# Fracture toughness of FCC High Entropy alloy by IIT

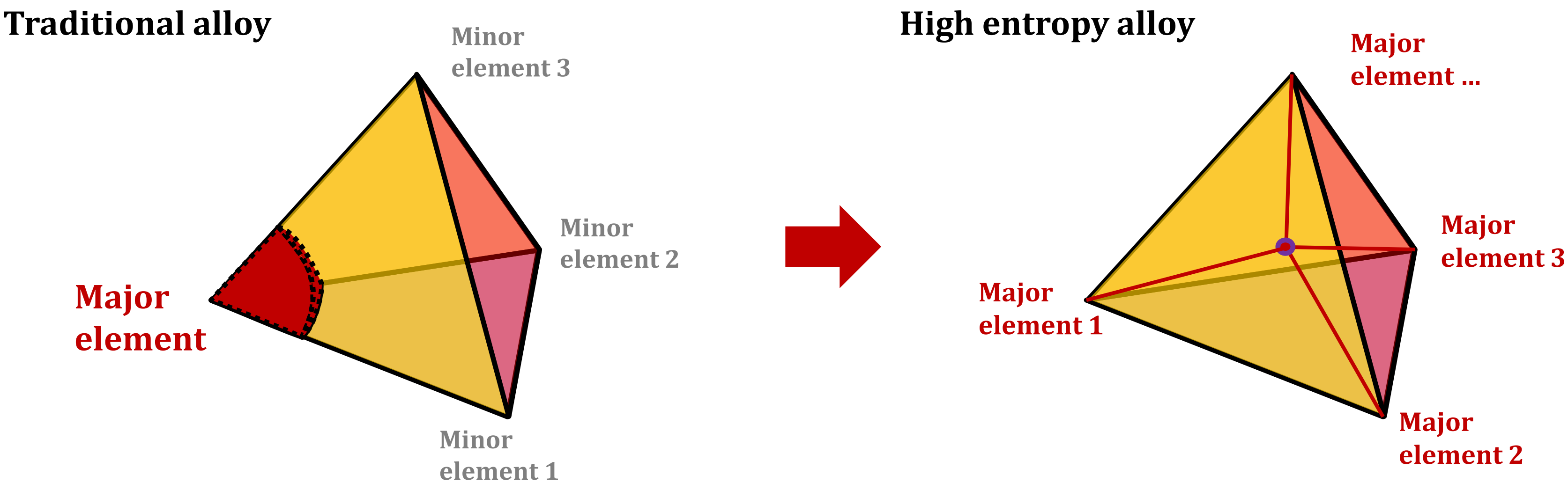
Current Status of Structural Materials

- Final presentation -

2018.06.20

원 종 호, 안 혜 상

# High Entropy Alloy




$$\textit{number of elements} \uparrow \leftrightarrow \textit{configurational entropy} \uparrow$$

$$\Delta S_{config.} = Rln(n)$$

*Y.Zhang et al., Prog.Mat.Sci. 61, 1 (2014)*

(1) Thermodynamic : high entropy

(2) Structure : severe lattice distortion

(3) Kinetics : sluggish diffusion

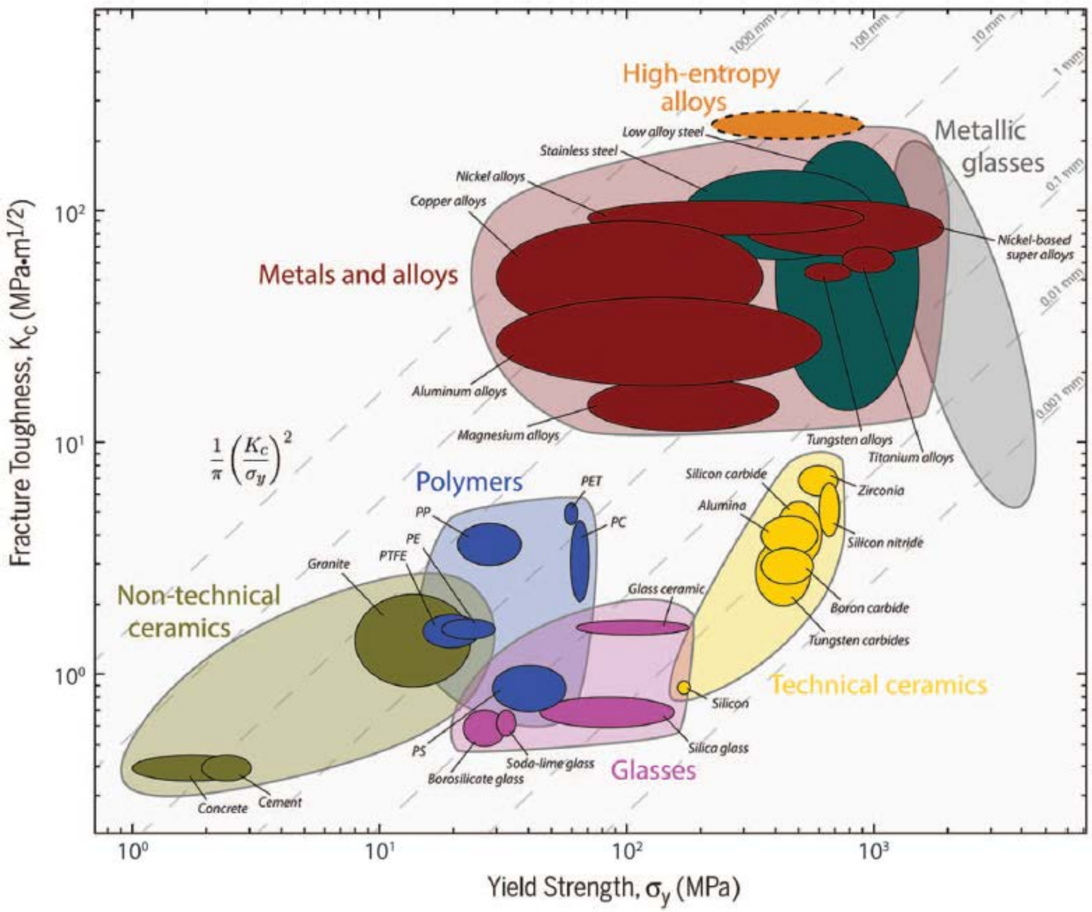
(4) Property : cocktail effect

**High entropy alloy**
**: Potential candidate material for extreme environment applications**

# Mechanical properties of HEA

- **Ashby map**

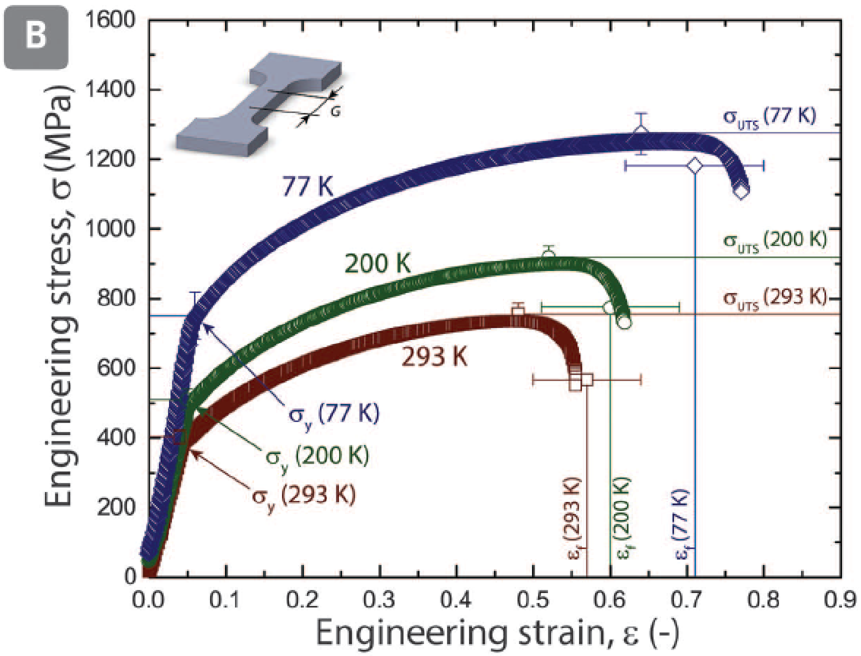
*Bernd Gludovatz / SCIENCE VOL 345 ISSUE 6201*

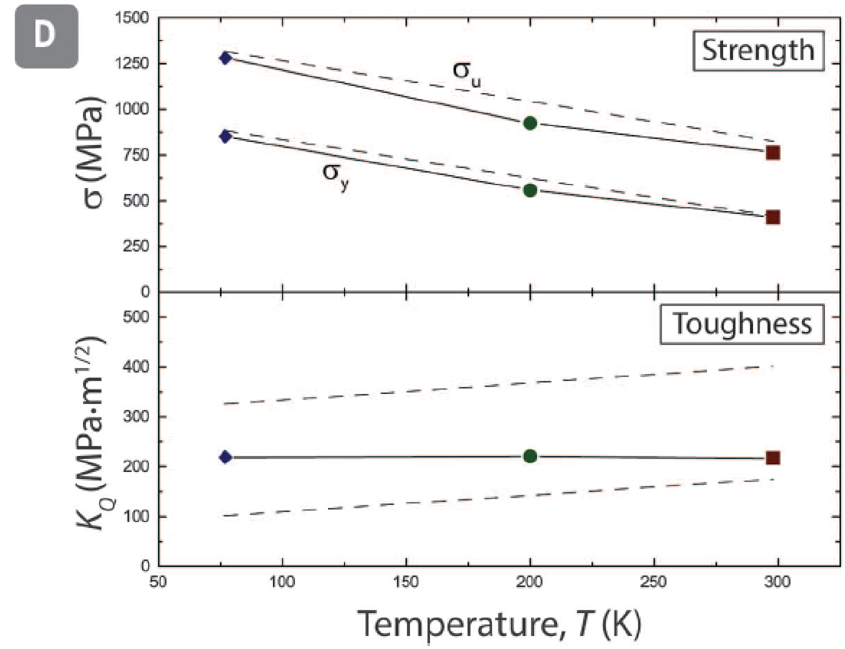



There are clearly stronger materials, which is understandable given that CrMnFeCoNi is a single-phase material, but the **toughness of this high-entropy alloy exceeds that of virtually all pure metals and metallic alloys.**

# Fracture toughness of HEA

*Bernd Gludovatz / SCIENCE VOL 345 ISSUE 6201*



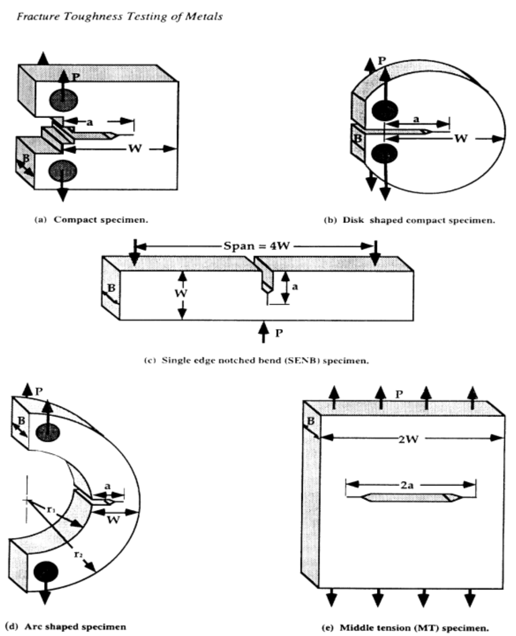




Although the toughness of the other materials decreases with decreasing temperature, the toughness of the high-entropy alloy remains unchanged.

**mechanical properties actually improve at cryogenic temperatures**

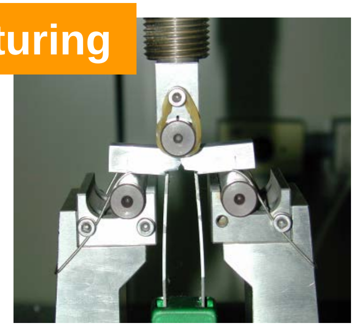
# Fracture Toughness Testing

## Compact tension test & Three point bending test




**Pre-cracking**

**Fracturing**



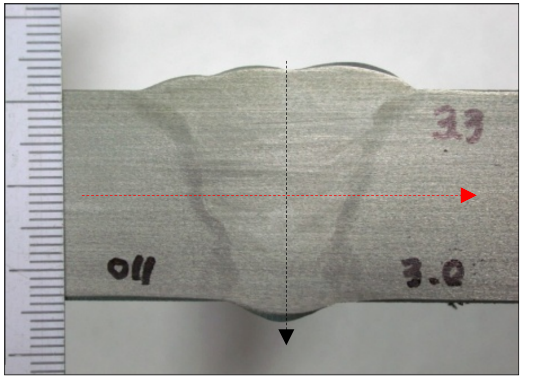
### Limitation

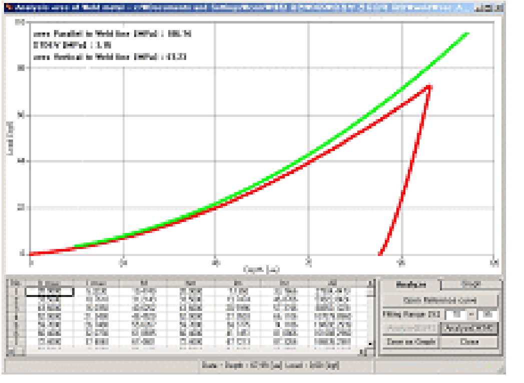
- **Destructive**
- **Complex testing procedure**
- **Cannot apply to in-service**

➡ **Development of testing method to measure the fracture properties more easily and more economically**

# Indentation Fracture Toughness

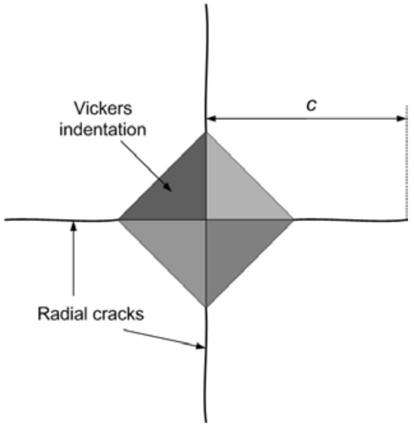
- **Instrumented Indentation Technique (IIT)**

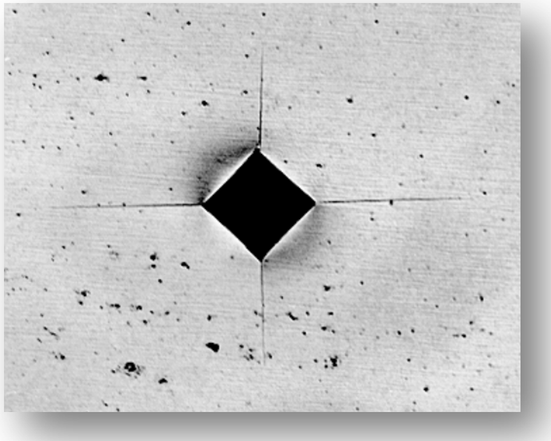




**➔ In-situ & In-field System, Non-destructive & Local test, Simple & fast**

- **Indentation Cracking Method (cracking in indentation)**



[Vickers indenter, Macroscale]

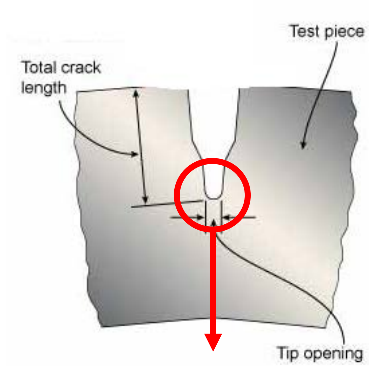
$$K_{IC} = \alpha \left( \frac{E}{H} \right)^{1/2} \left( \frac{P}{c^{3/2}} \right)$$

***➔ Only for ceramic materials (very brittle)***

# Indentation Fracture Toughness

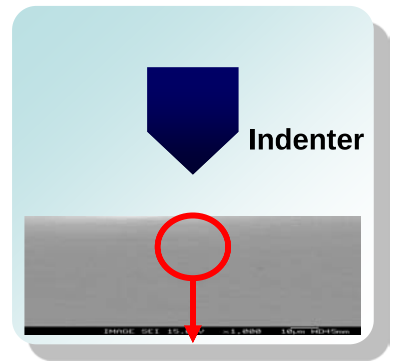
- In case of metallic materials (no cracking in indentation)

**Fracture test**



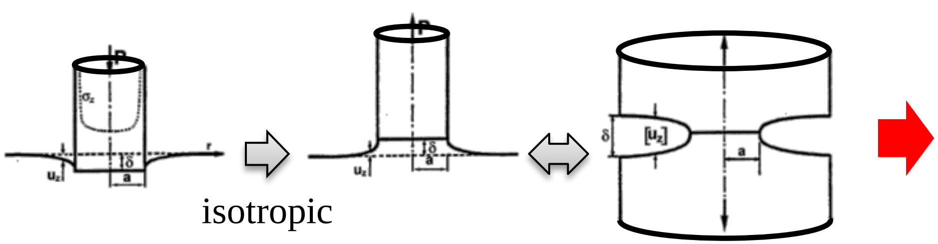

Crack propagation and fracture

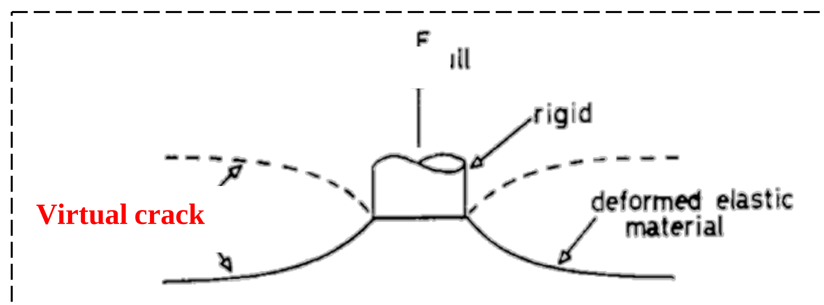
**IIT**



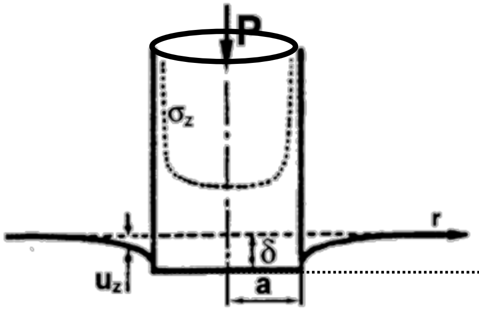

No crack and no fracture

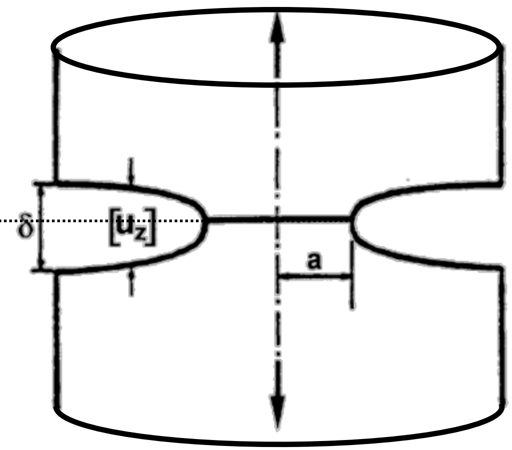
- How to correlate **flat punch indentation** with crack tip behavior



# How to correlate flat punch indentation with crack tip behavior




**Contact mechanics of flat punch indentation** ⟺ **Fracture mechanics of cracked round bar specimen**

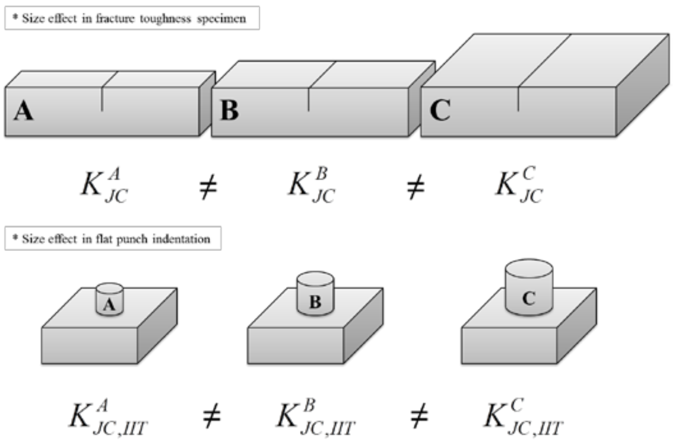
**Direct calculation of J from indentation curve**

**Determination of Fracture toughness**

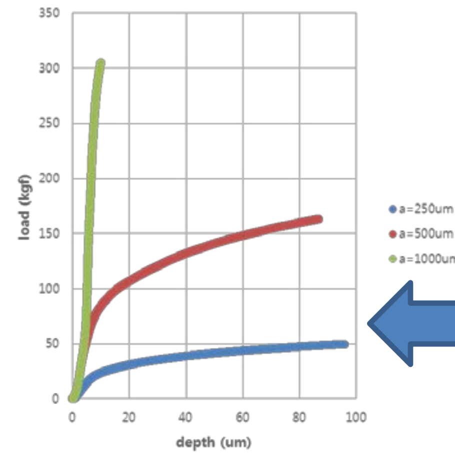
**Flat punch: regarded as a very deep cracked round bar specimen**

# How to correlate flat punch indentation with crack tip behavior

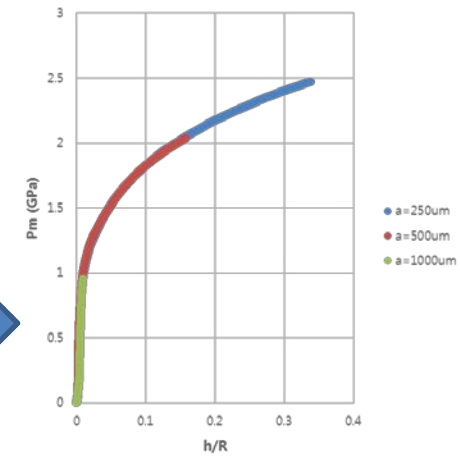
## * Size effect




* Load-depth curve

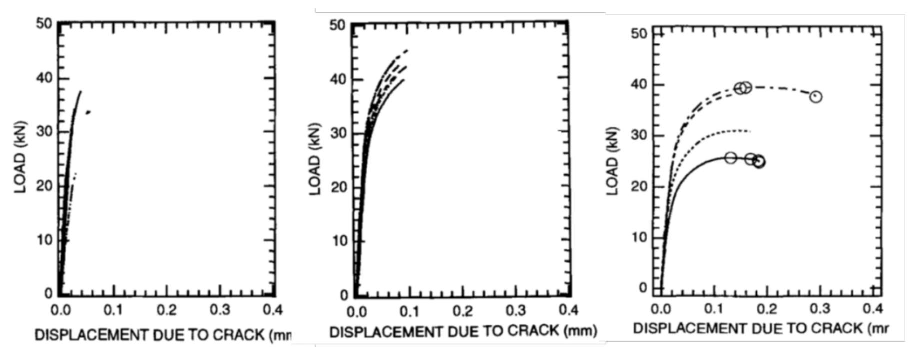



* Normalization

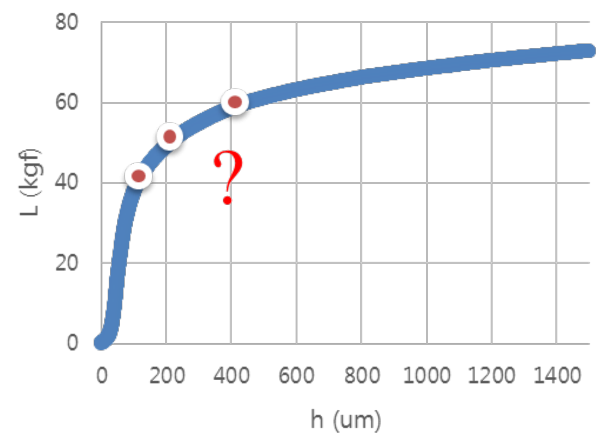



## * Crack initiation point

J. H. Giovanola et al., *ASTM STP 1244* (1995)



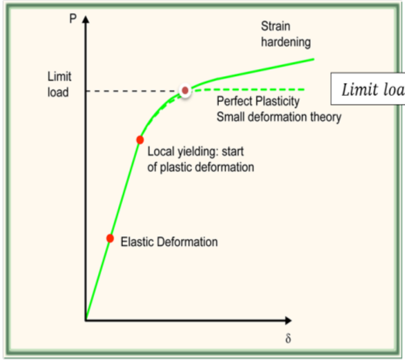

In flat-punch indentation test



# How to correlate flat punch indentation with crack tip behavior

## * Load criterion

* Limit load




Limit load = $f(geometry, \sigma_{YS})$

* For cracked round bar geometry

$$P_L = \pi b^2 \sigma_{YS} \begin{cases} 3.285 & for \quad \frac{a}{R} > 0.65 \\ \frac{R}{b} & for \quad \frac{a}{R} < 0.65 \end{cases}$$

Von mises yield criterion
a : crack length
b : ligament radius
R : specimen radius

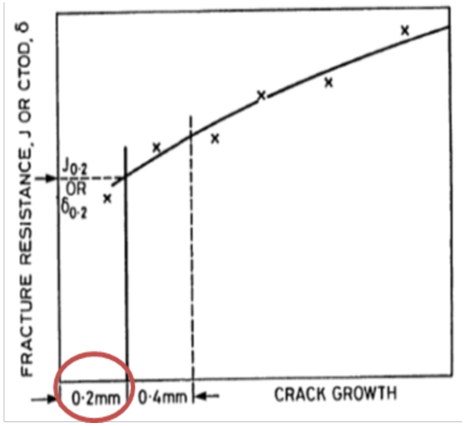
## * Depth criterion

**Geometrical similarity**

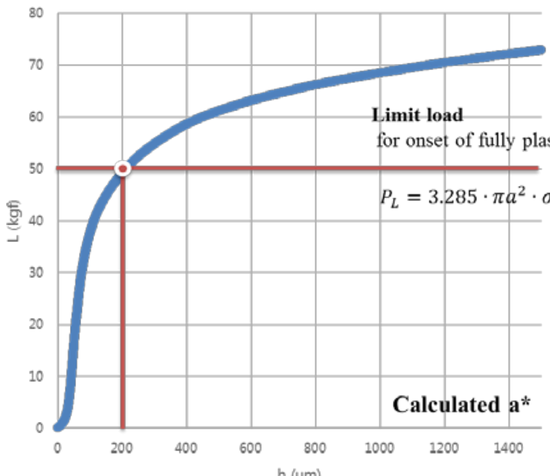
Indentation depth (h)
= ½ crack tip opening displacement (CTOD)
= crack extension (Δa)

**Standard condition**

Estimate of $J_{IC}$ = J at Δa = 0.2 mm
» h* ≈ 0.2 mm




## * Evaluation




**h* = 0.2 mm**
matching with engineering crack extension $h \approx \Delta a = 0.2\ mm$

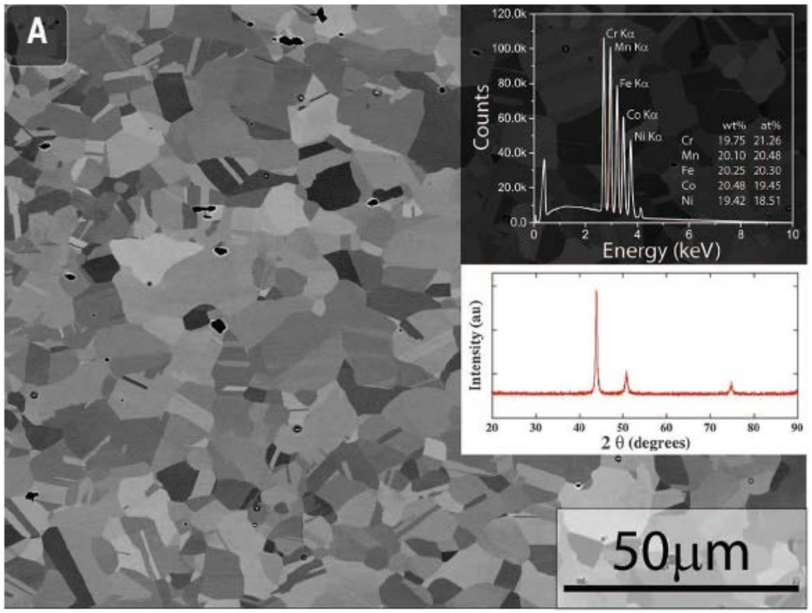
$$K_I = \frac{P}{2a\sqrt{\pi a}}$$

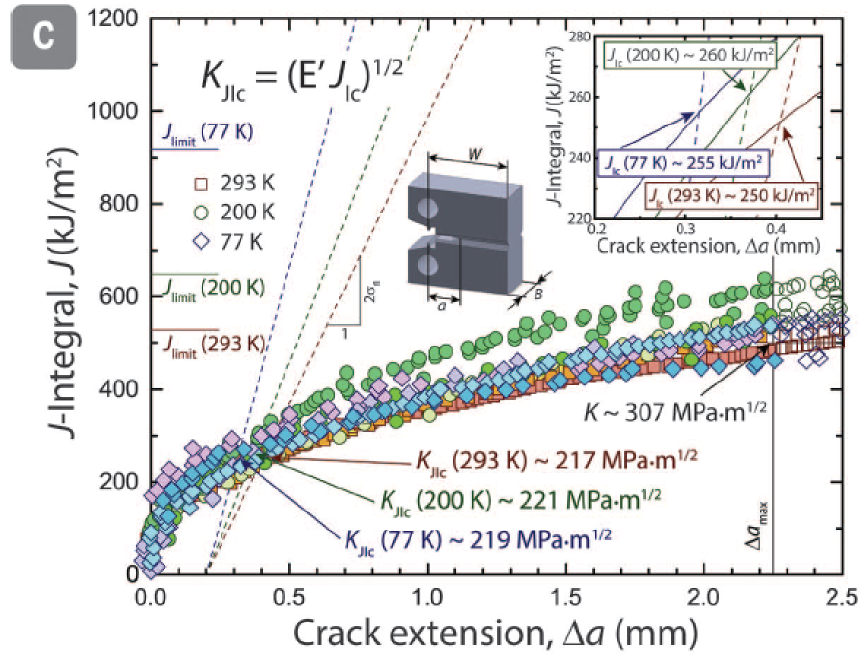
$$J = J_e + J_p = \frac{(1-\nu^2)K_I^2}{E} + \eta_{pl}\frac{A_{pl}}{\pi a^2}$$

# Fracture toughness of cantor alloy

## Reference alloy : Cantor alloy – $Cr_{20}Mn_{20}Fe_{20}Co_{20}Ni_{20}$

*Bernd Gludovatz / SCIENCE VOL 345 ISSUE 6201*



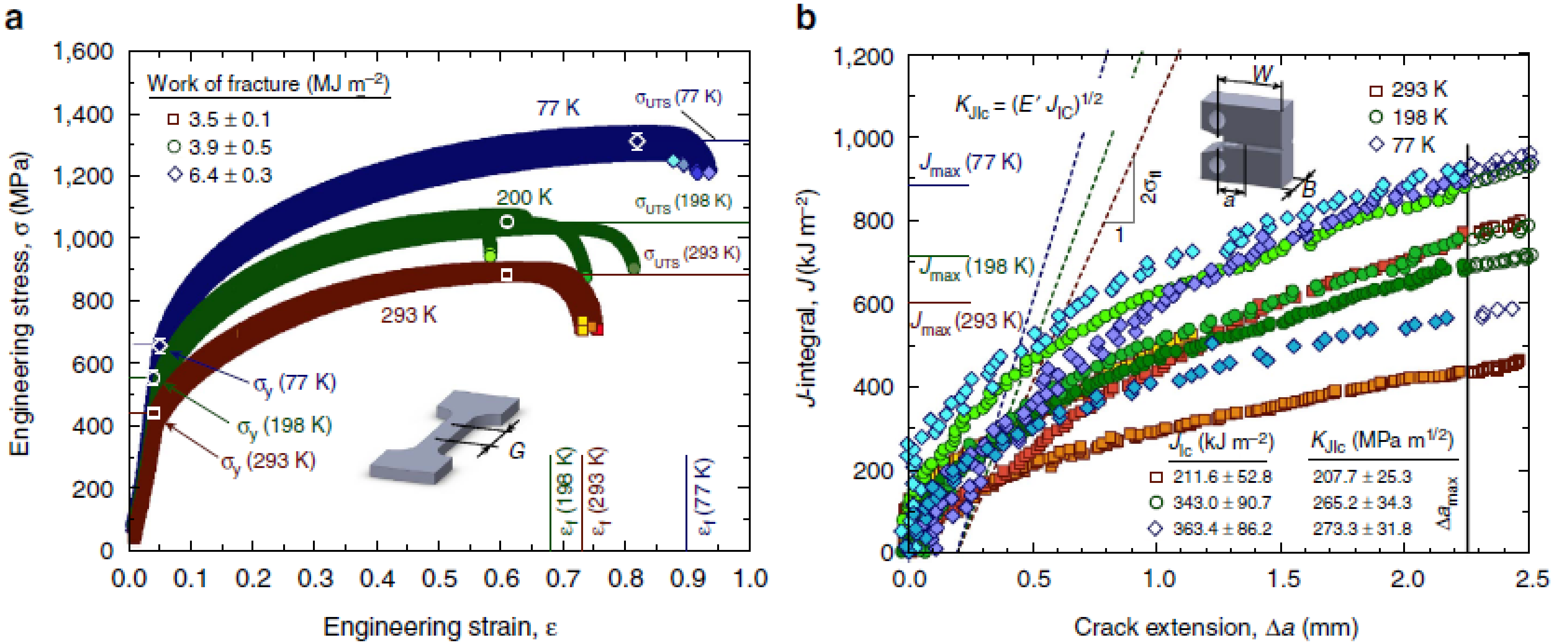

Process : Arc melting, copper mold drop casting, cold forging and cross rolling at RT, recrystallization

| KjIc | Ave. (Mpa·$m^{1/2}$) | Grain size (μm) |
|---|---|---|
| Cantor @293K | ~217 | ~ 6 |
| Cantor @200K | ~221 | ~ 6 |
| Cantor3 @77K | ~219 | ~ 6 |

# Fracture toughness of cantor alloy

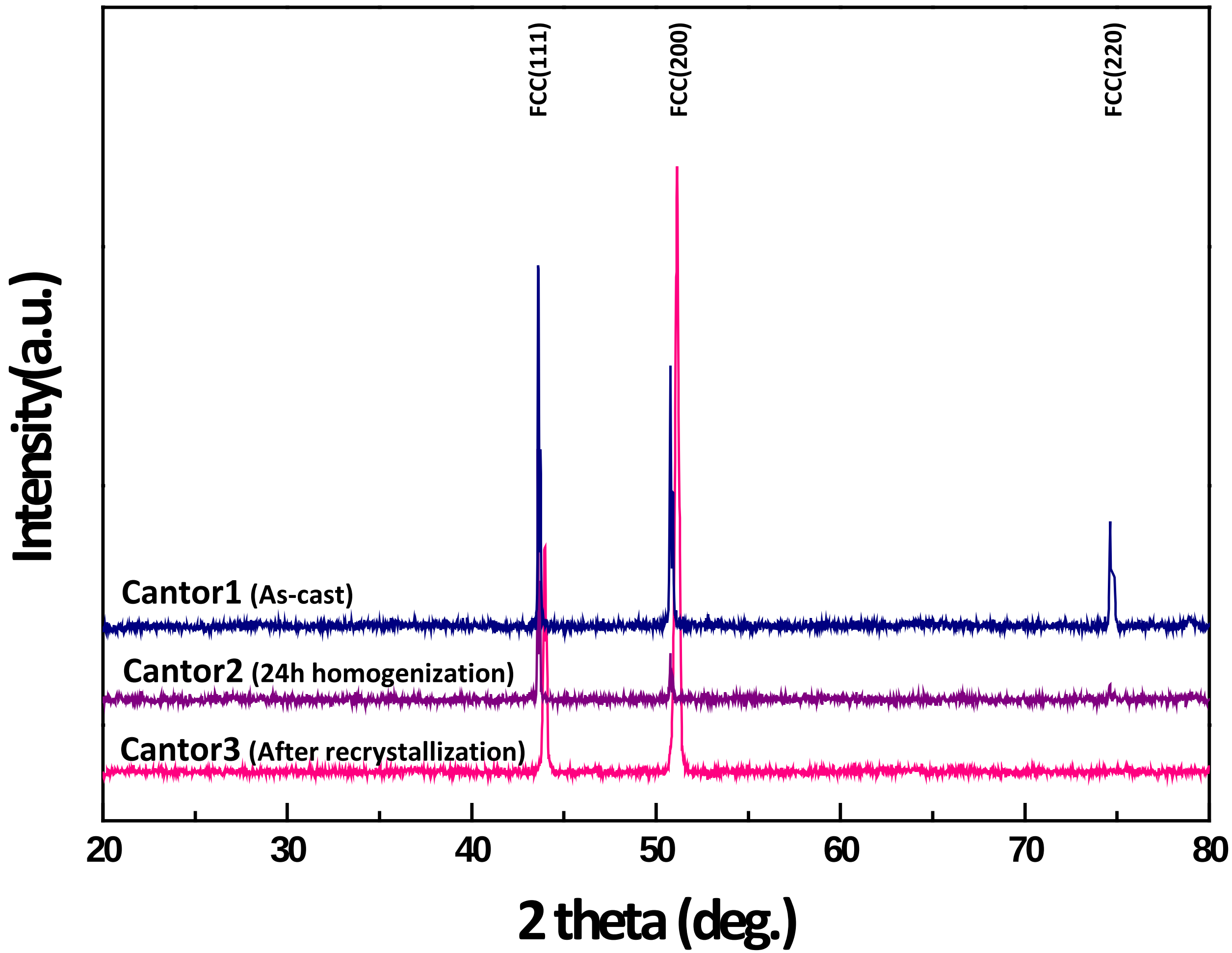
## Reference alloy : CrCoNi

*Bernd Gludovatz/ NATURE COMMUNICATIONS | 7:10602 | DOI: 10.1038/ncomms10602 |*



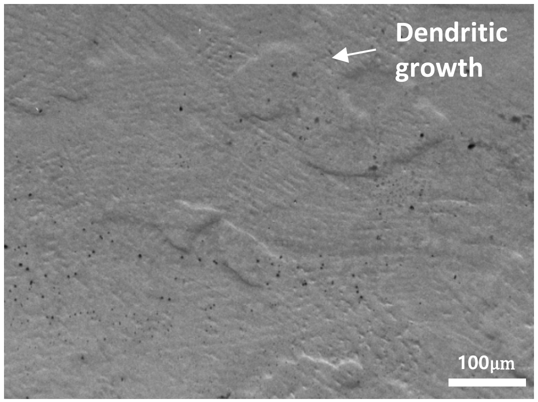
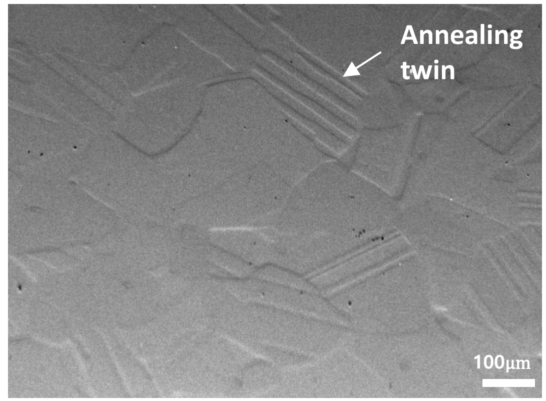
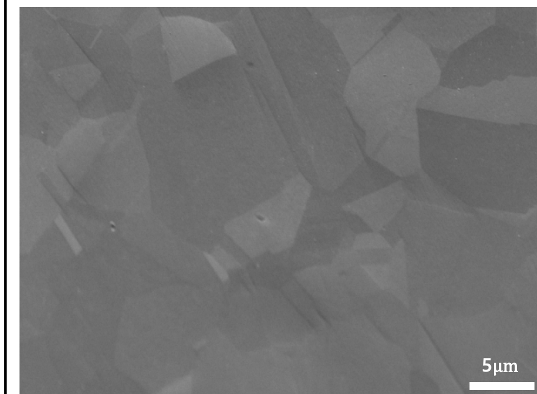
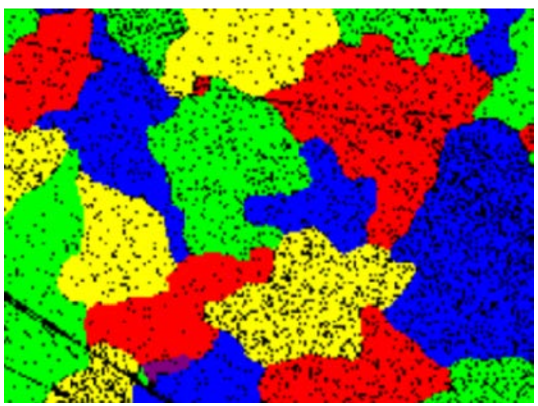
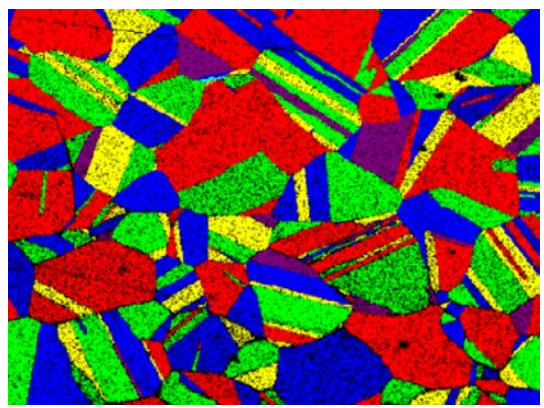
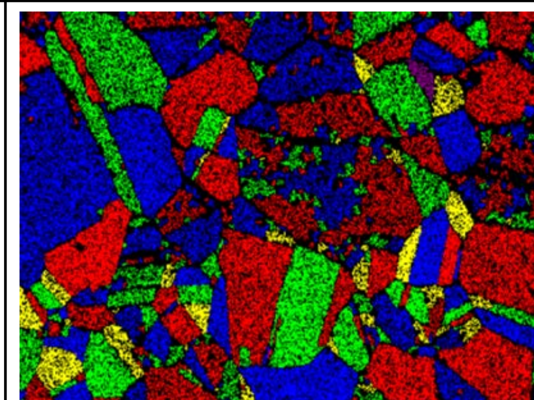

| KjIc | Ave. (Mpa·m$^{1/2}$) | Grain size (μm) |
|---|---|---|
| @293K | ~205 | ~ 5 |
| @200K | ~265 | ~ 5 |
| @77K | ~273 | ~ 5 |

# XRD data of cantor alloy




**XRD data shows that all specimen have FCC crystal structure**

# Microstructure of Cantor alloy

| Cantor 1<br>(as-cast) | Cantor 2<br>(24h homogenization) | Cantor 3<br>( + cold rolling + recrystallization) |
|---|---|---|
|  |  |  |
|  |  |  |

**Grain size :** ~ 97 μm     ~ 107 μm     ~ 4 μm

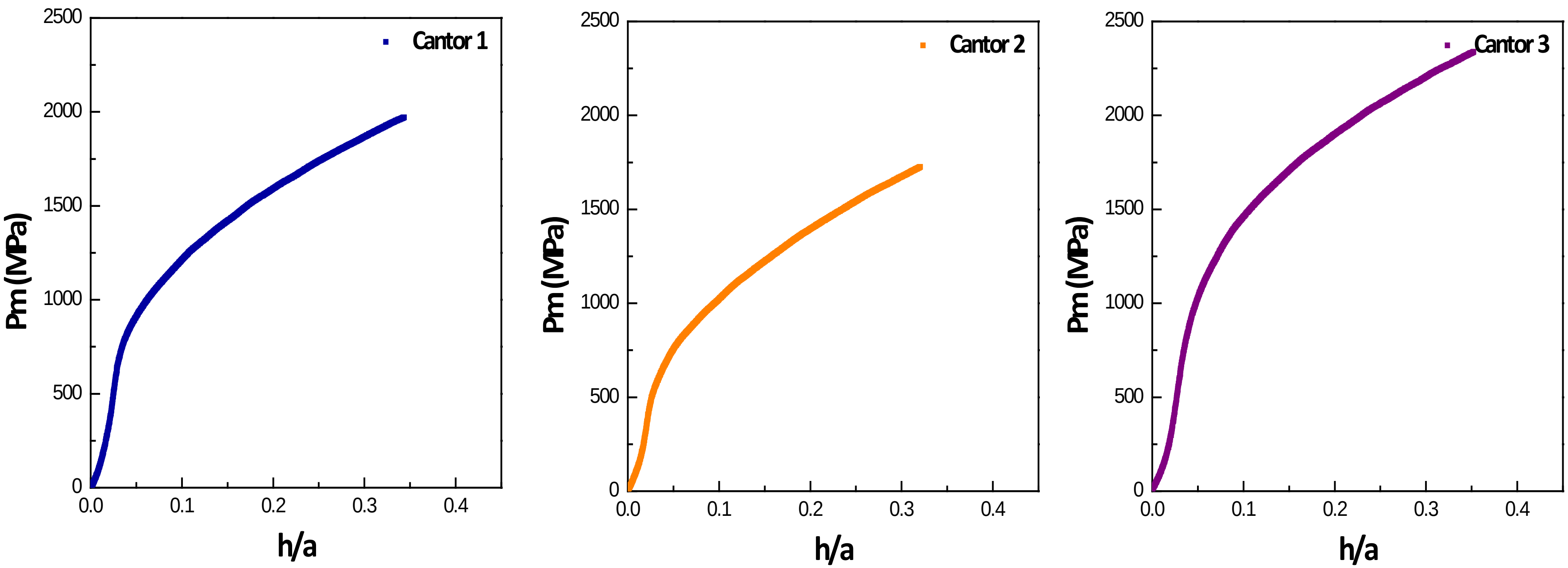
# Indentation test for toughness of cantor alloy

- **Test condition**

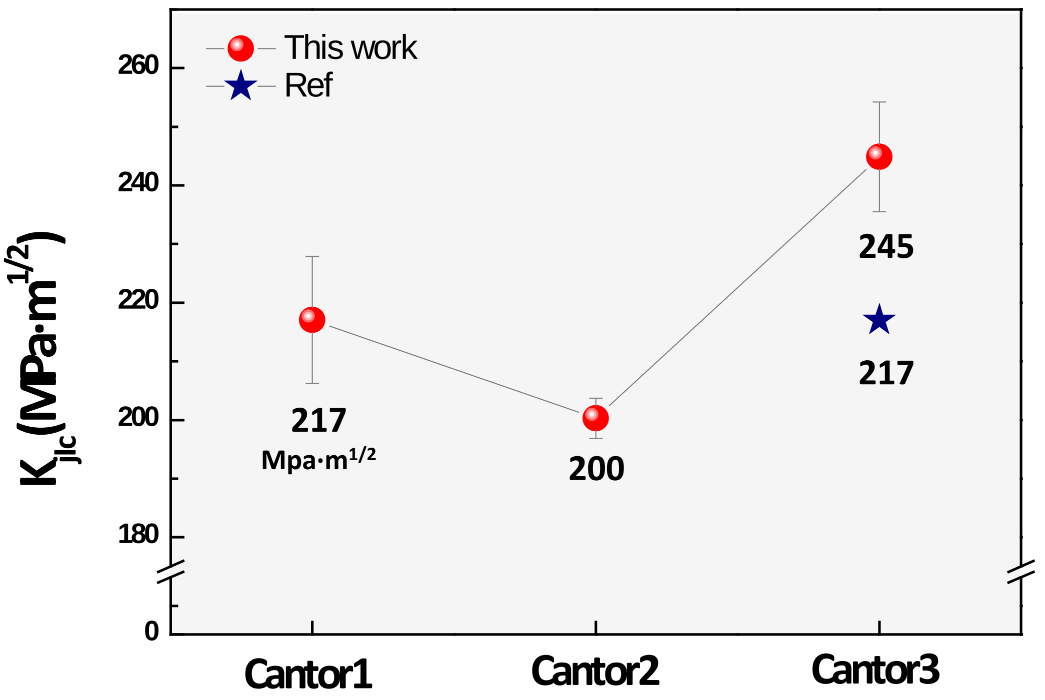
  Loading rate : 0.3mm/min,   Depth control : 100um,   Zero index : 0.1kgf

- **Normalized curve**

Cantor 1　　Cantor 2　　Cantor 3



# Result of indentation test

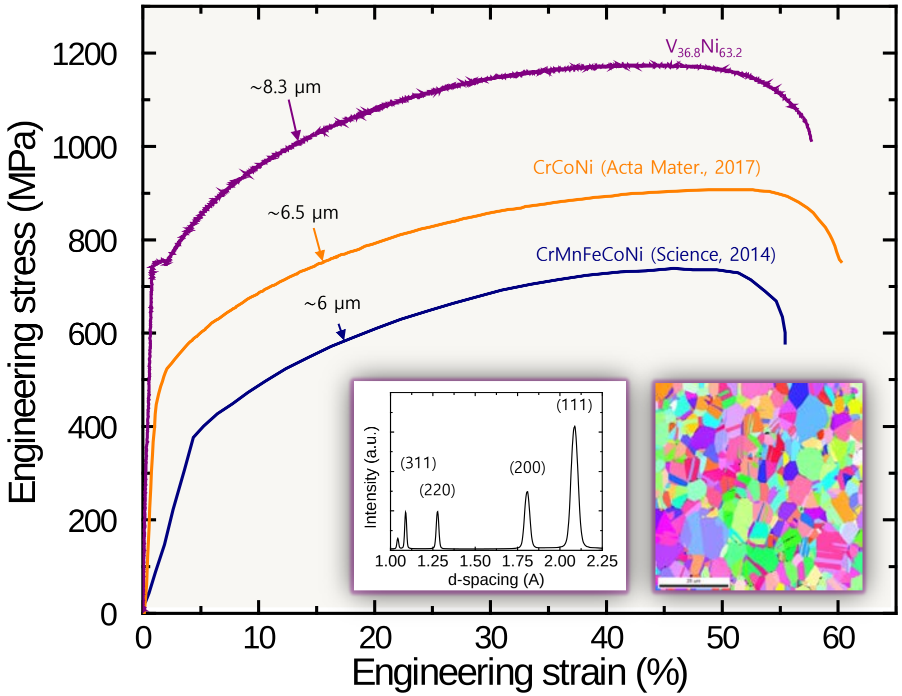


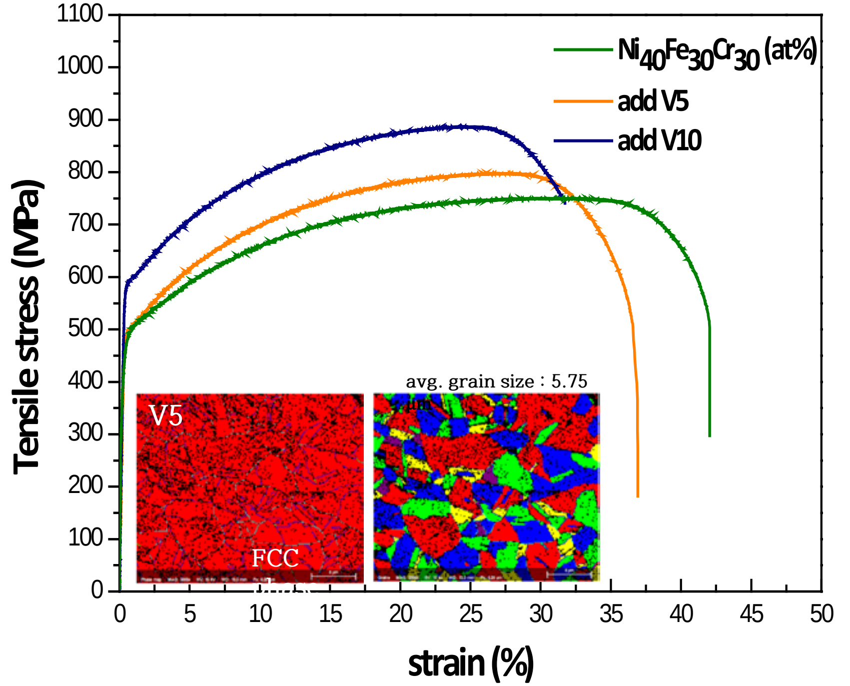

| Kjc | Test 1 | Test 2 | Test 3 | Test 4 | Ave. (Mpa·m$^{1/2}$) | Grain size (μm) |
|---|---|---|---|---|---|---|
| Ref. | - | - | - | - | 217 | ~ 6 |
| **Cantor1** | 229.913 | 225.478 | 204.192 | 208.74 | **217.0808** | ~ 97 |
| **Cantor2** | 195.559 | 203.835 | 198.548 | 203.291 | **200.3083** | ~ 107 |
| **Cantor3** | 230.931 | 241.726 | 254.396 | 252.385 | **244.8595** | **~ 4** |

**Ref. and cantor3 which are similar processing condition show 12% deviation**

# Strength of Ni-Fe-Cr systems

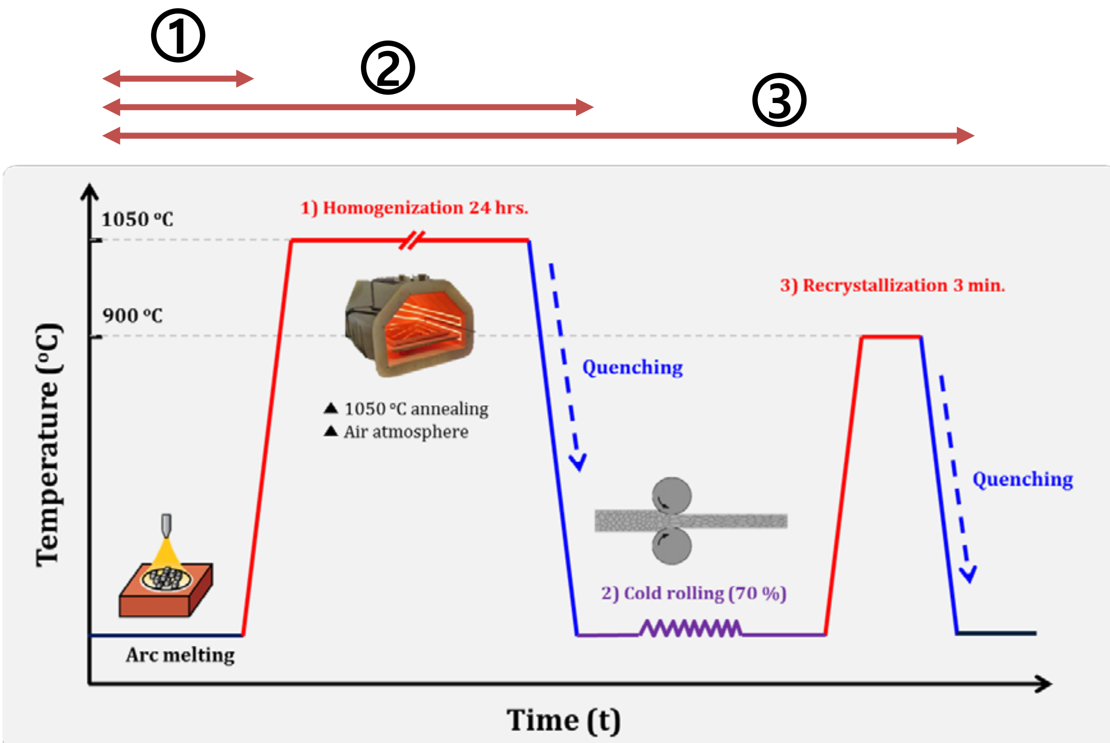
*H.S.Oh / ph.D graduation paper*




| at% | Y.S (MPa) | U.T.S (MPa) | Elongation (%) | Grain size (μm) |
|---|---|---|---|---|
| Cantor | 331.96 | 693.49 | 41.3 | ~ 6 |
| CoCrNi | 452.9 | 927.36 | 45.21 | ~ 6.5 |
| NiV | 743.49 | 1174.05 | 44.53 | ~ 8.3 |
| $Ni_{40}Fe_{30}Cr_{30}$ | 470.2 | 736.8 | 28.3 | ~ 3.65 |
| $Ni_{40}Fe_{30}Cr_{25}V_{5}$ | 476.4 | 785.7 | 28.6 | ~ 4.18 |
| $Ni_{40}Fe_{30}Cr_{20}V_{10}$ | 682.7 | 954.7 | 19.4 | ~ 5.75 |

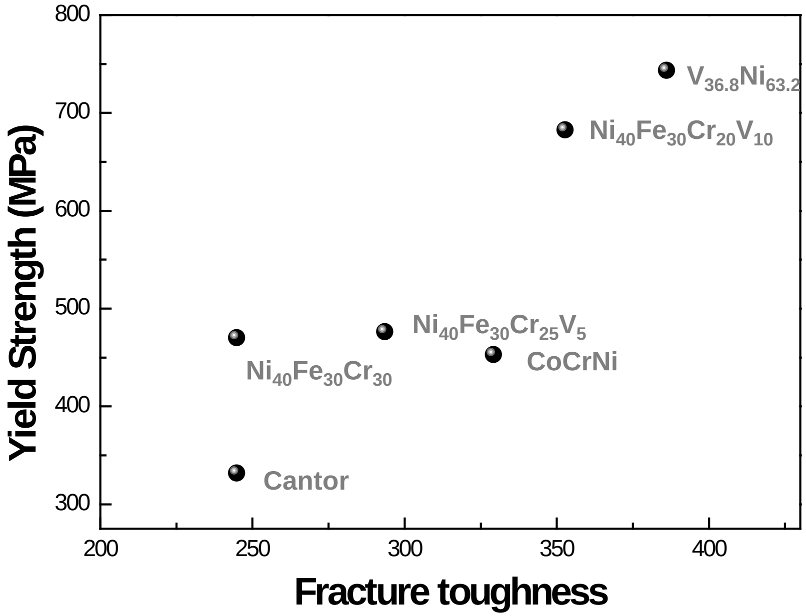
# Heat treatment of specimen



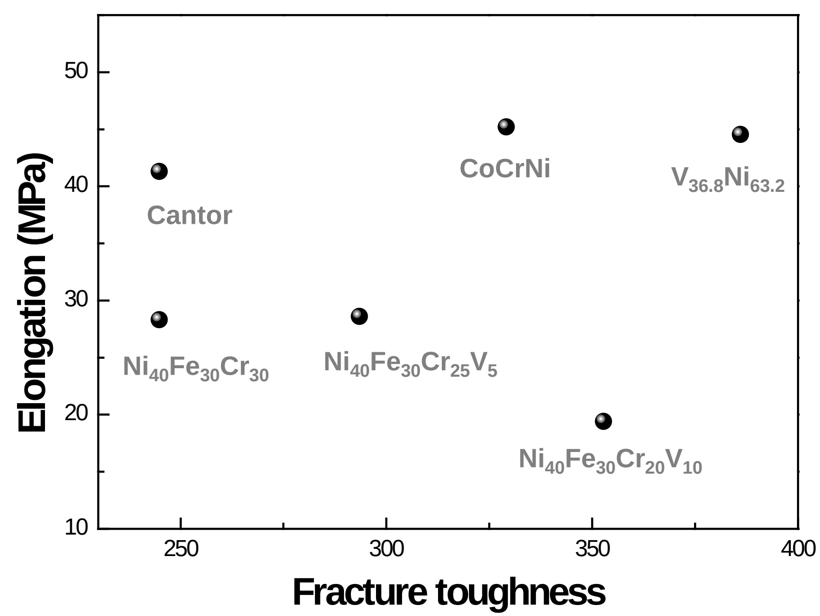

| | | Heat treatment | Phase | Grain size (μm) |
|---|---|---|---|---|
| 1 | $Ni_{40}Fe_{30}Cr_{30}$_1 | As-cast | FCC | - |
| 2 | $Ni_{40}Fe_{30}Cr_{30}$_3 | homogenization + Cold rolling + Recrystallization | FCC | ~ 3.7 |
| 3 | $Ni_{40}Fe_{30}Cr_{20}+V_{10}$_1 | As-cast | FCC | - |
| 4 | $Ni_{40}Fe_{30}Cr_{20}+V_{10}$_3 | homogenization + Cold rolling + Recrystallization | FCC | ~ 4 |

# Fracture toughness and tensile property

- **Yield Strength vs Fracture toughness**

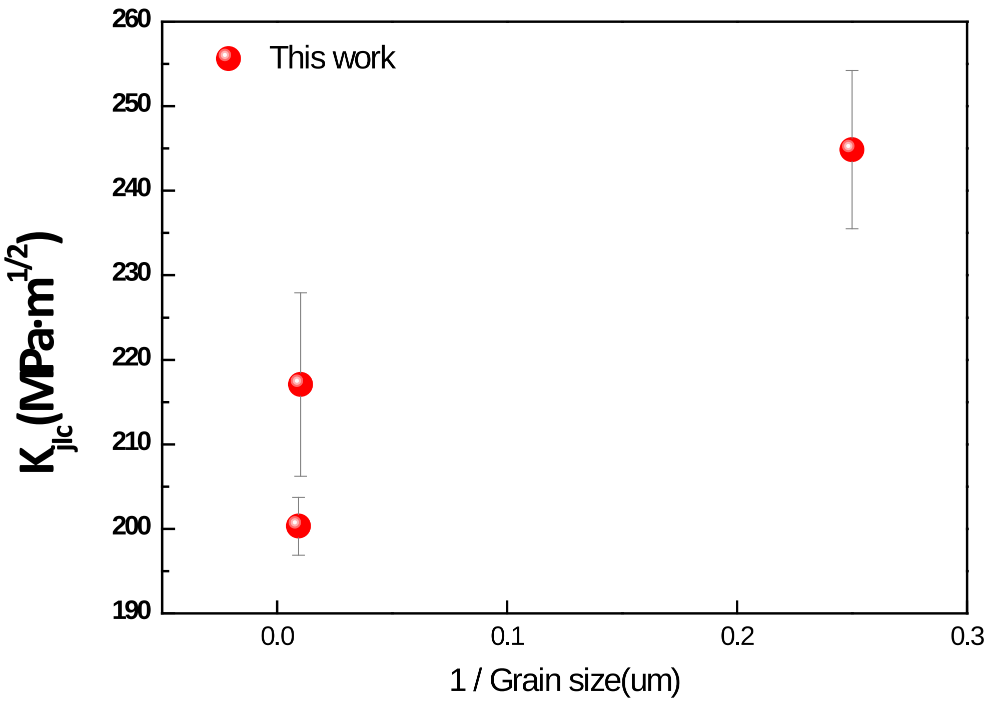



- **Ductility vs Fracture toughness**




**Fracture toughness through IIT yield strength에 의한 영향이 더 큰 것으로 보임.**

# Grain size effect on fracture toughness

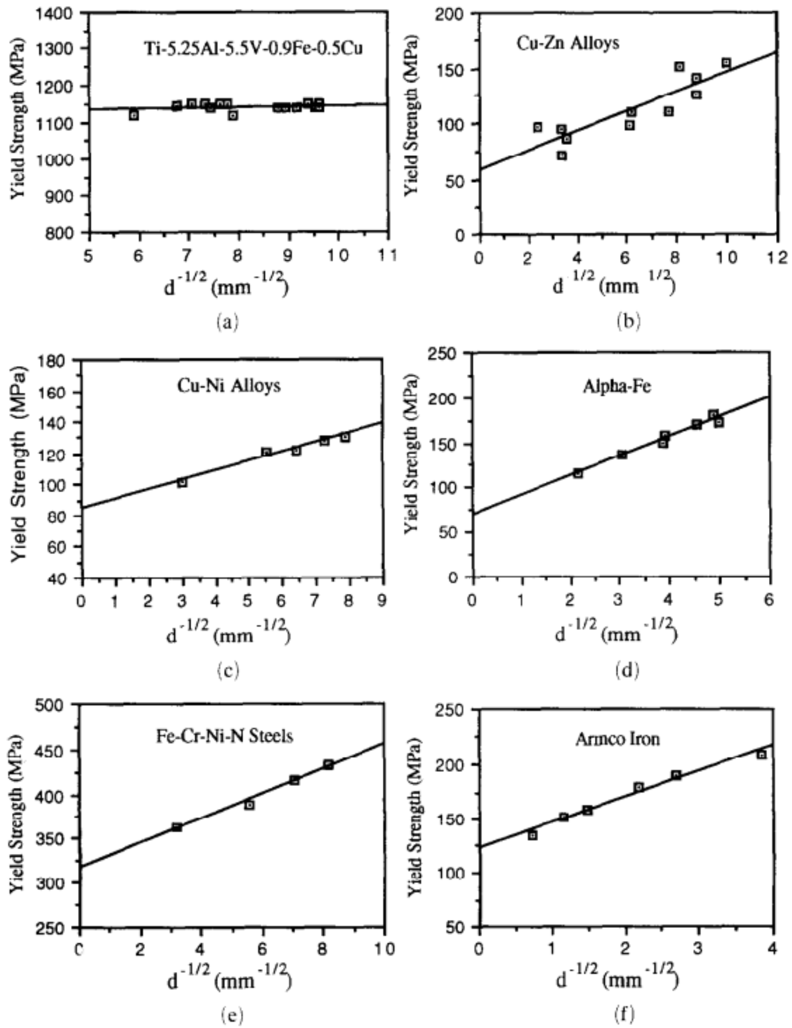



**Grain size 가 작아질 수록 Fracture toughness 가 증가하는 것으로 보임.**

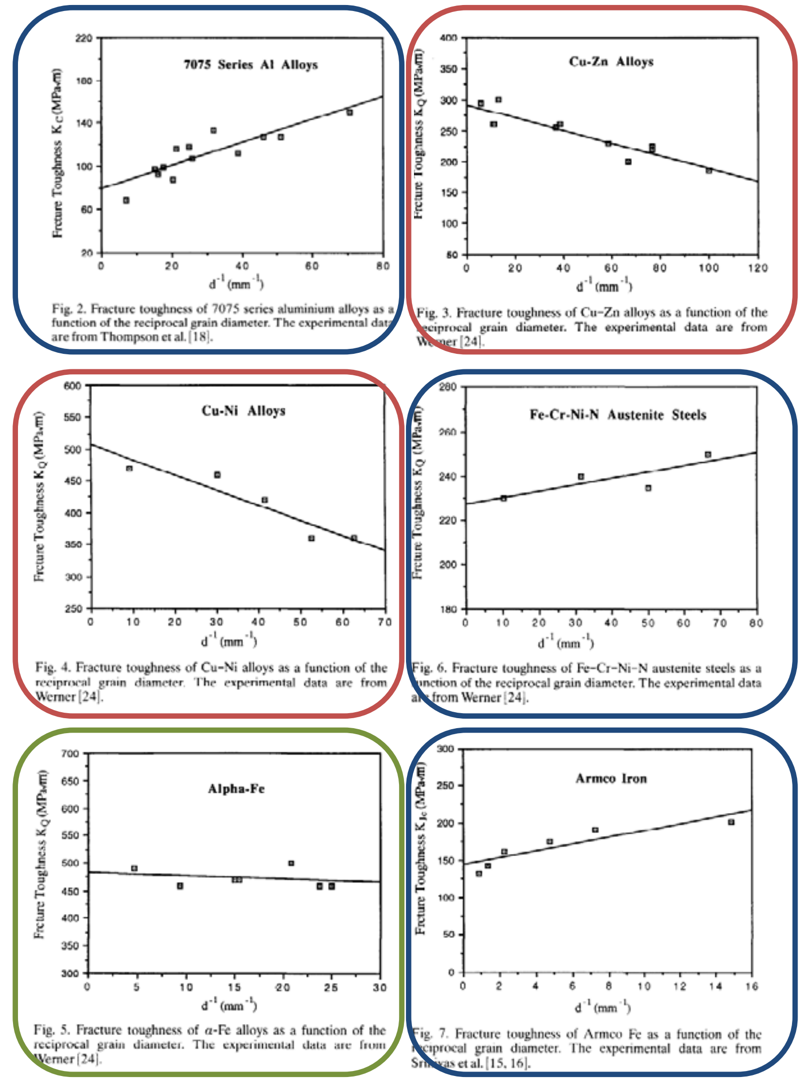
# Grain boundary and fracture toughness

Zhongyun Fan, Mat. Sci. Eng. A, *191* (1995)

- **Grain size vs Yield Strength**




- **Grain size vs Fracture toughness**




**Hall-petch relation :** $\sigma_y = \sigma^\circ_y + k_d d^{-1/2}$

# Grain boundary and fracture toughness

Zhongyun Fan, Mat. Sci. Eng. A, *191* (1995)

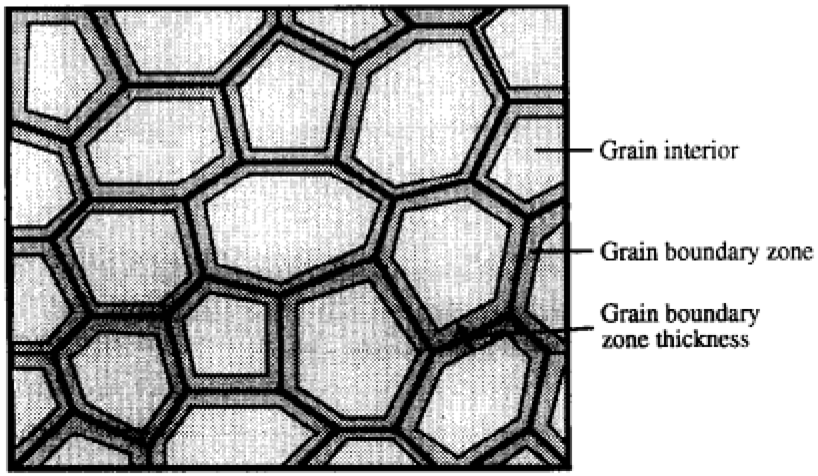



Fig. 10. A schematic illustration of the grain boundary zone and grain interior after deformation at large plastic strain.

$$K_{Ic} = K^{\circ}_{IC} + k_F d^{-1}$$

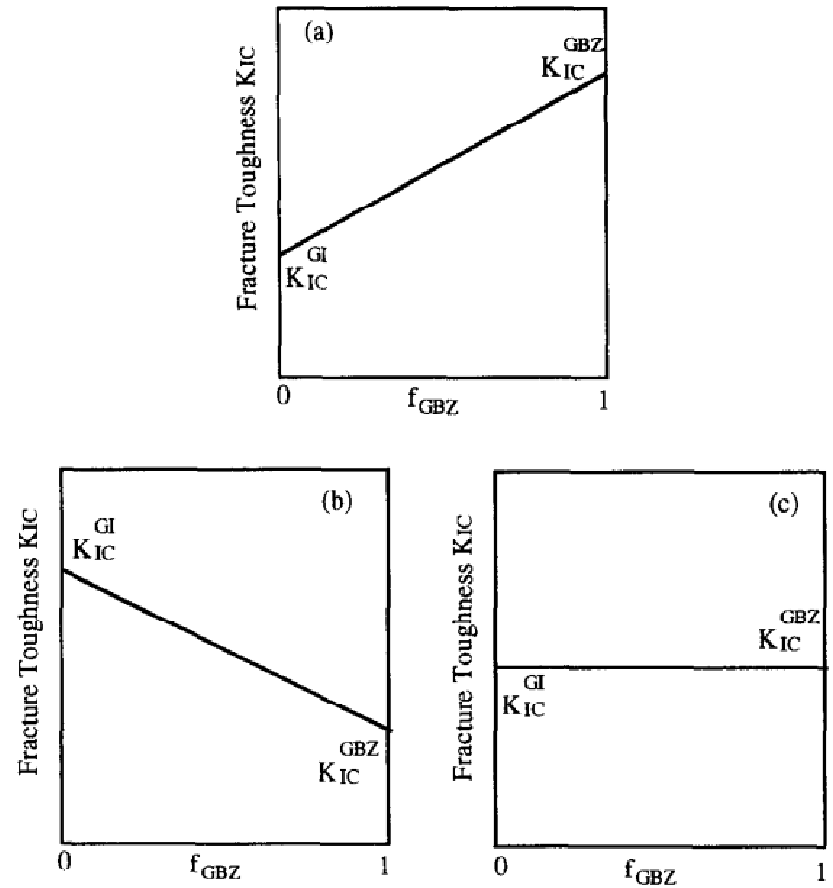



Fig. 11. Schematic illustration of fracture toughness as a function of volume fraction of the grain boundary zone: (a) $K_{IC}^{GBZ} > K_{IC}^{GI}$, the grain boundary toughening effect; (b) $K_{IC}^{GBZ} < K_{IC}^{GI}$, the grain boundary embrittling effect; (c) $K_{IC}^{GBZ} \approx K_{IC}^{GI}$, $K_{IC}$ is independent of grain size.

# Thank you