

**6th IEA & JUPITER Joint Workshop on  
Vanadium Alloys for Fusion Applications  
Loews Ventana Canyon Resort  
Tucson, Arizona  
June 21- 22, 2002**

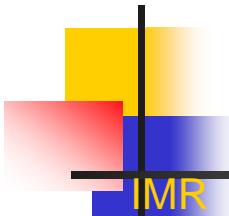
**Session II – Microstructural Evolution**

**Effect of impurities and alloying additions (binary V-X systems) on the formation of dislocation loops, voids, and precipitates during irradiation**



**H. Matsui**

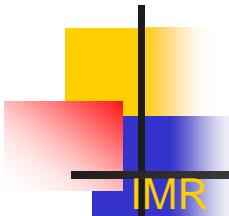
**IMR, Tohoku University**



# Contents

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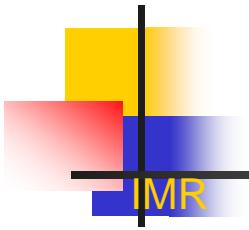
- Fundamental point defects parameters
- Dislocation loop formation
  - Effect of alloying additions (binary V-X systems)
  - Effect of impurities
- Voids
  - Effect of alloying additions (binary V-X systems)
  - Effect of impurities
- Precipitates
  - Effect of alloying additions (binary V-X systems)
  - Effect of impurities
- Variable temperature irradiation results



# Formation Energy *compiled by N. Nita*

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		Reference
<b>Formation Energy [eV]</b>		
n I-Cluster	$3.313n^{0.75}$	[237]
SIA	0.22, 4.58	[239][244]
n V-Cluster	$2.078n^{0.75}$	[237]
V	1.83, 1.2, 2.22, 2.2, 2.2, 2.1, 2.0, 2.1, 2.70-3.06, 3.07	[241][264][244][254][252][253][255][255][251][250]
2 V	3.99	[244]



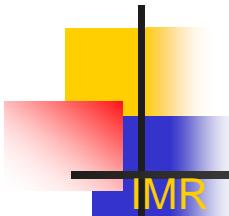
# Binding Energy

Binding Energy [eV]		
n I,V-Cluster	$E_{i,v}^b(n) = E_{i,v}^f(n-1) + E_{i,v}^f(1) - E_{i,v}^f(n)$	[237]
2 V	0.45	[244]
2 V	0.31	[263]
3 V	0.58	[263]
4 V	0.57	[263]
5 V	1.21	[263]
6 V	1.19	[263]
2 SIA	0.71	[262]
3 SIA	1.2	[262]
4 SIA	2.09	[262]
5 SIA	1.67	[262]
6 SIA	2.12	[262]
7 SIA	1.84	[262]
8 SIA	3.00	[262]
9 SIA	2.16	[262]
10 SIA	1.96	[262]

# Migration Energy

## Migration Energy [eV]

SIA	0.116, 0.03, 0.13, 0.6, 0.21, 0.56, 0.62, 0.55, 0.06	[237][262][239][243][259][260][260][244]
2 SIA	0.139, 0.067	[237][262]
3 SIA	0.156, 0.17	[237][262]
4 SIA	0.335	[237]
7 SIA	0.069	[237]
V	0.763, 0.83, 1.2, 1.25, 1.4, 0.76, 1.57, 1.02, 0.72-1.3, 1.0, 1.5, 1.5, 0.81, 1.3, 1.2,	[237][266][246][240][265][241][260][260][243][244][255][261][259][255][254]
2 V	0.818	[237]
3 V	1.227	[237]
He	<0.1	[242]
O	1.21, 1.26, 1.26, 1.26-1.28	[245][246][268][249]
C	1.18	[267]



# Other Parameters 1

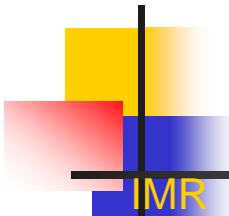
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## Self Diffusion Energy [eV]

Vanadium	3.2, 2.65-3.2, 4.64, 4.26-5.96, <3.5	[244][244][244][244][261]
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## Damage Efficiency

SDF	0.35	[269]
CDF <sub>i</sub>	0.02	[238]
CDF <sub>v</sub>	0.06	[238]



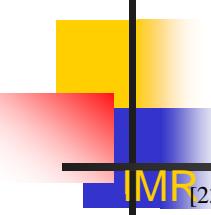
# Other Parameters 2

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<b>Stacking Fault Energy [J/m<sup>2</sup>]</b>		
	2.68	[237]

<b>Surface Energy [erg/cm<sup>2</sup>]</b>		
100 loop	1743, 2589	[256][257]
110 loop	1473, 1822	[256][258]
Flat Lattice	2.62 eV	[261]
Void	1.2-1.6 eV	[261]

<b>Dissociation Energy [eV]</b>		
He <sub>n</sub> VX	1.5	[247]
He <sub>n</sub> VO	1.42	[248]



# References

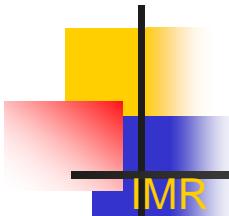
IMR

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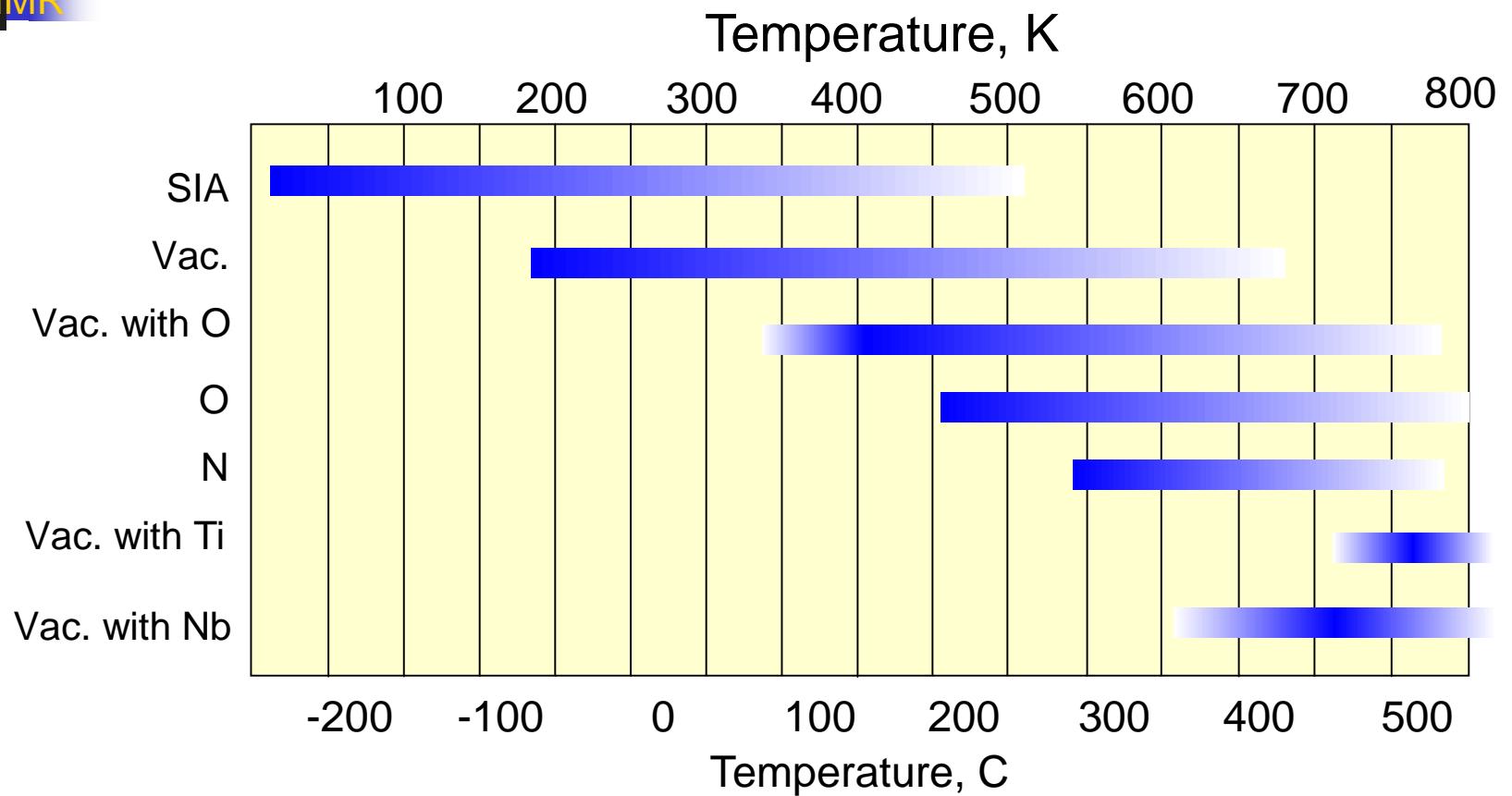
# Activation energies obtained from the HVEM experiment

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specimen	Interstitial impurity level and solute concentration (at.%)	$E_{\text{effec.M}}^{\text{I}}$ (eV)	Possible physical significance
Pure V	High purity (HP)	0.24	$E_M^{\text{I}} + E_b$ b.w. a SIA and an impurity
	Nominal purity (NP)	0.22	$E_M^{\text{I}} + E_b$ b.w. a SIA and an impurity
V-xFe	NP ( $X = 0.1, 0.2, 0.3, 5$ )	0.81	$E_M^{\text{I}}$ for solute dumbbells + $E_b$ b.w. a SIA and an interstitial impurity
	HP ( $X = 0.1$ and 5)	0.5 for V-5Fe	$E_M^{\text{I}}$ for solute dumbbells
V-yCr	NP ( $Y = 0.1$ )	N.D.	$E_M^{\text{I}}$ for solute dumbbells + $E_b$ b.w. a SIA and an interstitial impurity
	NP ( $Y = 1$ and 5)	0.65	$E_M^{\text{I}}$ for solute dumbbells + $E_b$ b.w. a SIA and an interstitial impurity
V-1Si	NP	0.99	?
V-5Ti	HP	0.38 in stage III'	$E_M^{\text{I}} + E_b$ b.w. a SIA and a solute (in concentrated alloy (?))
	NP	0.39 in stage III'	$E_M^{\text{I}} + E_b$ b.w. a SIA and a solute (in concentrated alloy (?))
V-4Cr-4Ti	NP	0.50 in stage III' 1.0 in stage IV	$E_M^{\text{I}} + E_b$ b.w. a SIA and a solute (in concentrated alloy (?))? ?
NIFS H-I	As-received	N.D.	Trapping by metal impurities
	HP	N.D.	Trapping by metal impurities



# Mobility of Defects in V-alloys



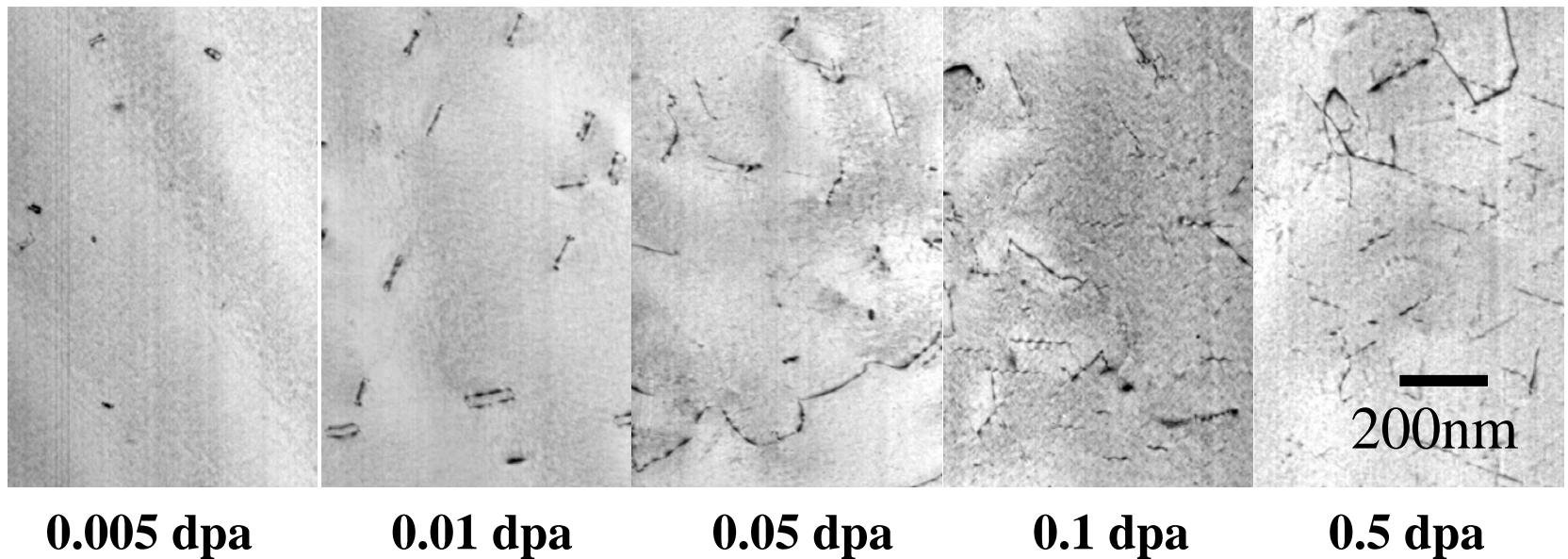
Compiled from available data of TEM, positron, Q<sup>-1</sup>

# Dislocation Loops

- Based on TEM observation:
  - Neutron irradiated in JMTR

JMTR, 350C

pureV

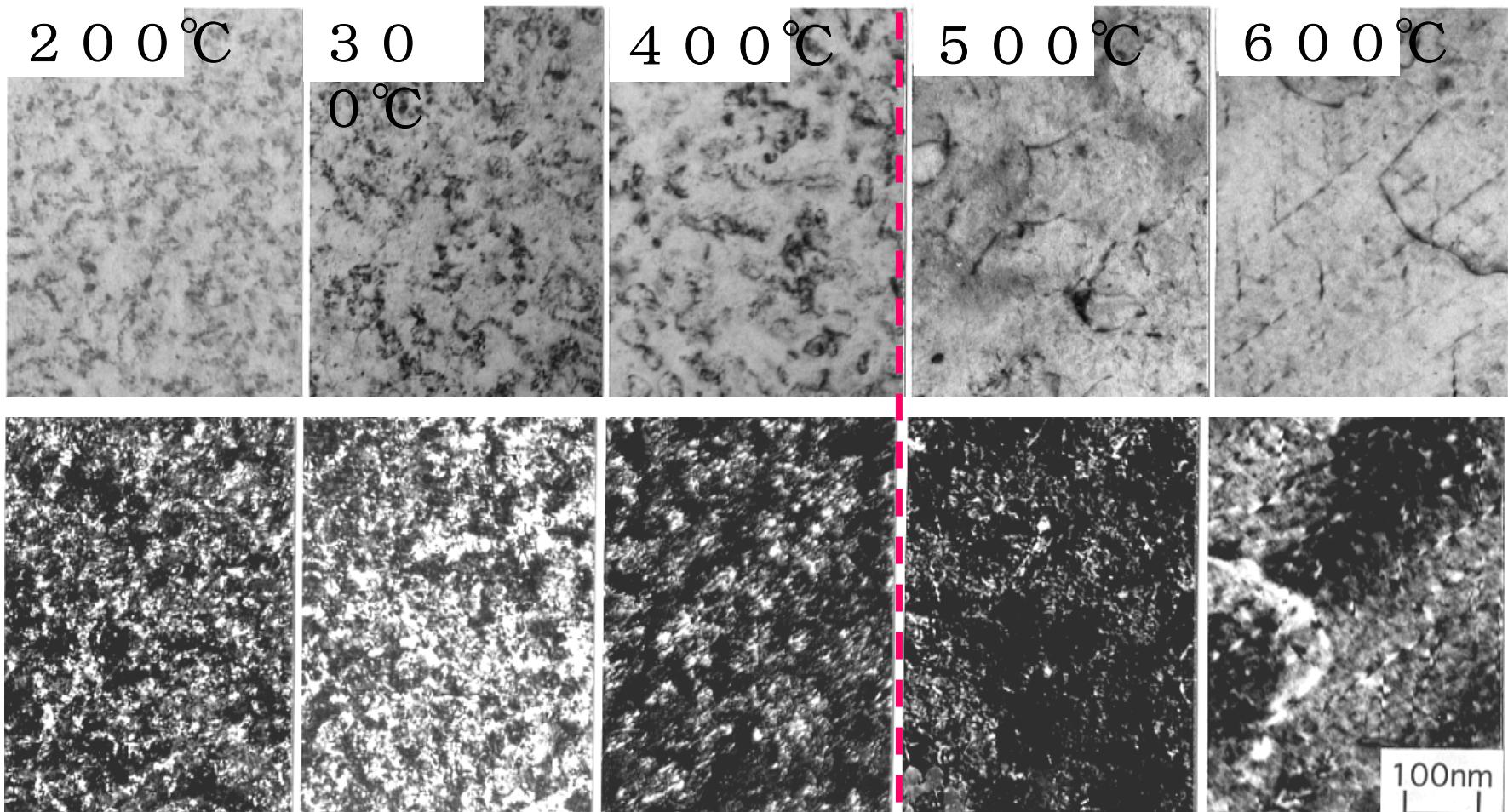


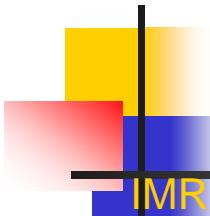
# V: Temp. Dependence of Damage

3MeV-Cu<sup>2+</sup>, 0.75dpa, 200-600°C

H. Watanabe & N. Yoshida

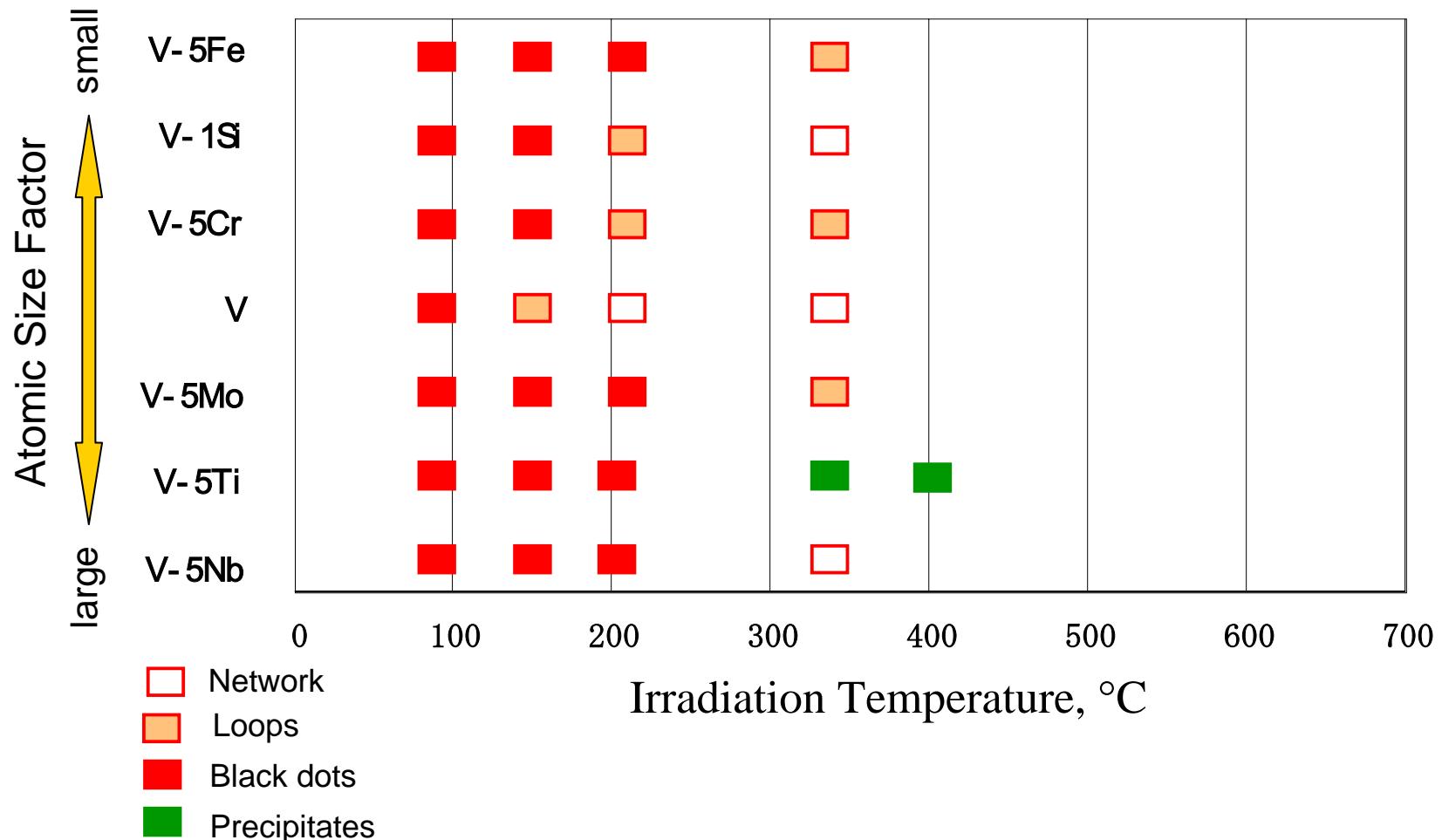
- **I-loops, V-clusters, precipitates, voids are formed depending on irradiation temperature.**



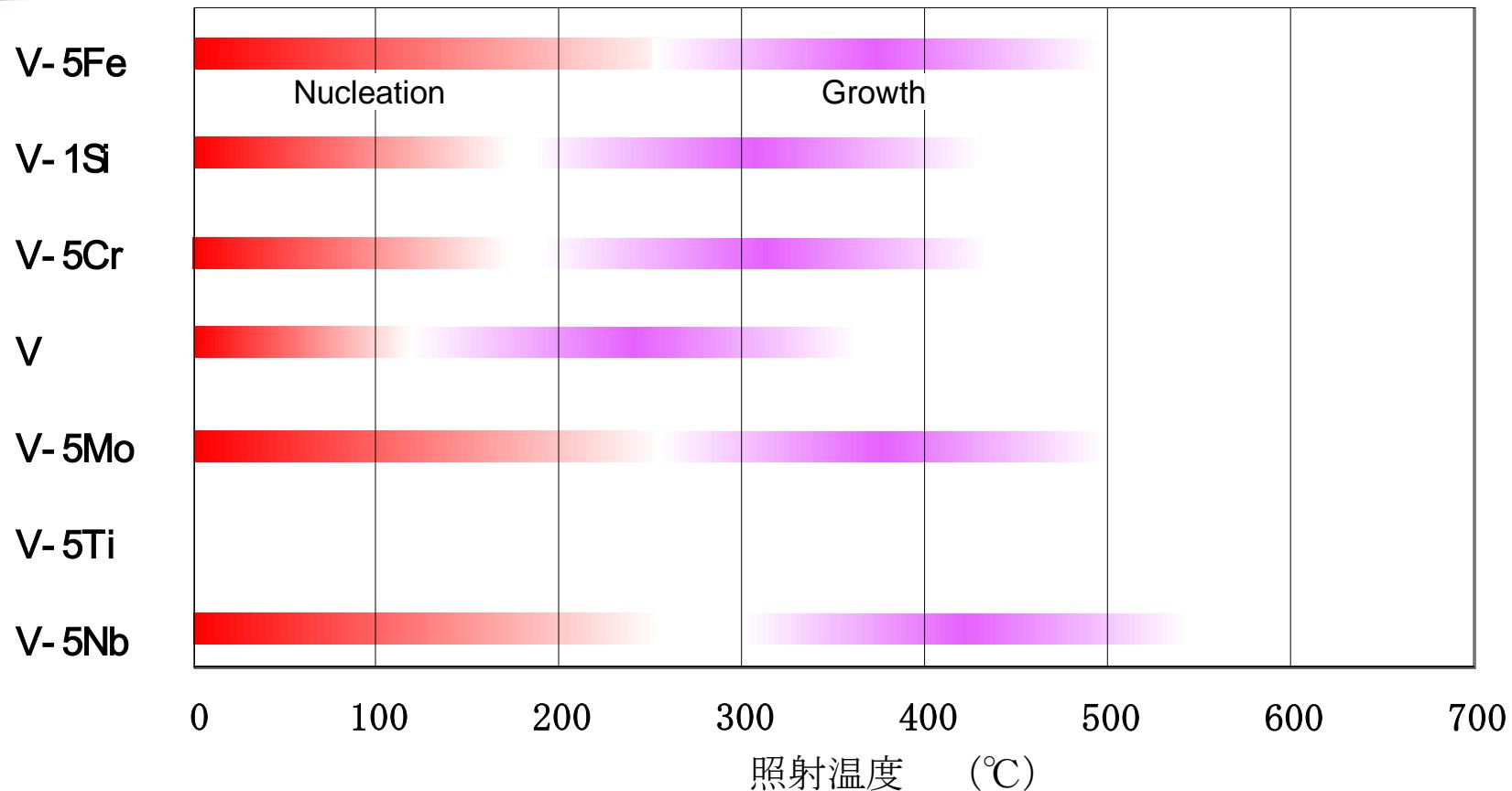


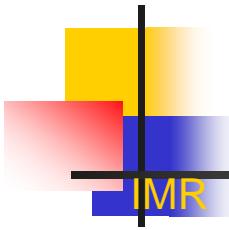
# Temp. Dependence of Dislocations

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# Nucleation and Growth of Dislocation Structure



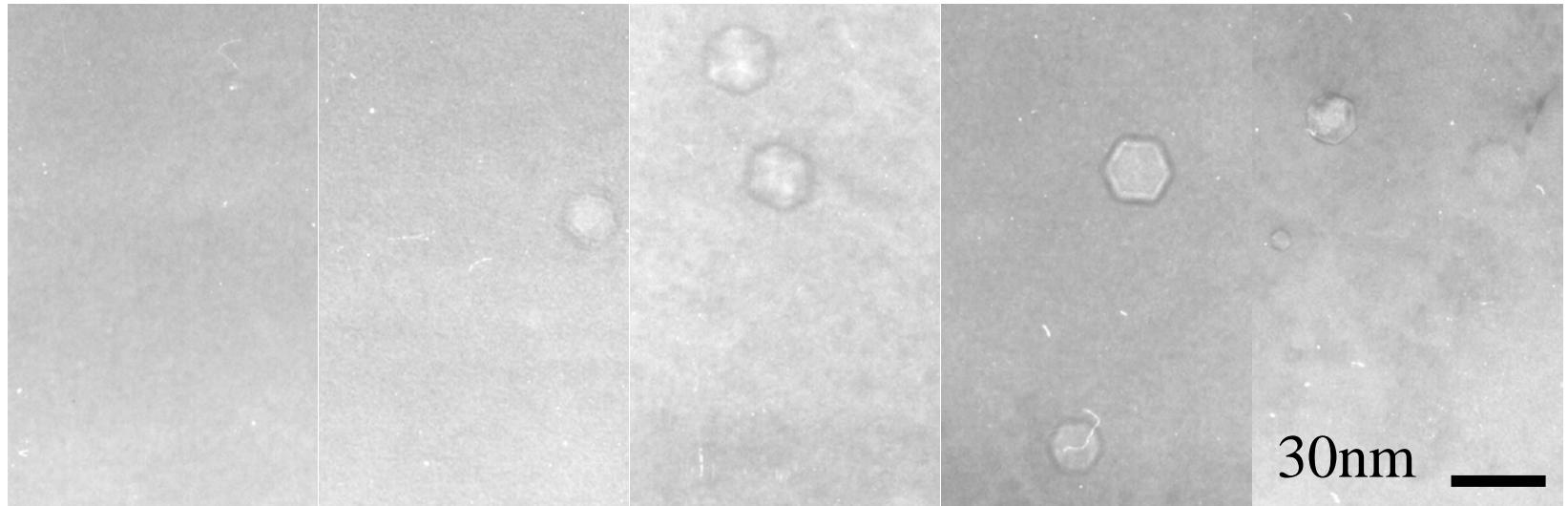


# Cavities

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- TEM Observation

pureV



**0.01 dpa**

**0.03 dpa**

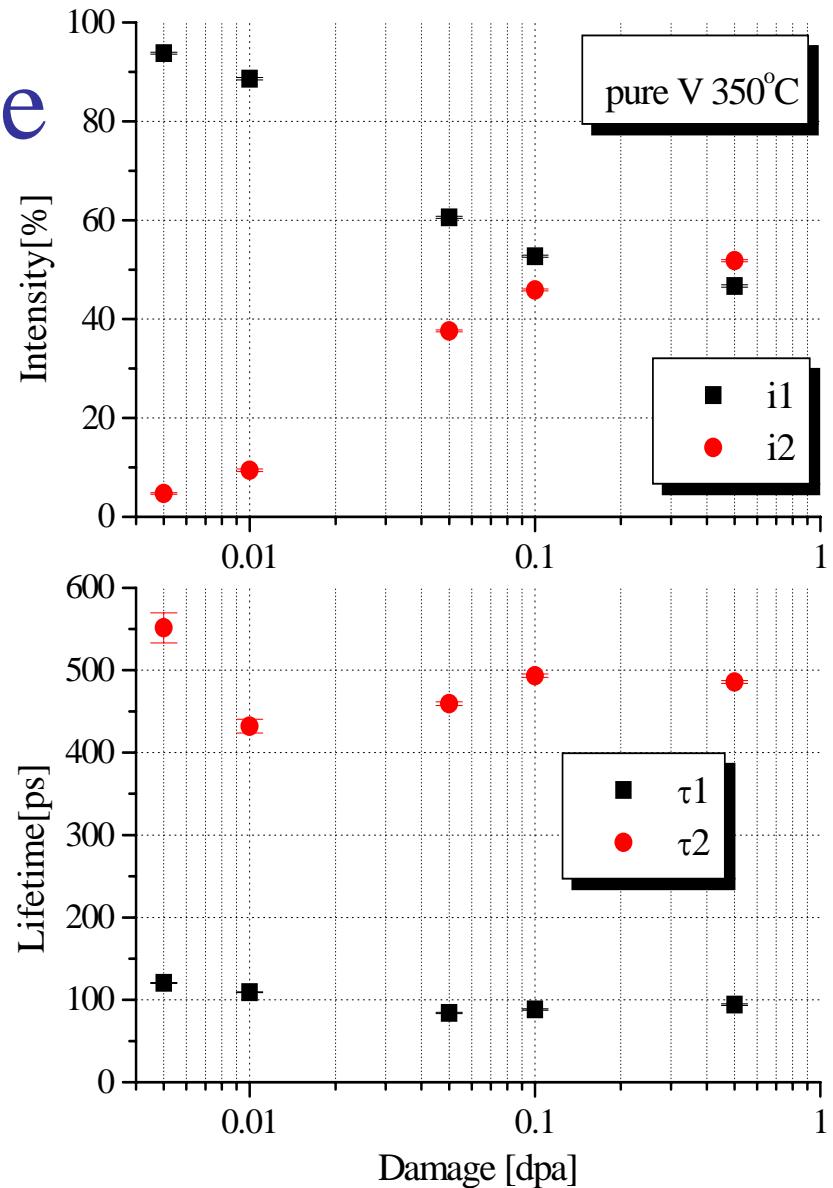
**0.1 dpa**

**0.3 dpa**

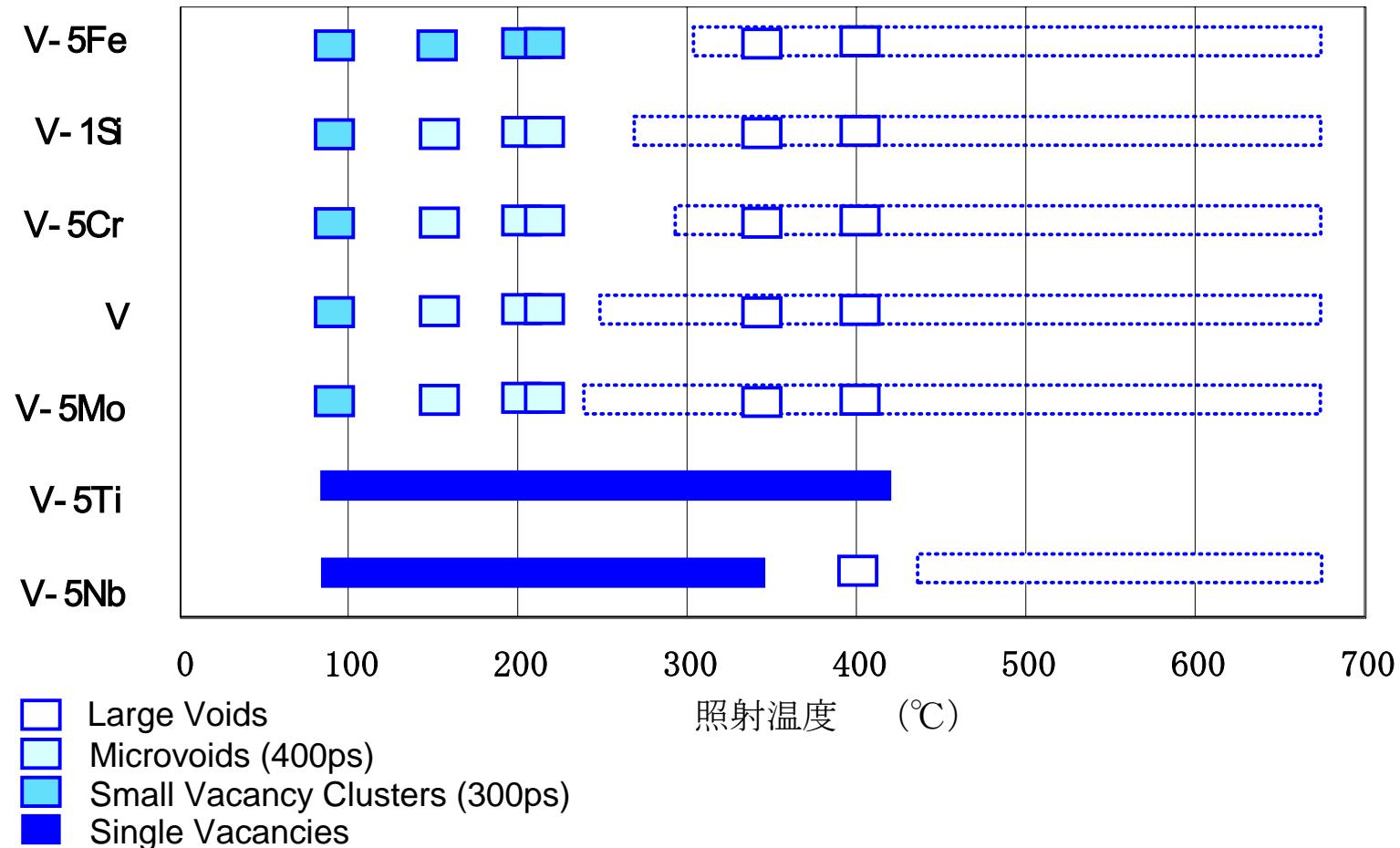
**1 dpa**

# Positron Lifetime

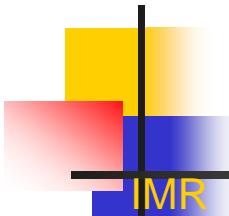
- Sensitive to V-type clusters



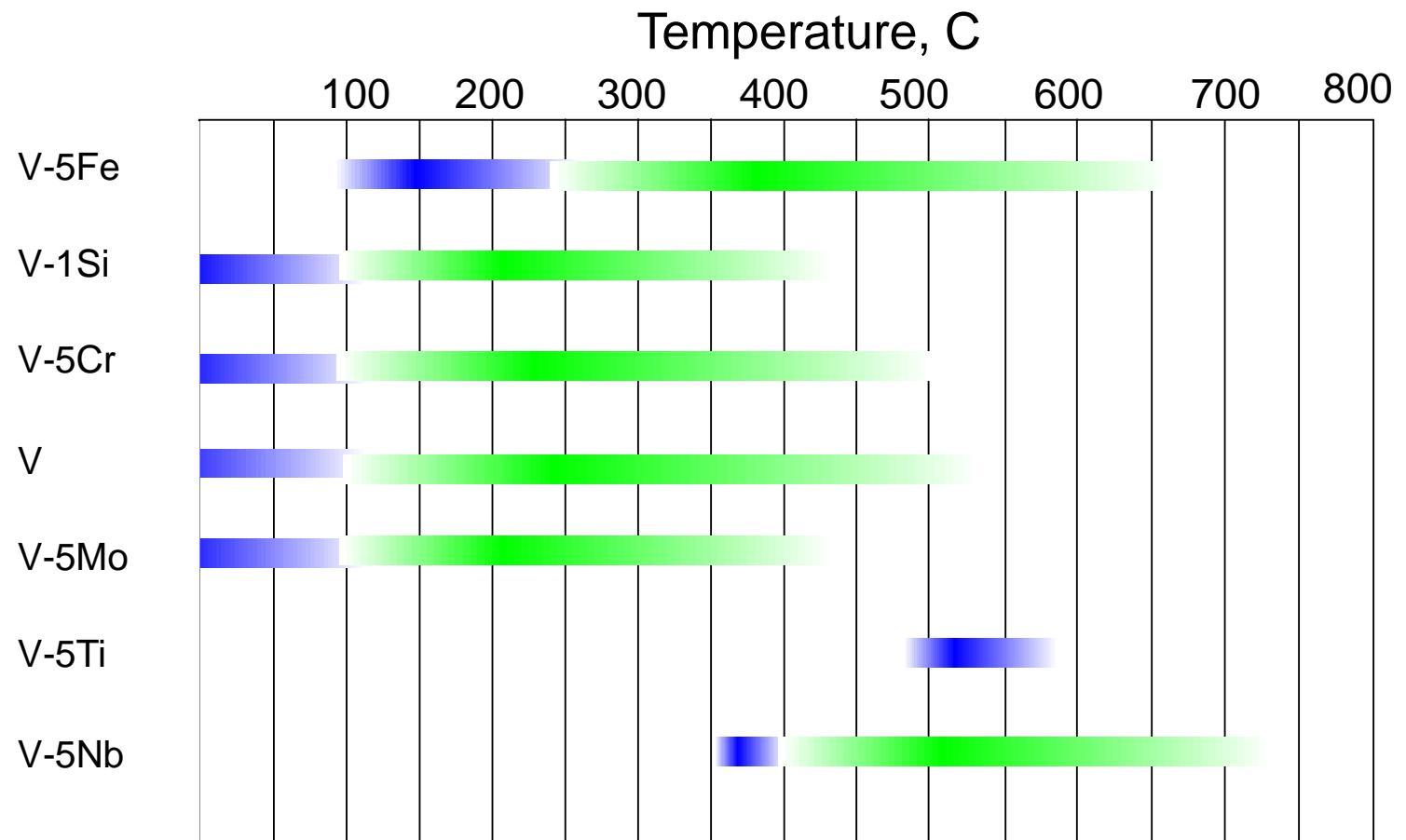
# V-clusters under Constant Temp. Irradiation



Data: Based on TEM and positron annihilation lifetime measurements



# Nucleation and Growth of V-clusters

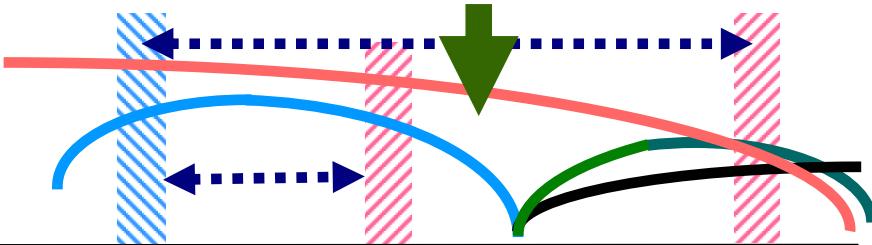


# Temp. Depend. of Defect Formation in V and V-Alloys

H. Watanabe & N. Yoshida

Pure V

Border temp.



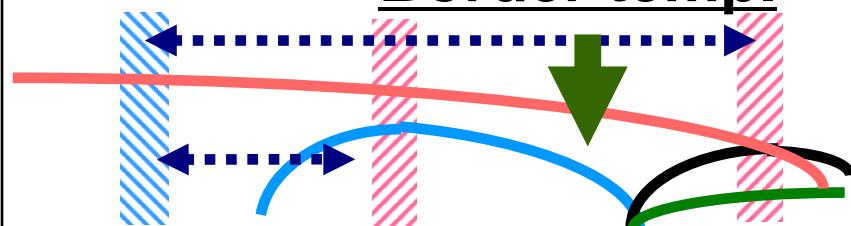
3MeV-Cu<sup>2+</sup>,  
0.75dpa, 200-600°C

Border temp.

(Namely, Stage V)

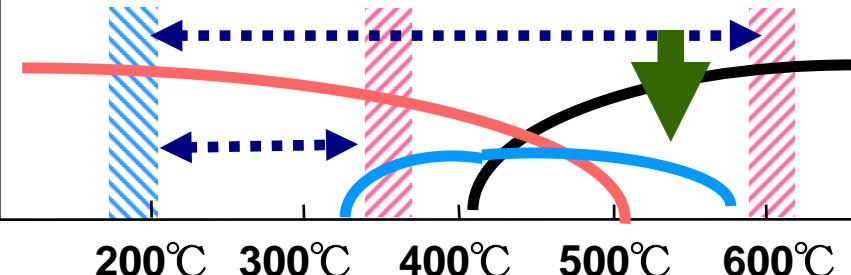
V-5Cr

Border temp.



Dissociation of small  
V cluster  
Significant effects on  
microstructure

V-5Ti, V-4Cr-4Ti Border temp.



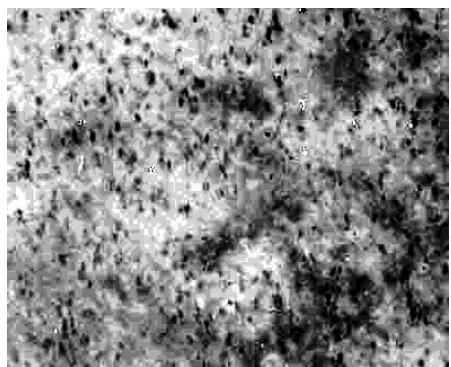
- I-loop
- V-cluster
- Void
- Precipitates

# Precipitates

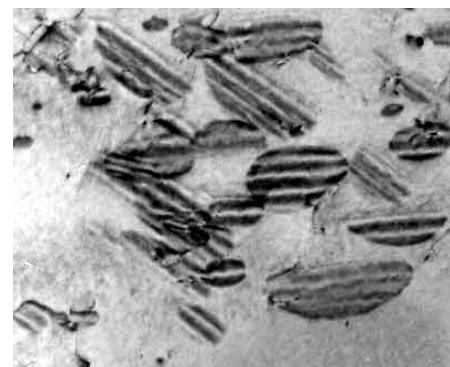
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- “Pure V”
  - VC
- V-5%Ti
  - $Ti_2O$ ,  $TiO$ ,  $TiO_2$
- V-Cr-Ti(-Si)
  - $TiO$ ,  $Ti(O,C,N)$  ,  $Ti_5Si_3$ , etc.

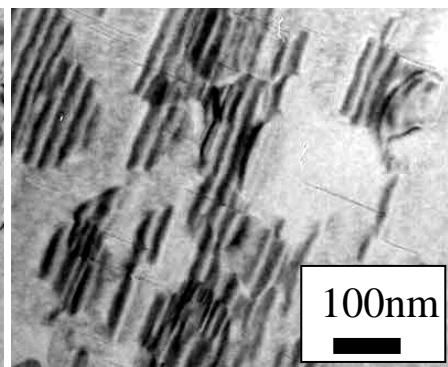
V-5Ti



400(0.1dpa)



500(0.1dpa)

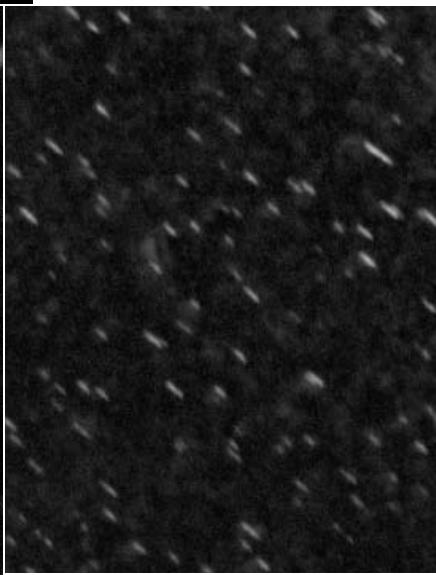
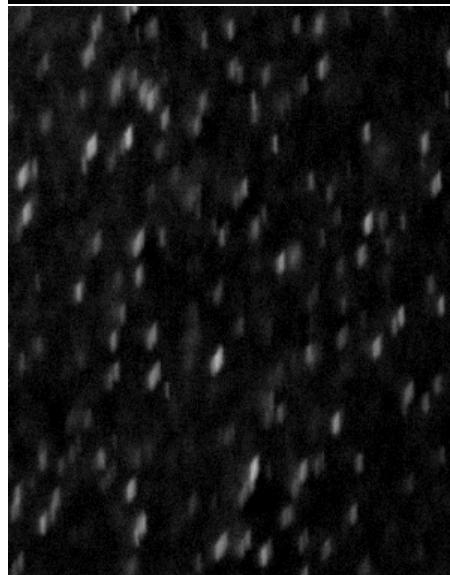
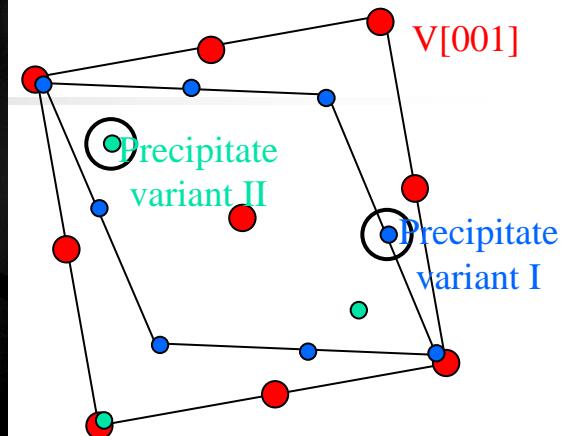
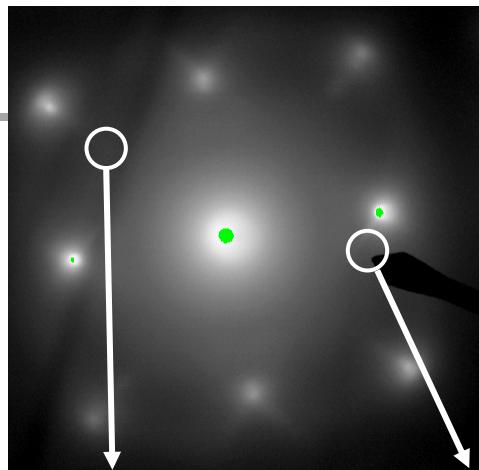


600(0.1dpa)

# Precipitates

- TiO precipitates
  - Needle like,  
along  $<100>$

V-3Fe-4Ti-0.1Si, 340°C, 0.061dpa



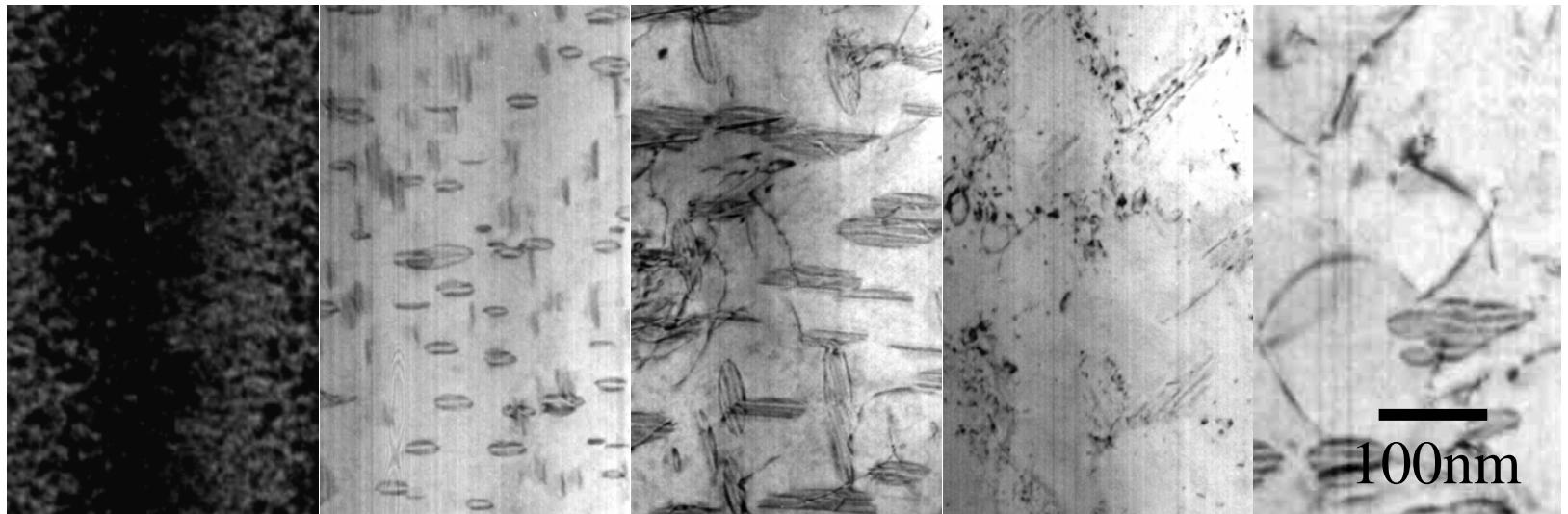
# Precipitates in V-4Cr-4Ti

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- Mostly TiO precipitates
- TiSiO

500°C

V-4Cr-4Ti-0.1Si



0.01 dpa

0.03 dpa

0.1 dpa

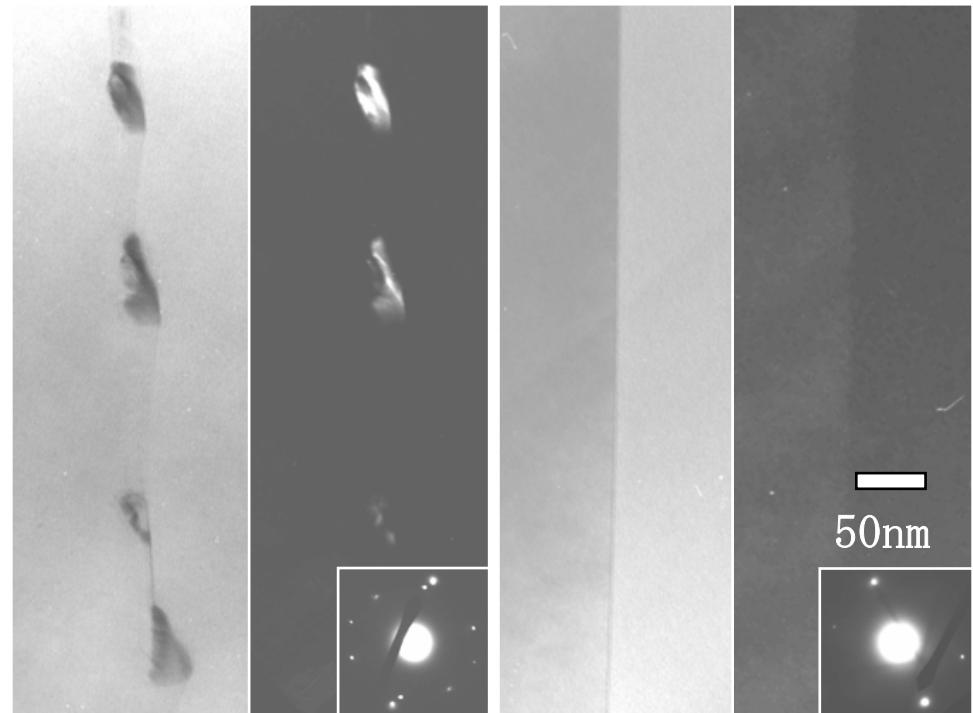
0.3 dpa

1 dpa

100nm

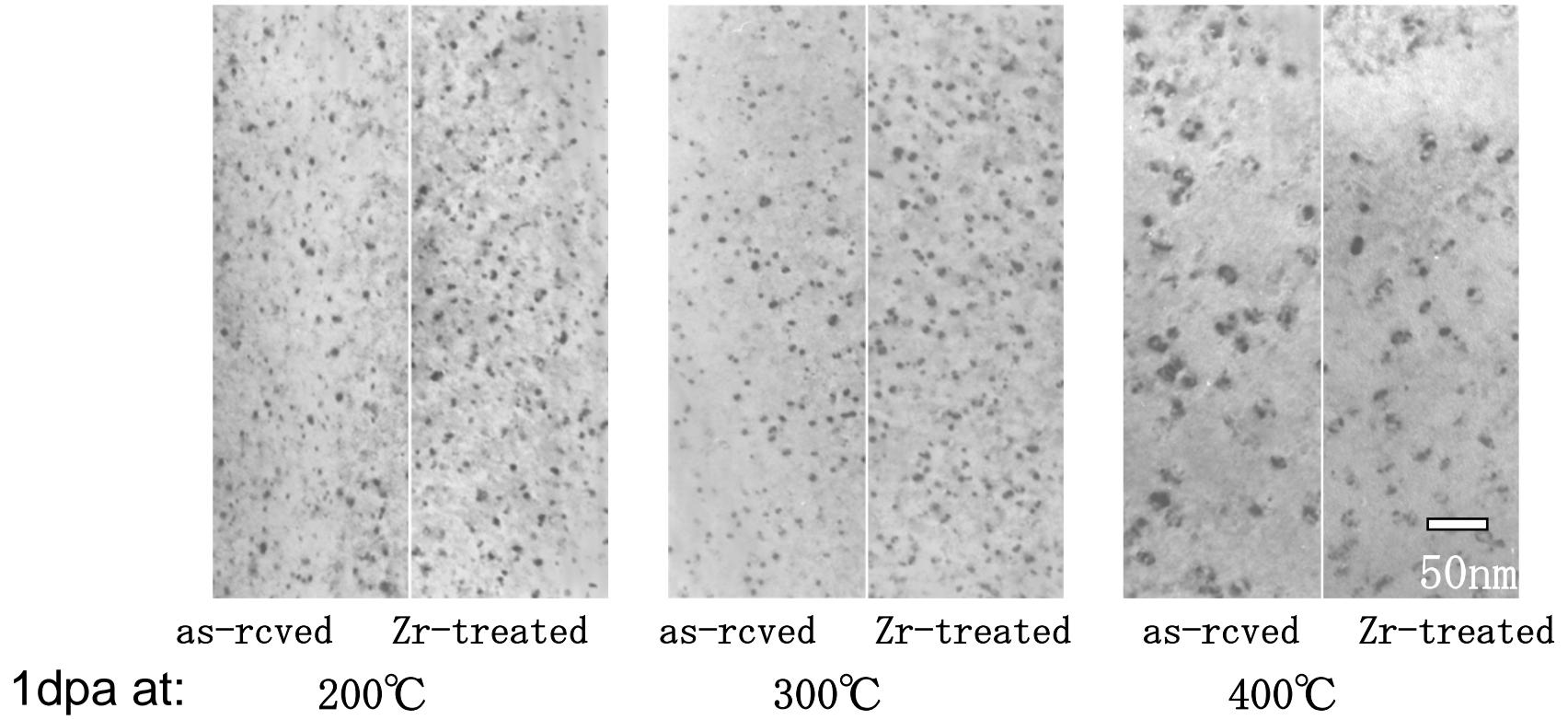
# Effects of Purification

- V-4Cr-4Ti
  - Grain boundary precipitates disappear by purification



O	t	144	wppm	2
N		124		6
C		54		3

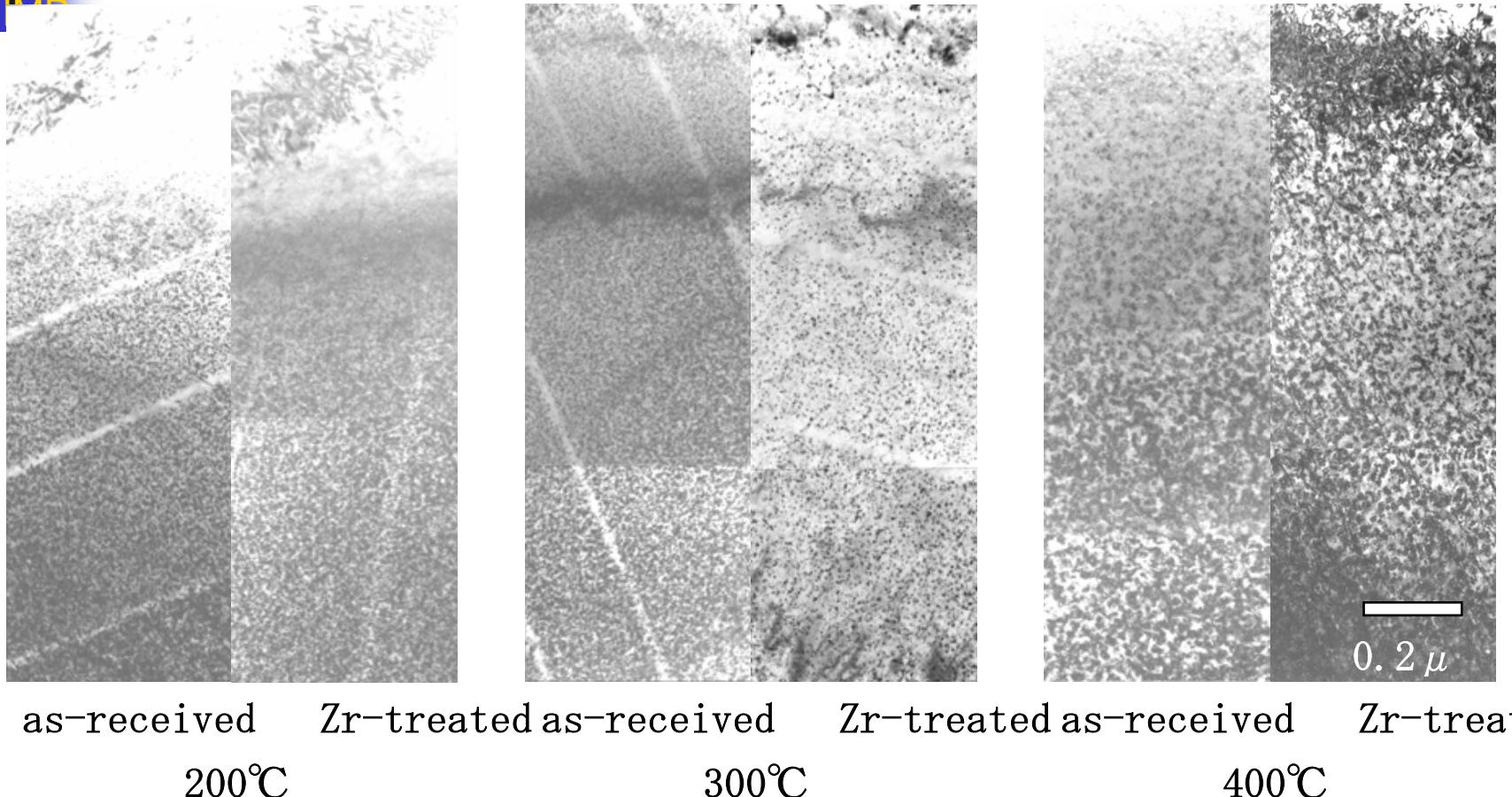
# Effect of Purification on Loops



- No significant effects of purification is observed

# Effects of Purification on Channeling

## V-4Cr-4Ti

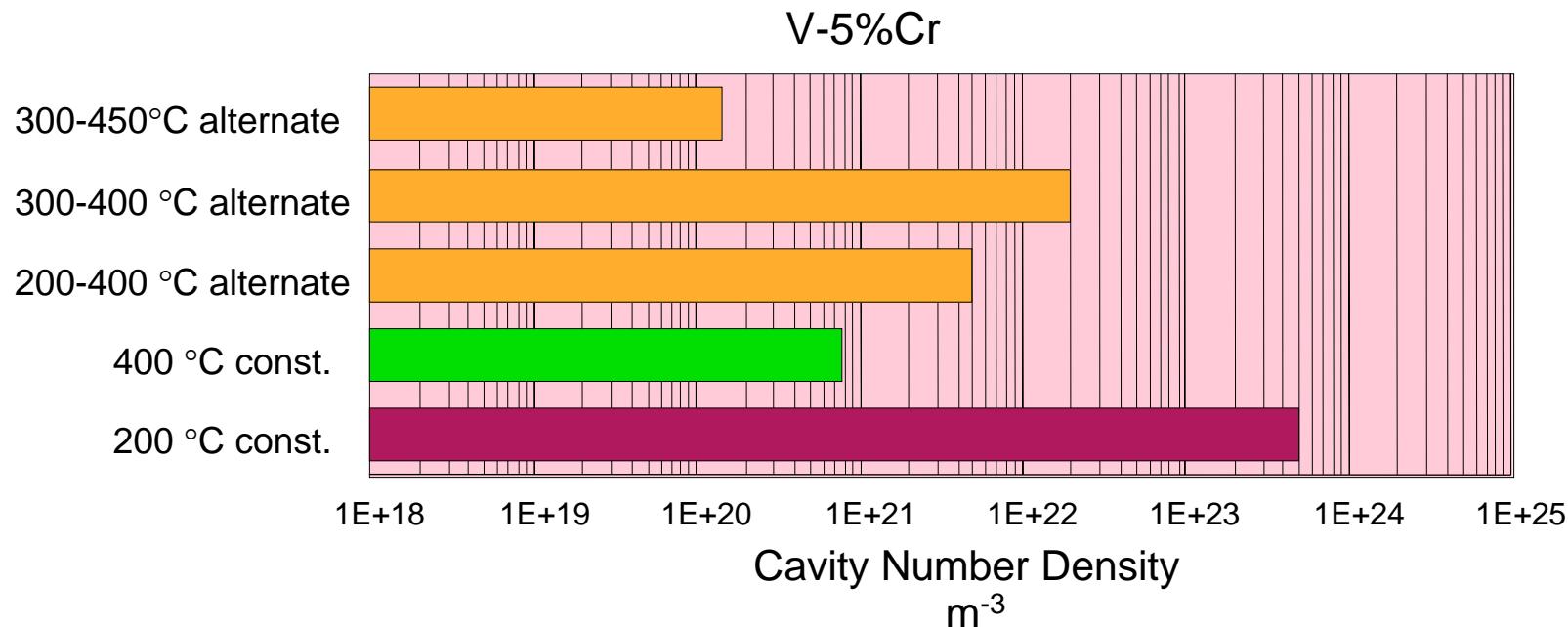
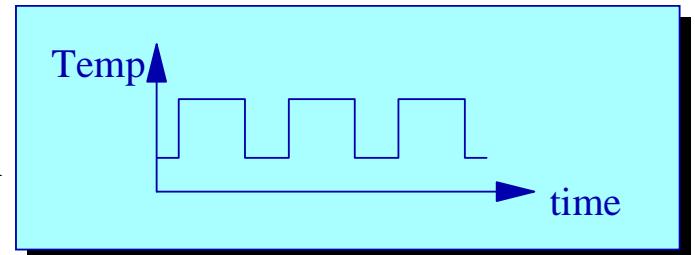


- Frequency of dislocation channeling is higher in as-received material, despite no apparent difference in dot density.

# Variable Temperature Irradiation

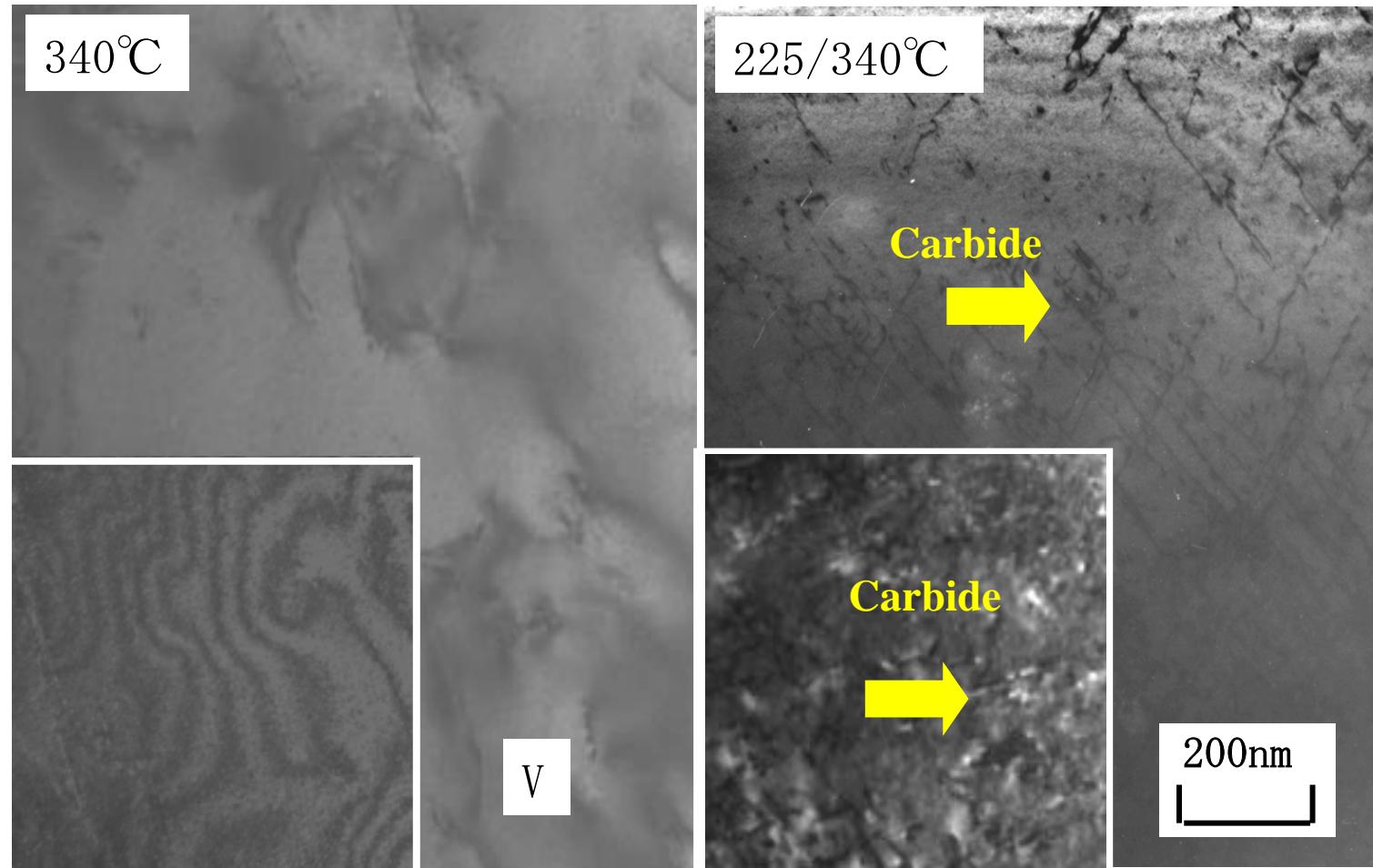
IMR

- Variable Temp. Irradiation:
  - Sometimes defect number density becomes more than an order of magnitude greater than constant temperature irradiation
  - Embrittlement, swelling, etc. will be also affected accordingly.



# Enhanced Carbide Formation in

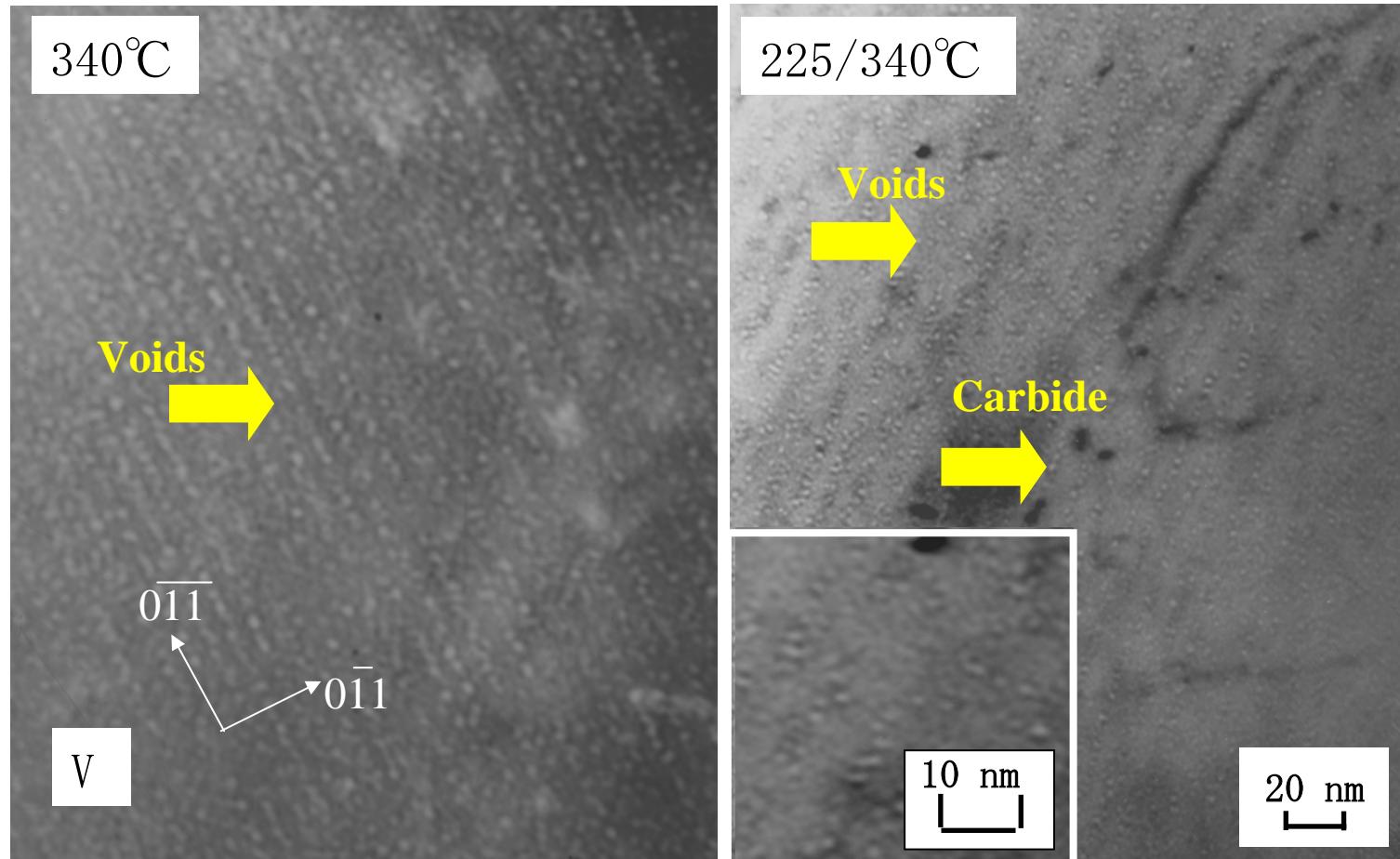
V



**Const. Temp.; Low dislocation density**

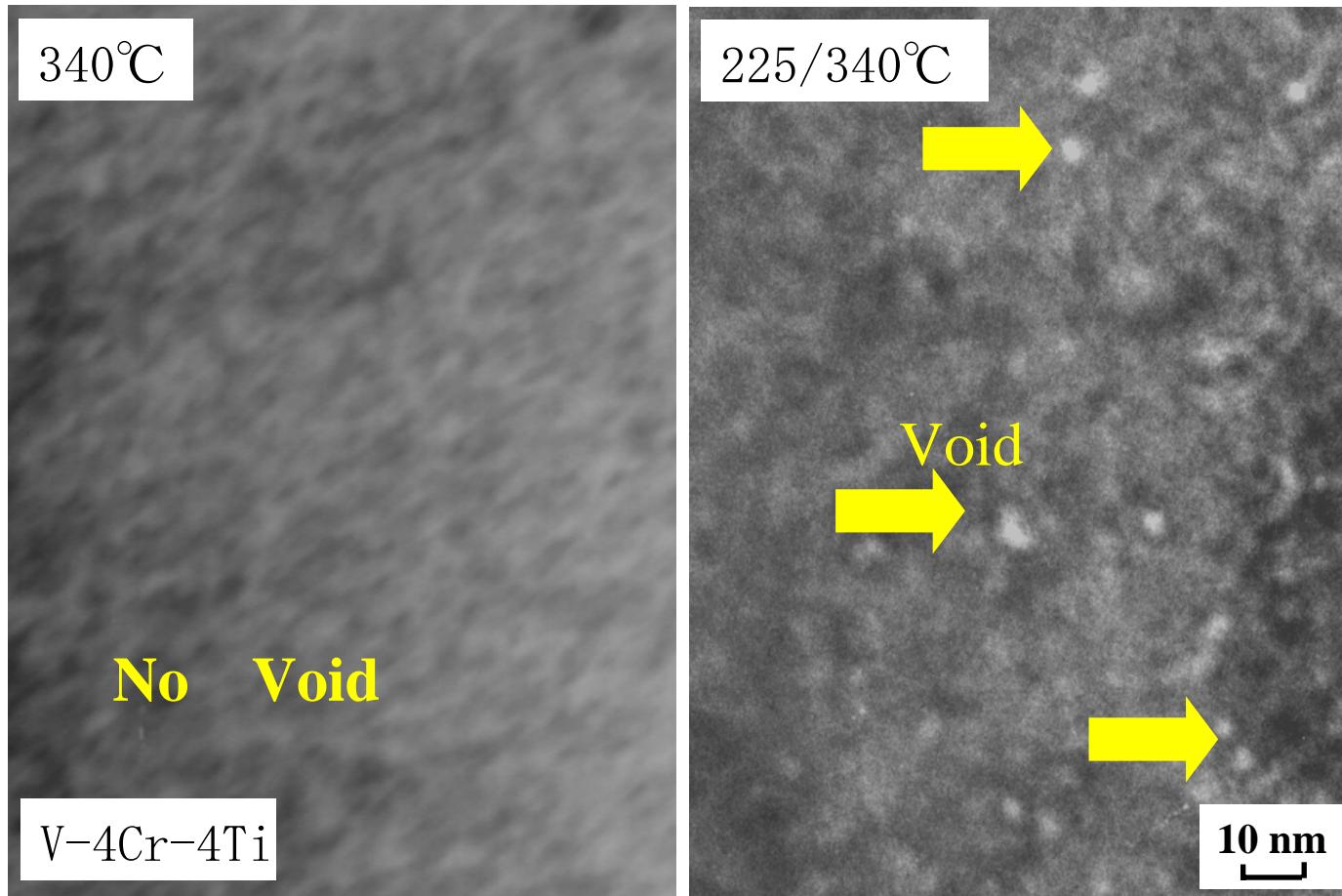
**Varying Temp.; They are oriented <100> direction,  
Carbide formation**

# Void Lattice Formation in V



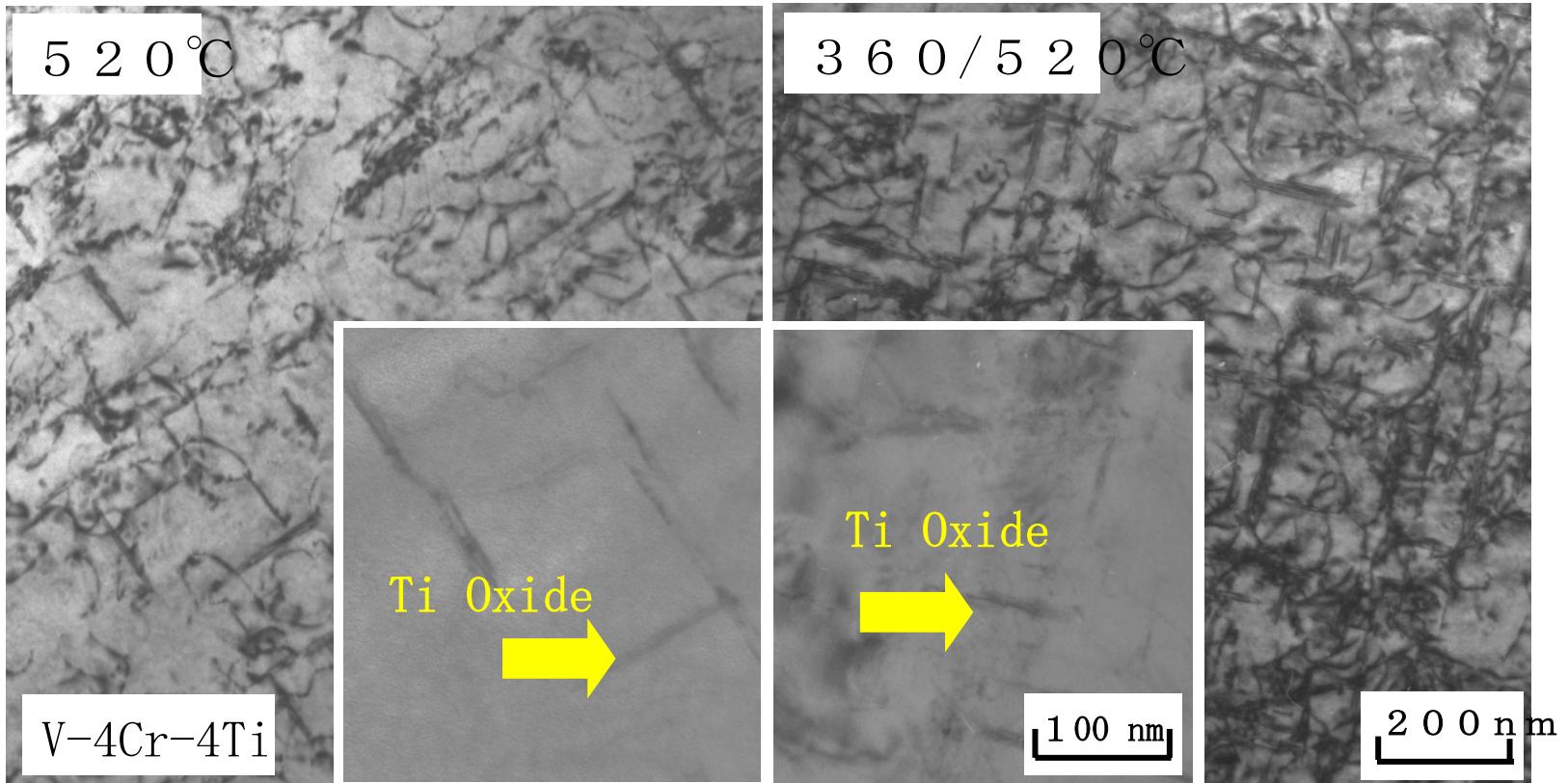
**Const. Temp.; Formation of Void lattice (Effects of O?)**  
Size; 2.8(nm) Density;  $1.1 \times 10^{24} (\text{m}^{-3})$   
**Varying Temp.; Random distribution (1.8nm,  $1.4 \times 10^{24} (\text{m}^{-3})$ )**

# Void Contrast Image of V-4Cr-4Ti

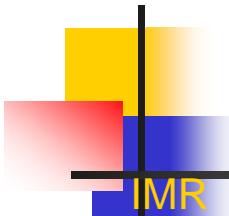


**Small voids were observed at varying temp. condition.**  
→ Enhanced nucleation of Void, Oxide related?.  
→ Unstable at higher dose levels?

# Microstructure of V-4Cr-4Ti

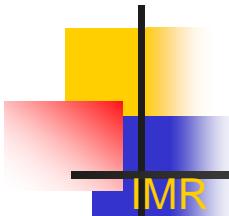


- 360/520°C irrad. produced higher Ti oxide number density
- Size of oxide; 250 → 160 (nm)



# Variable Temperature Irradiation

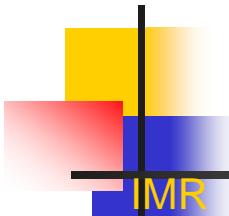
- Theory and Modeling of temperature variation is necessary:
  - -to make planning of irradiation experiments
  - -to understand the mechanism.
- To be fully covered in the swelling session.



# Summary

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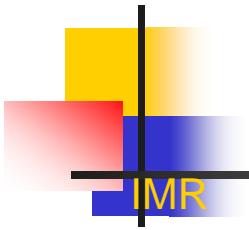
- Temperature ranges of loop and void formation are relatively well understood.
- Fundamental parameters of point defects are known in pure vanadium, while the effects of alloying and impurity elements are less well known.
- Precipitates are mainly originated from interstitial impurities, and are not controlled well.
  - Tests on low oxygen materials are necessary since oxygen will be eventually re-distributed to lithium. To narrow down oxygen concentration range will be beneficial in building up database.



# Summary -2

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- High dose irradiation tests are lacking.
- He and H effects are still need to be addressed intensely.
  - to be covered in the swelling session.
  - Qualitative understanding has been obtained, while quantitative evaluation is not possible with high accuracy.
  - Fusion neutron source is awaited; simulation technique, e.g. DHCE may be useful.

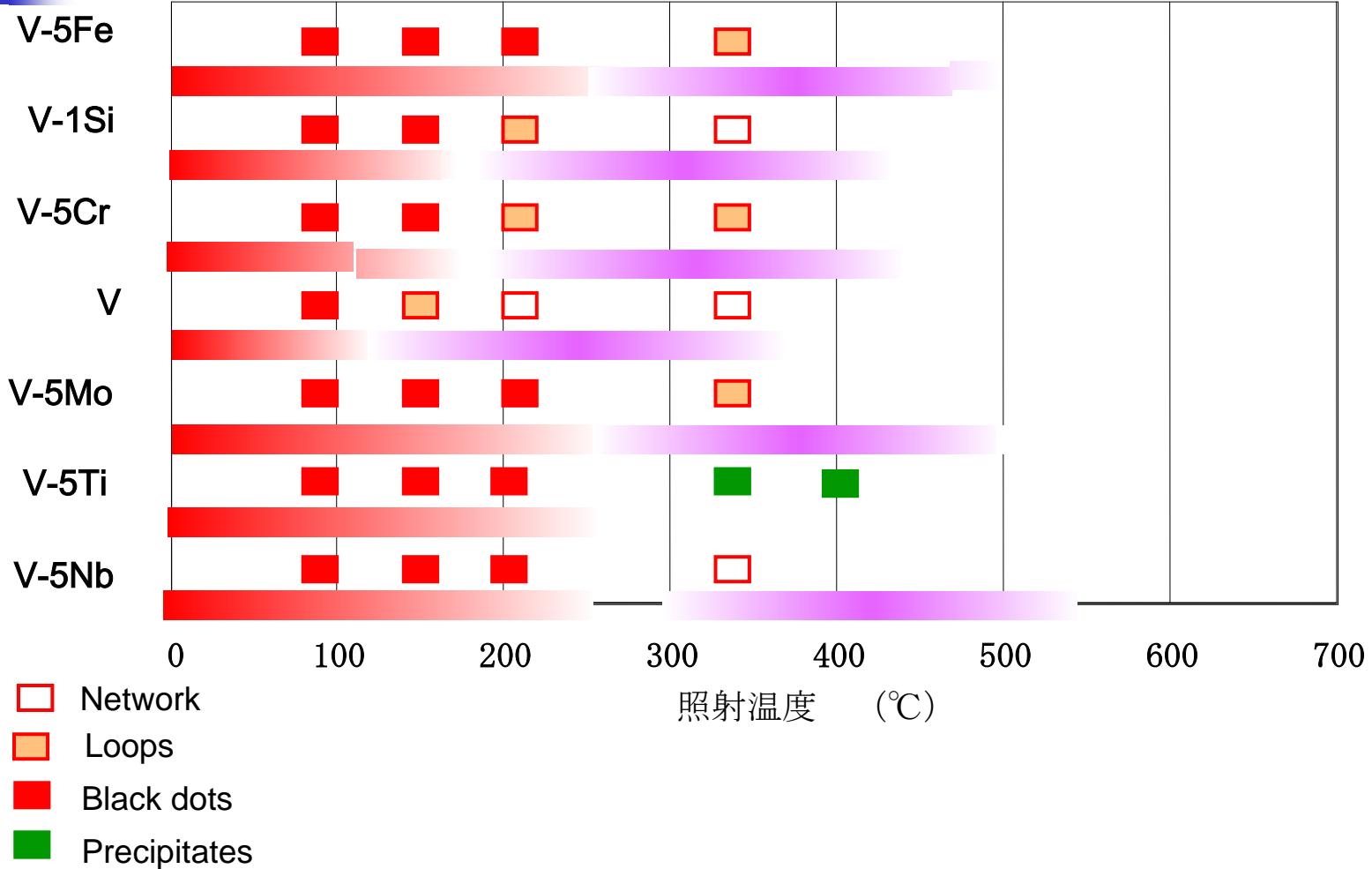


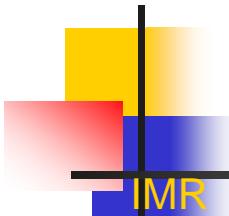
IMR

# Temp. Dependence of Dislocation Structure

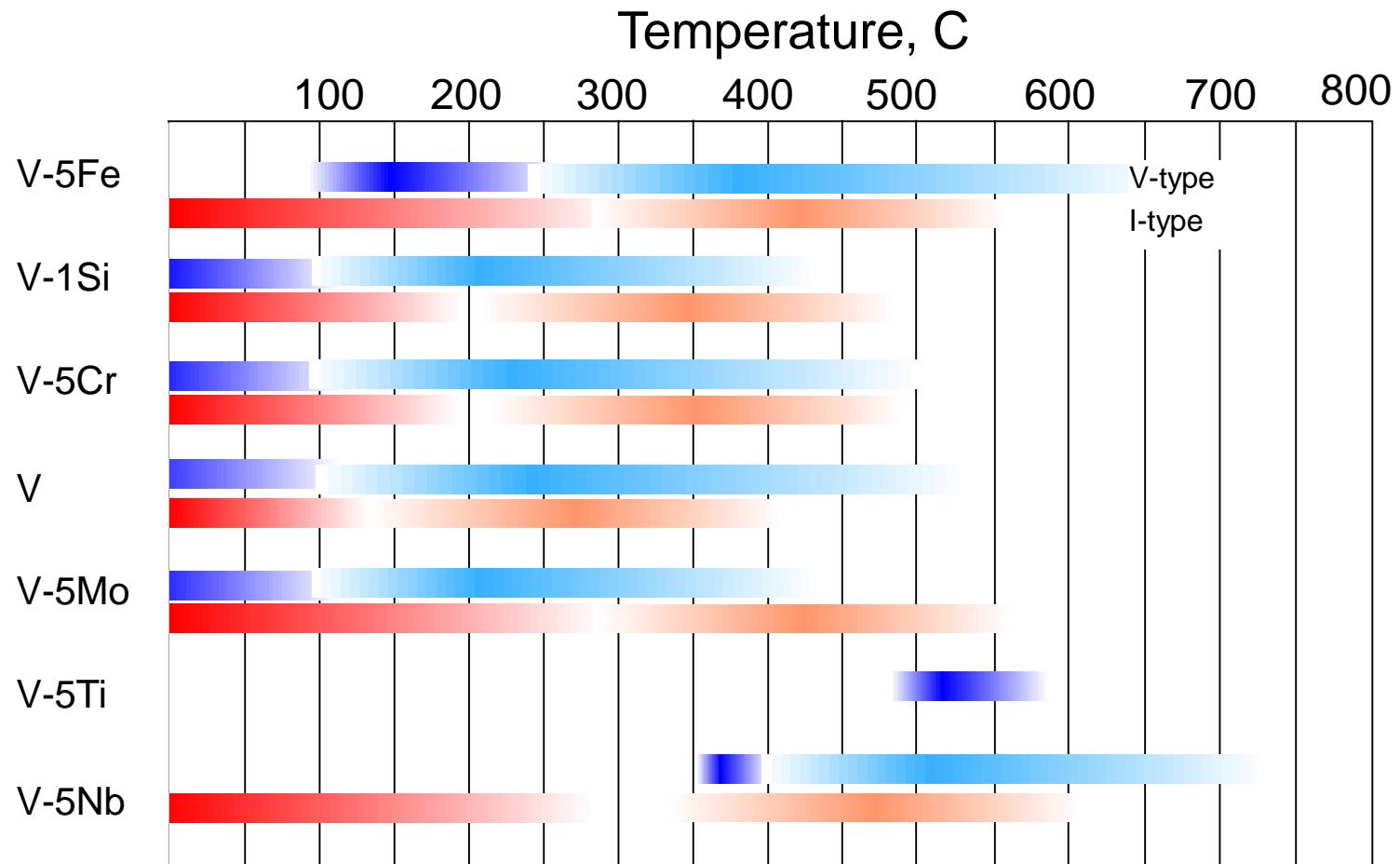


IMR





# Nucleation and Growth of Clusters

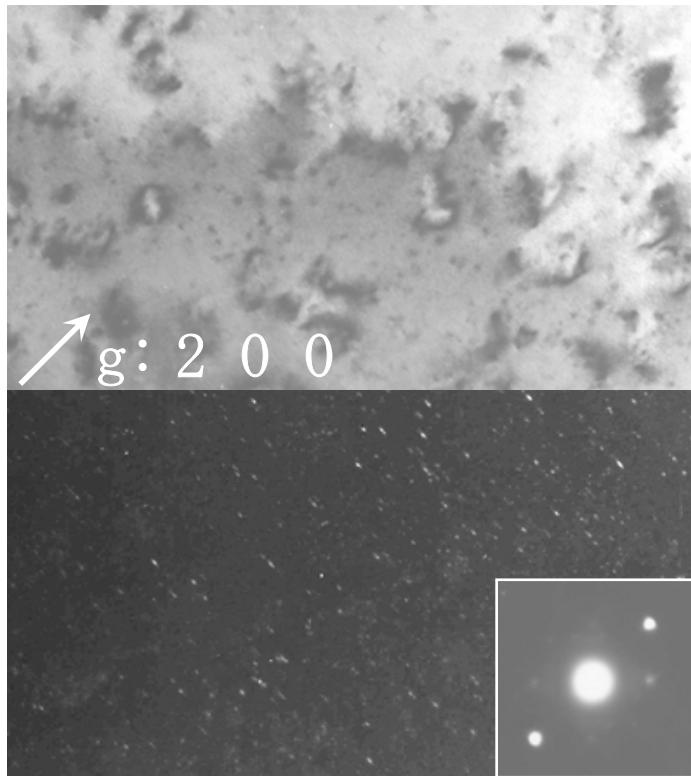


# Effects of Oxygen Reduction

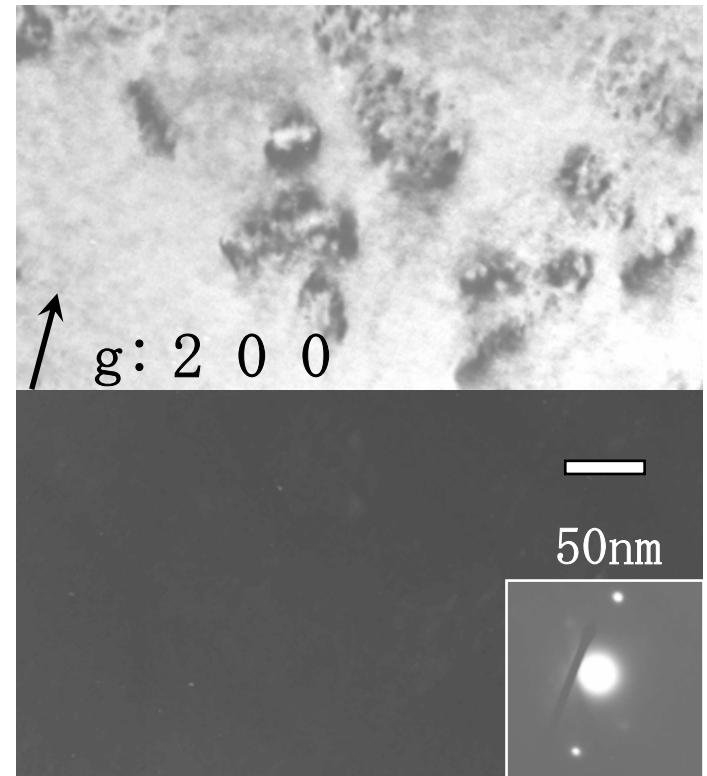
IMR

- Fine precipitates disappear by reducing oxygen

V-5%Ti  
400C, 1dpa



as-received ( $C_0=400\text{wppm}$ )



Zr-treated ( $C_0=33\text{wppm}$ )