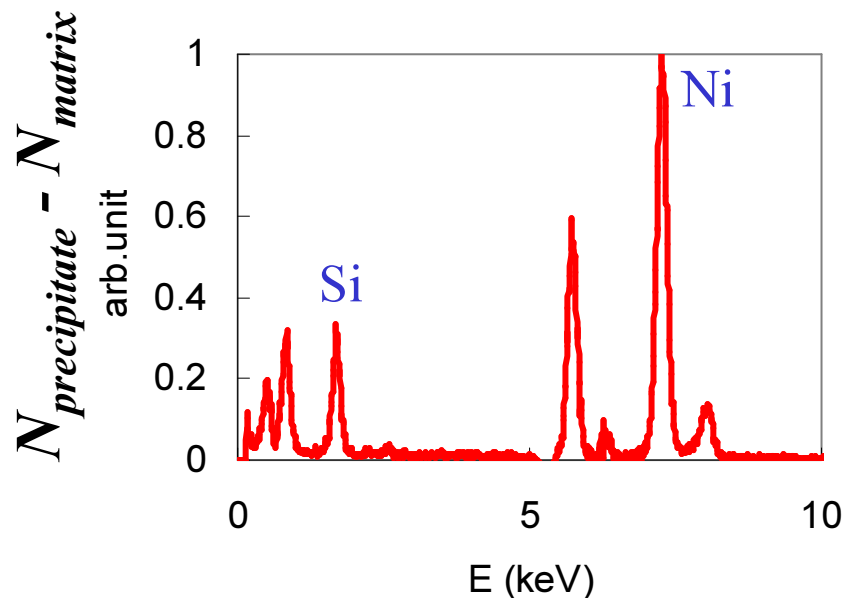
480°C, 35dpa



570°C, 48dpa



620°C, 33dpa



Si addition :

$\text{Ni}_3\text{Si} - \gamma'$ phase formation below 500°C
with cavity formation

Above 500°C, no typical precipitation.
Si segregation at the dislocation core.

EDS spectrum from dislocation at 570°C

Summary of solute addition effect for swelling behavior

C : Only C addition does not affect the precipitation and swelling behavior.
Synergistic effect with metallic minor elements (Ti, Nb...)

Mo : No effect of Mo addition on swelling behavior

Ti : Ti addition makes lower the stacking fault energy during irradiation.

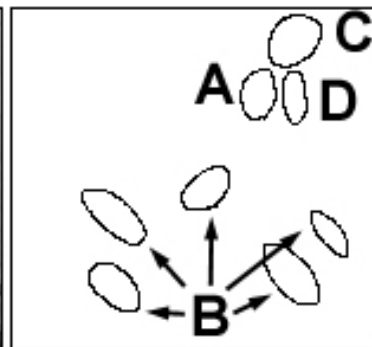
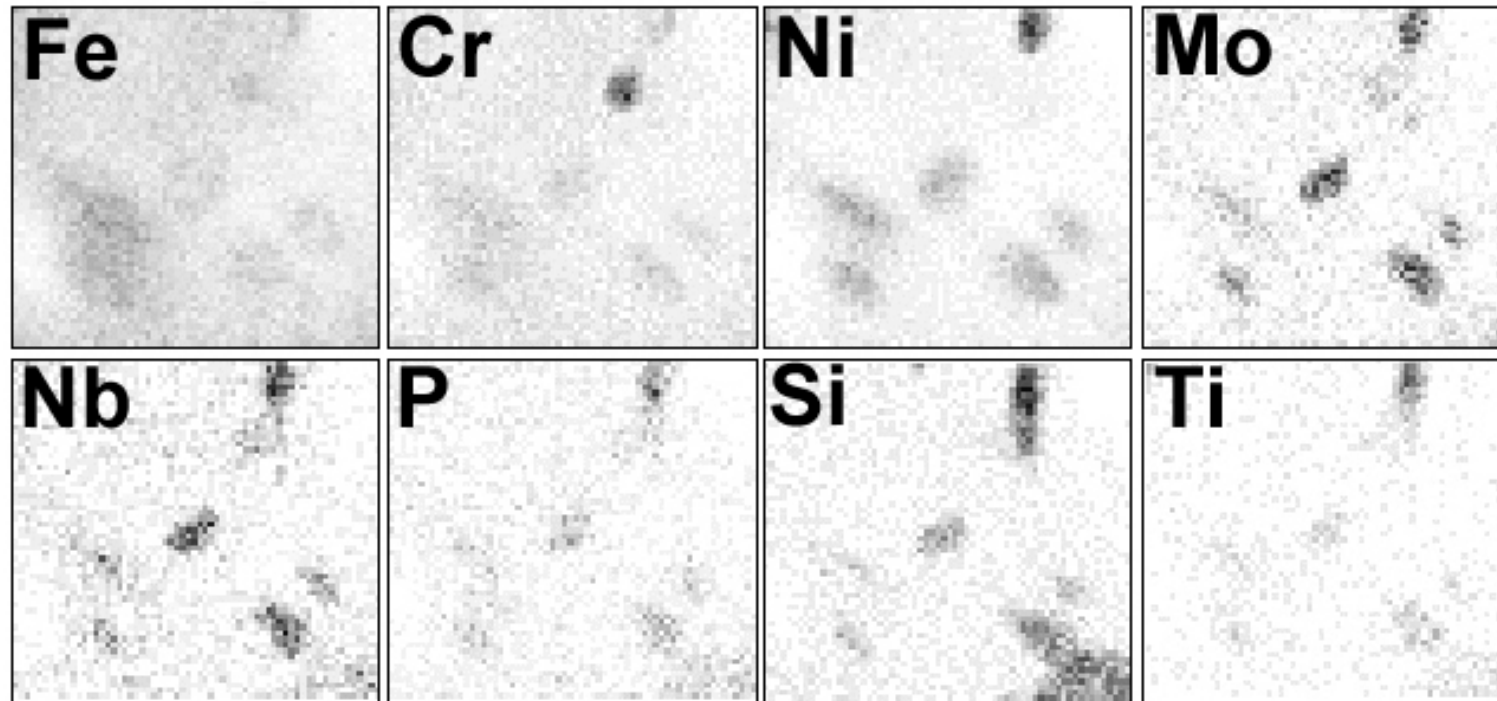
Nb: The incoherent MC precipitates are formed from 480°C.
MC precipitates suppress the swelling at higher temperature (>550°C)

P: MP precipitates and phosphors in solution suppress the swelling strongly.

B: All boron precipitates are burn away during irradiation.
The mechanism of swelling suppression at higher temperature due to B addition is unclear.

Si: Unstable γ' phase above 500°C, but effect of swelling suppression is little.

Characteristics of precipitates in PNC1520 steels by using EDS-2Dmapping



100nm

PNC1520 589°C 32dpa

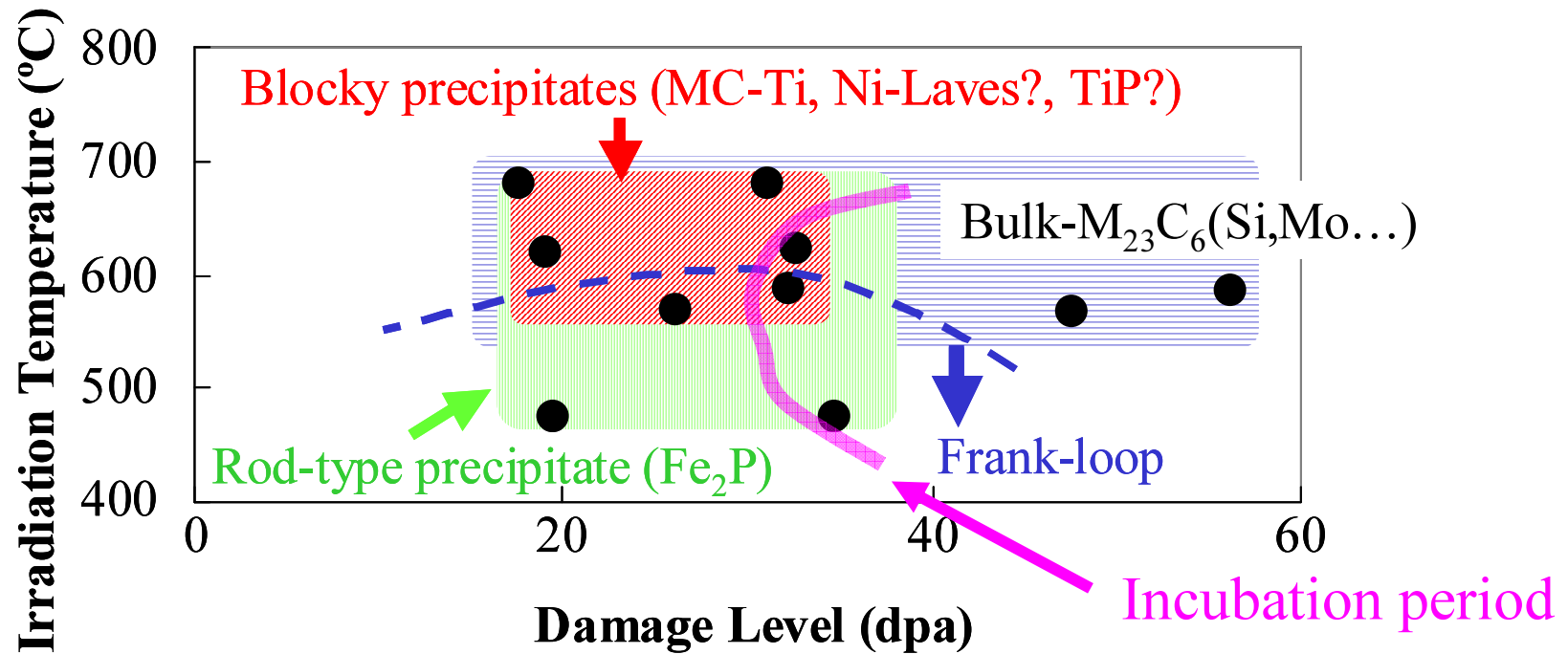
A: $M_{23}C_6$

B: MC

C: Laves

D: ? Si-rich Precipitate

Temperature – dose dependence of character of precipitates



Typical features

Fe₂P : strong effect for the suppression of swelling

Ti aggregate on Frank-loops in the temperature regime below 550°C

Dissipation of the MP and other precipitates at high dose levels

→ coarsening of the large M₂₃C₆ RMP

Summary

Swelling behavior of PNC1520

- Incubation damage levels is about 30 dpa for a PNC1520-SA steel
- Phosphor precipitates are effective for swelling suppression in PNC1520.

The effect of minor solute element for the swelling behavior

- Nb addition is stronger for the swelling suppression than Ti addition
- Ti addition makes stacking fault energy lower in a Fe-15Cr-20Ni alloy.
- P, Nb and B additions are effective for swelling suppression.
- Both Phosphor precipitates and P in solution is effective for swelling suppression.