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Ultra-High Temperature Ceramics: Materials For Extreme Environmental Applications II

Proceedings

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# Microstructures and thermal conductivities of hot-pressed ZrB2-SiC ceramics with a variety of SiC sources

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# Microstructures and Thermal Conductivities of Hot-pressed $ZrB_2$-SiC Ceramics with a Variety of SiC Sources

2012. 05. 17.

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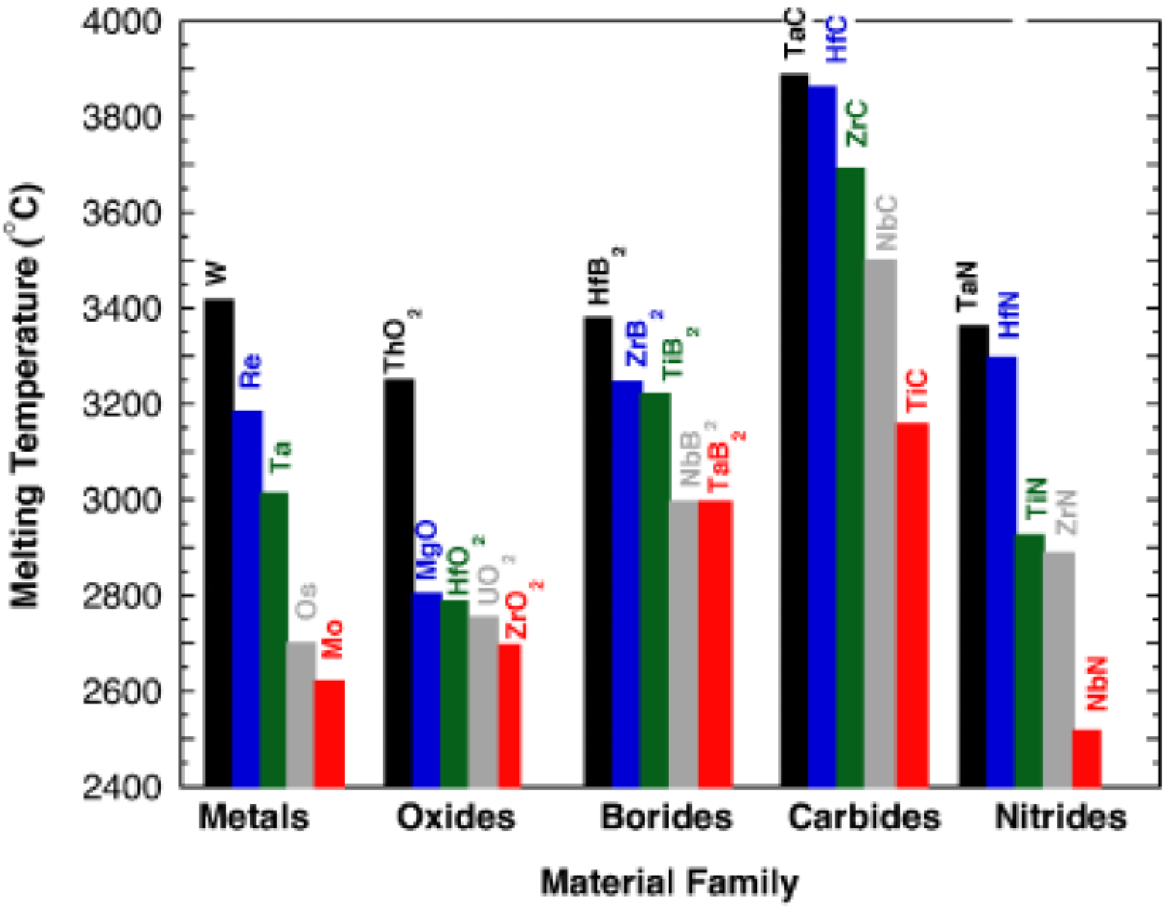
# Contents

- Introduction
- Objective and Experimental
- Results and Discussion
- Summary and Further Work

- Activities on UHTCs in Korea and KICET

# Examples of UHTCs

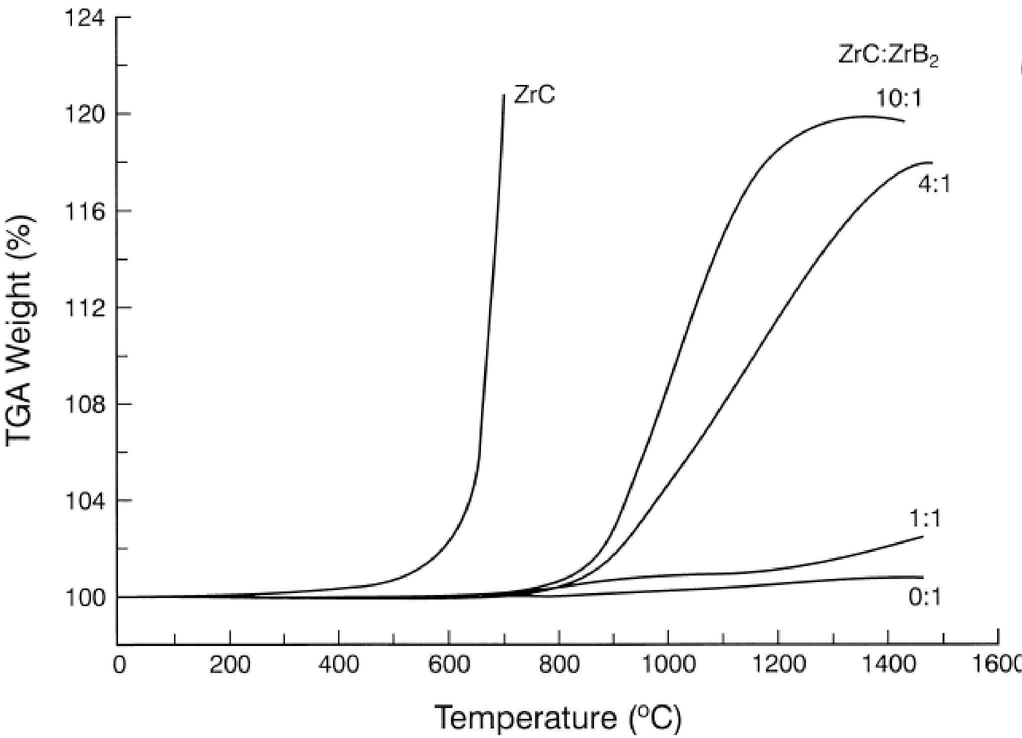


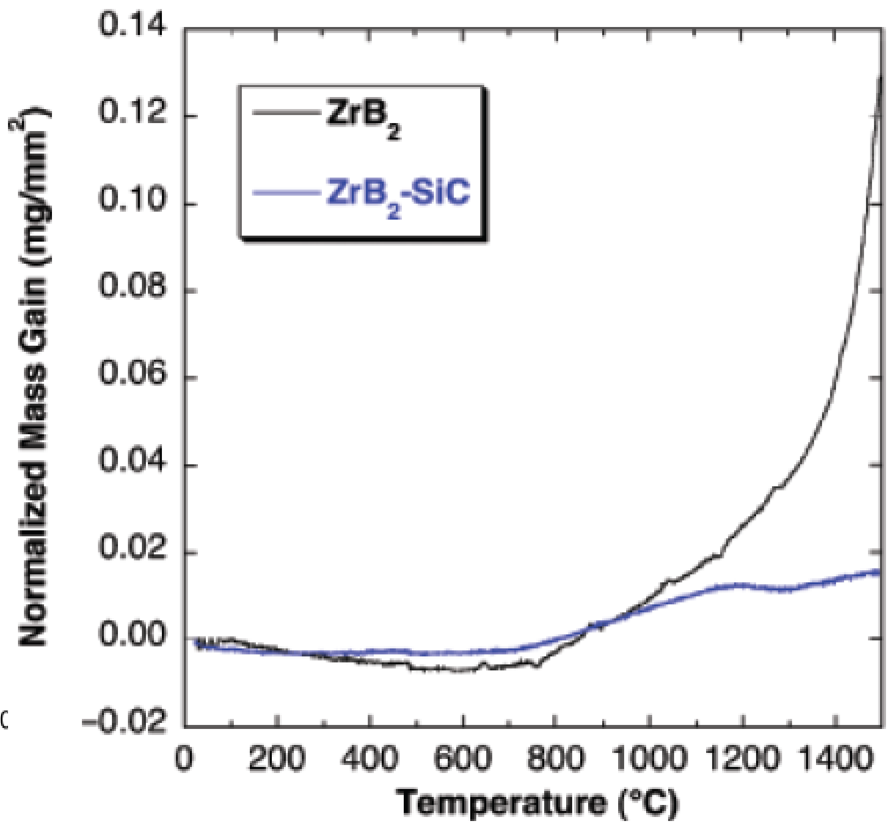

## Borides, carbides, nitrides of transition metals



* W.G. Fahrenholtz et al., J. Am. Ceram. Soc., 90 [5] (2007) 1347

# Oxidation Behavior of UHTCs



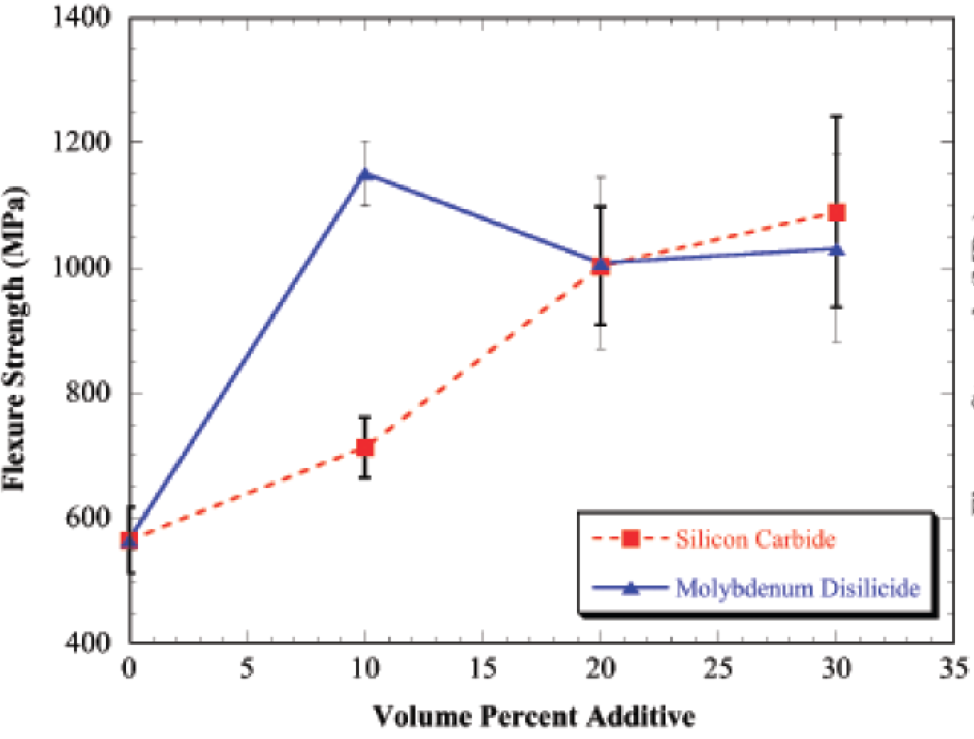

- ✓ Excellent oxidation resistance compared to carbides (nitrides)
  - → $B_2O_3$ passivation layer up to 1200°C
- ✓ Deterioration of oxidation resistance over 1200°C
  - → Addition of SiC

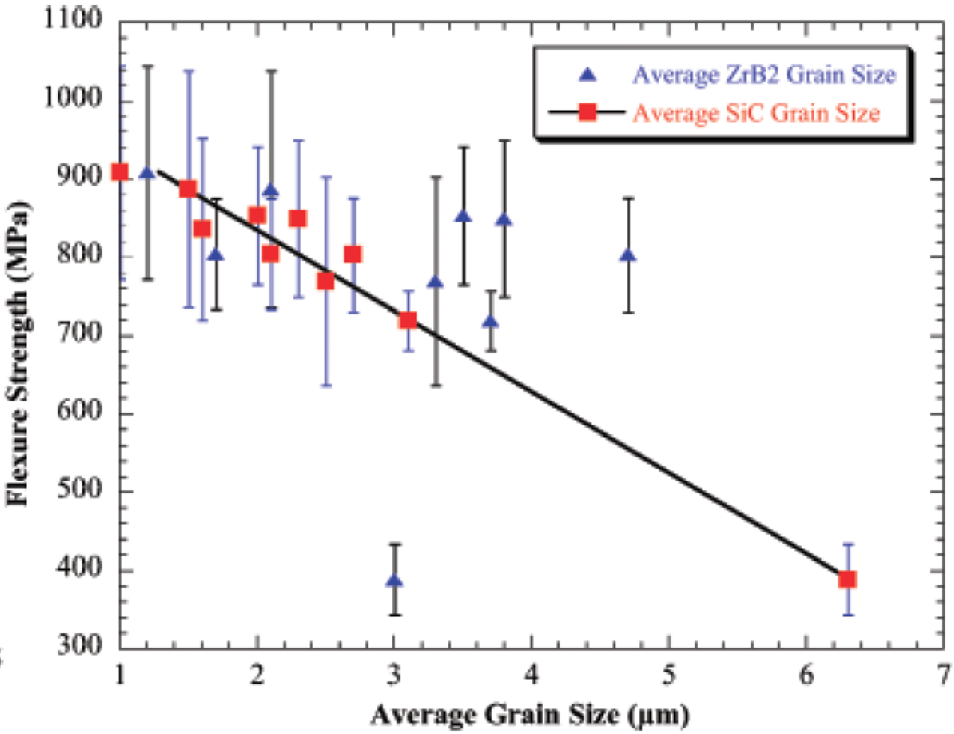
 * M. M. Opeka et al., JECerS,19 (1999) 2405

* W. G. Fahrenholtz et al., JACerS, 90 [5] (2007) 1347

# Mechanical Properties of $ZrB_2$-UHTCs

**with SiC addition** | **with grain size**



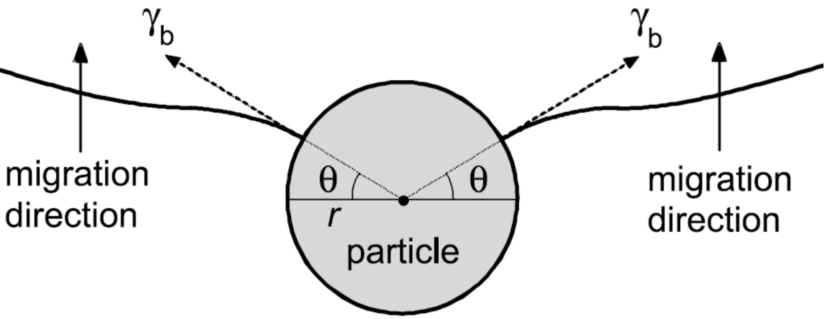




✓ Flexural strength of $ZrB_2$-based UHTCs increases with SiC addition or decrease in grain size.

* W .G. Fahrenholtz et al., J. Am. Ceram. Soc., 90 [5] (2007) 1347.

# Zener Effect : Grain Growth Inhibition

- **Grain growth Inhibition by second-phase particle**

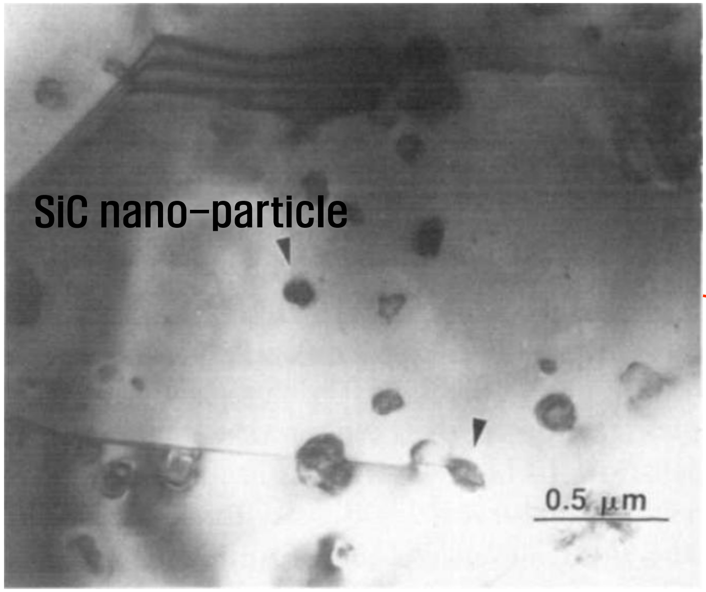


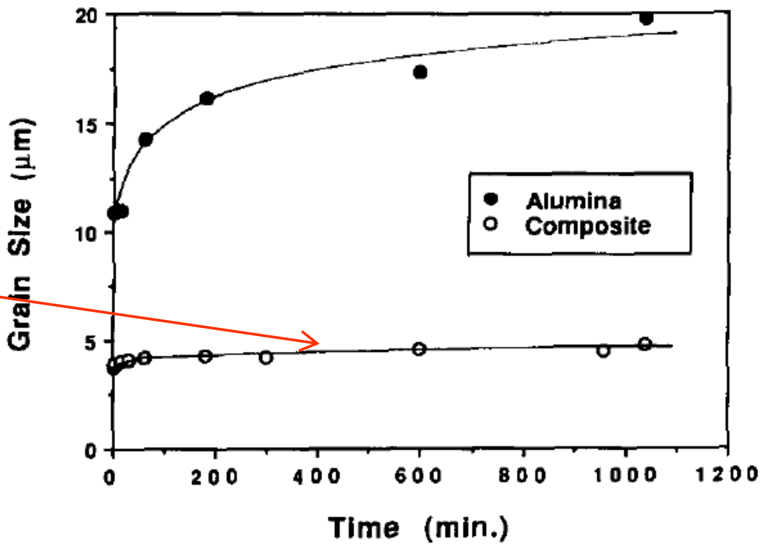

➢ Drag force of a particle against the grain boundary movement

$$F_d=\gamma_b \sin\theta \times 2\pi r \cos\theta=\pi r \gamma_b \sin 2\theta$$

* S.-J. L. Kang, Sintering; Densification, grain growth and microstructure (2005).

$Al_2O_3$-SiC 'Nanocomposites'

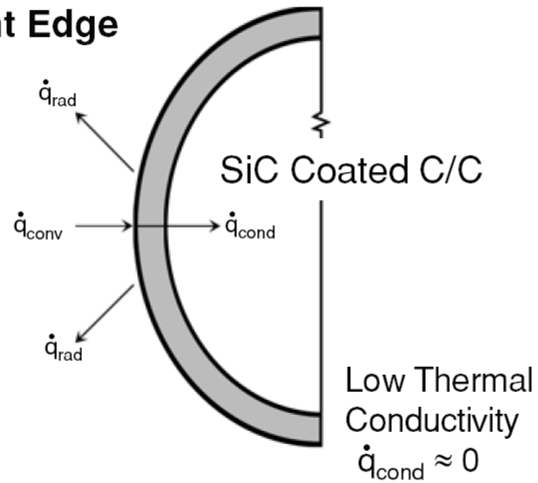



L. C. Stearns, et al, *J. Euro. Ceram. Soc., 10 (1992) 473.*

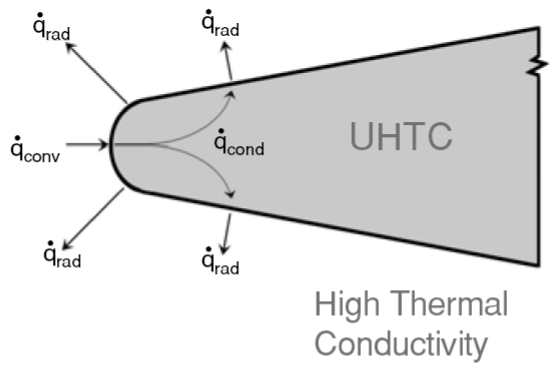
# Thermal Conductivities of UHTCs

**Blunt Edge**




$$\dot{q}_{conv} \approx \dot{q}_{rad}$$

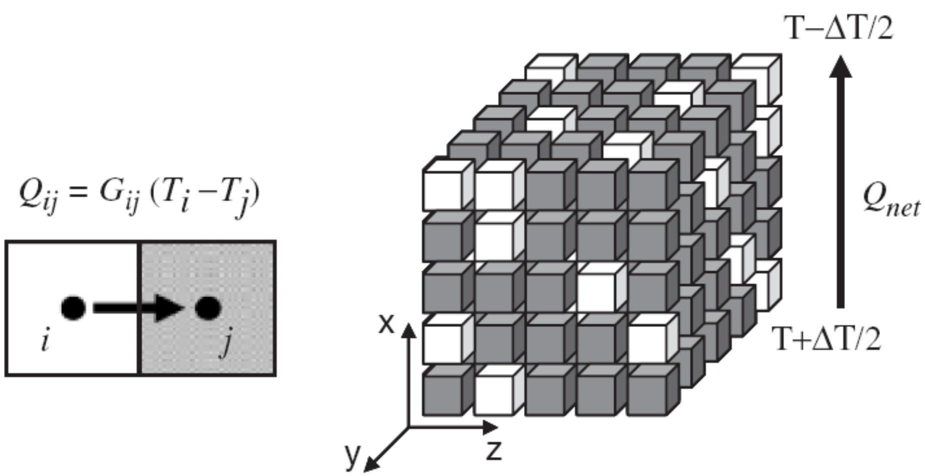
**Sharp Edge**




$$\dot{q}_{conv} = \dot{q}_{rad} + \dot{q}_{cond}$$

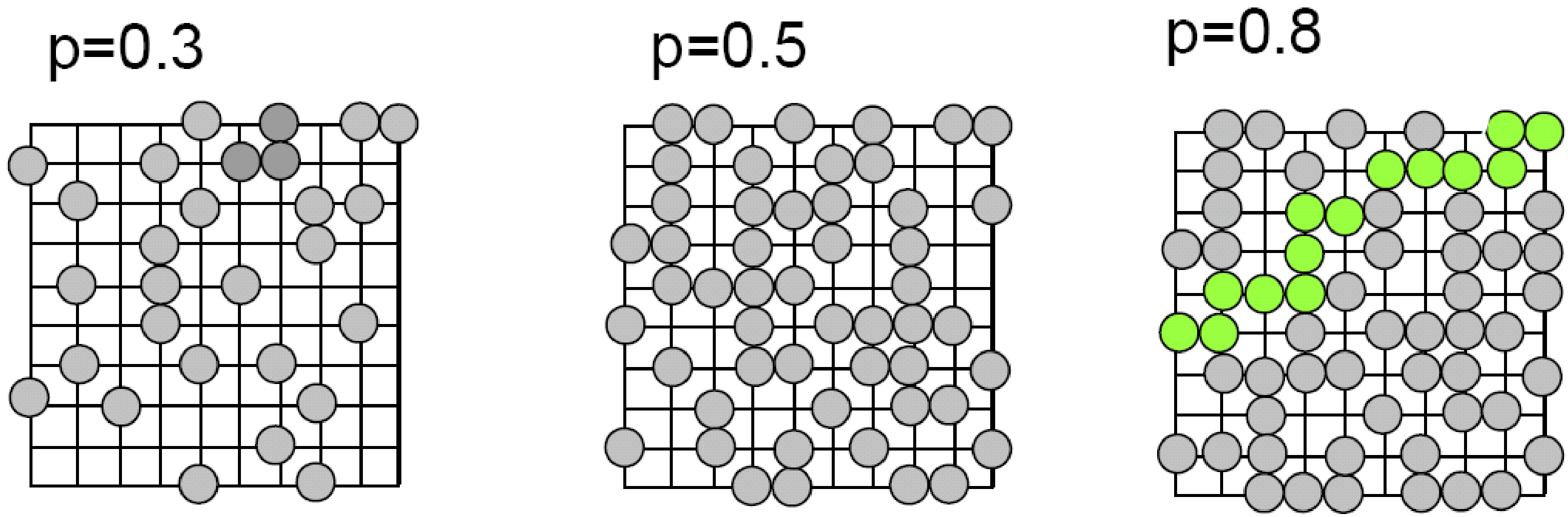
* Handbook of Ceramic Composites, Springer (2006)

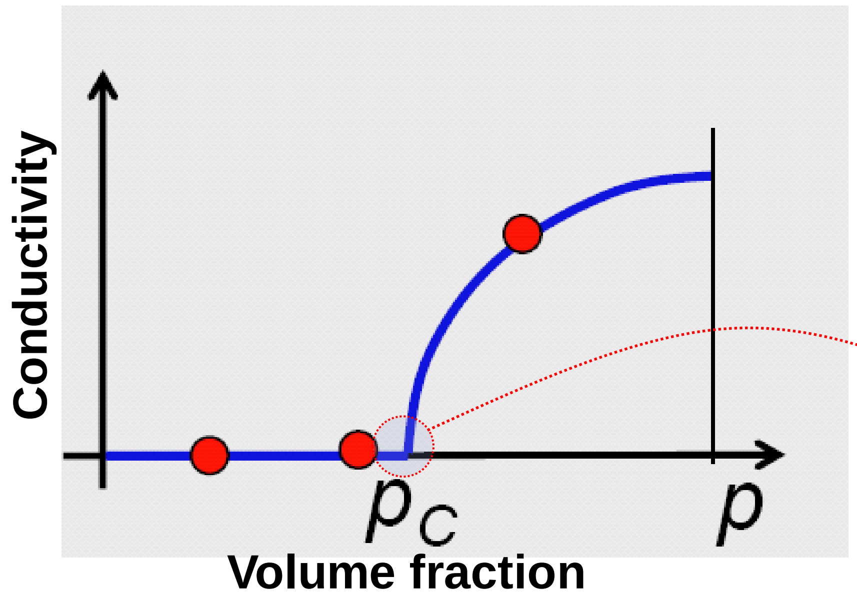
* A. Paul *et al,* Am. Ceram. Soc. Bull., **91**, (2012) 22.




* Gasch et al., J. Am. Ceram. Soc., 91 (2008) 1423.

# Percolation : Transport Properties




**$K_{ZrB2}$ : 60~80 W/mK**

**$K_{SiC}$ : 120~200 W/mK**

**Percolation threshold**

# Objective and Experimental

- **Objective**

: To examine the effect of SiC size on the microstructures and thermal properties of $ZrB_2$-SiC ceramics

- **Experimental**
  - ✓ **Composition : $ZrB_2$-x v/o SiC (x= 0, 10, 20, 30)**
  - ✓ **Powder : $ZrB_2$ (1.88㎛, Japan New Metals Co. Ltd., Japan)**

    **SiC (0.5㎛, SIKA Tech., Germany)**

    **Nano-SiC (<100㎚, SIGMA-ALDRICH, Germany)**

    **PCS (Polycarbosilane, TBM tech, Korea)**
  - ✓ **Mixing : Ball mill with SiC-ball for 24hrs**
  - ✓ **Sintering : hot pressing (1900°C/2hrs)**

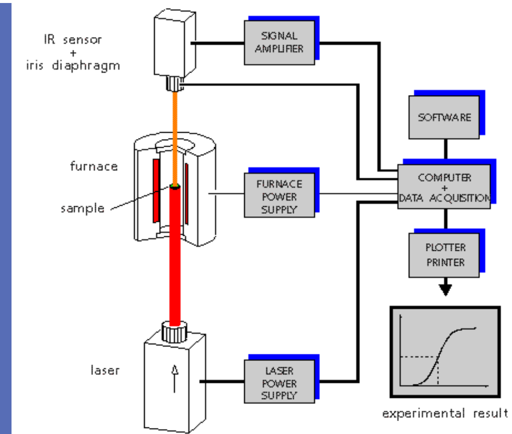
# Thermal Conductivity Measurement

$$\kappa(T) = \rho \cdot C_p(T) \cdot \lambda(T)$$

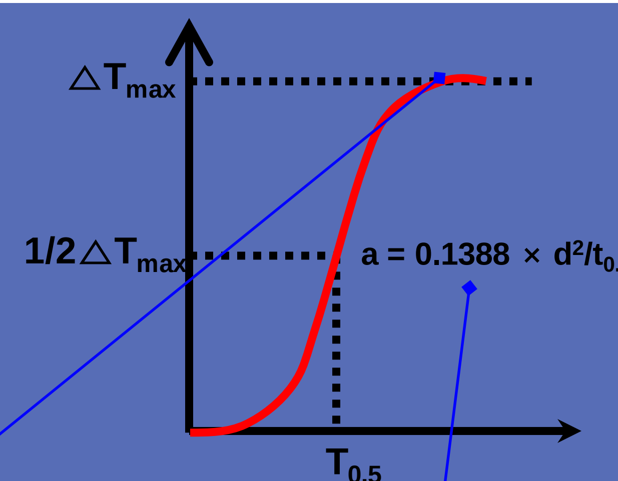
**ρ** : Apparent density

$C_p$ : Specific heat capacity

**λ** : Thermal diffusivity

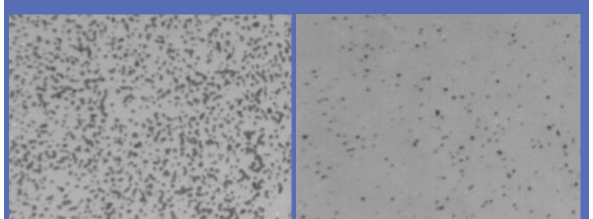



**Laser flash analysis**




- **Density**

$$k_{true} = \frac{k_{effective}}{\left(1 - \frac{4}{3}\phi\right)}$$



- **Specific heat capacity**

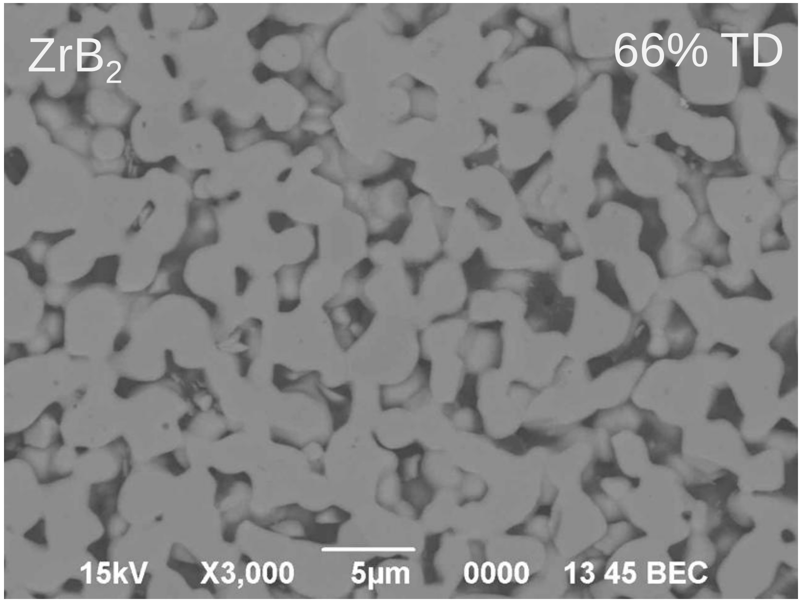
$$C_p = \left(\frac{\partial E}{\partial T}\right)_p$$

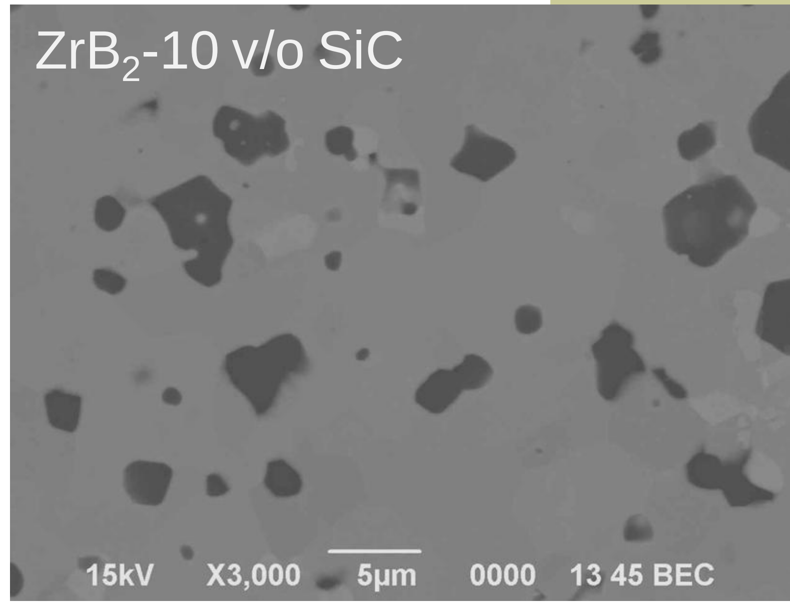
- **Thermal diffusivity**

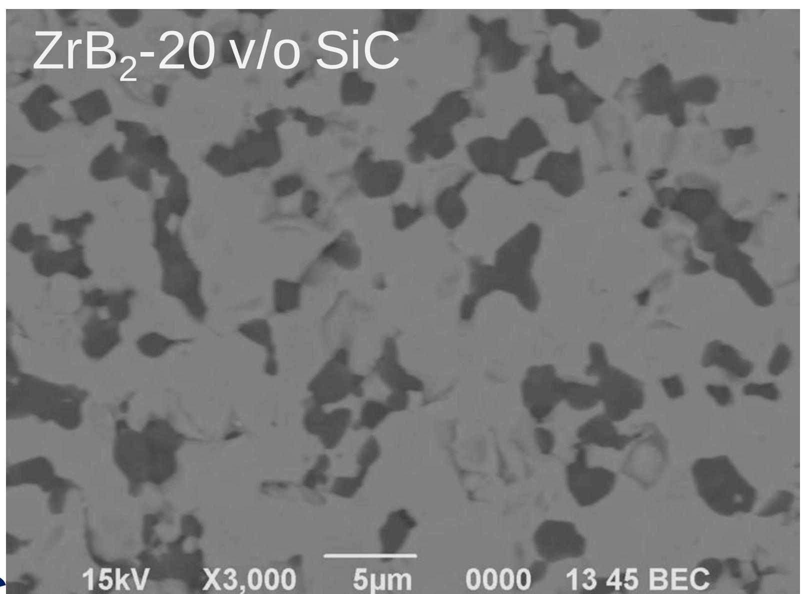
$$a = 0.1388 \times d^2/t_{0.5}$$

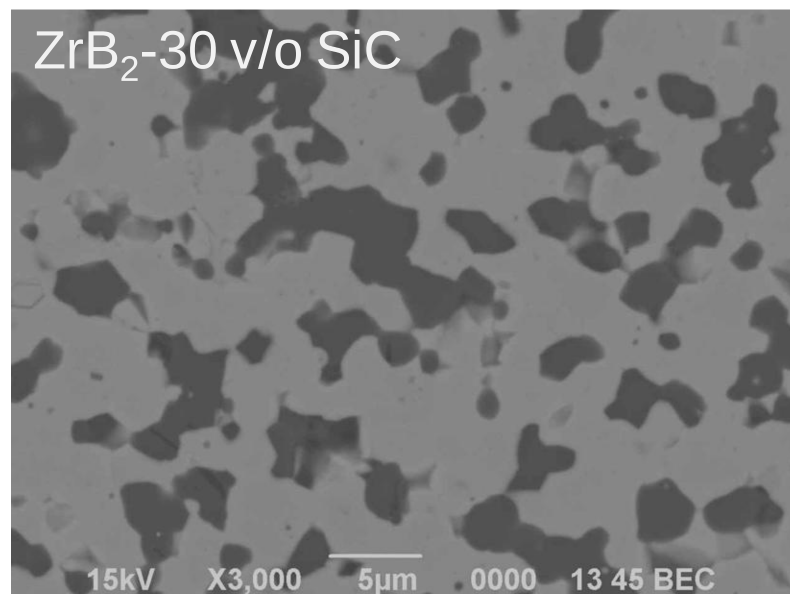
**a** : Temperature diffusivity ($cm^2/s$)
**d** : Thickness of the test piece(mm)
$T_{1/2}$ : Time 50% of the temperature increase, measured a t the rear of the test piece (s)

# Microstructure : Hot-pressed (1900℃/2h)



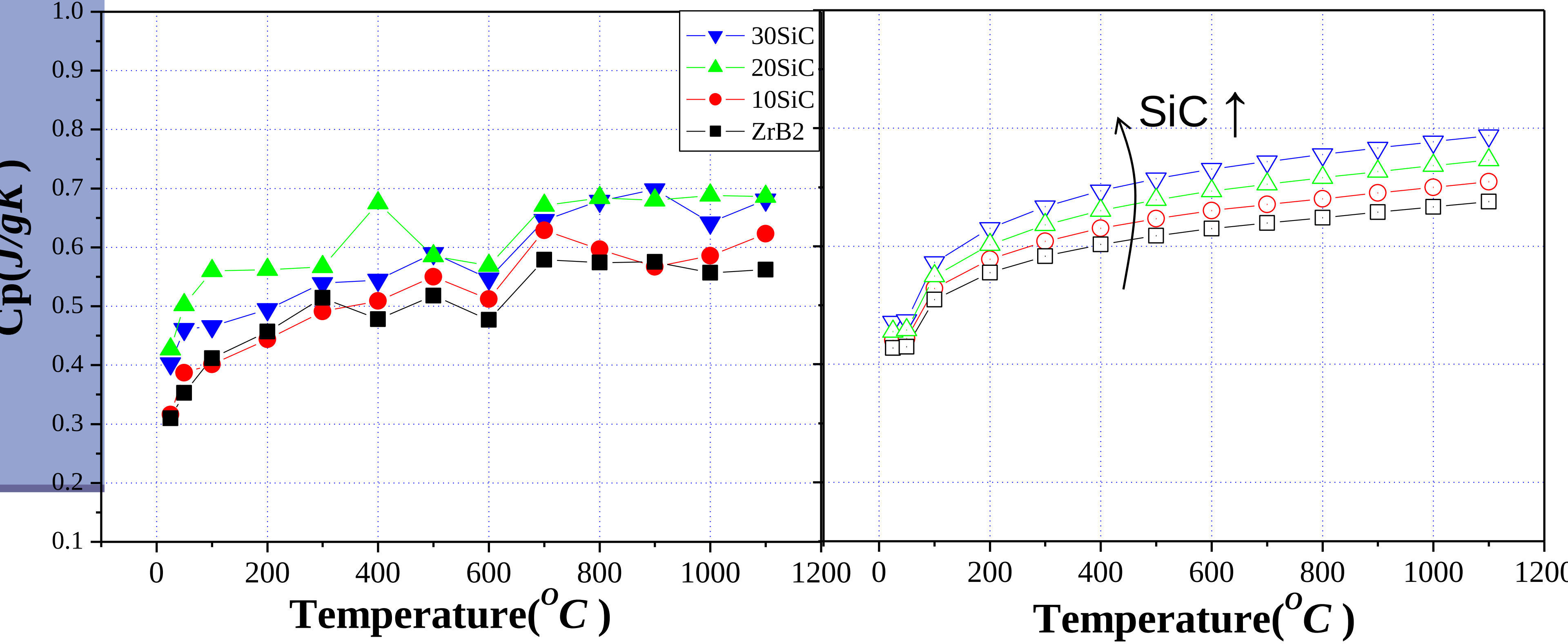



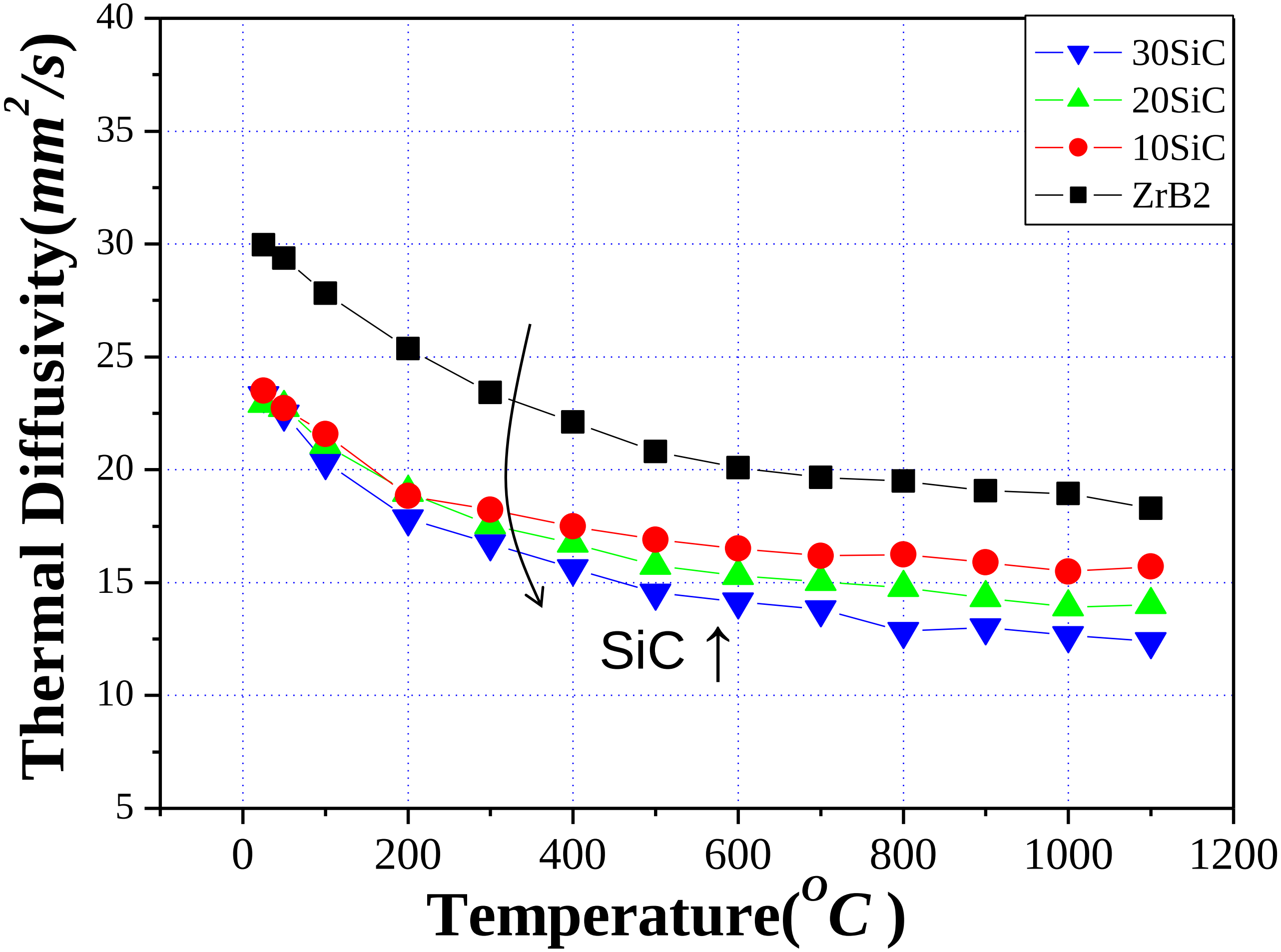
# Specific Heat Capacities

LFA measured

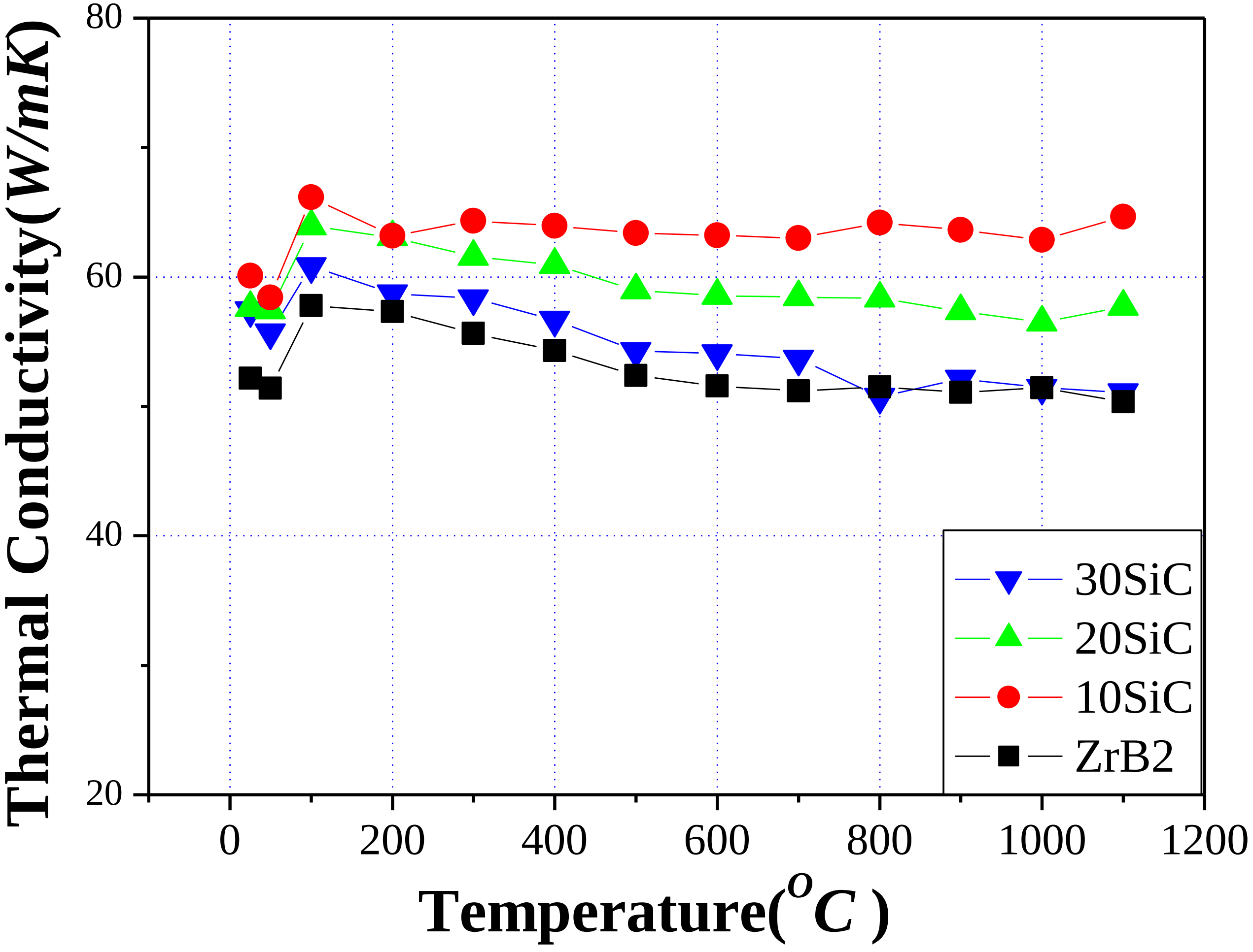
NIST-JANAF



# Thermal Diffusivities

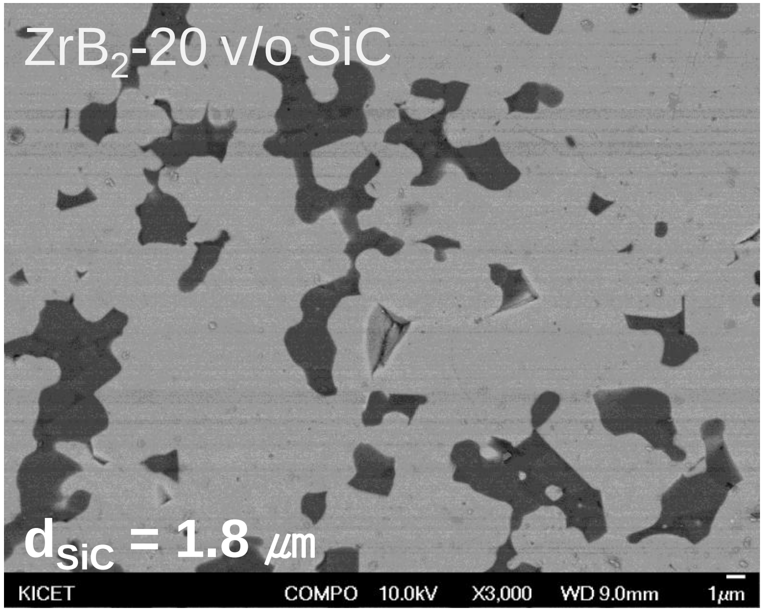


# Thermal Conductivities



2nd Set

# Microstructure : Hot-Pressed (1900℃/2hrs)

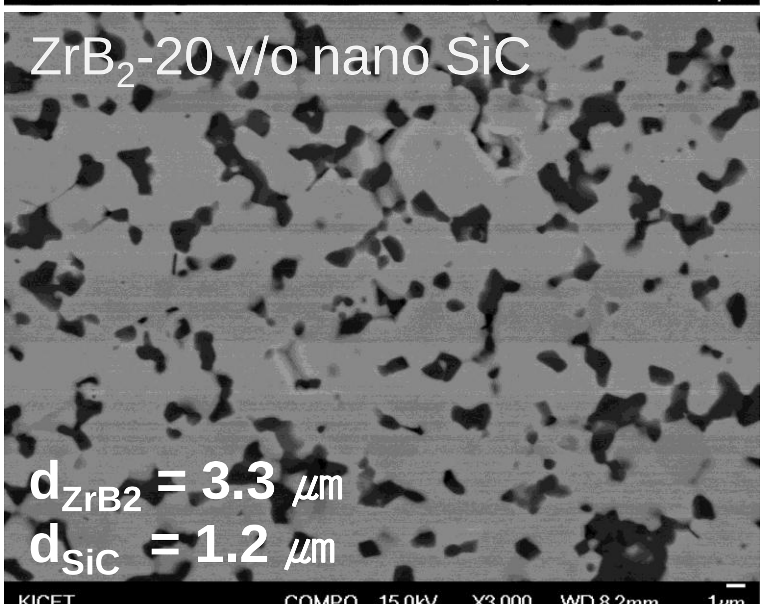


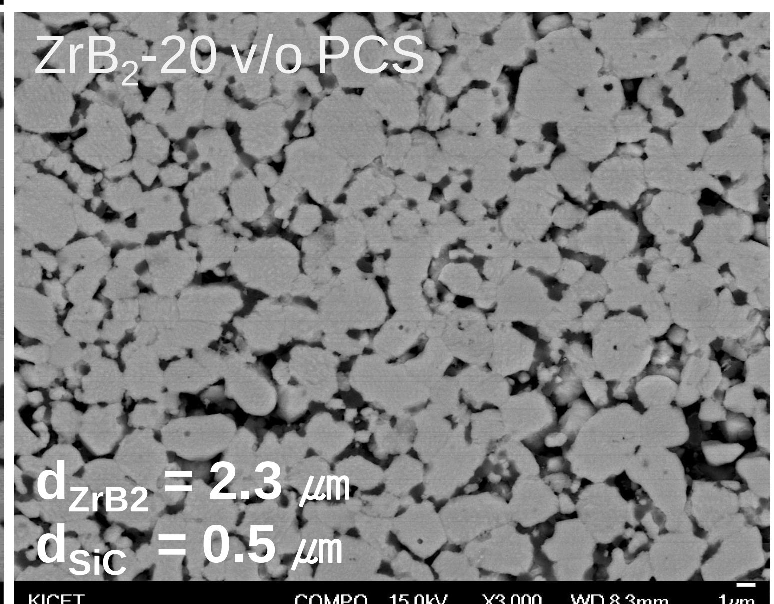

$ZrB_2$ (1.88㎛)

SiC (0.5㎛), nano-SiC(<100㎚)

polycarbosilane

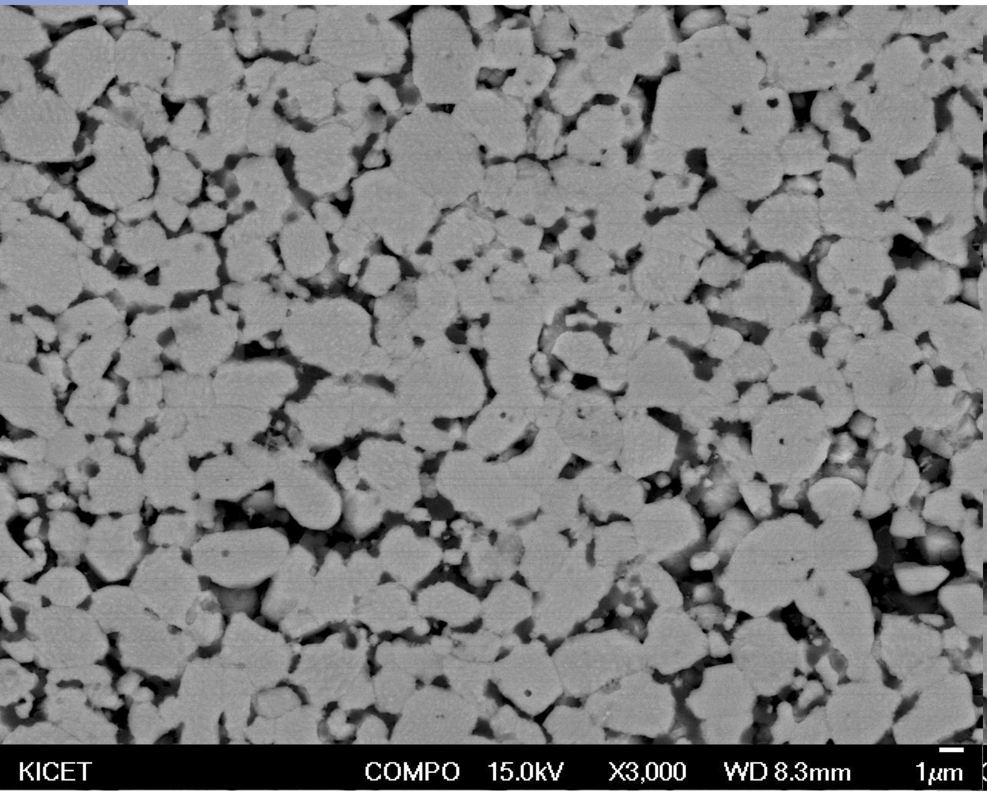
1900°C/2hrs hot pressing

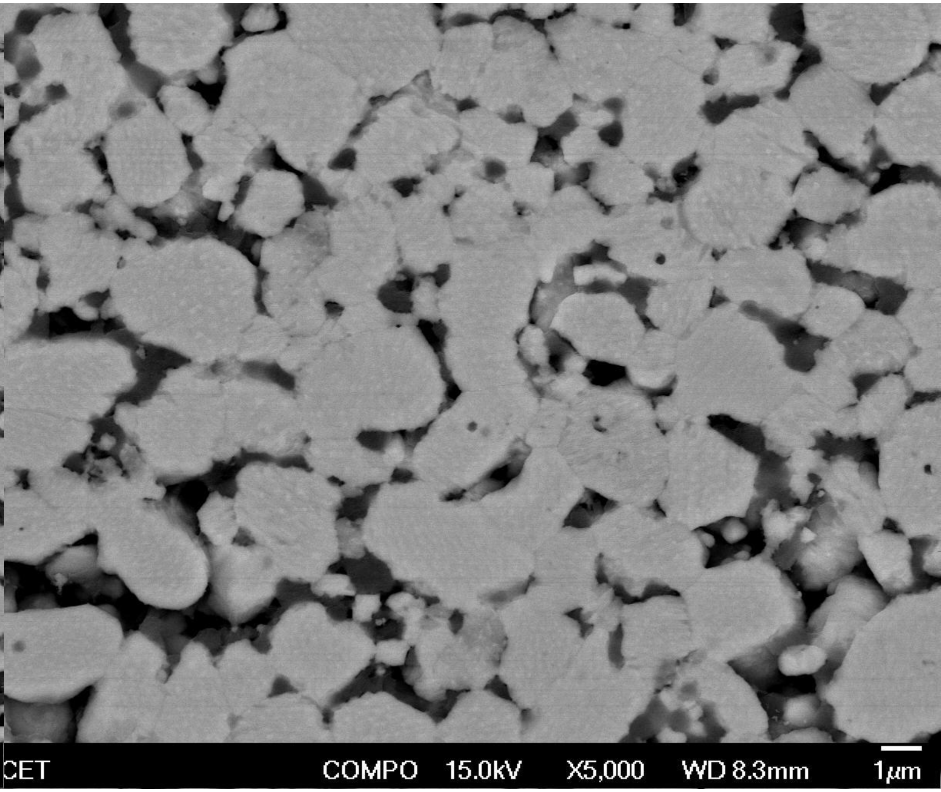


2nd Set

# Microstructure : $ZrB_2$-20 v/o PCS

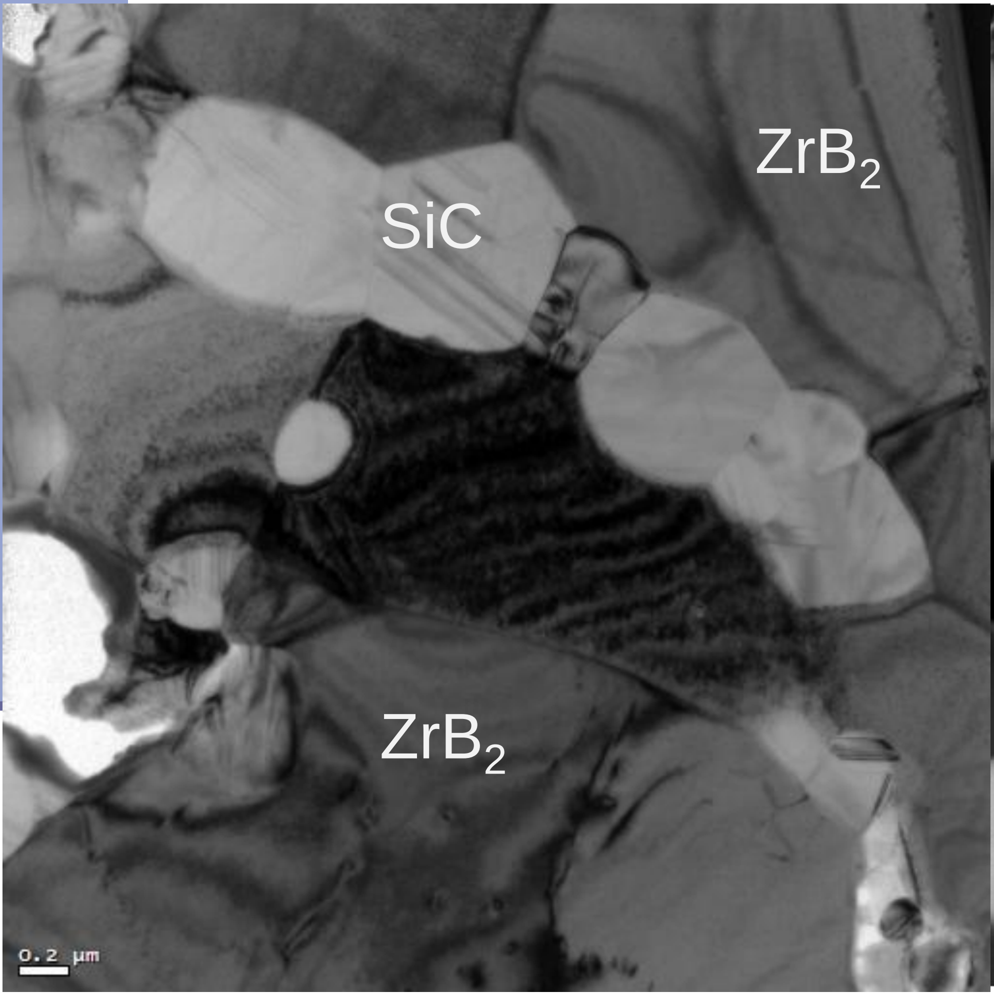


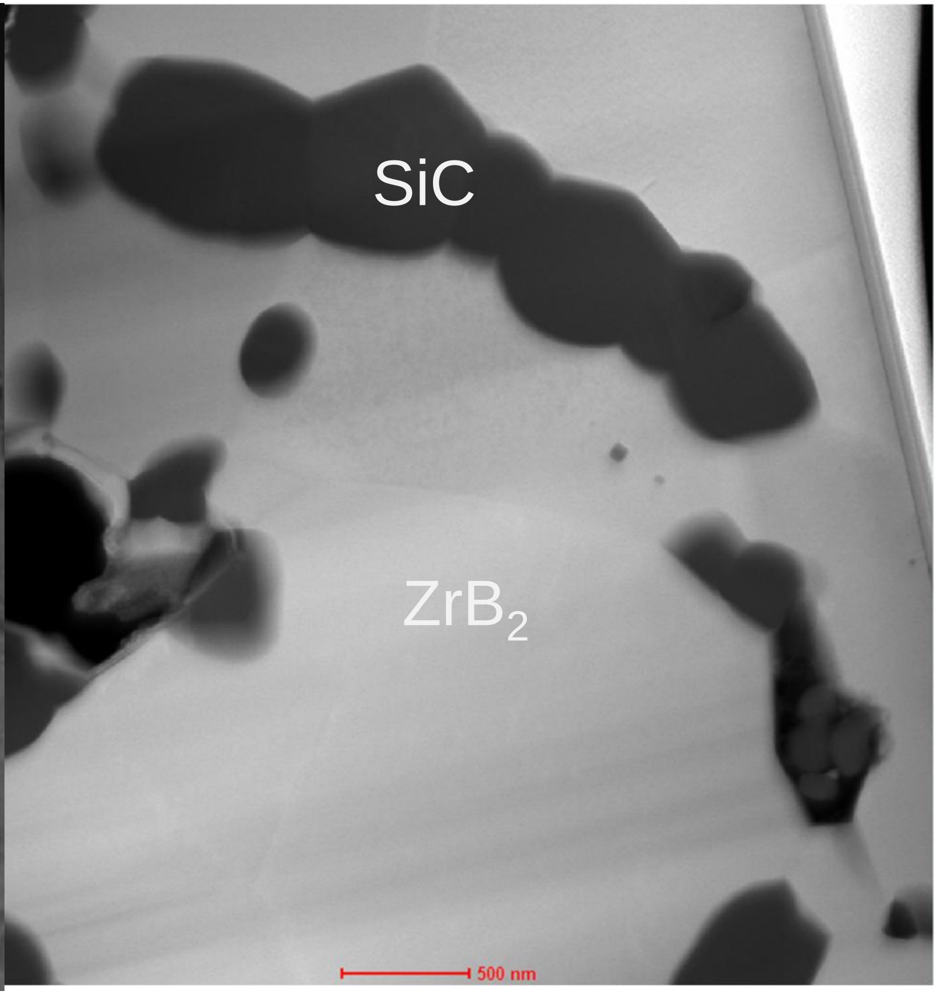




## Hot-pressed (1900℃/2hrs)
## (direct HP with holding at 1400℃/1hr)

2nd Set

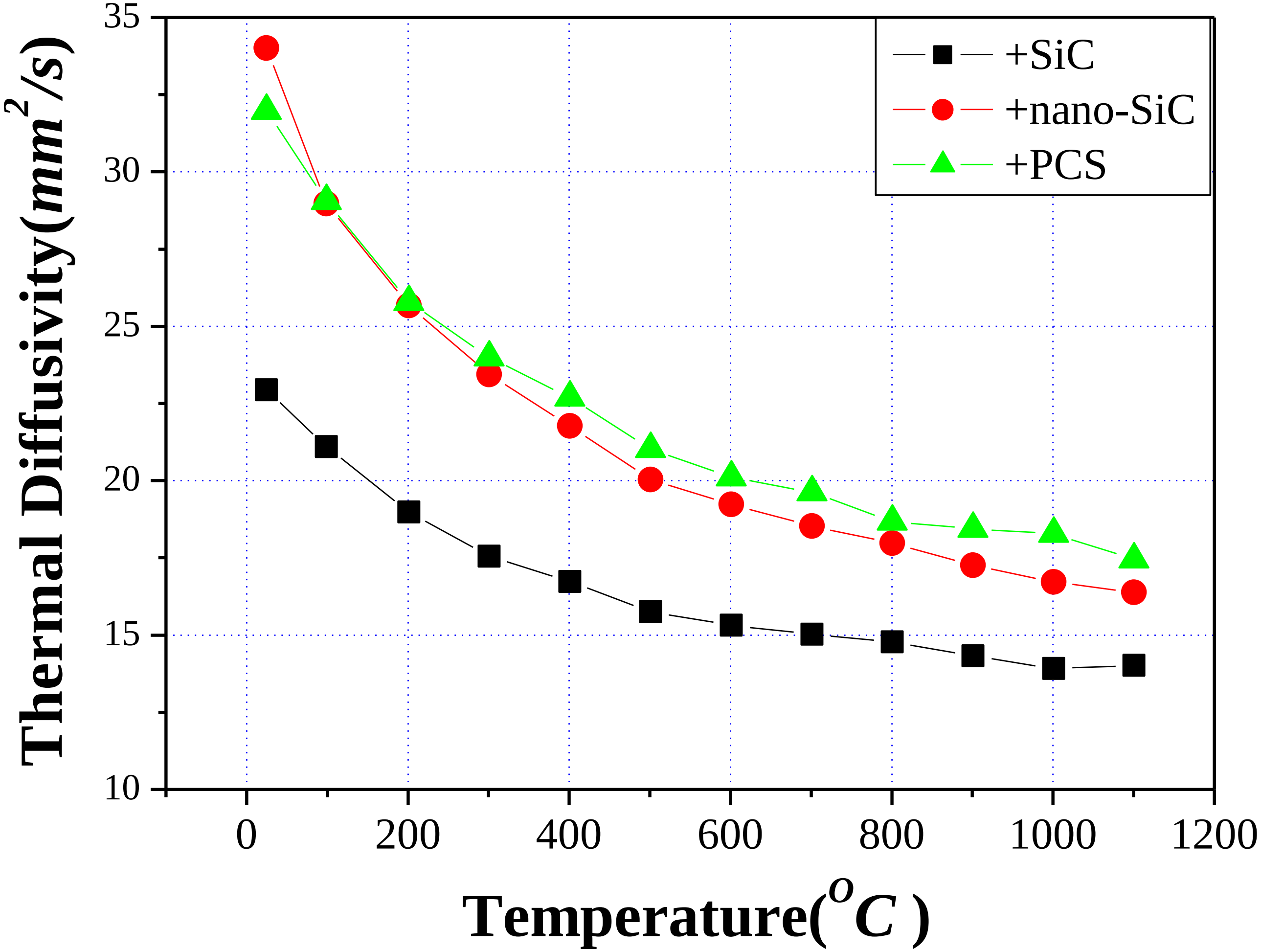
# TEM Microstructure : $ZrB_2$-PCS, Hot-pressed



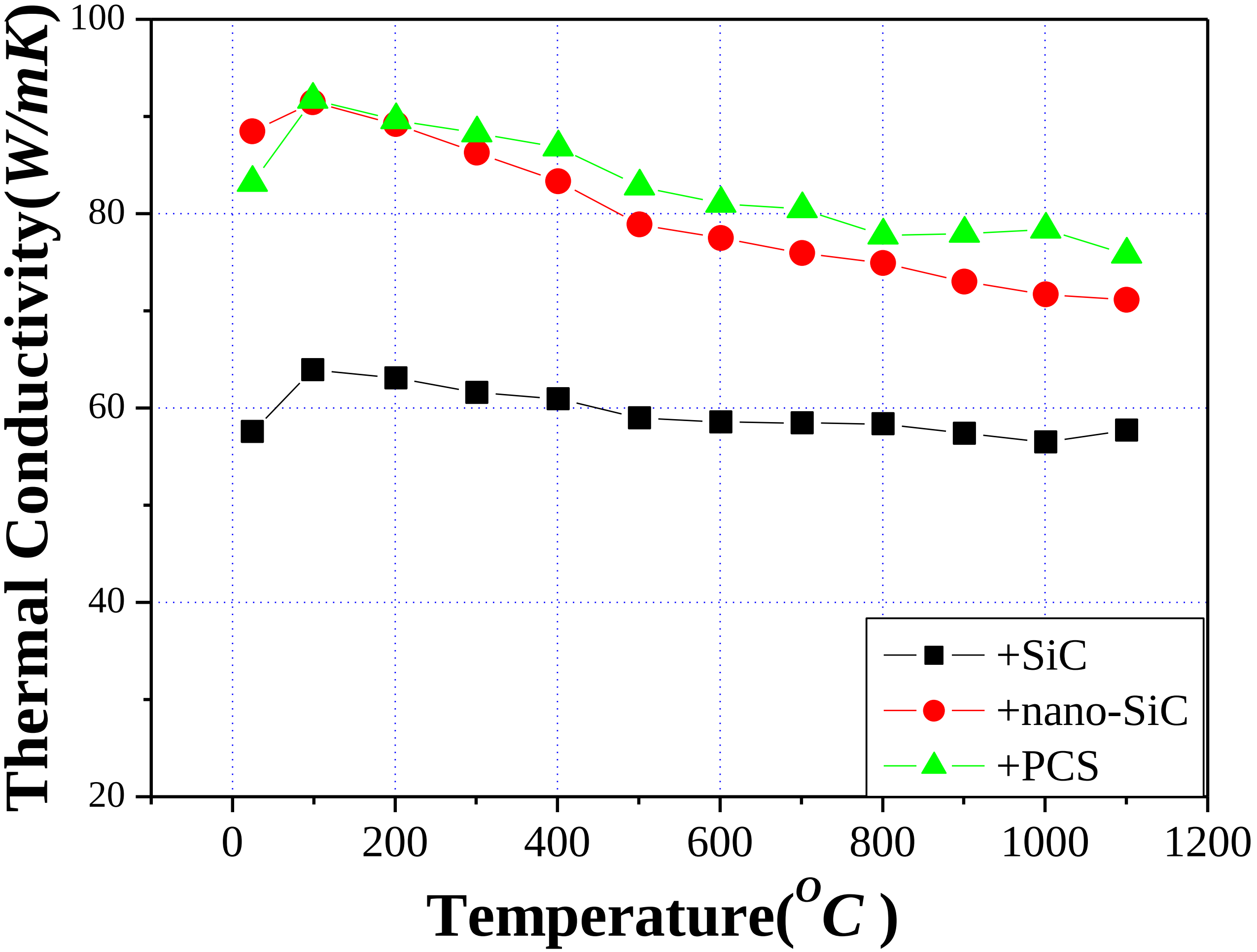



2nd Set

# Thermal Diffusivities



# Thermal Conductivities



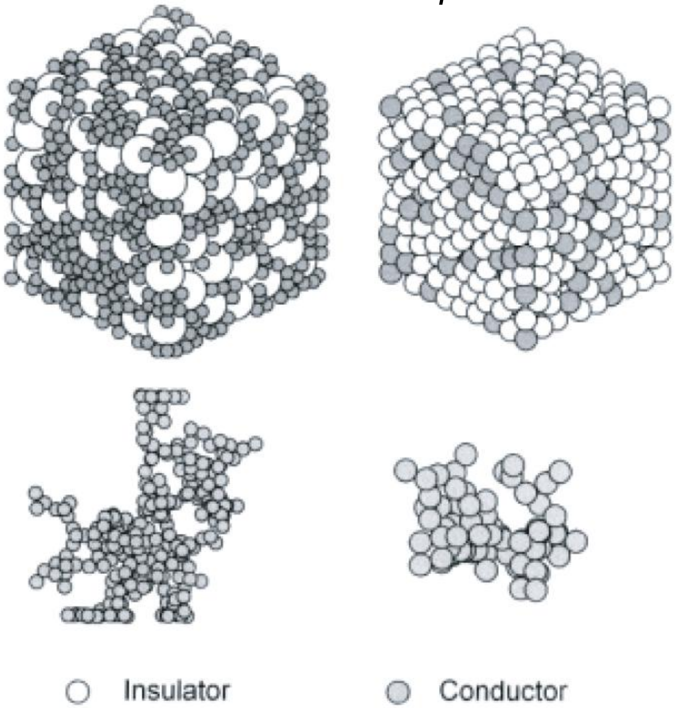
# Percolation Threshold

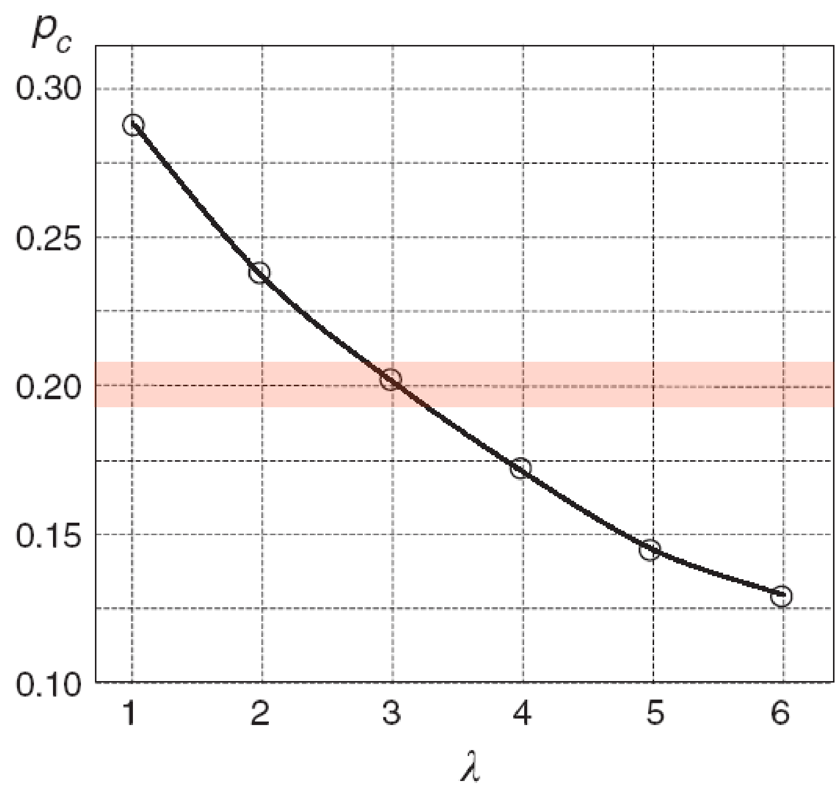
particle size ratio of the insulating to conductive powder

$$\lambda = \frac{d_i}{d_c}$$

$\lambda = 3$
$n = 1500$
$n_c = 1312$
$p = 0.205$

$\lambda = 1$
$n = 1000$
$n_c = 288$
$p = 0.288$




**particle size ratio ↑ → percolation threshold↓**

* D.He and N. N. Ekere, J. Phys. D: Appl. Phys. 37 (2004) 1848.

# Summary and Further Work

- ✓ The effect of SiC size on the microstructures and thermal conductivities of $ZrB_2$-SiC ceramics were examined.
- ✓ Nano-SiC or PCS addition inhibited grain growth of $ZrB_2$.
- ✓ $ZrB_2$-SiC ceramics with growth-inhibited grains showed enhanced thermal conductivities.
- ✓ Optimization of SiC dispersion needs further investigation.

# Thank you