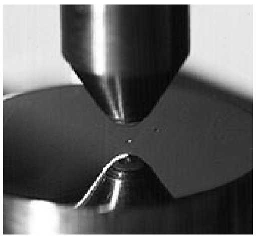
Evaluation of residual stress using Instrumented Indentation Technique

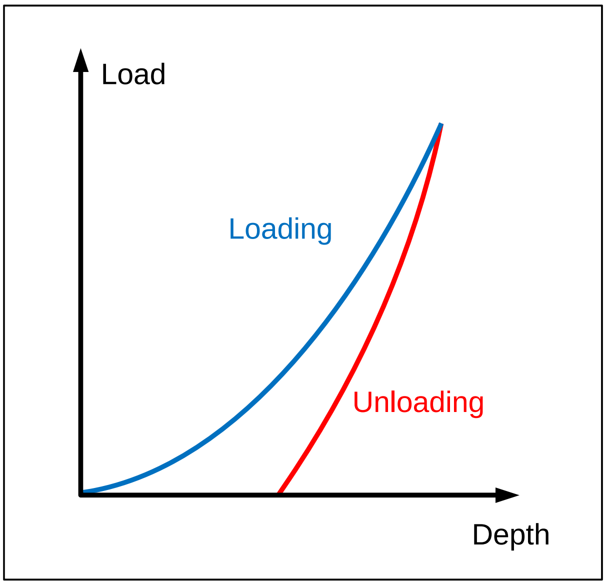
2014. 04. 01.

Jong hyoung Kim

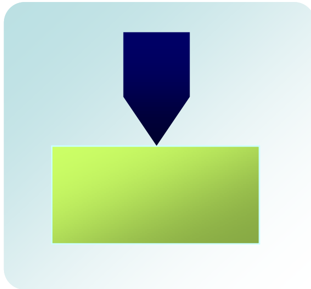
Contents

Instrumented indentation technique (IIT)





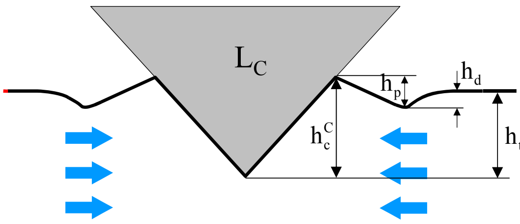
Indentation load-depth curve



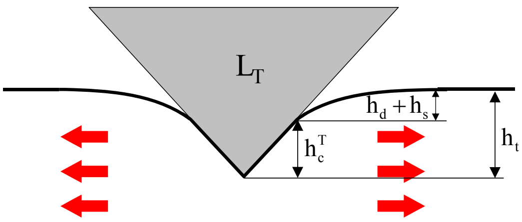
Hardness
Elastic modulus
Tensile properties
Fracture toughness
Residual stress
⋮

Easier and simpler to measure quantitative mechanical properties

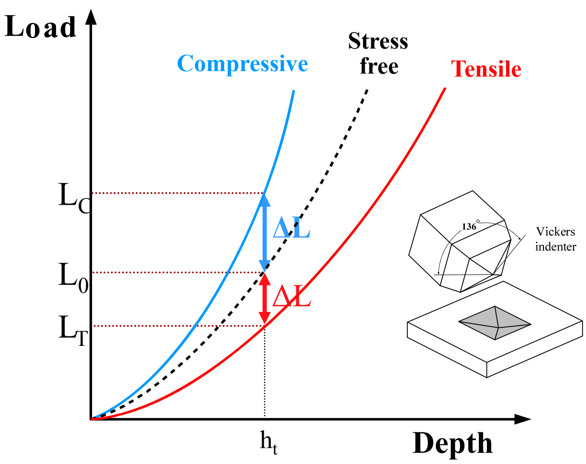
Evaluation of residual stress using IIT



Compressive stress



Tensile stress

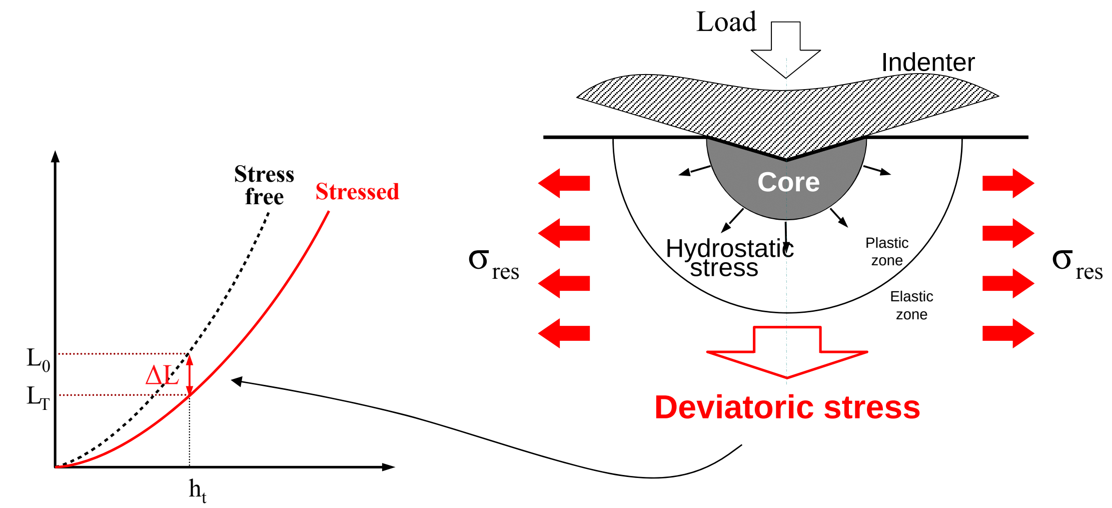


$$\sigma_{res} \propto \Delta L$$

- L_C = Indentation load in compressive stress state
- L_0 = Indentation load in stress-free state
- L_T = Indentation load in tensile stress state
- h_t = Indentation depth (experimentally measured)
- h_d = Elastic deflection height
- h_p = Pile-up height
- h_s = Sink-in height
- h_c^C = Real contact depth in compressive stress
- h_c^T = Real contact depth in tensile stress

Interaction of residual stress with indentation load

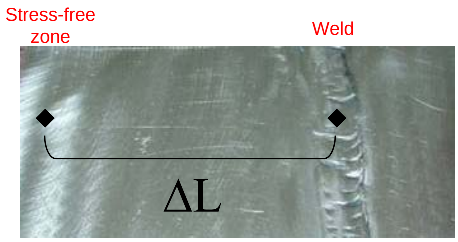
Indentation load ⟷ Residual stress, σ_{res}



Contents

Necessity of stress-free state estimation

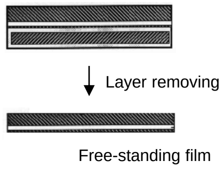
< Determination of stress-free zone >



< Stress relaxation – generation of stress-free state>

- Heat treatment -

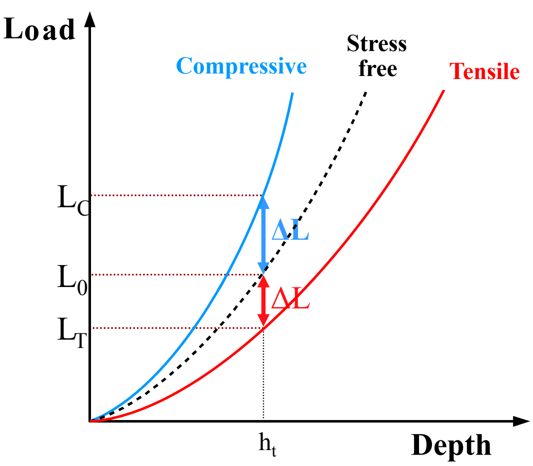
- Substrate etching -



(without determination of stress-free zone or generation of stress-free state)

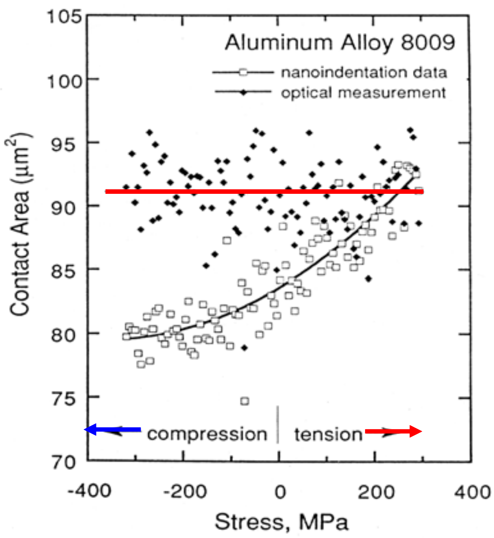
Estimation of load-depth curve of stress-free state from stressed load-depth curve

Stress effect to indentation curve and contact



< Stress effect to indentation curve >

[T.Y. Tsui et al., *J. Mater. Res.* (1996)]

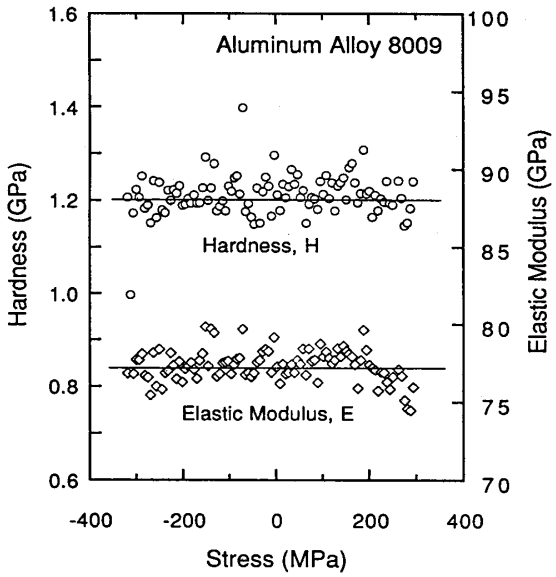


< Invariant real contact area >

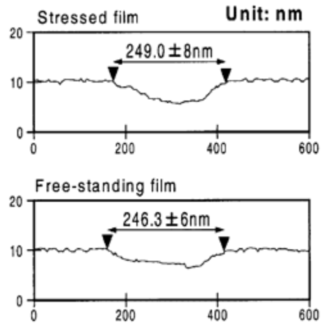
Invariant elastic modulus, hardness and contact area

$$H = \frac{L_0}{A_c}$$

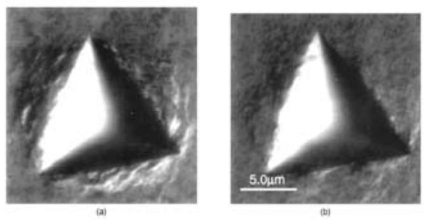
[T.Y. Tsui et al., *J. Mater. Res.* (1996)]



[Y.H. Lee, D. Kwon, *J. Mater. Res.* (2002)]



[T.Y. Tsui et al., *J. Mater. Res.* (1996)]



Compressive stress (-290 MPa)

Tensile stress (+251 MPa)

< Invariant hardness regardless of stress state >

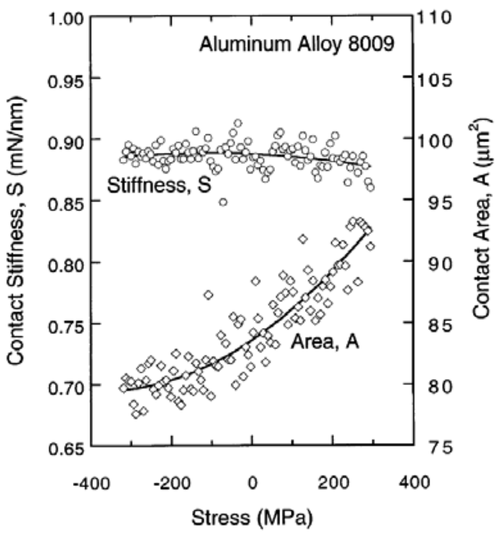
< Invariant contact area regardless of stress state >

Invariant stiffness

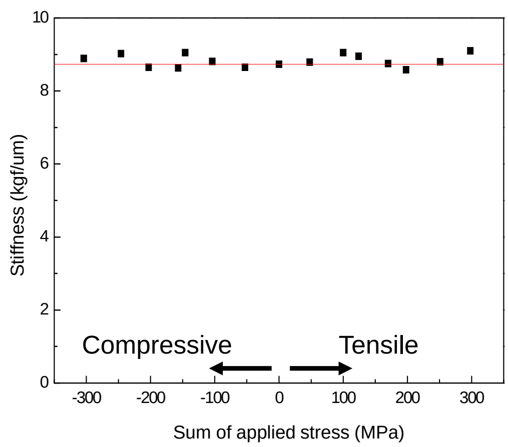
- **Elastic modulus**

$$E_{eff} = \frac{1}{\beta}\frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}}$$

[T.Y. Tsui et al., *J. Mater. Res.* (1996)]



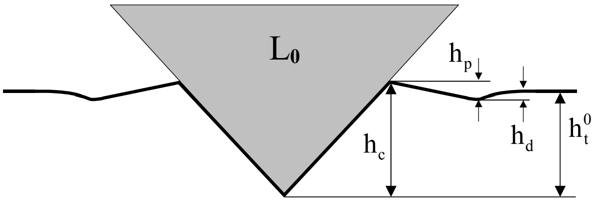
Experimental results



- Material : S20
- Maximum load : 50 kgf
- Stiffness at stress-free state : 8.73 kgf/um

Invariant stiffness → Invariant elastic modulus

Reason for invariant contact area



< Stress-free >

(h_d = Elastic deflection
h_p = Pile-up

$$h_c = h_t^0 - h_d + h_p$$

[M.F. Doerner, W.D. Nix, *J. Mater. Res.* (1986)]
[W.C. Oliver, G.M. Pharr, *J. Mater. Res.* (1992)]

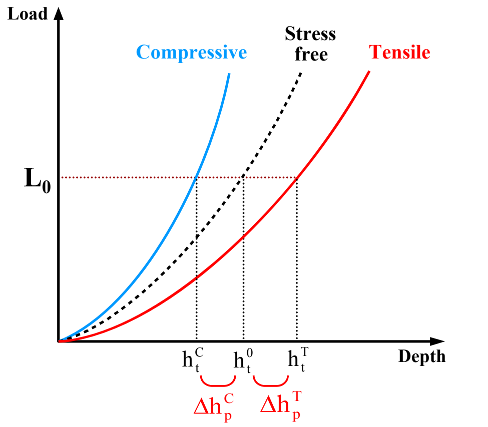
$$h_d = \varepsilon \frac{L_{max}}{S}$$

[Y.T. Cheng, C.M. Cheng, *Appl. Phys. Lett.* (1998)]
[S.K. Kang et al., *J. Mater. Res.* (2010)]

$$h_p = f\left(\frac{H}{E}\right) = f\left(\frac{W_e}{W_{total}}\right)$$

Parameters dependent on material properties

(No change by residual stress)

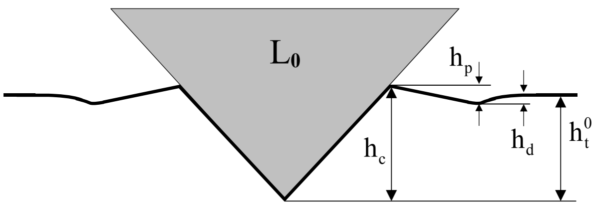


$$h_t^0 = h_t^C + \Delta h_p^C$$ Compressive

$$h_t^0 = h_t^T - \Delta h_p^T$$ Tensile

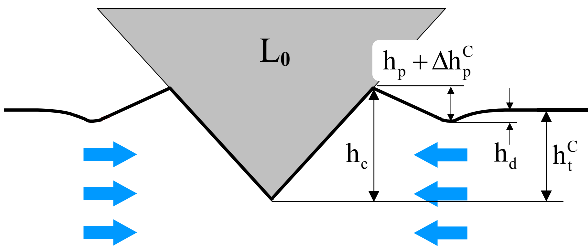
Change of pile-up height by residual stress

Reason for invariant contact area

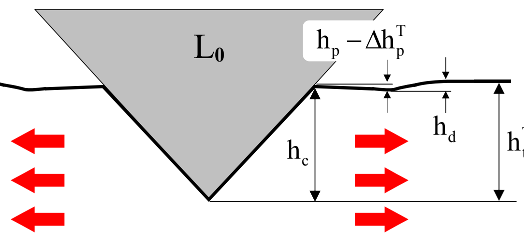


< Stress-free >

$$h_c = h_t^0 - h_d + h_p$$



< Compressive >



< Tensile >

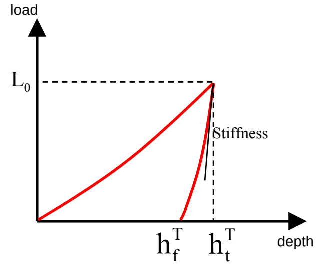
$$h_c = h_t^C + \Delta h_p^C - h_d + h_p$$

- Increased pile-up by compressive stress

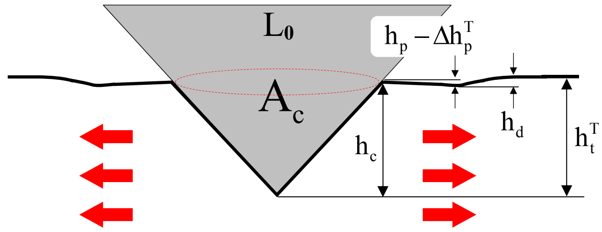
$$h_c = h_t^T - \Delta h_p^T - h_d + h_p$$

- Decreased pile-up by tensile stress

Concept of stress-free state estimation



< In case of tension >



** Assumption :*
- Invariant contact area
- Invariant stiffness

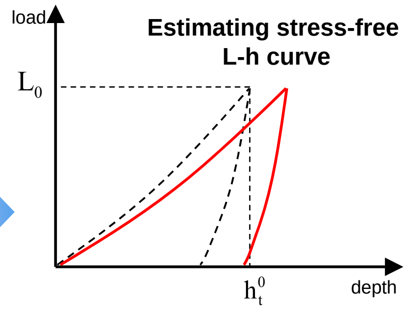
Measurement of contact area A_c

Calculation of real contact depth h_c

$A_c = 24.5(h_c)^2$ (by Vickers' geometry)

Determination of h_t^0

$$h_t^0 = f(h_c,\ h_t^T - h_f^T)$$



Calculation of real contact depth h_c

- **Measurement of real contact area A_c**

  → Optical measurement of indent

  - A_c is measured from stressed state.

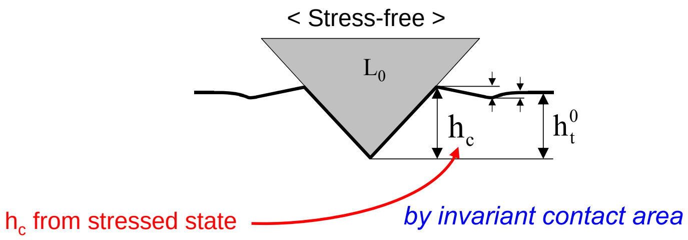
- **Calculation of real contact depth h_c**

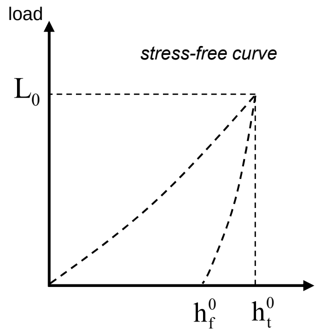
$$h_c = f(A_c)$$

- $A_c = 24.5(h_c)^2$ (by Vickers' geometry)

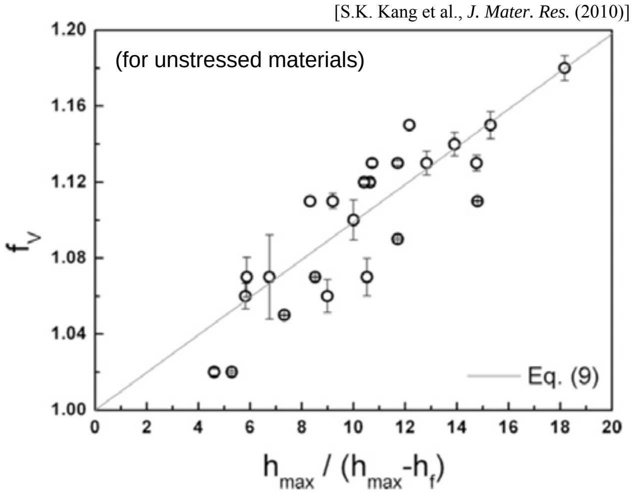
  - h_c is calculated from stressed state of A_c.

Relation between h_c and h_t^0 in stress-free state





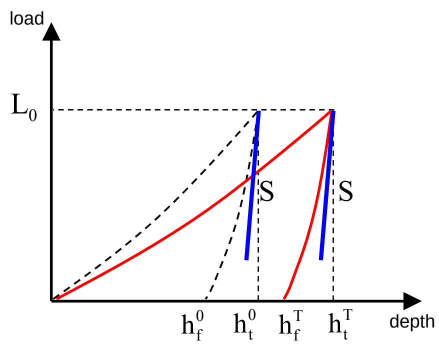
[S.K. Kang et al., *J. Mater. Res.* (2010)]



$$\frac{h_c}{h_t^0} = 9.9\times10^{-3}\frac{h_t^0}{h_t^0 - h_f^0} + 1.0$$

$$\rightarrow \quad h_t^0 = f(h_c,\ h_t^0 - h_f^0)$$

Estimation of h_t^0 using invariant stiffness



- Elastic deflection, h_d

$$h_d = \varepsilon\frac{L_0}{S} \quad \propto \quad h_t - h_f$$

- Stiffness S is invariant,
  so, elastic deflection is consistent.

$$\rightarrow \quad h_t^0 - h_f^0 = h_t^T - h_f^T = h_t^C - h_f^C$$

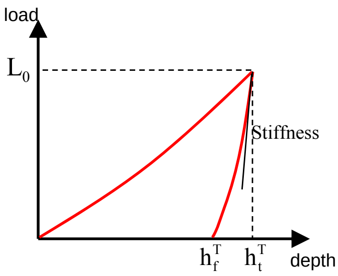
$$\frac{h_c}{h_t^0} = 9.9\times10^{-3}\frac{h_t^0}{h_t^0 - h_f^0} + 1.0$$

$$\frac{h_c}{h_t^0} = 9.9\times10^{-3}\frac{h_t^0}{h_t^T - h_f^T} + 1.0$$

$$h_t^0 = f(h_c,\ h_t^T - h_f^T)$$

$$\left(9.9\times10^{-3}(h_t^0)^2 + (h_t^0 - h_c)(h_t^T - h_f^T) = 0\right)$$

Estimation of stress-free state



- **Measurement of real contact area A_c**
- **Calculation of real contact depth h_c**

$$A_c = 24.5(h_c)^2$$ (by Vickers' geometry)

→ h_c from stressed state

- **Estimation of h_t^0**

$$\frac{h_c}{h_t^0} = 9.9\times10^{-3}\frac{h_t^0}{h_t^0 - h_f^0} + 1.0$$

$$h_t^0 = f(h_c,\ h_t^T - h_f^T) \quad \begin{pmatrix} h_t^0 - h_f^0 \\ = h_t^T - h_f^T \\ = h_t^C - h_f^C \end{pmatrix}$$

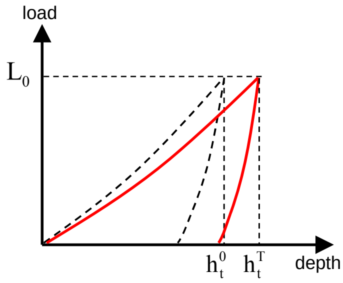
Invariant h_c *Invariant S*

- **Fitting the stress-free curve**

- k : fitting coefficient

$$L_0 = k(h_t^0)^2$$

(Kick's law)



Necessity of new approaches

- In case of difficult to measure contact area
- Small indentation depth (ex. nanoindentation)
- Nonmetal materials (ex. polymer)

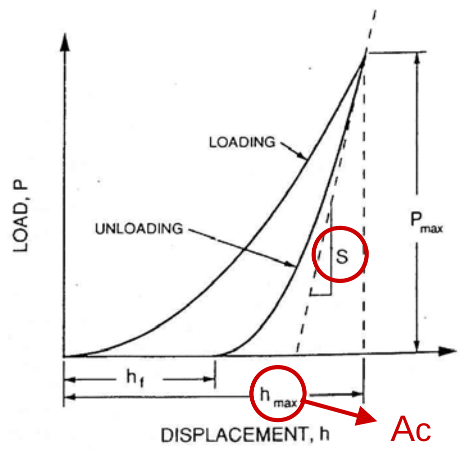
Without measurement of contact area

→ Calculation of contact area using **invariant properties**

Elastic modulus, Stiffness (invariant in stressed state) → Calculation → **Contact area**

Calculation of contact area

- Calculation of contact area using elastic modulus and stiffness



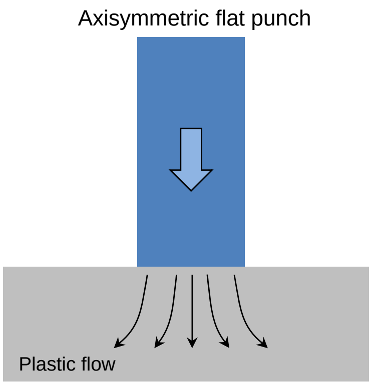
- Sneddon's relationship

Elastic modulus, $E_{eff} = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_C}}$

W. C. Oilver, G. M. Pharr 1992

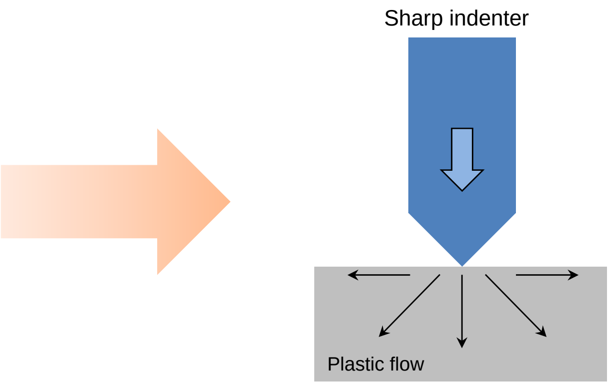
Correction factor (β≠1)

Sneddon's solution



$$E_{eff} = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}}$$

Application of Sneddon's solution



$$E_{eff} = \frac{1}{\beta} \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}} \quad (\beta \neq 1)$$

Correction factor (β≠1)

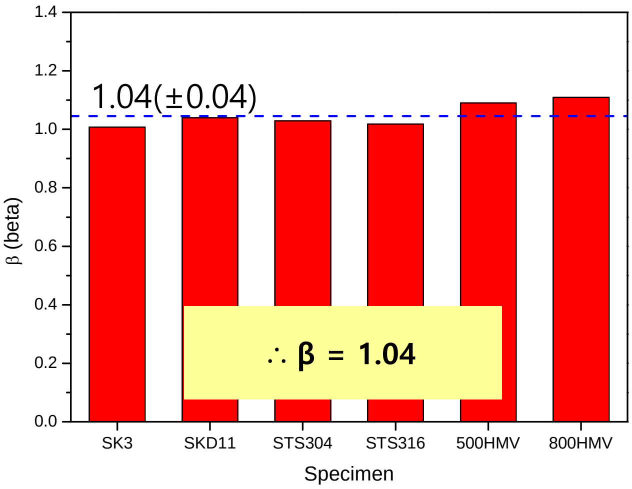
■ Evaluation of correction factor using real contact area and Sneddon's relationship

- β=1.04±0.04

$$E_{eff} = \frac{1}{\beta}\frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}}$$

↓

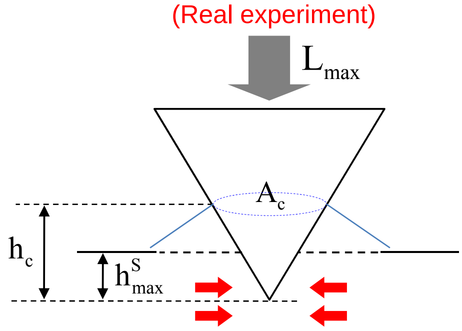
$$\beta = \frac{1}{E_{eff}}\frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}}$$



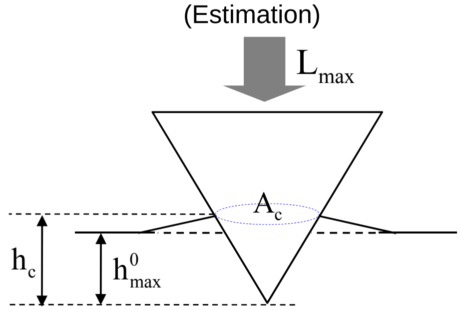
Good agreement with previous research
(β=1.0226~1.085, W. C. Oilver, G. M. Pharr 2003)

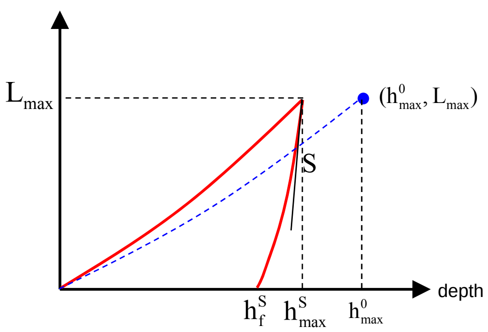
Estimation of stress-free state

Compressive Stress State
(Real experiment)



Stress-free State
(Estimation)





- **Step 1 : $E_{eff} \rightarrow A_c$** $\quad E_{eff} = \frac{1}{\beta}\frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}}$

- **Step 2 : $A_c \rightarrow h_c \rightarrow h^0_{max}$** $\quad A_c = 24.5(h_c)^2$

$$\frac{h_c}{h^0_{max}} = 1.06\times10^{-2}\frac{h^0_{max}}{h^0_{max} - h^0_f} + 1.00$$ *(f-function)*

$$(h^0_{max} - h^0_f = h^S_{max} - h^S_f)$$

- **Step 3 : L=kh²** $\quad L_{max} = k(h^0_{max})^2$

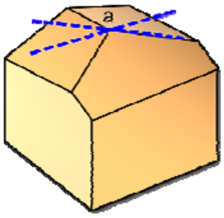
(Kick's law)

Input
Output

Experimental conditions

Machine & Indenter

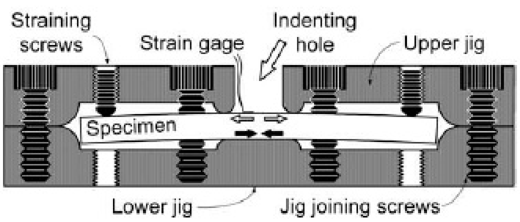
Model	NANO-AIS
Maximum load	60 mN
Method	Single indentation (load control)

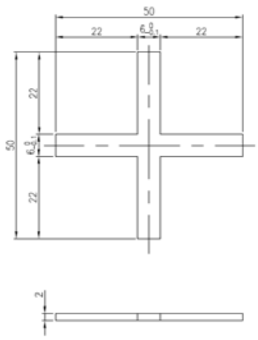


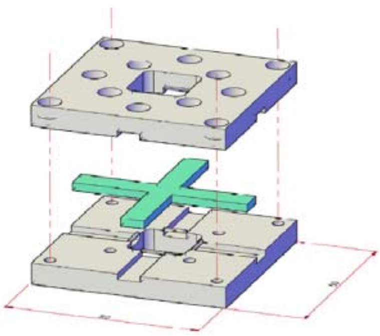
Indenter	Berkovich
Shape	Three-sided Pyramidal
Included angle	142.35º

Experimental conditions

Jig & Specimen





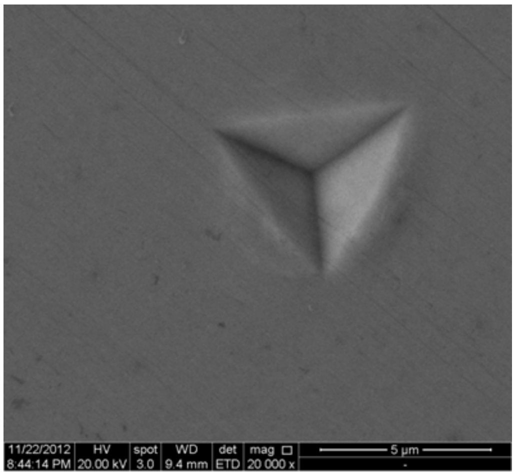


Materials	Elastic modulus(GPa)	Yield strength(MPa)
SKD11	217.35	342.87
SUS304	189.97	321.31
SUS316	203.56	282.74
Al6061	69.92	262.46

Strain gauge	Kyowa

Experimental conditions

Contact area measurement



Model	Quanta FEG 250
Magnification	15000x, 20000x
HV	5kV, 20kV

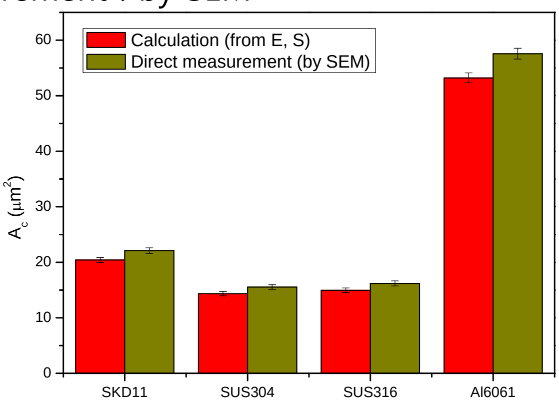
Calculation of contact area

■ Calculation of contact area using elastic modulus and stiffness

- Calculation : using elastic modulus and stiffness

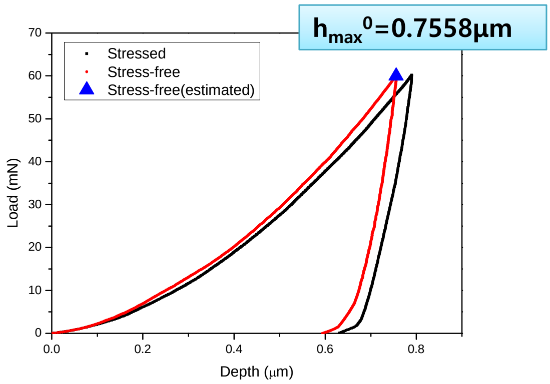
$$E_{eff} = \frac{1}{\beta}\frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \quad (\beta=1.04)$$

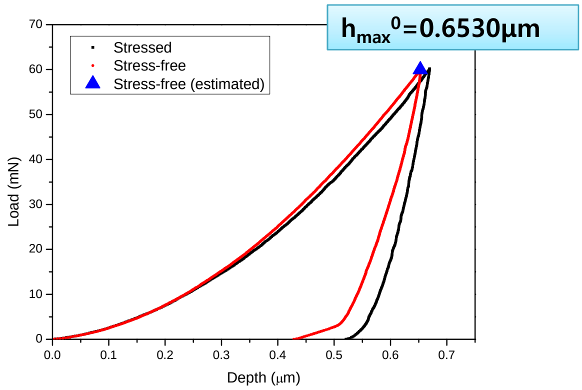
- Direct measurement : by SEM



➢ Average error : 7.54%

Estimation of stress-free state





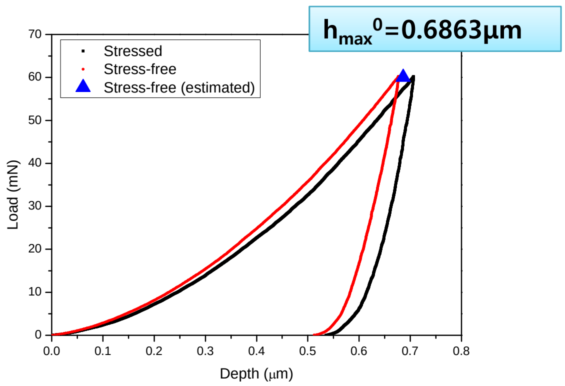
- Experimental condition : Tensile stress state

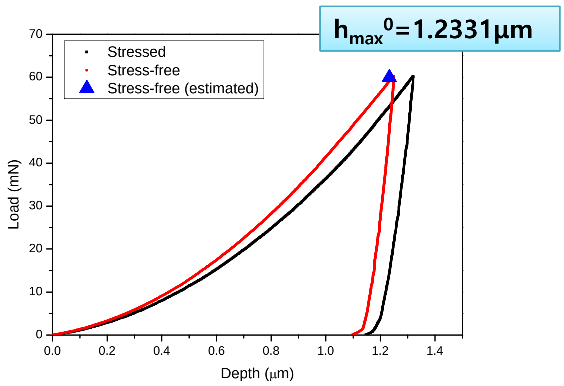
Materials	$\sigma_{res}^{x} + \sigma_{res}^{y}$ (MPa)
SKD11	138.24

- Experimental condition : Tensile stress state

Materials	$\sigma_{res}^{x} + \sigma_{res}^{y}$ (MPa)
SUS304	196.62

Estimation of stress-free state





- Experimental condition : Tensile stress state

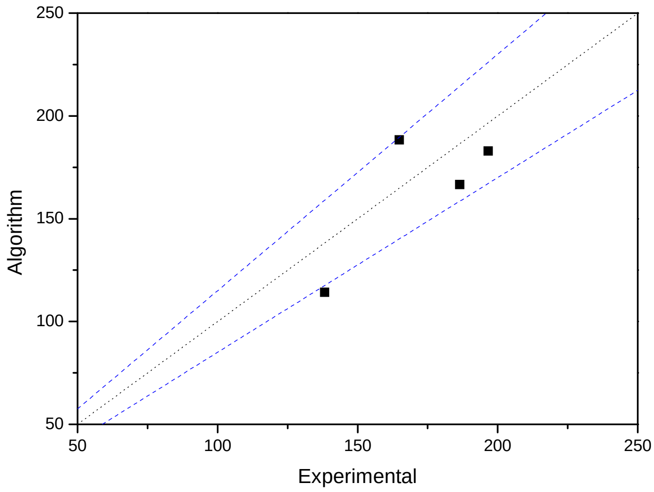
Materials	$\sigma_{res}^{x} + \sigma_{res}^{y}$ (MPa)
SUS316	164.88

- Experimental condition : Tensile stress state

Materials	$\sigma_{res}^{x} + \sigma_{res}^{y}$ (MPa)
Al6061	186.41

Evaluation of residual stress

$\sigma^x_{res} + \sigma^y_{res}$ (MPa)



➢ Average error : 12.3%

Experimental details (Macro scale)

Testing equipment : AIS3000

Specimens **(total 23 materials)**

* 14 power-law hardening materials (+ API steels)
* 6 linear hardening materials
* 3 nonferrous materials (Al alloys)

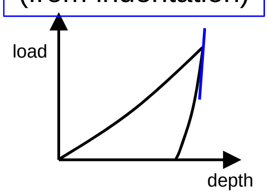
Analysis

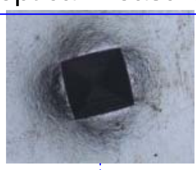
$$\beta = \frac{1}{E_r}\frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}}$$

Stiffness (from indentation)

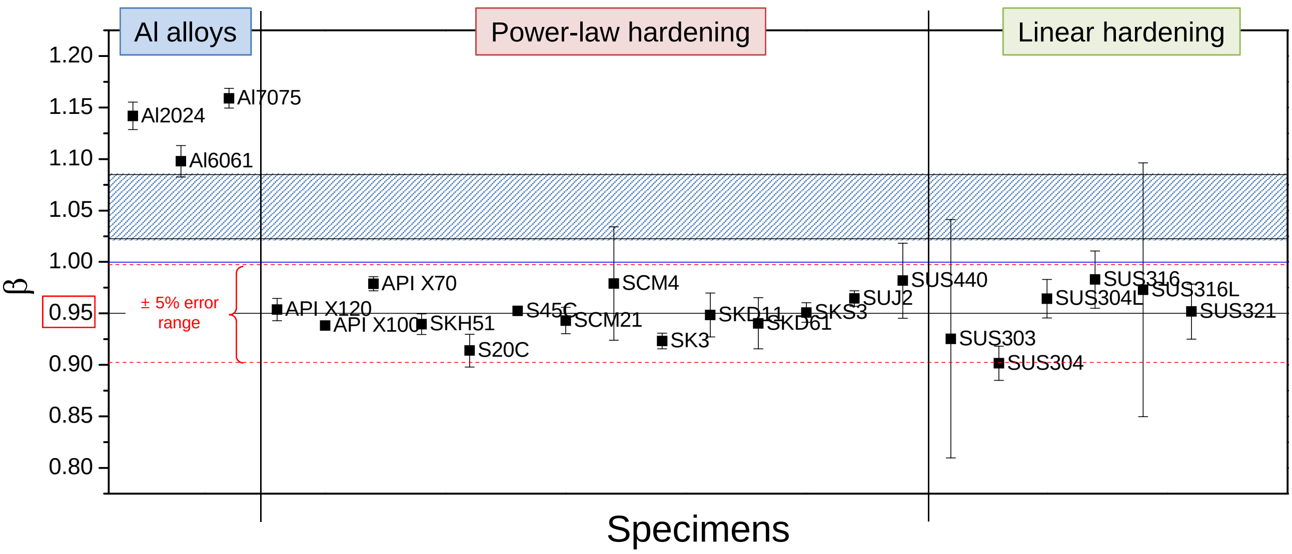
Contact area (from optical measurement)

Elastic modulus (from tensile test)





Results (Macro Scale)



Previous researches (1.0226 ~ 1.085)

Theoretical analysis (1, flat punch)

Experimental results

For most ferrous materials

$\underline{\beta = 0.95}$ (±5% error range)

Contents

1) Introduction (Evaluation of residual stress using IIT)

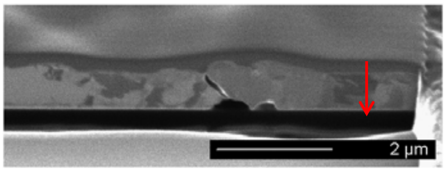
2) Estimation of stress-free state using Elastic modulus & Stiffness

3) Evaluation of through-thickness residual stress

Introduction

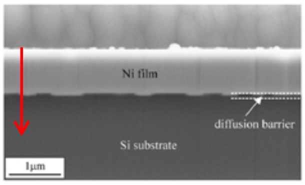
▶ Through-thickness Residual stress

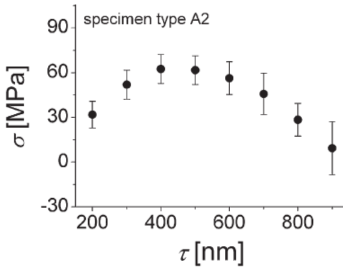
[M. Wohlschlögel, 2011]

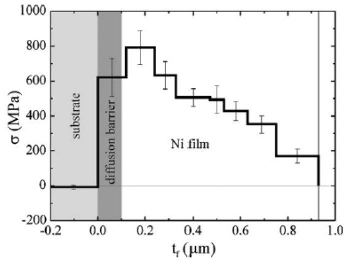


Through-thickness direction

[S. Massal, 2007]



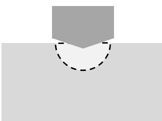




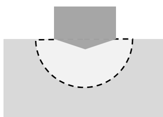
Through-thickness residual stress

Procedure

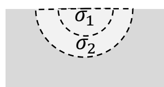
Step 1. Step 2. Step 3. Step 4.



→



→ $f(\sigma_1, \sigma_{1+2}) = \sigma_2$ →



Measure σ_1

Measure σ_{1+2}

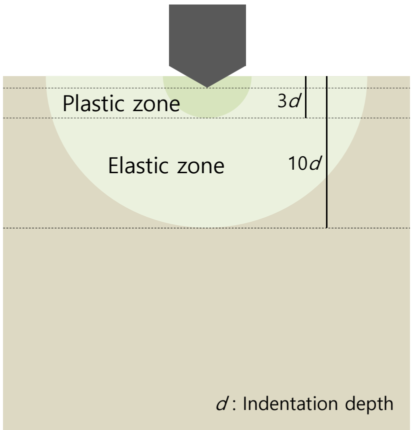
Issue

(1) Sensing depth (measurement of residual stress using IIT)

(2) Residual stress separation : $(\sigma_1, \sigma_{1+2}) \rightarrow \sigma_2$

Issue 1. Sensing depth (1)

$$\frac{1}{\psi}\frac{\Delta L_1}{A_{c,1}} = \frac{(1+p)}{3}\sigma_{res,1}$$ → z-direction Distribution area?



Indentation deformation

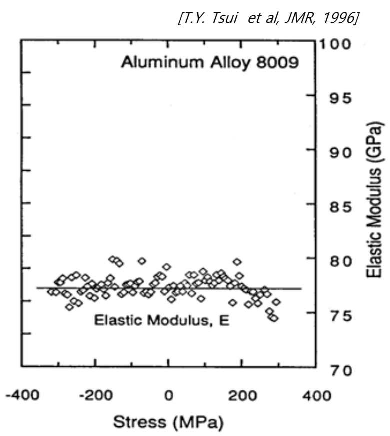
: Elastic deformation + Plastic deformation

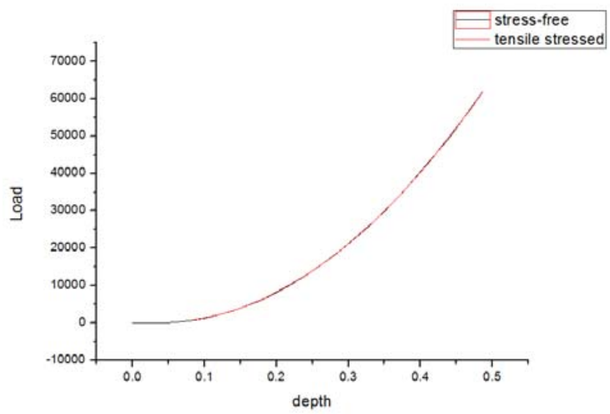
Residual stress
- Ineffective stress
- Effective stress
  - Hydrostatic stress
  - Deviatoric stress

Indentation depth ⟷ matching ⟷ z-direction Plastic zone size

Issue 1. Sensing depth (2)

Relation between **elastic deformation** and **residual stress**





FEM simulation : elastic property
Tensile residual stress : 300 MPa

Issue 1. Sensing depth (3)

Approach : sensing depth = plastic zone size

Issue : Effect of residual stress on plastic zone size

- Modifying Gao's equation

$$h_r = c = \left(\frac{1}{3}\frac{E}{\sigma_y}tan^2\gamma\right)^{\frac{1}{3}} h \quad \xrightarrow{\sigma_y \rightarrow \sigma_y^{app}} \quad h_r = c = \left(\frac{1}{3}\frac{E}{\sigma_y^{app}}tan^2\gamma\right)^{\frac{1}{3}} h$$

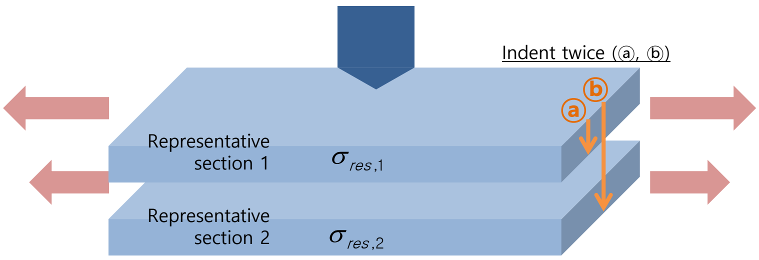
Compressive : $\sigma_y < \sigma_y^{app}$: c ↓

Tensile : $\sigma_y > \sigma_y^{app}$: c ↑

Compressive (300 MPa)	Stress-free	Tensile (300 MPa)
0.9	1	1.2

(relative size of plastic zone, using ABAQUS)

Issue 2. Residual stress separation (1)



Evaluation of Through-thickness residual stress

object residual stress

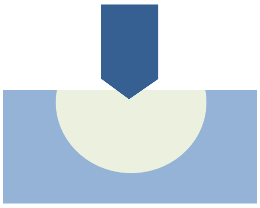
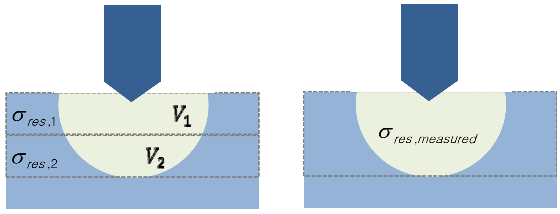
$\sigma_{res,1}$ $\sigma_{res,2}$

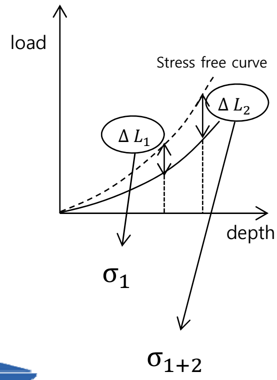
measured residual stress

$\sigma_{res,a}$ $\sigma_{res,b}$

measured		object
$\sigma_{res,a}$	=	$f(\sigma_{res,1})$
$\sigma_{res,b}$	=	$f(\sigma_{res,1}, \sigma_{res,2})$

Issue 2. Residual stress separation (2)

Diagram		
state	stress-free state	stressed state
work of Indentation	W	$W + \Delta W$ ΔW : change of the indentation work by residual stress



$$\Delta W(\sigma_{1+2}) = \Delta W(\sigma_1) + \Delta W(\sigma_2)$$

$$dW = \sigma \cdot d\varepsilon^p$$

$$\Delta W_{1+2} * (V_1 + V_2) = \Delta W_1 * V_1 + \Delta W_2 * V_2$$

measured	object
σ_1, σ_{1+2}	σ_2

Conclusion

- Load-depth curve of stress-free state could be estimated by the parameters which were measured from the stressed curve including real contact area under stressed state with the concept of invariant contact area and stiffness.
- But we could not measure the real contact area when the contact depth is very small in nanoindentation or for some nonmetal materials.
- By Sneddon's relationship, contact area was estimated using elastic modulus and stiffness.

$$E_{eff} = \frac{1}{\beta}\frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \quad \text{(Correction factor } \beta = 1.04\text{)}$$

- Algorithm for estimation of stress-free state has good agreement with the experimental results.
- When we evaluate the residual stress, the sensing area of the residual stress is the plastic deformation area and can be predicted by Gao's equation.

Thank you for your attention