Identification of mechanical characteristics of materials using diminutive specimen—an empirical study

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Abstract

Small punch test technique on circular shape of diminutive specimen is considered in the present study. The small punch tests are conducted on diminutive specimen of circular shaped. Four different steels are chosen for this purpose. The load on diminutive samples is quasi-statically applied using different tip diameters hemispherical headed indenter. The experimental outputs of small punch (SP) test on diminutive specimen are obtained in the form breakaway loads, load vs. displacement curve/data and reduced thickness of tested diminutive samples is measured at failure. The resulted SP experimental data are analyzed used to establish the empirical relations to predict the mechanical behaviour of materials. For this purpose, the empirical correlations are proposed for the estimation of the yield stress ductility (fracture strain) and fracture toughness and are used to predict the behaviour of four different materials under study. The standard tensile and fracture toughness tests are also carried out for comparison. The small punch test on diminutive specimen is demonstrating the strong potential for the determination of mechanical behaviour.

Keywords: Diminutive specimen, small punch test, hemispherical headed indenter, load vs. displacement, mechanical behavior, yield stress, ductility, fracture toughness, conventional standard test.

1. INTRODUCTION

There various test standards are available for the determination of mechanical properties such as yield strength, ultimate tensile strength, ductility, ductile/brittle transition temperature and fracture toughness, which are required for the structural integrity assessment of plant components. However, there is no widely recognized test procedure for sub-sized or miniature specimen. There are many advantages in using a sub-sized or small test specimen. A sub-sized specimen would facilitate the assessment of material properties of components with service damage without the removal of a large amount of material, which might compromise the components integrity [1-4]. Specimens extracted from aged structures and components can be used to assess their degraded mechanical properties and estimate their remaining life. Miniature specimen test technique provides a way of obtaining mechanical properties of components while consuming an amount of materials that is very small to that required for full-size conventional specimens. In
most situations, full-size specimen cannot be obtained without destroying the component, whereas extraction of miniature samples can be virtually nondestructive [5-7].

2. EXPERIMENTAL PROCEDURE AND MATERIALS

In the present study, for circular disk (10mm diameter, 0.50mm thick) shaped diminutive specimen, the empirical correlations have been proposed for the evaluation of yield stress, ductility (fracture strain), and fracture toughness. The developed correlations are based on the SP experimental load vs. displacement curve and reduced thickness measured from tested specimen. The materials used are medium carbon (MS), Chromium hot-worked (H11), Non-shrinking die (D3) and structural (STS) steels. The diminutive specimens are tested on INSTRON machine and the data obtained from the small punch tests are used to establish the appropriate empirical correlations for the prediction of yield stress, ductility, and fracture toughness. The estimated values from small punch test, and mechanical properties determined from the conventional standard specimen tests are compared. Materials used in the present study, which are widely used in various industrial applications. Chromium hot work steel (H11) is a low allow steel with Manganese (0.40%), Silicon (1%), Chromium (5%), Molybdenum (1.1%), and Carbon (0.36%). Medium carbon steel (MS) contains carbon about 0.5% and Manganese & Silicon are 0.63% and 0.217% respectively. Other alloying elements are S, P, Cu, Ni, Al, Cr, Mo, are present in very small quantities i.e. less than 0.05%. The chemical compositions for various steel are given in Table 1. These materials are used to check the versatility of small punch test on diminutive samples and to predict (virtually non-destructive manner) the yield stress, ductility, and fracture toughness.

Table 1. The Nominal Chemical Composition (% by wt.) of Different Steels.

| Materials | C  | Mn  | Si  | S   | P   | Cu   | Ni | Cr  | Mo  | Al |
|-----------|----|-----|-----|-----|-----|------|----|-----|-----|----|
| H11       | 0.36 | 0.40 | 1.00 | 0.00 | 0.00 | 0.00 | 5.00 | 1.10 | 0.00 |
| D3        | 2.00 | 0.30 | 0.30 | 0.00 | 0.00 | 0.00 | 12.0 | 0.0  | 0.00 |
| MS        | 0.472 | 0.633 | 0.217 | 0.037 | 0.029 | 0.004 | 0.004 | 0.021 | 0.01 | 0.001 |
| STS       | 0.07 | 1.22 | 0.22 | 0.003 | 0.014 | 0.02 | 0.01 | 0.02 | 0.0  | 0.03 |

Fig. 1 shows the developed fixture for small punch test [8]. Three different rigid punches (hemispherical headed) are used to impart the quasi-static loads on diminutive specimens. The fixture contains a specimen holder (with specimen), punch holder (with punch) and connecting rods with guide pins. The specimen holder consists of a pair of dies in the centre of the fixture to support a diminutive specimen, rigidly clamped by six screws. The specimen is subjected to a central load applied by a punch. The upper and lower dies and the six clamping screws uniformly tighten the specimen. With the use of this specimen holder, the specimen with rigid fixed boundary condition is prevented from cupping upward during punching and plastic deformation is therefore concentrated in the region below the punch.

![Specimen holder](image)

![Punch & Punch holder](image)

![Fixture](image)

![Connecting rod](image)

![Guide pin](image)

Fig. 1 Details of small punch test fixture

The small punch tests were conducted with a constant crosshead speed of 0.2mm min⁻¹. The diminutive specimens are tested in air, at room temperature. In the experiment, 4-6 specimens are tested and the average of these resulted loads vs. displacement curves is reported for each material (see Fig. 2). The resulted load vs. displacement curve/data
is used for the analysis and prediction of mechanical properties. The conventional tensile test for standard specimen and three points bend test are also conducted on these steels for comparisons.
2.1 Estimation of Mechanical Properties
Different investigators have proposed different empirical relations for the estimation of mechanical properties (such as yield stress, ductility and fracture toughness etc.) using miniature specimen.

2.2 Existing Relations For The Estimation of Yield Stress
The displacement of the small punch specimen is assumed to be governed primarily by elastic bending and the effect of denting formed under the tip of rigid hemispherical headed punch is negligible, which can validate the simple bending theory. The load at the breakaway ($P_y$) from the linearity can be used to estimate the primary strength ($\sigma_y$) of the material. The methodology to determine the yield stress, using empirical relation suggested by Xu and Zhao [9] is based on the analysis of elasto-plastic bulge deformation behavior of the thick circular disc loaded at center.

$$\sigma_y = 0.477 \frac{P_y}{t_0^2}$$  \hspace{1cm} (1)

Where $t_0$ is the diminutive specimen thickness

Mao and Takahashi [10] established another empirical correlation between the yield stress $\sigma_y$ (MPa) and $P_y$ (kN).

$$\sigma_y = 0.36 \frac{P_y}{t_0^2}$$  \hspace{1cm} (2)

2.3 Existing Relations For The Estimation of Ductility & Fracture Toughness
The small punch tested samples are cut into two pieces and the reduced thickness is measured to determine the fracture strain by the equation

$$\varepsilon_{qf} = \ln \left( \frac{t_0}{t^*} \right)$$  \hspace{1cm} (3)

Where $t^*$ is the minimum thickness at fracture point, and $t_0$ is original thickness of the specimen

The biaxial fracture strain can be estimated from the empirical relation using small punch test, suggested by Kameda [11] is as follows.
\[
\varepsilon_{gf} = 0.12 \left( \frac{\delta^*}{t_0} \right)^{1.72}
\]

where \(\delta^*\) is the maximum punch displacement in mm at fracture.

Similarly, another empirical relationship proposed by Mao et al. [3] is as follows

\[
\varepsilon_{gf} = 0.15 \left( \frac{\delta^*}{t_0} \right)^{1.5}
\]

The experimental correlation between equivalent fracture strain and fracture toughness \((J_{IC})\), based on the single specimen technique proposed by Takahashi et al. [12], is linear, as follows

\[
J_{IC} = 280 \varepsilon_{gf} - 50 \quad (for \quad \varepsilon_{gf} \geq 0.2)
\]

Where \(J_{IC}\) is in \(\text{kJ/m}^2\)

Similarly, another relation for fracture toughness \((J_{IC})\), suggested by Mao et. al. [10]

\[
J_{IC} = 354 \varepsilon_{gf} - 113 \quad (for \quad \varepsilon_{gf} \geq 0.4)
\]

3. NEWLY DEVELOPED EMPIRICAL CORRELATIONS

The above-proposed correlations are independent of the geometry of specimen as well as the geometry of indenter. Hence the results are not very accurate. The new empirical correlations are developed for the prediction of yield stress, ductility (fracture strain), and fracture toughness, \((J_{IC})\). In developing the new correlations, the shape of the specimen and the geometry of the indenter are taken into consideration.

3.1 New Correlation For the Prediction of Yield Stress

The empirical correlations for the prediction of primary strength for circular diminutive specimen is established. These correlations are based on geometry of specimen, boundary condition, and the punch load up to yielding of diminutive specimen or the breakaway load. The breakaway load is obtained by analyzing the SP experimental load vs. displacement curves using 0.20% offset method. In this case the value of 0.20% displacement (corresponding to maximum load under the tip) of punch is identified from the SP experimental load vs. displacement curves. In case of circular shaped specimen, it is assumed that only 20% of the tip radius of the hemispherical headed punch \(r_0\) comes in contact with miniature sample up to the yielding, when the indenter is pressed slowly.

3.2 Proposed Empirical Correlation For Circular Shaped Specimen

The generalized empirical relation takes into picture the size of the miniature specimen as well as tip radius of the hemispherical headed indenters. Hence these empirical relations can be used for different size of the specimen as well for different diameter of pinch radius.

\[
\sigma_y = 1.50 \frac{P_y}{\pi t_0^2} \left(1 + \nu \right) \left[ \ln \frac{R}{r} + \left( \frac{r}{2R} \right)^2 \right]
\]

Where, \(\nu\) =Poisson’s ratio, \(R=\)radius of bore hole in dies
\(r = \)contact tip radius of rigid punch = 0.80 \(r_0\), \(r_0 = \)radius of the head of the punch
\(P_y = \)breakaway load, \(t_0 = \)original thickness of specimen

The breakaway load (or yielding load) was obtained by analyzing the SP experimental load vs. displacement curves using 0.20% offset method.

3.3 Correlation For the Prediction of Fracture Strain and Fracture Toughness

The equivalent fracture strain is obtained by measuring the thickness ratio \((t_0/t^*)\) at the fracture point in the diminutive specimen by cutting the specimen into two pieces. On the basis of original thickness \((t_0)\) and the reduced thickness \((t^*)\) of the diminutive specimen, a power law empirical logarithmic correlation is established. The proposed
correlation depends upon the shape factors $S_a$ and $S_b$. The existing empirical relation does not take into consideration the shape factor. The proposed empirical relation is

$$
\varepsilon_{qf} = S_b \left[ \ln \left( \frac{t_0}{t^*} \right) \right]^{S_a}
$$

(9)

Where $t^*$ is the minimum thickness at the fracture of specimen and $t_0$ is the original diminutive specimen thickness (i.e. 0.50mm), $S_a = 1.24$ and $S_b = 1.688$

During the small punch test study on diminutive specimen, it is noticed that the fracture strain influences the fracture toughness more strongly than any other material parameter. In this study, the equivalent fracture strain, $\varepsilon_{qf}$, and the fracture toughness, $(J_{IC})$, are empirically correlated.

$$
J_{IC} = S_A \left( \varepsilon_{qf} \right)^{S_B}
$$

(10)

Where $J_{IC}$ is in (kJ/m$^2$). $S_A = 722.28$ and $S_B = 2.837$ are the shape factor used for the estimation of fracture toughness.

4. RESULTS

The load versus displacement curves as obtained from the small punch test on diminutive samples of four different steels are shown in Fig 2. The yield load $P_y$ (Load at breakaway) is calculated by the offset method as given in Table 3. In this case the value of 0.2% displacement (at maximum load) under the punch load is calculated. This displacement ($\delta$) value is used to draw an offset line parallel to the slopes at the origin and $P_y$ value is determined from the intersection point with the curve. The yield load ($P_y$) values obtained from the plots are used to evaluate primary strength ($\sigma_y$) by using existing empirical equations (i.e. Eqn. (1) & Eqn. (2)) and newly developed equations (i.e. Eqn (8) for circular shaped specimen, using hemispherical headed punches of different diameters (2.309mm, 1.633mm & 1.115mm). The primary strength results are compared with the values obtained from standard tensile tests for the all four materials (Table 4).

Table 3. Breakaway loads (N) calculated from SP experimental load vs. displacement curves using indenter of different tip diameter.

| S. No. | Different Indenters | 2.309mm (P1) | 1.633mm (P2) | 1.155mm (P3) |
|--------|---------------------|--------------|--------------|--------------|
| 1.     | H11                 | 230          | 170          | 140          |
| 2.     | D3                  | 240          | 165          | 130          |
| 3.     | MS                  | 160          | 115          | 90           |
| 4.     | STS                 | 235          | 165          | 130          |

Table 4 Estimated primary strength of circular shaped specimen using different punch diameters.

Medium carbon steel (MS)

| S. N o. | Empirical Equations given by | $\sigma_y$ (MPa) estimated, using different punches for diminutive specimen test | $\sigma_y$ (MPa) Standard tensile test |
|---------|------------------------------|---------------------------------|----------------------------------------|
| 1       | Eqn. 1                       | 305.3                           | 219.4                                  |
|         |                               | 171.7                           | 323.32                                 |
| 2       | Eqn. 2                       | 230.4                           | 165.6                                  |
|         |                               | 129.6                           |                                        |
| 3       | Eqns. 8                      | 328.0                           | 327.2                                  |
|         |                               | 330.1                           |                                        |

Low alloy steel (H11)

| S. N o. | Empirical Equations given by | $\sigma_y$ (MPa) estimated, using different punches for diminutive specimen test | $\sigma_y$ (MPa) Standard tensile test |
|---------|------------------------------|---------------------------------|----------------------------------------|
| 1       | Eqn. 1                       | 438.8                           | 324.4                                  |
|         |                               | 286.2                           |                                        |
| 2       | Eqn. 2                       | 331.2                           | 244.8                                  |
|         |                               | 216.0                           | 484.02                                 |
3 Eqns. 8  471.6  513.5  513.5
Die steel (D3)

|       | Eqn. 1 | Eqn. 2 | Eqns. 8 |
|-------|--------|--------|---------|
| 1     | 457.9  | 345.6  | 492.0   |
| 2     | 314.8  | 237.6  | 465.5   |
| 3     | 248.0  | 187.7  | 476.7   |
| Total | 478.0  | 471.6  | 478.0   |
Structural Steel (STS)

|       | Eqn. 1 | Eqn. 2 | Eqns. 8 |
|-------|--------|--------|---------|
| 1     | 448.4  | 338.4  | 481.8   |
| 2     | 314.8  | 237.6  | 469.4   |
| 3     | 248.0  | 187.2  | 476.8   |
| Total | 475.0  | 471.6  | 475.0   |

The equivalent fracture strain is calculated by using the existing relations i.e. Eqn. 3, Eqn. 4, & Eqn. 5, and also by proposed correlation (i.e. Eqn. 9). The reduced thickness for each tested diminutive specimen is measured for all four steels. Fracture strain values obtained by using different correlations are shown in Table 6.

Table 6 The Equivalent fracture strain values calculated by different equations for all four steels.

| Specimen shaped | Equivalent fracture strain calculated by different Equations |
|-----------------|-----------------------------------------------------------|
|                 | Eqn. 3 | Eqn. 4 | Eqn. 5 | Proposed Eqn. 9 |
| MS              | MS     | MS     | MS     | MS           |
| H11             | H11    | H11    | H11    | H11          |
| Circular        | 0.325  | 0.618  | 0.319  | 0.626        |
|                 | 0.494  | 0.618  | 0.319  | 0.626        |
| D3              | STS    | STS    | STS    | STS          |
|                | 0.186  | 0.298  | 0.920  | 0.330        |
|                | 0.430  | 0.298  | 0.920  | 0.330        |

The fracture toughness, JIC, has been estimated using the newly developed empirical correlation in conjunction with equivalent fracture strain. Empirical correlation for different diminutive specimens using respective shape factor is employed for all the four materials to estimate the values of fracture strain and the fracture toughness, JIC. The fracture toughness values for all the steels are also calculated using existing (in literature) empirical correlations. The fracture toughness, JIC is also obtained for validation by performing the standard conventional fracture toughness (three point bend specimen) test. The estimated values of fracture toughness obtained by SP experimental equivalent fracture strain using proposed empirical equations and by existing empirical correlations are reported in Table 7.

Table 7 Fracture toughness values obtained by SP test method based on fracture strain values for Circular shaped diminutive specimen

| S. No. | Equations used for estimating JIC | Fracture toughness, JIC \( \left( \frac{k\sqrt{J}}{m^2} \right) \), using fracture strain values obtained by diff. Eqns | Eqn. 3 | Eqn. 4 | Eqn. 5 | Eqn. 9 |
|--------|----------------------------------|-----------------------------------------------------------------------------------------------------------------|--------|--------|--------|--------|
|        |                                  |                                                                                                                 | MS H11 | MS H11 | MS H11 | MS H11 |
| 1.     | Eqn. 6                           | 41.0                                                              | 123.0  | 42.12  | 125.0  | 51.36  | 66.2   | 147.1  |
| 2.     | Eqn. 7                           | 0.875                                                             | 102.0  | 0.505  | 102.6  | 11.89  | 30.2   | 129.8  |
| 3.     | Proposed Eqn. 10                 | 29.74                                                             | 184.3  | 30.83  | 191.1  | 40.43  | 59.6   | 266.6  |
|        |                                  | D3                                                               | STS    | D3     | STS    | D3     | STS    |
| 1.     | Eqn. 6                           | 2.080                                                             | 33.44  | 207.6  | 42.40  | 189.40 | 8.520  | 115.7  |
| 2.     | Eqn. 7                           | 48.83                                                             | 10.19  | 204.4  | 0.850  | 194.3  | 40.89  | 91.24  |
| 3.     | Proposed Eqn. 10                 | 6.113                                                             | 23.28  | 570.1  | 31.09  | 520.6  | 8.510  | 163.2  |

Standard three point Bend test

|        | MS | H11 | D3 | STS |
|--------|----|-----|----|-----|
|        | 64.80 | 230.63 | 8.40 | 211.20 |
5. CONCLUSIONS

The small punch tests are performed on circular shaped diminutive specimens made from medium carbon steel (MS), chromium hot work steel (low alloy steel, H11), die steel (D3) & structural steel (STS). The breakaway loads and load vs. displacement curves/data are obtained. The data obtained from the tests of diminutive specimen using small punch test technique are analyzed and then the empirical equations for the prediction of primary strength have been proposed. The empirical equations correlates the breakaway loads with tip radius of hemispherical headed punches (in contact with disk specimen), specimen’s geometry and the borehole radius of specimen holder (dies). The yield stress values predicted for four different steels using proposed correlations and as well by using existing relations are shown in Table 4 & Table 5. The empirical correlations for the prediction of fracture strain ($\varepsilon_{fr}$) and fracture toughness ($J_{IC}$) are based on diminutive specimens shape factors, as defined in the proposed correlation. It is noticed that the equivalent fracture strain estimated by proposed empirical equation is more accurate then the equivalent fracture strain calculated by existing empirical equations. It is observed that the fracture toughness values obtained by using equivalent fracture strain, and proposed equation based on SP experimental data, are quite close to the values obtained from standard fracture toughness (Table 7). It is concluded from the present study, that the prediction of yield stress, ductility (fracture strain), and fracture toughness using proposed empirical correlations are very close to the values obtained from standard conventional test specimen.

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