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Evaluation of cryogenic mechanical properties of aluminum alloy using small punch test

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Abstract

The Small Punch Test (SPT) was developed to evaluate the softening and embrittlement of materials such as power plants and nuclear fusion reactors by taking samples in the field. Specimens used in the SPT are very thin and small disk-shaped compared to specimens for general tensile test, and thus have economic advantages in terms of miniaturization and repeatability of the test. The cryogenic SPT can also be miniaturized and has a significantly lower heat capacity than conventional universal test machines. This leads to reduced cooling and warm-up times. In this study, the cryogenic SPT was developed by modifying the existing room temperature SPT to be cooled by liquid nitrogen using a super bellows and a thermal insulation structure. Since the cryogenic SPT was first developed, basic experiments were conducted to verify the effectiveness of it. For the validation, aluminum alloy 6061-T6 specimens were tested for mechanical properties at room and cryogenic temperature. The results of the corrected tensile properties from the SPT experiment results were compared with known room temperature and cryogenic properties. Based on the correction results, the effectiveness of the cryogenic SPT test was confirmed, and the surface fracture characteristics of the material were analyzed using a 3d image scanner. In the future, we plan to conduct property evaluation according to the development of various alloy materials.

Keywords: small punch test(SPT), cryogenic, mechanical properties, 3d image scanner

1. INTRODUCTION

In recent years, there has been a growing concern surrounding the aging of materials attributed to the prolonged operation of thermal power generation facilities and the imperative to ensure the safety of pressure components within nuclear power plants [1-3]. Additionally, the assessment of mechanical properties in extreme environments, such as very low temperatures and high pressures, has become increasingly vital. This is particularly relevant in the context of emerging environmentally friendly energy sources, such as hydrogen energy and high-temperature superconducting magnets. Notably, there has been a significant emphasis on predicting hydrogen embrittlement behavior in structural materials exposed to high-pressure H2 environments [4, 5]. Likewise, for pressure vessels and pipes subjected to diverse extreme environmental conditions, evaluating the mechanical performance of materials is indispensable for ensuring overall structural integrity. Conducting mechanical property evaluations and projecting the lifespan of structural materials through the collection of on site structural samples under diverse equipment and system operating conditions for standardized testing encounters certain limitations. For instance, in the context of tensile testing aimed at assessing a material's yield strength and tensile strength, as well as Charpy impact testing to evaluate material toughness, obtaining test specimens directly from the site necessitates additional welding repairs to the damaged area. This, in turn, poses a potential threat to the structural integrity of the overall system. Consequently, there is a pressing need for non-destructive testing methods within the applicable scope that do not compromise the strength of on-site components [6, 7].

The Small Punch Test (SPT), developed in the 1980s, serves as a testing device designed to assess the mechanical properties of structural materials and determine their residual lifespan. It presents a non-destructive approach owing to the diminutive size of the disc-shaped specimens employed, usually with a diameter of a few millimeters and a thickness of 0.5 mm. The compact dimensions facilitate on-site sample collection without necessitating additional repairs.

Nevertheless, as of the current state, the Small Punch Test has not yet achieved a consensus on fully standardized procedures [5]. Various factors, such as specimen size, fabrication methods, the configuration of test machine jigs, and testing conditions, show variations. Furthermore, the standardization of assessment methods for mechanical properties and the residual lifespan of materials is incomplete, with research under diverse temperature conditions, including extremely high and cryogenic temperatures, still being insufficient.

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In this study, we designed and constructed a cryogenic SPT device using a liquid nitrogen cooling method with the purpose of evaluating the properties of structural materials in cryogenic environments. To assess the validity of the developed test device, we evaluated the mechanical properties using Aluminum 6061 T-6 material and observed and analyzed specimen surface fracture characteristics in both room temperature and cryogenic environments.

2. Estimation of mechanical properties through SPT

2.1. Force-Displacement Curve

Fig. 1. (a) represents a generalized configuration of the SPT, which has been developed and improved by many researchers. It consists of a mechanism where a disk-shaped specimen is placed between two dies, fixed in position, and force is transmitted from the load punch to the specimen.

Fig. 1. (b) shows the Force-Displacement curve obtained through the SPT. The force-displacement curve is typically divided into four distinct regions. Region I is the elastic bending region, influenced by the material's elastic modulus and Poisson's ratio. Region II is the plastic bending region, where the material's behavior deviates from linearity and exhibits a change in slope due to yielding. The yield load, P_y, is defined at the boundary between Region I and Region II. Region III is the membrane stretching region, where biaxial stress causes specimen elongation, and strain hardening leads to an increase in the curve's slope. Region IV is the plastic instability region, where as deformation continues, the specimen's thickness significantly decreases, leading to radial cracking and ultimately, failure. This region represents the maximum load and corresponding deformation.

2.2. Mechanical property estimation

SPT applies a force to the specimen through a steel ball, causing the force distribution to vary depending on the displacement. Unlike conventional tensile testing, it is difficult to accurately calculate the stress applied to the material. Therefore, various stress estimation methods are used. Because there are no defined specific guidelines for evaluating mechanical properties, ongoing standardization efforts are ongoing by the European Committee for Standardization (CEN Workshop Agreement) and ASTM WK61932 [9,10]. Consequently, many researchers have undertaken extensive studies to infer yield strength and tensile strength using the Force-Displacement curve obtained through the test.

Equation (1) is a commonly used equation for estimating yield strength, and equations (2), (3), and (4) are equations for estimating tensile strength.

$$\sigma_{YS} = \alpha_1 \cdot \frac{p_y}{t^2} + \alpha_2 \qquad (1)$$

$$\sigma_{UTS} = \beta_1 \cdot \frac{p_m}{t^2} + \beta_2 \qquad (2)$$

$$\sigma_{UTS} = \beta_1' \cdot \frac{p_m}{t} + \beta_2' \qquad (3)$$

$$\sigma_{UTS} = \beta_1'' \cdot \frac{p_m}{t \cdot d_m} + \beta_2'' \qquad (4)$$

Where, σ_{YS} represents yield strength, p_y is the yield load, t is the initial specimen thickness, and α_1, α_2 are defined as test constants. σ_{UTS} denotes tensile strength, p_m is the maximum load, d_m is the displacement at maximum load, and β_1, β_2 are defined as test constants. The experimental coefficients α, β used to estimate the mechanical properties are obtained through the normalization process of p_y, p_m, and d_m derived through SPT based on the mechanical properties derived through uniaxial tensile test.

Fig. 2 illustrates representative methods for estimating yield load, such as the Two-tangent, CWA, and Offset methods.

P_{y_Mao}[11] is defined as the intersection of the tangents defined in region I and region II of the F-D curve. At this time, the tangent in region I is defined as the point with the maximum slope, and the tangent in region II is defined as the point with the minimum slope. P_{y_CEN}[10] defines the intersection of two tangent lines obtained by the two-tangent method as the value projected perpendicularly to the curve.

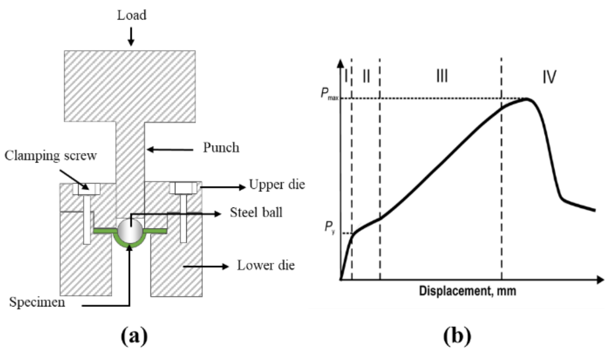

Fig. 1. (a) Schematic of SPT (b) General F-D curve [8].

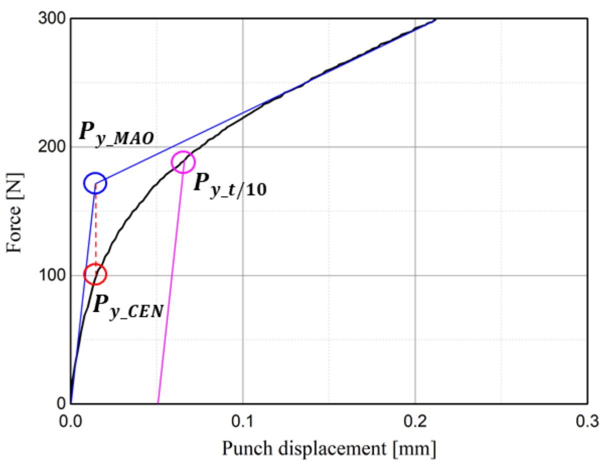

Fig. 2. Yield load estimation methods.

Offset method [12] is similar to the offset method in tensile testing. A line is drawn parallel to the tangent in the elastic section and passes through the point at t/10, and the point where it meets the curve is defined as the yield load.

In the case of ultimate strength, it has been reported that the influence of specimen thickness parameters is significant [13]. Equation (4), unlike equations (2) and (3), indirectly represents the effect of reducing the thickness of the specimen by considering the displacement at the maximum load.

3. Experimental setup

3.1. Cryogenic Small Punch Device

Fig. 3 illustrates the configuration of a cryogenic SPT device designed for liquid nitrogen boiling cooling, along with a view during the experiment. Liquid nitrogen is injected into the liquid nitrogen dewar to cool all internal structures of the device. During this process, the internal structures, initially at room temperature, undergo boiling heat transfer with the cryogenic liquid nitrogen. The cooling process persists until the surface boiling of the LN2 dewar filled with liquid nitrogen stabilizes, and the head part of the extension load is cooled until the boiling stabilizes.

The constructed cryogenic Small Punch Test (SPT) device transmits force sequentially through the extension load, load stick, and steel ball, resulting in force being applied to the specimen. The displacement of the deformed specimen during the test is measured by transmitting the displacement from the connection stick to the bellows flange. Subsequently, the strain rod, connected to the flange of the bellows, transmits the displacement to the strain gauge. The upper part of the bellows is welded in the form of a flange, preventing liquid nitrogen leakage and ensuring the strain gauge operates under the specified temperature conditions.

Additionally, the G-10 rod coupled to the extension load and the G-10 block at the bottom of the device limit conduction heat load from the testing machine into the specimen. The size of the specimen is 10 mm in diameter and 0.5 mm in thickness, and the size of the steel ball is 2.5 mm in diameter. Test speed is 0.5 mm/min, which is the generally recommended test speed for SPT [2]. Detailed experimental settings are shown in Table 1.

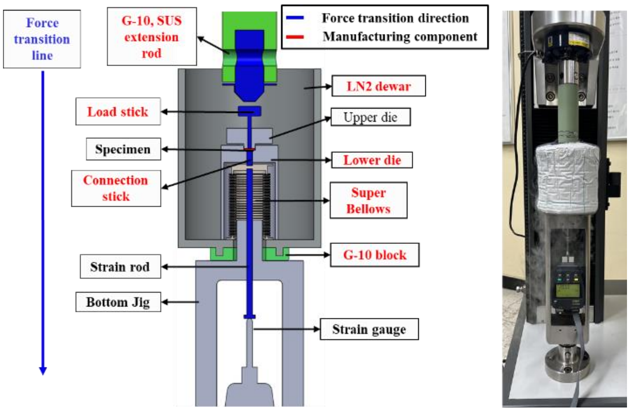

Fig. 3. Configuration of cryogenic SPT device.

TABLE 1.
TEST CONFIGURES.

Properties	Value
Temperature	296 K (RT), 77 K (CR)
Specimen diameter	10 mm
Specimen thickness	0.5 mm
Steel ball	2.5 mm
Test Speed	0.5 mm/min
Max. test load	5 kN
Load measurement	Load cell
Strain Measurement	Strain gauge

4. Experiment results

4.1. Small Punch Test Device Validation

Fig. 4 is the F-D curve at room temperature(RT) and cryogenic temperature(CR) of Al 6061 T6 material tested using a cryogenic small punch teste device. In the F-D curve, the x-axis was expressed as Deflection to observe

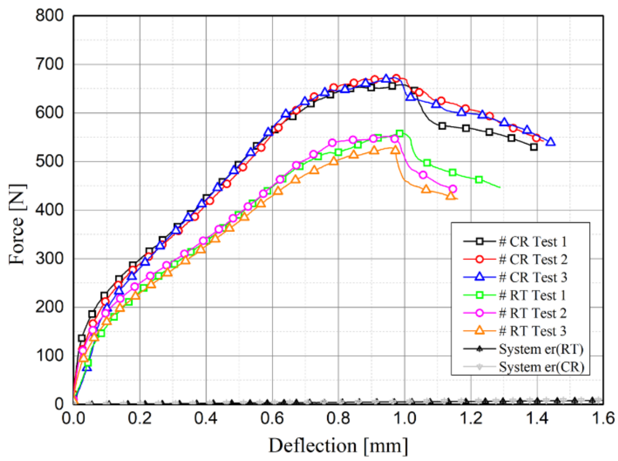

Fig. 4. F-D curves at RT and CR of Al 6061 T6.

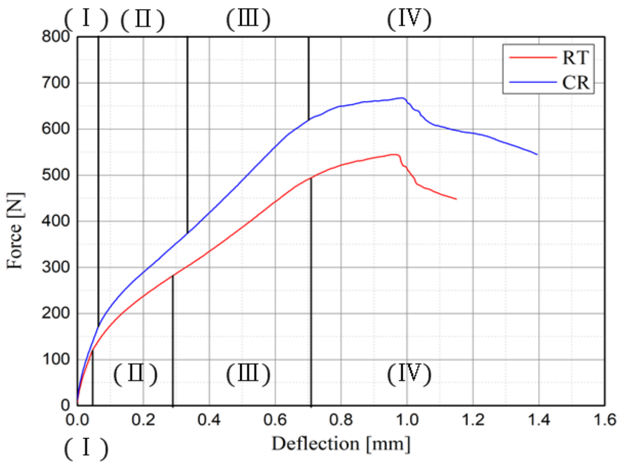

Fig. 5. Representative curves at RT and CR.

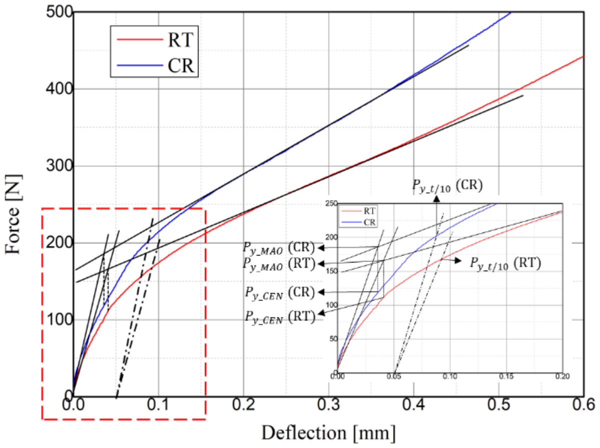

Fig. 6. Determination of $\boldsymbol{P_y}$ using estimation methods.

TABLE 4
RESULTS OF YIELD STRENGTH

Approach	Yield strength, σ_{YS} $[MPa]$			
	Two tangent	CEN	t/10 offset	Tensile test
RT Test	293.5	213.2	232.5	238
CR Test	328.8	225.6	280.3	280

TABLE 5
RESULTS OF ULTIMATE STRENGTH

Approach	Ultimate strength, σ_{UTS} $[MPa]$			
	P_m/t^2	P_m/t	$P_m/(t\cdot d_m)$	Tensile test
RT Test	410.5	427.3	314.6	274
CR Test	442	458	376.9	366

the specimen's inherent deformation. As can be seen in Figure 5, the reproducibility of the tester was confirmed as a result of three repeated experiments at RT and CR. In addition, system error represents the load according to deformation caused by the stiffness of the bellows structure, and the evaluation results showed 1% at RT and 1.4% at CR. These values were deemed negligible.

Fig. 5 is a representative curve at RT and CR using the average value of Al 6061 T6 data obtained through repeated experiments. When compared with the characteristics of each region of the general F-D curve, in the case of CR, the slope of membrane stretching increased in region 3, which showed that the increase in load was attributed to the effect of low-temperature hardening of the material.

4.2. Mechanical properties evaluation

Fig. 6 shows the yield load at RT and CR determined using the two-tangent, CEN, and t/10 offset methods from the F-D curve of Al 6061 T6 material derived through SPT. Table 2 shows the test constants used in general metal alloys, and the yield strength can be estimated from the previously determined yield load and test constants value [13]. Table 4 is the yield strength derived from equation (1) with reference to the test constants value (Table 2). When comparing the error with the yield strength of Al 6061 T6 obtained through a tensile test in a RT experiment, two-tangent method was 23.3%, the CEN method was 10.4% , and the t/10 offset was 2.3%.
In the case of CR, the two-tangent method was 17.4%, the CEN method was 19.4%, and the t/10 offset method was 0.09%. At both RT and CR, the estimated yield strengths using the t/10 offset method were similar to the tensile test results.

Table 3 is the test constants used in ultimate strength estimation equations (2), (3), and (4) [13]. Table 5 is the ultimate strength calculated utilizing constant values. Similarly, when comparing the error with the ultimate strength obtained from the tensile test in a RT, $P_m/t^2$was 49.8%, P_m/t was 55.9%, and $P_m/(t\cdot d_m)$ was 14.8%. In the case of CR, P_m/t^2 was 20.85%, P_m/t was 25.37%, and $P_m/(t\cdot d_m)$ was 3%. At both RT and CR, the estimated ultimate strengths using the $P_m/(t\cdot d_m)$ were similar to the tensile test results.

TABLE 2
YIELD STRENGTH ESTIMATION CONSTANTS.

Approach	Factor	Value
Two-tangent	α_1	0.442
	α_2	0
CEN	α_1	0.476
	α_2	0
t/10 offset	α_1	0.346
	α_2	0

TABLE 3
ULTIMATE STRENGTH ESTIMATION CONSTANTS.

Approach	Factor	Value
P_m/t^2	β_1	0.065
	β_2	268.81
P_m/t	β_1'	0.129
	β_2'	286.7
$P_m/t\cdot d_m$	β_1''	0.277
	β_2''	0

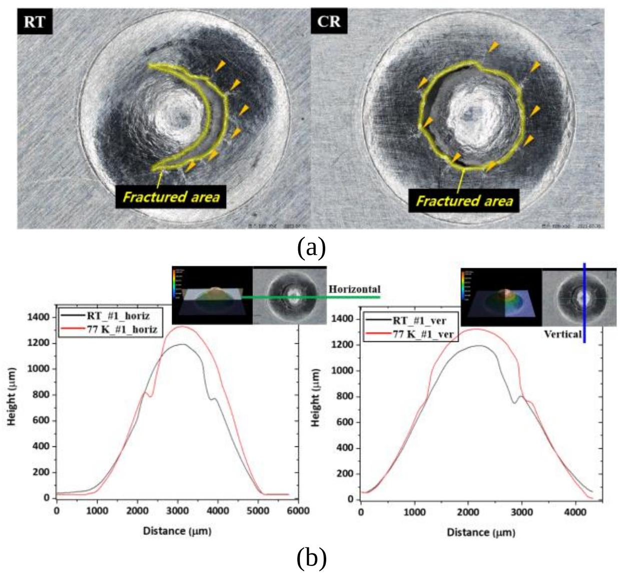

Fig. 7. Analysis of specimen fracture surface using 3D image scanner (a) Specimen fracture surface at RT,CR (b) Height of specimen indentation at RT, CR.

4.3. Surface fracture characteristic of specimen

Fig. 7 shows the surface fracture characteristics of Al 6061 alloy observed using a 3D scanner at RT and CR. In the case of the fracture surface at CR, the fracture area is generally wide unlike RT, and it may be seen that the strain energy (U) has a larger CR result in the Force-Deflection curve. Fig. 7 shows the specimen indentation depth at RT and CR. From the results of the horizontal and vertical line profiles, it could be observed that the width of the indentation was similar at RT and CR, but the height was remarkably high at 77 K

5. Conclusion

In this study, we developed a cryogenic small punch test device that can utilize the liquid nitrogen boiling cooling method and conducted evaluations of the mechanical properties of aluminum alloy 6061 T6 material at room temperature(300 K) and cryogenic temperature(77 K) to validate the effectiveness of the test device. The mechanical properties obtained through the small punch test were compared with the results from tensile tests performed on the same material.

Upon evaluating the system error of the developed small punch test device, it was determined that overall test device deformations remained negligible, measuring at 1% at room temperature and 1.4% at cryogenic temperature. Additionally, the reproducibility of repeat test results was confirmed.

Given the absence of a specific method for evaluating the mechanical properties of the small punch test, we evaluated these properties using test constants established for general metal alloys, as referenced in the literature. For yield strength, with the application of the t/10 offset method, the smallest error was confirmed at 2.3% at room temperature and 0.09% at cryogenic temperature, relative to the tensile test results. For tensile strength, utilizing the $P_m/(t \cdot d_m)$ method resulted in the smallest error at 14.8% at room temperature and 3% at cryogenic temperature, compared to the tensile test results. These results were confirmed to be consistent with previous research results [13].

As a result of observing the fracture characteristics of the specimen surface using a 3D image scanner, it was confirmed that the fracture area was widely distributed throughout the specimen in the case of cryogenic temperatures. This phenomenon is attributed to the increased strain hardening rate and higher strain energy (U) observed in the small punch test, as compared to the results obtained from the force-deflection curve.

The developed cryogenic small punch test for liquid nitrogen boiling cooling can be used to evaluate mechanical properties by manufacturing various alloy materials into miniaturized specimens. In the future, hydrogen withdrawal characteristics can be evaluated through small punch tests on alloys exposed to a hydrogen environment.

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