Evaluation of ductile-brittle transition temperature of anisotropy material by small punch test with U-shaped notch

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Abstract: Miniature and standard specimens, were cut from the anisotropy materials with axial, central and radial directions to study the mechanical property. In the paper, main research focused on the small punch test (SPT) with un-notched and U-shaped notched specimen in low temperature. Through the small punch energy variation with temperature, the ductile-brittle transition temperature by the small punch test (TSP) can be determined. The results indicated that there was no obvious difference among three different directions in transformation temperature of SPT with un-notch specimens, and it cannot represent upper plateau impact energy of three different directions. And the SPT with U-shaped notched specimens can determine the differences of upper plateau fracture energy of three different directions. Therefore, SPT with U-shaped notch specimens was more useful to evaluate the material anisotropy.

Keywords: Small punch test; Low temperature; Ductile-brittle transition temperature; U-notch grooves specimens; Anisotropy

1. Introduction

The small punch test (SPT) originated in the 1980s and was proposed by Baik [1] et al, by which various material properties could be obtained from a fairly small disk specimen with an almost nondestructive. The small-punch testing method has been applied successfully to evaluate a variety of material mechanical properties, such as yield strength, rupture strength, ductile-brittle transition temperature, fracture toughness [2] and creep performance [3]. The index of material embrittlement commonly was represented by the ductile-brittle transition temperature by the small punch test (TSP), which is associated with standard ductile to brittle transition temperature by Charpy Impact test (TCVN) [4]. Due to the bidirectional force, the specimens were insensitive to the anisotropy by SPT. The mechanical properties of each anisotropic material cannot be obtained in different directions by SPT. This paper study the A350 flanged forging by changing the conventional pattern specimens of SPT, in order to effectively characterizing the anisotropic materials, which has certain guiding significance for evaluating the toughening brittle characteristics of anisotropic materials and also has a certain help for the improvement of the technology of small punch test in the future research.

2. Test procedure

2.1 Chemical composition and mechanical performance test

In order to compare the correlation with the small punch test, the chemical composition analysis, tensile test and impact test of the material must be carried out.

2.1.1. Chemical composition analysis

The test material in this paper is A350 flanged forging, and its chemical composition was shown in Table 1, which conformed to the ASTM A350 standard.
Table 1. Chemical composition of A350 steel (wt%).

| Material  | C   | Mn  | Si  | Cr   | Mo  | Ni  | Cu   | V    | Nb   | S   | P   |
|-----------|-----|-----|-----|------|-----|-----|------|------|------|-----|-----|
| A350      | 0.20| 0.86| 0.2 | 0.12 | 0.05| 0.098| 0.14 | ≤ 0.001 | ≤ 0.001 | 0.015 | 0.025 |
| Standard  | ≤ 0.30| 0.60~| 0.15~| ≤ 0.030 | ≤ 0.12 | ≤ 0.040 | ≤ 0.030 | ≤ 0.02 | ≤ 0.040 | ≤ 0.035 |

2.1.2. Tensile test

Tensile test specimens were respectively carried along the axial direction L, circumferential direction C and radial direction R. According to Table 2, the mechanical properties on the materials of circumferential and radial basic are quite, while properties of axial direction on mechanical are the worst. The axial yield strength and tensile strength are slightly smaller, and the percentage elongation after fracture is obviously inadequate.

Table 2. The properties comparison of A350 flange material for three different directions.

|                      | Yield Strength (Rel) | Tensile Strength (Rm) | Percentage elongation after fracture A (%) |
|----------------------|----------------------|-----------------------|-------------------------------------------|
| Axial direction L    | 240.9                | 474.5                 | 10.8                                      |
| Central direction C  | 261.8                | 502.2                 | 26.4                                      |
| Radial direction R   | 258.4                | 498.6                 | 25.6                                      |
| Standard A350-LF2-1  | ≥250                 | 485~655               | ≥22                                       |

2.1.3. Impact test

In order to investigate the anisotropy of the impact toughness of A350 flanged forging, the flange should be sampled according to the fracture surface with different notch direction. For the impact samples, the axial direction (L), circumferential direction (C) and radial direction (R) are respectively used to the length direction of the impact sample. The Charpy impact test specimens sampling are shown in Figure 1, which cover the impact toughness of flange in all directions.

Figure 2 showed that CVN curves at three different temperatures of A350 samples. The Boltzmann function was used to fitting the ductile-brittle transition temperature curve. The ductile-brittle transition temperature in the three different directions of the A350 material was determined:
- C-L direction $T_{CVN}$=205.4K;
- R-L direction $T_{CVN}$=193.8K;
- L-C direction $T_{CVN}$=199.4K.

From the ductile-brittle transition temperature, the result was R-L > L-C > C-L, which had a little difference. From the upper shelf impact energy, the result was C-L > R-L > L-C, which had obvious difference.
2.2 Disk samples of SPT

SPT samples are disks with a diameter of 10 mm and thickness of 0.5 ± 0.005 mm with a good finish (P1200). Figure 3 showed the specimen with U-shaped notch. Both surfaces are parallel. The specimen with U-shaped notch was processed by Electric discharge method on the basis of sample without gap with a diameter of 0.18 mm. The size of the sample with U-shaped notch was shown in Figure 4.
2.3 SPT at low temperature

The experiments were carried out in three different directions at low temperature (0℃, -25℃, -50℃, -75℃, -100℃, -125℃, -135℃, -145℃, -155℃, -175℃, -185℃ and -196℃), and the experimental results were processed.

As shown in Figure 5, the samples of the small punch were carried out in three different directions of the A350 material respectively, and the directions were the same as that of the Charpy impact sample.

Finarelli [5] observed that in the process of the small punch test, the crack appears near the peak load point (F_m) and gradually expand, until the break. According to the GB for SPT [6], the fracture energy of small punch test specimen is abbreviated as E_{SP} is commonly derived from integrating the load-displacement curve along the X axis with defining 80% of F_m point for integral upper limit. In the impact test, the relationship between impact energy and test temperature is more commonly used by Boltzmann function and hyperbolic tangent function [7]. Some studies showed that the former has a good correlation coefficient and a little error. Therefore, Boltzmann function is used to fit the relationship between the fracture energy of the small punch and the experimental temperature.

\[
A = \frac{A_2 - A_1}{1 + e^{\frac{X_0 - X}{\Delta}} + A_2}
\]

Among them:
A_2 is the upper shelf of the ductile-brittle transition curve.
A_1 is the lower shelf of the ductile-brittle transition curve.
X_0 represents the energy transition temperature ETT50.

There are two methods to determine the ductile-brittle transition temperature of the small punch test [8-10].

1. T_{SP} is identified as the half of peak energy point in the energy curve.
2. T_{SP} is identified as the temperature of the average value of the peak energy and the lower shelf if the lower shelf can be appeared.
The first determination was used in this article. And the first half of the curve was fitted with Boltzmann function, and the second half was fitted with a straight line. The fracture energy fitting curve of three different directions was shown below.

![Fracture Energy Curve](image)

**Figure 6.** Temperature dependence of fracture energy for A350 material with disk specimens.

Figure 6 showed the fracture fitting curves in three different directions with disk specimens. And the T_SP of A350 material was determined by the energy curve method.  
Axial direction: T_SP=138.2K;  
Central direction: T_SP=135.2K;  
Radial direction: T_SP=139.2K.

As shown in Figure 6, the curves in three different directions of the A350 materials are almost the same. And the upper shelf energy and T_SP have a little deference in three different directions.

2.4 U-notched samples of SPT at low temperature

Due to conventional small punch test results cannot evaluate the variation of the upper shelf energy for anisotropic material, the small punch test at low temperature was carried out by using the U-notched shape samples.

![Fracture Energy Curve](image)

**Figure 7.** Temperature dependence of fracture energy for A350 material with U-notched specimens.
Figure 7 showed the fracture fitting curves in three different directions with U-notched specimens. And the T_sp of A350 material was determined by the energy curve method.

Axial direction: T_sp=114K;
Central direction: T_sp=142K;
Radial direction: T_sp=150K.

As shown in Figure 8, the upper shelf energy and T_sp in three different directions of the A350 materials are significantly different.

Figures 8-10 are respectively the fracture energy-temperature relationship curves of the A350 material in three different directions. As can be seen from the Figures, the fracture energy-temperature curves of the U-notched and un-notched specimens are similar. Both have the upper shelf and the transition zone, and the samples of the three different directions have no lower shelf. With the U-notched sample, the fracture energy of the sample reduced, especially on the upper shelf. The fracture energy decreases as the temperature decreases. The difference of the two samples is that the range of transition zone is widened with the U-notched sample.

**Figure 8.** Temperature dependence of fracture energy in axial direction for A350 material.

**Figure 9.** Temperature dependence of fracture energy in central direction for A350 material.
Figure 10. Temperature dependence of fracture energy in radial direction for A350 material.

2.5 Comparison and correlation of small punch test and standard impact test

Table 3 was the results of the $T_{SP}$ of small punch test and $T_{CVN}$ of Charpy impact test for the A350 material. With U-notched specimens, the differences of transition temperature among three different directions are larger than disk specimens. Especially the transition temperature of axial direction is much smaller than the other two different directions, which means the axial direction performance is best. In this case, the force of axial specimen is the combined force of radial and circumferential tensile stress, which is exactly the same as the tensile strength. The transition temperature obtained by small punch test with disk specimens can only be used in empirical correlations with the ductile-brittle transition temperature by the Charpy Impact test [11] and is unable to evaluate the upper shelf impact energy on impact test curves. However, the small punch test with the U-notched specimens can show the difference in the ductile-brittle transition temperature of anisotropic materials in different directions.

### Table 3. The comparison of SPT transition temperature of A350 by disk and U-notched specimens and impact transition temperature.

| Material            | Disk samples $T_{SP}$/K | U-notched samples $T_{SP}$/K | Impact test samples $T_{CVN}$/K |
|---------------------|-------------------------|-----------------------------|-------------------------------|
| A350 Axial direction (L-C) | 139.2                   | 114                         | 199                           |
| A350 Central direction (C-L)  | 135.2                   | 142                         | 205                           |
| A350 Radial direction (R-L)   | 138.2                   | 150                         | 194                           |

3. Discussion

1. The results of conventional tensile test in three different directions of the A350 showed that the A350 has obvious anisotropy and the axial tensile properties is the worst, but the results of Charpy impact test showed that the difference in ductile-brittle transition temperature is a little among three different directions, while the upper shelf impact energy have obvious differences, which is the lowest in axial direction, and is consistent with the tensile properties.

2. The transition temperature and upper shelf impact energy obtained by small punch test and Charpy impact test are consistent in three different directions. So the conventional small punch test can be used to link the $T_{SP}$ and the $T_{CVN}$.
3. The force of small punch specimens in three different directions is complex. The force of axial direction is the combined force of the radial direction and circumferential direction tensile stress. The force of radial direction is the resultant force of the axial direction and circumferential direction tensile stress. The force of circumferential direction is the combined force of the radial direction and axial direction tensile stress. According to the tensile results, the intensity in axial direction is weaker than the circumferential direction and the radial direction, so the axial sample has a good performance, and the axial Tsp should be the lowest. The fracture energy of the sample decrease with the U-notched specimens and the tendency of decline is getting smaller. The fracture energy of the U-notch specimens shifted to the right with the temperature changing, and the transition temperature of the U-notch specimens is obviously changed.

4. Conclusion

1) The study for the anisotropy of A350 material found that the small punch test with disk specimens can be used to link the TSP and the TCVN.
2) The fracture energy of the U-notch specimens shifted to the right with the temperature changing, and the transition temperature of the U-notch specimens is obviously changed.
3) The small punch test with U-notched specimens can effectively reflect the differences of the impact energy in three different directions.

Reference

1. Baik, J.M.; Kameda; J. and Buck, O. "Small punch test evaluation of intergranular embrittlement of an alloy steel." Scripta Metallurgica 17.12(1983):1443-1447.
2. Misawa, T.; et al. "Fracture toughness evaluation of fusion reactor structural steels at low temperatures by small punch tests." Journal of Nuclear Materials 169.12(1989):225-232.
3. Li, Y. and Šturm, R. "Determination of Creep Properties From Small Punch Test." ASME 2008 Pressure Vessels and Piping Conference 2008:739-750.
4. Linse, T., et al. "Usage of the small-punch-test for the characterisation of reactor vessel steels in the brittle—ductile transition region." Engineering Fracture Mechanics 75.11(2008):3520-3533. D
5. Finarelli, D.; Roedig, M. and Carsugh, F. "Small punch tests on austenitic and martensitic steels irradiated in a spallation environment with 530 MeV protons." Journal of Nuclear Materials 328.2–3(2004):146-150.
6. GBT 29459.1-2012. "Metallic material- Small punch testing for in-service pressure equipment in Chinese.” 2012.
7. Huang, Q. "Fitting Curves to Impact Toughness Date of Nuclear Structure Steel by Computer Program.” Journal of Sichuan Union University (1995).
8. Zhao, J.P.; Zhang, X.M. and Shen, S.M. "On the method of data processing for ductile-brittle transition temperature." Petro-chemical Equipment (2004).
9. Zhou, C. and Xia, X. "Regression Analysis of Ductile -Brittle Transition Temperature Curve for CrMo Steel.” Pressure Vessel Technology 20.6(2003):13–18.
10. Matsushita, T.; et al. "DBTT Estimation of Ferritic Low Alloy Steels in Service Plant by means of Small Punch Test.” Key Engineering Materials 51-52(1991):259-264.
11. Matsushita, T.; et al. "Correlation Between a Charpy V-notch Impact Test and a Small Punch Test in Ductile-Brittle Fracture Mode Transition Behavior.” Nihon Kikai Gakkai Ronbunshu A Hen/transactions of the Japan Society of Mechanical Engineers Part A 55.515(1989):1619-1622.