Instrumented Indentation Technique
(IIT)

Prepared by

[Head Office] #407, Byucksan Digital Valley 6, 481-4,
Gasan-dong, Guemcheon-Gu, Seoul, 153-803, Korea
TEL: 82-2-884-8025 / FAX: 82-2-875-8018
E-Mail: sales@frontics.com/ info@frontics.com

[Research center] 3rd floor, Research Institute of Advanced Materials
Seoul National University, San 56-1,
Shinrim-dong, Kwanak-gu, Seoul 151 -742, Korea
www.frontics.com

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FRONTICS, Inc

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1. Outline of IIT(Instrumented Indentation Technique)

Structures constructed in the rapid economic growth age of 1970s have been seriously deteriorated by continuous use for more than 30 years, and this may cause large industrial disasters.

Therefore, for safe use of such aged facilities, it is necessary to estimate precise life by evaluating properties on the materials regularly.

For safe use and maintenance of obsolete equipment, it is necessary to adopt the technology that allows monitoring material characteristics according to age-related degradation in industry fields in addition to the existing laboratory based mechanical properties evaluation method. It is also urgently needed to complement the system, the system obtains/collects data of material degradation and life prediction to determine the risk and periodic time of inspection of the equipment and it then determines time to replace and repair.

For industry utilization of developed materials, it is important to obtain reliability of materials and parts based on properties accumulation by applying appropriate evaluation method. However, existing standard method for material property assessment of uniaxial tensile and fracture mechanics provide considerable information on transformation and destruction behavior, but it require bulk specimen which can be damaged on facilities and structures with notch while collecting specimen from, so it ironically can harm the safety of structures, and even worse, specimen taken on properties may be different from original properties of structure at the site as a result of relaxed stress and damage made while being collected.

To consider the requirements of test method to apply properties evaluation in micro materials, it should be able to evaluate hundreds of Å thin film thickness alone, and it should be able to limit the properties evaluation area to inner part of grain to evaluate mechanical properties of each phase.

To solve the said problems, automated indentation testing method which is semi non-destructive, and simple to operate directly on site and nano indentation test method which holds nm indentation depth with extreme low load are proposed. [1,2]

Unlike existing hardness test method that measures hardness alone, these testing methods obtain the indentation load-depth curve and measures various mechanical properties related to deformation and fracture with the indentation load-depth curve. With automated indentation testing method, it is able to measure not only hardness but also the flow properties including yield strength, tensile strength and strain hardening index [1] and also, it is able to evaluate characteristics of fracture with appropriate modeling. [3,4]

Researches regarding nano indentation testing method were mainly focused on evaluating hardness and elasticity modulus on thin film and Nano phase by using a sharp indenter with certain angle [2, 5]. But recently, researches are on progress to assess high-level properties like flow curve by integrating analyzed technology from automated indentation testing method.

Furthermore, properties evaluation methods mentioned above are able to evaluate weak local area like welded area by varying size and shape of indenter [3]. Besides, it is able to evaluate material behavior under various degradation situations by adding thermal atmosphere device.
In this page, we have outlined research cases and application of advanced indentation technique to evaluate mechanical properties in macro and nano scale, and went over the direction forward in the areas of material properties evaluation and diagnosing the safety.

2. Application of Advanced Indentation System

Advanced indentation method, an improved method of existing hardness test method, is a various mechanical properties evaluation method by setting actual load and depth displacement during the indentation as an initial data. There is no restriction in size and preparation of specimen due to use of same form of indenter and fine spherical indenter of 1mm in diameter. It is also able to evaluate detailed properties on minute weak area of mm scale. [1]

Therefore, it is able to collect hyperfine specimen without harm to large structure and evaluate degradation properties on the specimen. However, there are problems to have precise evaluation with collecting hyperfine specimen from the structure in case of there is serious deformation between the surface and the inside of the material or alleviated residual stress occurred

As it is necessary to have in-situ evaluation of material properties, a non-destructive testing device is attached to on-site structure. At this point, Frontics Inc. has developed portable Advanced Indentation System to attach to on-site structure as Fig. 1.

Advanced Indentation System 1000 is run by a motor which is able to precise location control in micron scale, and it also features sensors for load and displacement.
Therefore, as it takes motor caused load and displacement signal concurrently, it shows initial test result as shown in Fig.2. Test results are analyzed with flow curve evaluation software to evaluate strength properties related to deformation.

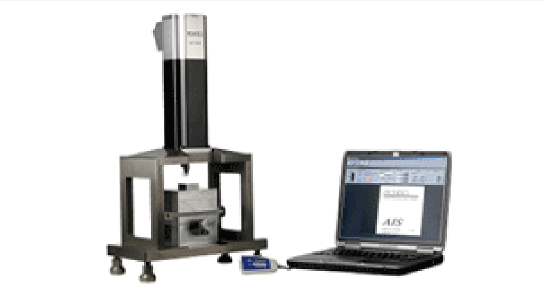


Fig. 1. Advanced indentation system developed by Frontics, Inc.

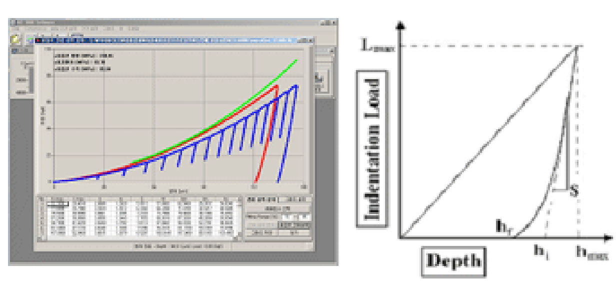


Fig. 2. Analysis of indentation load-depth curve.

The portable AIS is emphasized on its small size, weight and easy operation on-site, so it is made to allow user to move the entire system easily, and remote control of the system is considered as well. For lighter weight of the tester, low specific gravity high strength alloy was used for its frame, and interface board and driver are separated from the tester by using a control box. A high performance laptop is included to collect and analyze test materials, and for performing on-site test on various structures including pipe and plate surface, the V-block, magnetic systems and mechanical way are applied to securely fix the system to structures.

For the practice of advanced indentation technique, nano indentation test with minimized indentation load depth was used for evaluating hardness and elasticity modulus index on thin film only [2,5]. Recently, researches about evaluating electronic parts and analyzing damages on uneven thermal steel have been progressed by various use of advanced indentation technique.[6,7,8,9]

Concretely, it is not single thin film evaluation, it is applied to evaluate multi-layered thin film [7] with multi-indentation test varies by indentation depth or consistent evaluation of contact-properties by loading pulse signal on the load at the same time.[6]

To analyze deformation phenomenon of thin film, it evaluates flow properties of thin film using uniaxial indentation flow strength testing technique which used in automated indentation testing technique.[8] It is also able to investigate determination of grain boundary, occurrence of fine structure during the heat treatment, overall transformation and thermal equipment by determining fine structure of steel, and evaluating strength properties on grain and transgranular of each test material.

And for the residual stress, a problem area in thin film, theoretical and experimental researches are in progress, too. [9] Looking at the specific development direction of nano indentation tester hardware, improvement in quality of actuator related to load/displacement measurement and sensors are in progress, and researches related to observe indentation trace and various properties evaluation under various condition by adding Atomic Force Microscopy (AFM) module, indenter movement interfacial adhesion measurement module fatigue load module and high temperature testing module are in progress as well.

Fig. 3 and Fig. 4 show nano indentation system and observation result of indented surface of 15 ㎛ by using AFM that confirms indentation trace of 2 line nano indentation test across each grain.

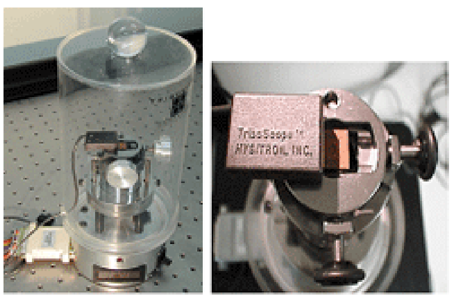


Fig. 3. Nanoindentation system combined with atomic force microscope.

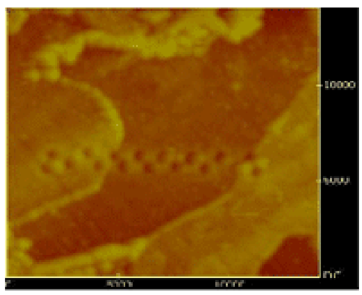


Fig. 4. Atomic scale surface morphology observed using AFM.

3. Macro scale mechanical properties evaluation

Unlike existing hardness properties evaluation, mechanical properties evaluation of bulk material using Advanced Indentation System is focused on various strength properties evaluation related to deformation which can be obtained from uniaxial indentation test and researches to predict brittle fracture and transition fracture. In this study, theory of flow properties evaluation and experiment results obtained from spherical indentation test are introduced, and yield properties and tensile strength related researches are presented as below.

A. Contact properties

Fig.2 show indentation load depth curve obtained from Advanced Indentation System, and generally user gets experiment data of each step of indentation from single indentation test by using multi-indentation testing method.

Indentation depth is determined with movement of indenter which is measured with displacement measuring sensor and contact depth can be different from measured indentation depth according to elasticity, plastic deformation around the indenter.

Contact depth deformation caused by elastically bended phenomenon around the indenter can be determined by using S value, the initial slope of the tangent of indentation load depth curve. S in Fig.2 represents stiffness, and contact depth hc* with elastic deformation can be determined as follow: [2]

$$h_c^* = h_{max} - \omega \frac{L}{S} \quad (1)$$

L is a load, ω is a constant related to geometric shape of indenter, 1 is used for flat punch, 0.75 is used for spherical indenter and 0.72 is used for Vickers indenter.

As Fig.5 shows, pile-up, a phenomenon piles deformed materials around the indenter or sink-in, a phenomenon sinks in deformed materials under the indenter may occur as plastic deformation is produced around the indenter.[10]

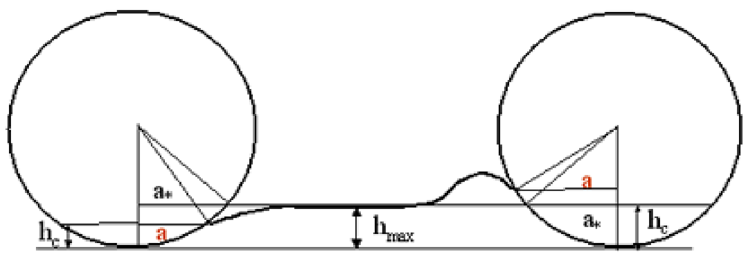


Fig. 5. (a) surface sink-in and (b) pile-up phenomena around spherical indenter.

In this case, contact depth deformation by pile-up/sink-in phenomenon can be determined as follow. [11]

$$a^2 = \frac{5}{2}\frac{2-n}{4+n}a_*^2 = \frac{5}{2}\frac{2-n}{4+n}(2Rh_c^* - h_c^{*2}) \quad (2)$$

n represents strain hardening index, R represents the radius of the indenter, and a represents the radius of contact. The radius of contact obtained from formula (2) is used for determining hc, the contact depth by using geometric relation, and this will be used to evaluate subsequent average contact pressure and indentation strain.

B. **Flow properties**

In subsequent yield zone of tensile curve obtained from uniaxial tensile test, true stress and true strain have relation as follow. [12]

$$\sigma = K\varepsilon^{n}$$

$$(\sigma : \text{True stress},\ K : \text{Stress factors},\ \varepsilon : \text{True strain}) \quad (3)$$

Strain in indentation test using spherical indenter represents a term the radius of contact and contact depth as formula (4) by applying the closest constant to maximum value of the strain(α, usually 0.1) which is determined by computing material depth directional displacement and then differentiated.

$$\varepsilon = \frac{\alpha}{\sqrt{1-(a/R)^2}}\frac{a}{R} \quad (4)$$

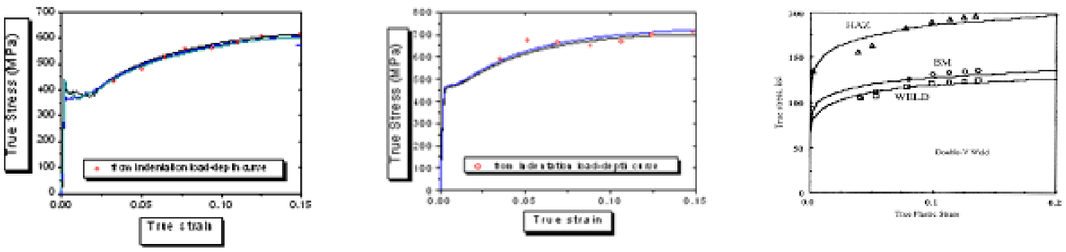
Deformation on the bottom of spherical indenter is divided into three stages of elasticity, plasto-elasticity and plastic deformation. In elasticity stage, it does not leave indentation trace with reversible deformation, and plasto-elasticity is a stress field occurs on the spot of 0.5a in the bottom of indenter and it expands to surface.

However, usually expansion to surface is very rapid due to surface friction, material on the bottom of the indenter stays in the perfect plasticity deformation zone. In this perfect plasticity zone, the stress on the bottom of the indenter has average pressure Pm, the indentation load to contact area and a constant ratio (Ψ, usually 3). [12]

$$\sigma = \frac{P_m}{\Psi} \quad (5)$$

Contact depth and the radius of contact are determined by using Indentation load depth-displacement curve obtained from muti load removal test method. With this, real stress and real strain are determined.

Based on above, evaluations on various materials have been performed, when it is compared to uniaxial indentation test results, it is able to evaluate flow properties precisely in perfect plasticity zone. The flow curve can be applied to evaluate various properties such as yield strength, strain hardening index, tensile strength by adopting Hollomon equation, optimizing and extrapolation. Fig.3 shows flow curves obtained from tests on a structural steel of SM50 and pressure vessel steel of SA508 [1] and result of flow curve by applying existing research on welded area.[3]



(a) SM 50 (b) SA508 cl. 3 (c) welding joint of PV steel

Fig. 6. Comparisons between flow properties calculated from advanced indentation (dotted line) and those from standard tensile tests(solid line).

C. **Yield strength**

Researches to evaluate yield strength by using indentation test are two. A simple method is to assume the indentation hardness and yield strength ratio as approximately 3, and the other method is to use flow curve obtained from spherical indentation test. In other words, yield strengthσ_y is calculated as formula (6) by using constant M which is obtained by presenting indentation test result as Meyer formula and constant β obtained from uniaxial tensile test.

$$\sigma_y = \beta M \quad (6)$$

Although we obtained appropriate yield strength of various carbon steel, additional tensile test is still needed. Therefore, we have evaluated yield strength by extrapolating the flow curve to yield spot, obtained cross point at 1% which is yield strain of general material, and we confirmed that it is well agreed with that of uniaxial indentation test. [1]

D. **Strain hardening exponent**

Meyer's formula (7) can independently determine strain hardening exponent in indentation test,[13], but it has large margin of error due to its difficulty to evaluate accurate contact properties.

$$P_m = K'\left(\frac{a}{R}\right)^n \quad (7)$$

Therefore, first we considered pile-up and sink-in to use corrected contact properties formula, then we applied indentation strain formula which used for obtaining flow curve, developed average contact pressure by indentation load method and suggest the formula to determine strain hardening index (8) as material constant term of A and term related contact factors and we have evaluated strain hardening index which is well agreed with uniaxial indentation test on various steel as shown in Fig.7. [14]

$$L = A\frac{(h^c)^n}{a^{n-2}} \quad (8)$$

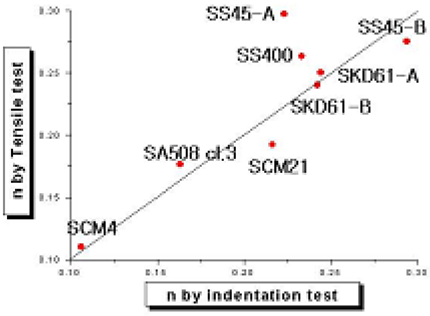


Fig. 7. Comparison of work hardening exponents from advanced indentation and from tensile tests.

E. **Indentation strength**

When the flow curve is evaluated, uniaxial indentation strength test can be determined as formula (9) by using the theory that supports strain hardening index and uniform elongation εu are same, and indentation strength on various materials are evaluated.

$$\sigma = K\varepsilon_u^n = Kn^n \quad (9)$$

F. **Non-uniform elongation**

Uneven deformation occurs right after the yield on some steel, may decrease machinability and elongation. Therefore, uneven deformation has estimated in indentation test, we have set quantification of non-uniform elongation εL from pile-up occurred during uneven indentation deformation as shown in Fig. 8. In other words, we measured height of pile-up, hm which occurred around indentation trace after the spherical indentation and by relating pile-up area with plastic modulus E, a material property and hardness value Hv, we determined quantification non-uniform elongation that occurs on most materials as formula (10). [15]

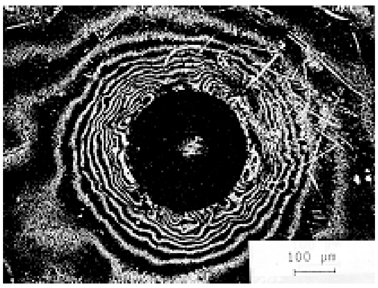


Fig. 8. Crown-like fringe pattern for 13-3-11 Ti alloy with Lüders strain.

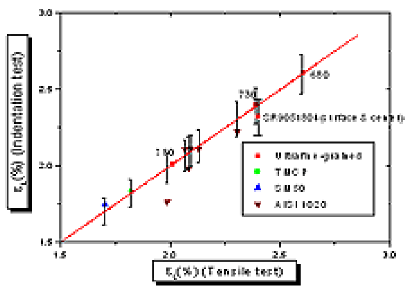


Fig. 9. Comparison of Lüders strain from advanced indentation and from tensile tests.

G. **Fracture toughness**

As existence of micro-crack may cause rapid destruction of structure, it is necessary to have fracture toughness (J_{IC}) test to evaluate characteristics related to fracture. Because of problem in collecting specimen and complexity of test procedure, safety of structure has been evaluated with simple impact test. However, as the impact test is result of dynamic properties evaluation, it is rarely related to static fracture toughness. Therefore, to solve said problem, researches using automated indentation testing technique has been processed, we made a modeling to evaluate fracture toughness by applying the automated indentation technique to lower self energy zone of brittle nature and transition zone.

$$K_{JC} = C\sqrt{\varepsilon_f^* \cdot l_0^* \cdot E \cdot \sigma_y} = C_0\sqrt{n \cdot l_0^* \cdot E \cdot \sigma_y} \quad (11)$$

εf* represents fracture strain, 0* represents microstructure characteristics distance which is size of grain, E represents elastic modulus and σy represents yield strength.

To determine fracture strain from above formula, an indentation test is needed. Therefore, automated indentation test itself enables to evaluate fracture characteristics by replacing term of fracture strain is with a proportional property of strain hardening index.

However, the above mentioned formula is not practical with its necessity of complex and various data about the properties so that researches on determining fracture toughness by indentation test itself are in progress.

In other words, we assumed that virtual fracture will occur when stress in the strength of fracture stress obtained from indentation test is occurred on the bottom of the indenter. Considering friction between indenter and specimen, we decided critical load Lf for imaginary fracture to reach fracture stress and depth hf, and imaginary fraction indentation strain energy (WIEF) was calculated by pent-up integral for the bottom of indentation load curve. [4]

In addition, in cryogenic state of lower shelf area, cleavage fracture energy that applied brittle Griffith energy balance concept is existing, fracture energy on transition area and upper shelf area Wf as formula (12) and WT energy which is used with cleavage fracture energy W0 are exist. As there is no crack in indentation, imaginary fracture indentation strain and overall deformation energy were considered equally. [14]

$$W_f = W_0 + W_T = W_0 + W_{IEF} \quad (12)$$

The relation between fraction energy and fraction toughness K_{JC} is presents as formula (13).

$$W_f = \frac{K_{JC}^2}{2E} \quad (13)$$

Therefore, when data about cleavage fracture energy and indentation strain energy to the imaginary fracture point are obtained, fracture toughness at temperature under below transition zone can be evaluated.

However, researches about fracture properties at upper shelf region where plastic deformation is predominant using indentation test are still needed. Researches on integrating automated indentation technique with ductile fracture behavior which is related to void growth are underway.

4. Micro mechanic properties evaluation

For the cases of nano indentation test technique of Advanced Indentation technique when decrease the indentation load to extreme low, it usually used in researches measuring plastic modulus or hardness of thin film based material. In recent years, it is now able to analyze down to minutest detail of deformation of indenter shape, flow properties evaluation and residual stress analysis those were applied to bulk material with existing method, are now able to analyze. Furthermore, in case of multiple phases are existing, or when evaluation on strength or degradation micro-structure is complicated, nano indentation technique can be applied to analyze each phase including nano phase. Detailed researches are as below.

A. Elasticity modulus and hardness of thin film

Evaluation on elasticity modulus and hardness of thin film can be determined according to the formula (1) of the relation to obtain depth between indenter and specimen by analyzing indentation load removal curve that was mentioned above in regard to contact properties evaluation. [2,5] We evaluated elastic recovery behavior of load removal curve as term of stiffness from slope evaluation, and with this, elasticity modulus and hardness are evaluated using each formula (14). Fig. 10 shows evaluation results of elasticity modulus and hardness according to thickness of W buffer layer of DLC thin film.

$$S = \frac{2E_r\sqrt{A}}{\sqrt{\pi}} \qquad (14)$$

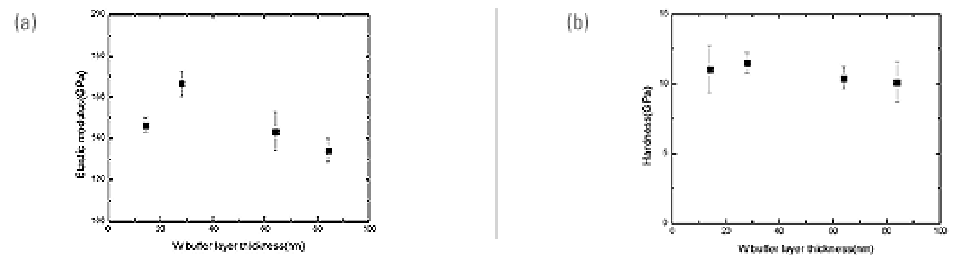


Fig. 10. (a) Elastic modulus and (b) hardness of DLC by nanoindentation method.

B. Flow properties of thin film

To evaluate strength characteristics of thin film, the theory used for existing spherical indenter was applied. However, existing 60 degree conical indenter was applied to evaluate flow properties because it is difficult to make spherical diamond in nano indentation situation.

In other words, even for conical indenter, bluntness may occur at the end of the tip with processing matter. Depends on this bluntness, deformation area according to indentation depth of conical indenter in response to deformation area according to spherical indentation depth can be analyzed.

For precise analyze in form of indenter, we have performed multiple indentation test using a standard specimen of fused quartz. As shown is Fig. 11, we performed 3 dimensional analyze of indenter form under the stable condition of elasticity modulus and decided valid radius of curvature to 1700㎛ for advanced conical indenter in the similar concept of spherical indenter as shown in Fig. 12

We confirmed increase in average contact pressure in relation to indentation depth by performing multiple nano indentation test for Au thin film of 0.56㎛ as shown in Fig. 13 and evaluated indentation stress and strain using flow properties evaluation formula (4) and (5) of automated indentation technique for bulk material. With this, flow properties of Au thin film was evaluated and it is shown in Fig. 14.

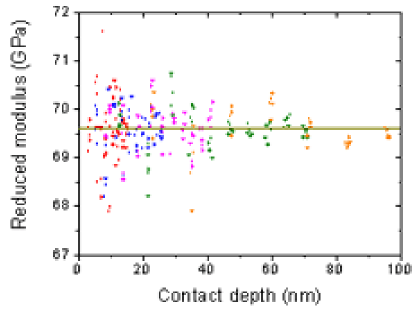


Fig. 11. Indenter tip calibration using the constant elastic modulus concept

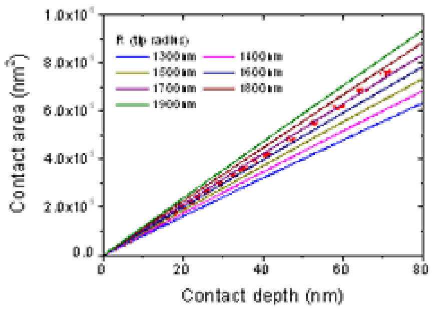


Fig. 12 Effective curvature radius from conical indenter

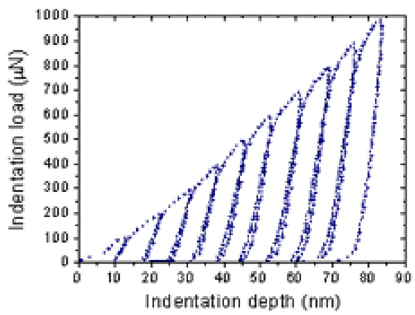


Fig. 13. Multiple indentation testing on 0.56㎛ thick Au thin film.

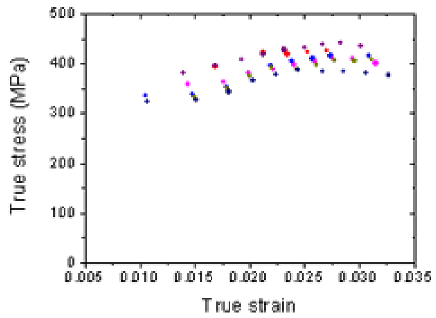


Fig. 14 Flow properties of Au thin film evaluated from nanoindentation

C. Residual stress of thin film

Residual stress on thin film analyze is evaluated by analyzing relationship between biaxial isotropy residual stress and indentation load.

First, we investigated dependence of indentation parameters depend on residual stress. Then we confirmed that hardness is not related to residual stress through experimental result and FEM simulation. [16]. Base on this, the modeling of deformation during indentation test pattern is presented as shown in Fig.15 to satisfy the change of indentation depth according to tensile and compressive residual stresses and no change in hardness and contact area at constant load.

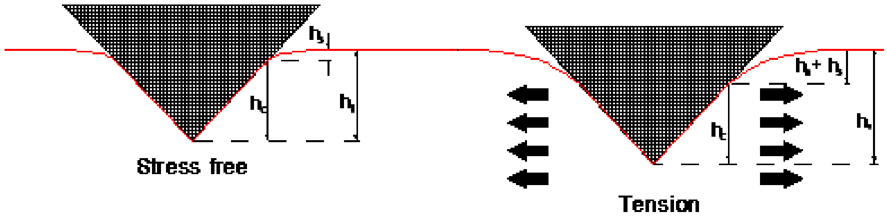


Fig. 15. Variation of surface morphology by the effect of residual stress using the concept of constant contact depth and hardness.

We have superimposed the common curve of contact load curve and indentation load curve at stress free situation and tensile stress situation

At this stage, we considered indentation load at each stress to have constant indentation depth, tensile residual stress brought same indentation depth due to its lower load than stress-free situation. As shown in Fig. 16, when residual stress is removed while keeping indentation depth at residual stress, contact depth and indentation load are increased as much as residual stress effect. Therefore, a relation between indentation load increase L+σ, average contact area of Acavg and residual stress σres is presented as formula (15).

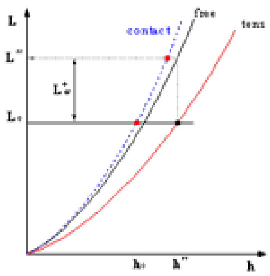


Fig. 16. Variation of loading curve by the effect of residual stress.

D. Analyzing nanophase

Nano indentation test with AFM is used for research to evaluate mechanical properties of multiphase materials.

We polished surface of API X65 steel, then etched the surface with nital etchant to distinguish the phase optically. In nano indentation test's case, indentation depth is limited in surface only, to compare the influence on surface stiffness layer that may occurred during the polishing, 50% Perchloric acid and 50% distilled water were used for electro-polishing as well, but the effect was insignificant.

First, we analyzed hardness of API steel on each phase by nano indentation test at 1000μN indentation load, and measured in standard method of MicroVinkers indentation test at 10gf of indentation load. Results of residual stress indentation trace are shown in Fig 17 and 18, and as Berkovich indenter that used in nano indentation test has same depth to area ratio with that of Vikers indenter, we compare them directly.

As Fig. 19 shows, the result presented fluid pattern for nano indentaion hardness according to distance from grain boundary, but average result showed that it is matched with that of MicroVickers which is indentation load is approved for multiple grains.

$$L_c^{+} = \sigma_{yes} \cdot A_c^{ave} \quad (15)$$

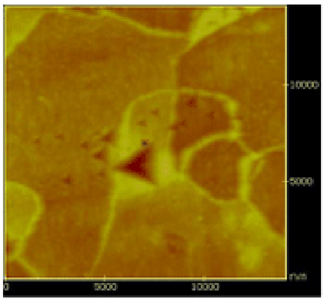


Fig. 17. Nanoindentation mark of API steel observed using AFM.

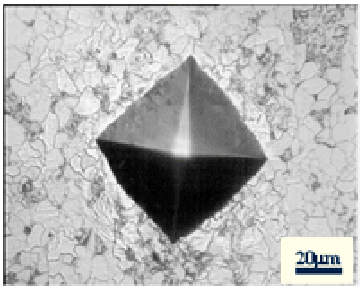


Fig. 18. MicroVickers indentation mark of API steel.

For the cases like refined grains into ㎛ scale through complicated hot rolling process like ultrafine grained steel we distinguished nanophase from its mechanical properties and we measured strength of each phase as well.

Unlike API steel, we could see not only ferrite but also 2nd phase such as bainite, Martensite on ultrafine grained steel by using AFM. Obtained various hardness values are shown in Fig. 20. For ferrite, we detected different type of ferrite (deformation-induced ferrite, DIF) and this ferrite has higher hardness value than regular cooling ferrite.

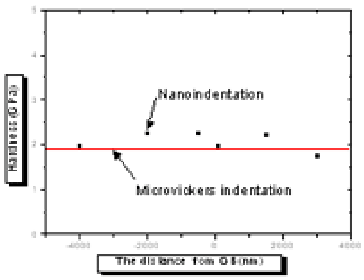


Fig. 19. Comparison between nanoindentation and micro Vickers hardnesses of API steel.

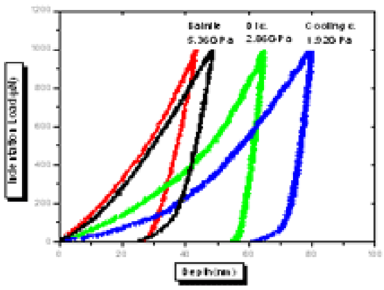


Fig. 20. Comparison of indentation load-depth curves and hardness values of nanophases in multiphase ultra-fine grained steels.

5. Summary & References

A. Summary

We have looked through the research cases on application of Advanced Indentation System to various structural steel and micro materials, and every property was matched with those from uniaxial indentation test within margin of error, and we confirmed that when we use automated indentation technique, it is able to evaluate fracture toughness in brittle and transition temperature zone with some data on material property.

In addition to above, we have confirmed that it is able to evaluate detailed deformation strength of thin film, residual stress and toughness properties according to fine structure through analyzing nanophase. From this, if Advanced Indentation System 1000 by Frontics, Inc., precise controllable hardware is used in conjunction with improvement in deformation and fracture related properties evaluation the automated indentation technique can be used for overall safety diagnosis and estimating structure life. Furthermore, the automated indentation technique can be substituted uniaxial indentation and fracture mechanics test, and for nano indentation test, it may be able to diagnose ultra-small sized part during its operation by setting various test condition.

B. References

01. Jeonghoon Ahn, Dongil Kown, Publication of the Korean Institute of Metals and Materials, 2000, in press

02. Oliver, W.C. and Pharr, G.M., J. Mater. Res., 1992, vol. 7, no.6, pp.1564

03. Murty, K.L., Mathew, M.D., Miraglia, P.Q., Shah, V.N. and Haggag, F.M., Mater. Res. Soc. Symp. Proc. vol. 503, 1998, pp.327.

04. Taeksang Byun, Jinwon Kim, Junhwa Hong, The 11th Strength of Material Papers, 1997, pp.467

05. Doerner, M.F. and Nix, W.D., J. Mater. Res., vol. 1, 1986, pp.601.

06. Page, T.F., Pharr, G.M., Hay, J.L., Oliver, W.C., Lucas, B.N., Herbert, E. and Riester, L., Mater. Res. Soc. Symp. Proc. vol. 522, 1998, pp.53.

07. Verdier, M., Niewczas, M., Embury, J.D., Hawley, M., Hawley, M., Nastasi, M. and Kung, H., Mater. Res. Soc. Symp. Proc. vol. 522, 1998, pp.77.

08. Field, J.S. and Swain, M.V., J. Mater. Res., vol. 10, 1995, pp.101.

09. Jarausch, K.F., Kiely, J.D., Houston, J.E. and Russell, P.E., Mater. Res. Soc. Symp. Proc.vol. 522, 1998, pp.119.

10. Cheng, Y.T. and Cheng, C.M, Phil. Mag. Letters, vol. 78, 1998, pp.115.

11. Norbury, A.L. and Samuel, T., J. Iron steel Inst., vol.117, 1928, pp.673.

12. Dieter, G.E., "Mechanical Metallurgy", McGraw Hill press, 1986, pp.286.

13. Tirupatiaiah, Y. and Sundararajan, G., Mater. Sci. Eng., vol. 91, 1987, pp.169.

14. EUnchae Jeon, Jeonghoon Ahn, Dongil Kown, Mechanical Society in Autumn proceedings The Korean Society of Mechanical Engineers, 2000, in press

15. Yeol Choi,, Dongil Kown, Publication of the Korean Institute of Metals and Materials, 2000, in press

16. Tsui, T.Y., Oliver, W.C. and Pharr, G.M., J. Mater. Res. vol. 11, 1996, 752.

17. Randall, N.X., Julia-Schmutz, C., Soro, J.M., Stebut, J. and Zacharie, G., Thin Solids Films, vol. 308-309, 1997, pp. 297.