Evaluation and analysis of uncertainty in tensile experiment results of modified PPR at elevated temperature

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Abstract: A high temperature tensile experiment of modified random copolymerized polypropylene was carried out by ASTM D 638-2014. It analyzed the factors influencing the accuracy of the high temperature mechanical properties of modified random copolymer polypropylene and discussed the causes of the uncertainty of measurement standards from the sample size measurement, the indication error of force value of experiment machines, its calibration, data acquisition of the experimental software, the temperature control, the numerical correction, and the material nonuniformity, etc. According to JJF 1059.1-2012, class A and class B evaluation were conducted on the above-mentioned uncertainty components, and all the uncertainty components were synthesized. By analyzing the uncertainty of the measurement results, this paper provides a reference for evaluating the uncertainty of the same type of measurement results.

1. Introduction
Random Copolymerized Polypropylene (PPR) is a general-purpose plastic which has been widely applied. However, its disadvantages greatly limit its wider application, such as the strength decline under the high temperature, obvious brittleness under the low temperature, high gap sensitivity. The pipe produced by PPR is brittle under the low temperature and has weak anti-pressure capability under the high temperature[1]. The modified PPR is to add modifier in the PPR to improve its crystalline properties and its mechanical properties, which is particularly important for the expansion of PPR applications in China[2]. In order to characterize the mechanical properties of PPR under the high temperature, it is necessary to carry out high-temperature tensile test to measure the tensile strength and elongation at break. To decide the accuracy of the experimental data and the credibility of the results, we need to evaluate and analyze the uncertainty of measurement of high-temperature tensile strength of the sample, aiming to find out the methods to reduce various system errors and improve the accuracy of the experiment. According to the definition of measurement uncertainty, there is dispersion in measurement results of PPR performance indicators no matter how advanced the detection methods and testing equipment are. The dispersion of the measurement results is evaluated and expressed by the uncertainty of measurement. The smaller uncertainty indicates the smaller dispersion of the measurement results, the greater credibility, and the higher accuracy of the measurement results as well as the greater value-in-use, and vice versa.

At present, there are no domestic standards for the high-temperature tensile test of such materials in China. The high-temperature tensile test is carried out according to ASTM D 638-2014 and the analysis and evaluation are conducted with the high-temperature tensile strength as the key indicators, coupled with other experimental indicators such as elongation at break. The evaluation of measurement uncertainty is carried out according to JJF 1059.1-2012 and related literature[3-5] from the perspective of the sample cross-sectional area, experimental machine indication error, experimental data revision
and temperature control precision, holding time and so on. Then the modified PPR high-temperature tensile strength indicators are taken as an example to have a comprehensive evaluation and discussion of the uncertainty of the measurement results, which could provide data support for the improvement of the modified PPR process as well as its high-temperature mechanical properties.

2. Experimental Part

2.1 Raw materials and equipment
PPR, R200P, pellets are produced by Hyosung Company from South Korea; ETM204 microcomputer-controlled high-temperature tensile testing machine is produced by the MTS Company from the United States.

2.2 Experimental methods
Conduct vacuum drying for the PPR pellets at 80 °C for 8 h and then use the sophisticated injection molding machine to mold it into a standard dumbbell type test piece. The size of the parallel area is 50 mm × 13 mm × 4 mm. Heat the sample to 100° C using the high temperature tensile tester according to ASTM D 638-2014, and hold the temperature for 15-30 min. Then stretch it under it fractures.

The tensile strength is calculated as the following equation (1).

\[ \sigma = \frac{W_m}{D \times H} \]  

(1)

Where: \( \sigma \) is the resistance strength. \( W_m \) is the maximum force value during the whole tensile test, N. D is the width of the parallel area of the sample, mm. \( H \) is the thickness of the parallel area of the specimen, mm.

3. Analysis of the Source of the Uncertainty
The evaluation of the uncertainty components can be divided into two categories, namely, Class A and Class B. The former one is to evaluate the measured value with the statistical analysis under the specified measurement conditions. The latter one is to evaluate the uncertainty components with a method different from Class A. 1) The relative standard uncertainty component \( u_{rel}(W_m) \) introduced by \( W_m \) contains the uncertainty \( u_{rel}(W_1) \) introduced by the indication error of the high-temperature tensile test machine tension sensor, \( u_{rel}(W_2) \) introduced by the force standard used in the calibration of the test machine, and \( u_{rel}(W_3) \) introduced by the computer-controlled experiment data collection. They are all the Class B uncertainty components; 2) The relative standard uncertainty component \( u_{rel}(A_0) \) introduced by the original cross-sectional area \( (A_0) \) of the dumbbell test, coupled with the uncertainty introduced by the width and thickness test of the parallel area of the specimen, is the Class B uncertainty component; 3) the relative standard uncertainty component \( u_{rel}(\text{rep}) \) introduced by the repeated test of the high-temperature tensile strength is the Class A uncertainty component; 4) the relative standard uncertainty component \( u_{rel}(\Delta t) \) introduced by the temperature control is the Class B uncertainty component; 5) The relative standard uncertainty component \( u_{rel}(S_{xy}) \) introduced by the experimental data revision is the Class B uncertainty component.

Based on the above analysis, the model to measure the uncertainty is established, as shown in equation (2).

\[ U_{rel}(\sigma) = \sqrt{u_{rel}(W_m)^2 + u_{rel}(A_0)^2 + u_{rel}(\text{rep})^2 + u_{rel}(\Delta t)^2 + u_{rel}(S_{xy})^2} \]  

(2)

\( U_{rel}(\sigma) \) is the relative standard uncertainty of high-temperature tensile strength.
4. Evaluation of Uncertainty Components

4.1 Evaluation of $u_{rel}(W_m)$

$u_{rel}(W_1)$ is evaluated according to equation (3). The indication error of 1.0 high-temperature tensile test machine is $\pm 1\%$ and the factor $k=\sqrt{3}$ is taken into consideration based on the uniform distribution.

$$u_{rel}(W_1) = \frac{a}{k} \times 100\% = 0.577\% \tag{3}$$

In the equation, $a$ is the indication error of the high-temperature tensile test machine.

$u_{rel}(W_2)$ is evaluated according to equation (4). Then use the standard force test at 0.3 level to verify the high-temperature tensile testing machine and measure the repeatability = 0.3%, $k = \sqrt{3}$.

$$u_{rel}(W_2) = \frac{R_1}{\sqrt{3}} \times 100\% = 0.173\% \tag{4}$$

$R_1$ is the measured repeatability.

To evaluate $u_{rel}(W_3)$, the relative uncertainty of Class B introduced by computer-controlled experimental software data acquisition is determined as $0.3\times10^{-2}$ according to previous experimental experience, and then $u_{rel}(W_3) = 0.3\%$.

Since the above three factors have no irrelevance, the relative standard uncertainty component $u_{rel}(W_m)$ of the maximum force value in the whole experiment is evaluated according to equation (5).

$$U_{rel}(W_m) = \sqrt{u_{rel}^2(W_1) + u_{rel}^2(W_2) + u_{rel}^2(W_3)} \times 100\%$$

$$= 0.670\% \tag{5}$$

4.2 Evaluation of $u_{rel}(A_0)$

According to ASTM D 638-2014, the width and thickness of the parallel area of the specimen are 13,000 and 4,000 mm. The uncertainty of $A_0$ consists of two parts: the uncertainty introduced by the indication error of the vernier caliper and the one introduced by the manual reading error.

To measure the width of the parallel area of the dumbbell specimen, the relative standard uncertainty introduced by the indication error of the vernier caliper is evaluated according to equation (6). The indication error of the vernier caliper is 0.020 mm. Conduct calculation based on the uniform distribution, $k = \sqrt{3}$.

$$u_{rel}(D) = \frac{R_3}{k \times 13} \times 100\% = \frac{0.020}{\sqrt{3} \times 13} \times 100\% = 0.088\% \tag{6}$$

$u_{rel}(D)$ is the relative standard uncertainty introduced by the indication error of the vernier caliper. $R_3$ is the indication error of the vernier caliper.

To measure the width of the parallel area of the dumbbell specimen, the relative standard uncertainty introduced by the operator (by the reading error) is evaluated according to equation (7). As the resolution is 0.010 mm, calculate it based on the uniform distribution, $k=\sqrt{3}$.

$$u_{rel}(D) = \frac{R_3}{k \times 13} \times 100\% = \frac{0.010}{\sqrt{3} \times 13} \times 100\% = 0.044\% \tag{7}$$

$u_{rel}(D)$ is the relative standard uncertainty introduced by the operator (by the reading error); $R_3$ is the resolution.

The total relative standard uncertainty components introduced by the width of the sample in the parallel area is evaluated by equation (8).

$$\text{The total relative standard uncertainty components introduced by the width of the sample in the parallel area is evaluated by equation (8).}$$
\[
\sigma_{rel}(D) = \sqrt{\sigma_{rel}^2(D) + \sigma_{rel}^2(D)} \times 100% = 0.098\% \quad (8)
\]
\(\sigma_{rel}(D)\) is the total relative standard uncertainty components introduced by the width of the parallel area of the specimen.

To test the thickness of the parallel area of the dumbbell specimen, the relative standard measurement uncertainty introduced by the micrometer indication error is evaluated according to equation (9). The indication error of the micrometer is 0.003 mm. Calculate it based on the uniform distribution, \(k = \sqrt{3}\).

\[
u_{rel}(H) = \frac{R_4}{k \times 4} \times 100% \\
= \frac{0.003}{\sqrt{3} \times 4} \times 100% = 0.043\% \quad (9)
\]
\(\nu_{rel}(H)\) is the relative standard measurement uncertainty introduced by the micrometer indication error; \(R_4\) is the indication error of the micrometer.

To test the thickness of the dumbbell specimen, the relative standard measurement uncertainty \([\nu_{rel}(H)]\) introduced by the operator (by the reading error) is evaluated according to equation (10). As the resolution \(R_5\) is 0.001 mm, calculate it based on the uniform distribution, \(k = \sqrt{3}\).

\[
u_{rel}(H) = \frac{R_5}{k \times 4} \times 100% \\
= \frac{0.001}{\sqrt{3} \times 4} \times 100% = 0.014\% \quad (10)
\]
\(\nu_{rel}(H)\) is the relative standard measurement uncertainty \([\nu_{rel}(H)]\) introduced by the operator (by the reading error); \(R_5\) is the resolution.

The total relative uncertainty component introduced by the thickness of the parallel area of the specimen is introduced by equation (11).

\[
u_{rel}(H) = \sqrt{\nu_{rel}^2(D) + \nu_{rel}^2(H)} \times 100% = 0.050\% \quad (11)
\]
\(\nu_{rel}(H)\) is the total relative standard uncertainty component introduced by the thickness of the parallel area of the specimen.

As there are no correlations between the uncertainty components introduced by measuring the width and thickness of the parallel area, \(\nu_{rel}(A_{0})\) is evaluated according to equation (12).

\[
u_{rel}(A_{0}) = \sqrt{\nu_{rel}^2(D) + \nu_{rel}^2(H)} \times 100% = 0.110\% \quad (12)
\]

4.3 The evaluation of \(\nu_{rel}(rep)\)
Test 10 modified PPR dumbbell specimens produced at the same time and the tensile strength is respectively 25.40, 25.50, 26.10, 26.30, 26.60, 23.70, 25.30, 25.20, 25.60, 25.80 MPa. Evaluate \(\nu_{rel}(rep)\) according to equation (13).

\[
u_{rel}(rep) = S_{rel}(\sigma) \times 100% = 0.980\% \quad (13)
\]

\(S_{rel}(\sigma)\) is the standard deviation of the high-temperature tensile strength test results for 10 samples.

4.4 Evaluation of \(\nu_{rel}(\Delta t)\)
The uncertainty of the temperature control consists of three parts. The uncertainty \([\nu_{rel}(\Delta t)]\) introduced by the indication error of the thermocouple used for temperature measurement, the relative standard uncertainty \([\nu_{rel}(\Delta t)]\) and \(\nu_{rel}(\Delta t)\) introduced by the temperature control accuracy specified by the testing methods and regulations and of the relative standard uncertainty.

\(\nu_{rel}(\Delta t)\) is evaluated by equation (14). The high-temperature tensile testing machine is equipped with the high-and-low temperature test box. The thermocouple calibration certificate shows that the
thermocouple uncertainty is 0.5 °C at a temperature of 100.0 °C. In the case of the normal distribution, the coverage factor is evaluated as \( k = 2 \).

\[
U_{\text{rel}}(\Delta t) = \frac{R_t}{k \times 100.0} \times 100\% \quad (14)
\]

\( R_t \) is the maximum allowable error of the temperature control system of the high-temperature box in the high-temperature tensile test at a temperature of 100.0 °C.

\[
u_t(\Delta t) = \frac{R_t}{k \times 100.0} \times 100\% = 1.732\%
\]

\( R_t \) is the maximum allowable error of the temperature control system of the high-temperature box in the high-temperature tensile test at a temperature of 100.0 °C.

\[
u_t(\Delta t) = \frac{R_t}{k \times 100.0} \times 100\% = 1.732\%
\]

\( R_t \) is the maximum allowable error of the temperature control system of the high-temperature box in the high-temperature tensile test at a temperature of 100.0 °C.

4.5 Evaluation of \( u_{\text{rel}}(S_{xy}) \)

\( u_{\text{rel}}(S_{xy}) \) is evaluated according to formula (17). According to ASTM D 638-2014, the standard of the rounding off unit in the experiment is 0.10 MPa. In accordance with JJJ 1059.1-2012, it can be estimated that \( u_{\text{rel}}(S_{xy}) \) is 0.29 times of the rounding off unit, which is shown as follows:

\[
u_{\text{rel}}(S_{xy}) = \frac{R \times 0.29}{\bar{\sigma}} \times 100\% = 0.110\%
\]

In the formula, \( R \) refers to the minimum acceptable rounding off unit, which is 0.10 MPa; \( \bar{\sigma} \) represents the average value of 10 groups of high temperature tensile strength, which is 25.60 MPa.

5. Evaluation Of \( U_{\text{rel}}(\sigma) \)

Since the above uncertainty components are not correlated with each other, the sensitivity coefficients of the components are all 1, then \( U_{\text{rel}}(\sigma) \) is evaluated according to formula (18).

\[
U_{\text{rel}}(\sigma) = \left[ u_{\text{rel}}^2(W_m) + u_{\text{rel}}^2(A_0) + u_{\text{rel}}^2(rep) + u_{\text{rel}}^2(S_{xy}) + u_{\text{rel}}^2(\Delta t) \right]^{1/2} \times 100\% = 2.120\%
\]

6. Evaluation of \( U_{\text{rel}}(\Sigma) \)

According to JJJ 1059.1-2012, the normal distribution is considered in the daily testing work on the tensile strength. Let be k=2, the confidence probability is about 95%, then the relative expansion uncertainty of the tensile strength is 2\( U_{\text{rel}}(\sigma) \) is 4.240%. The extended uncertainty is calculated by Formula (19).

\[
U(\sigma) = \bar{\sigma} \times U_{\text{rel}}(\sigma) = 25.60 \times 4.240\% = 1.09 \text{ Mpa}
\]

In the formula, \( U_{\text{rel}}(\sigma) \) is the relative expansion uncertainty of tensile strength and \( U(\sigma) \) is the extended uncertainty.

Finally, the tensile strength is \( (25.60 \pm 1.05) \text{ MPa} \), k = 2.

7. Results and Discussion

It can be seen from Table 1 that the uncertainty introduced by the original cross-sectional area measurement, standard dynamometer, data repair and other factors has a low contribution to the uncertainty
of the measurement results, that is, the effect on the dispersion of the measurement results is not noticeable. The factors such as the error of the experiment machine, the repeated measurement and the temperature control precision are the most important influencing factors in the uncertainty of the measurement result.

The confidence ratio of the truth value of tensile strength in the range of (25.60 ± 1.09) MPa is 95%, which indicates the degree of dispersion of the measurement results. When the σ of modified PPR is 25.60 MPa, $U_{\text{rel}}(\sigma)$ is 4.240%, which indicates that the dispersion of the experimental results is small.

Fixing other conditions, the experiment machines with precision measuring level of 0.5 level is used. Improve the temperature control accuracy to ± 3.0 ℃ and obtain $U_{\text{rel}}(\sigma)$ by increasing the number of measurements of the sample to 20 times. Its value is respectively 1.01 % and 2.020%, suggesting that the relative uncertainty of the measurement results is significantly reduced. It can be seen from Fig. 1 that when the tensile force is different $U_{\text{rel}}(\sigma)$ remains essentially unchanged, indicating that $U_{\text{rel}}(\sigma)$ is not affected by the measurement result; the extended uncertainty [U(σ)] of the measurement result will decline with the decrease of σ, but basically at a low level.

![Fig.1 Curves of U (σ), U_{\text{rel}}(σ) and σ](image)

| Standard Uncertainty Component | Source types of Uncertainty | Types | Distribution category | Coverage factor | Relative standard uncertainty, % |
|-------------------------------|-----------------------------|-------|-----------------------|----------------|---------------------------------|
| $u_{\text{ref}}(W_1)$         | Indication error of experiment machines | B     | Uniform distribution | $\sqrt{3}$     | 0.577                           |
| $u_{\text{ref}}(W_2)$         | Standard dynamometer        | B     |                       |                | 0.173                           |
| $u_{\text{ref}}(W_3)$         | Data acquisition system     | B     |                       |                | 0.300                           |
| $u_{\text{rel}}(A_0)$         | $A_0$ measurements          | B     | Uniform distribution | $\sqrt{3}$     | 0.110                           |
| $u_{\text{ref}}(\text{rep})$  | Measure repeatability       | A     |                       |                | 0.980                           |
| $u_{\text{ref}}(\Delta t)$    | Thermocouple calibration    | B     | Normal distribution   | 2              | 0.250                           |
| $u_{\text{ref}}(\Delta t)$    | Temperature control accuracy| B     | Uniform distribution | $\sqrt{3}$     | 1.732                           |
| $u_{\text{ref}}(S_{xy})$      | Data rounding               | B     |                       |                | 0.110                           |
### Synthesis

| $U_{\text{rel}}(\sigma)$ | Extension | 2.120 |
|------------------------|-----------|-------|
| $U_{\text{rel}}(\sigma)$ |           | 4.240 |

### 8. Conclusion

a) The main causes of $U(\sigma)$ of the modified PPR are indication errors of experiment machines, the measurement repeatability and the temperature control accuracy.

b) Fixing experimental conditions, $U_{\text{rel}}(\sigma)$ is basically the same in different tensile strength. The use of the experiment machines with higher precision, the improvement of the accuracy of temperature control and the increase of the number of measurements of the same batch can significantly reduce the relative uncertainty of the measurement results and the dispersion of the experimental results.

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