Experimental Study on Aging Performance of Polyethylene Gas Pipelines

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Abstract. Currently, there is a lack of adequate standards for evaluating the integrity inspection cycle of polyethylene (PE) gas pipelines. In this study, a series of photo-oxidation accelerated-aging experimental tests for PE80 gas pipe samples were performed. The attenuation laws of several critical performance parameters, such as micro-morphology, tensile strength, and thermal stability tests, were established. These parameters were determined at different aging times. A novel comprehensive performance evaluation method for pipeline integrity was developed using the analytic hierarchy process, and a reasonable inspection period for pipeline integrity was identified. The results showed that the samples experienced two stages of aging: rapid aging stage and aging platform stage. During the rapid aging stage, the comprehensive evaluation index for the aging performance decreased rapidly. However, the comprehensive evaluation index for the aging performance remained almost unchanged during the aging platform stage.

Keywords: polyethylene gas pipe; photo-oxidation accelerated-aging test; overall aging performance; analytic hierarchy process; evaluation cycle.

1. Introduction

Polyethylene (PE) pipes are gradually being used as a replacement material for steel pipes owing to their excellent corrosion resistance, construction characteristics, and economic performance, and they are becoming the first choice for fuel gas transmission and distribution. However, the effects of factors, such as light, heat, and oxygen, cause the slow aging of the pipe material, and consequently, reduces the material performance [1]. The integrity management of gas pipelines is aimed at synthetically managing all the internal and external factors that affect the safe and reliable operation of pipelines in five basic steps: data collection and integration, risk assessment, integrity assessment, repair and maintenance, and efficiency assessment. These steps involve a continuous cyclic process as well as continuous improvement [2]. As an essential aspect of pipeline integrity management, the integrity assessment involves how to determine the prior integrity testing and evaluation plans.

Various evaluation indexes are used for assessing the integrity of PE gas pipelines. Examples of such indexes include the tensile strength, oxidation induction time (OIT), melt mass-flow rate (MFR), resistance towards slow crack growth, longitudinal shrinkage, crystallinity, carbonyl, and hydroxyl indexes [3]. Currently, the attenuation laws of tensile strength, OIT, crystallinity, and MFR are mainly
adopted to assess pipe integrity [4]. However, significant differences exist in the variation trends of different performance indexes during aging, which makes it impossible to evaluate the pipe integrity synthetically using a single-performance index [5]. Therefore, it is necessary to integrate previously developed single-performance indexes and construct a comprehensive evaluation index suitable for evaluating the integrity of PE pipes for distributing fuel gas.

In this study, PE pipe, which is widely used in the gas industry (Classification PE80), was selected as the research object, and photo-oxygen accelerated-aging experimental tests on the pipe samples were performed. The aging characteristics of the PE80 pipes were determined through surface micromorphology, tensile strength, thermal stability, crystallinity, and melting property tests, and these indexes have a crucial impact on the pipe integrity. A comprehensive evaluation method for assessing the pipe integrity was constructed using the analytic hierarchy process (AHP), and the integrity was comprehensively evaluated to determine a reasonable integrity inspection and evaluation cycle for PE gas pipelines.

2. Experimental Tests

2.1. Sample preparation
A PE80 pipe produced by Chinaust Plastics (Xian) Co, Ltd. was utilized in this study. It was machined into a dumbbell shape (total length = 150 ± 1.0 mm; width ≥ 22 mm), as shown in Fig. 1.

2.2. Photo-oxygen accelerated-aging test
The outer wall of the sample was adopted as the exposure surface, and accelerated-aging tests were carried out at 40 °C for 0, 168, 336, 504, 672, 840, 1008, 1176, and 1344 h using the LUV-II ultraviolet (UV) aging test box with three 20 W UV-B lamps (Fig. 2). For the tests, sampling was conducted in batches corresponding to each aging time, and a total of nine batches were designed.

2.3. Aging performance test
(1) Scanning electron microscopy (SEM) of microscopic morphology
Cuboid samples having dimensions of 1 cm × 1 cm were cut off from the samples at different aging times, and the microscopic morphology of the samples was observed using a scanning electron microscope of Type EVOMA15. Before the experimental tests were performed, the surface of each sample was processed by spraying gold on the surface, and the sample was then placed in a tank for vacuum treatment.

(2) Mechanical performance test
A microcomputer-controlled electronic universal testing machine (CMT4304) was used to determine the mechanical properties of the samples at different aging times. The test speed was 50 mm/min, and the distance between the marking lines was 50 mm.

(3) Thermal stability and crystallinity tests
Disc-shaped samples with a mass of 15±0.5 mg each were cut out from the pipe for different aging times, and the OIT and crystallinity of the samples were determined using a DSC131 differential scanning calorimeter. The operating temperature was 200 °C, the nitrogen flow rate was 50±5 cm³/min, and the oxygen flow rate was 50±5 cm³/min.
(4) Melting property test
Samples were cut into small sizes for different aging times, and an MFR instrument (XNR-400A) was used to measure the MFR of the samples at 190 °C.

3. Analysis of Integrity Test Results

3.1. SEM result analysis
The samples were magnified to 2,000 times during the accelerated-aging tests (Fig. 3). The images showed that at the early stage of the accelerated-aging process, the surfaces of the samples were smooth and flat. After 672 h of accelerated aging, the surfaces of the PE80 gas pipe became relatively coarse with the existence of white round particles and debris, while gullies and holes appeared after aging of 840 h. After aging of 1008 h, the depth of the gullies became further extended, the width of the gullies widened, and the number of holes increased. After 1176 h, the variation trend of the gullies and holes tended to be stable.

During the early stage of accelerated aging, oxide films with a sufficient thickness formed on the sample surface. Under the UV irradiation from a xenon lamp, the samples were rapidly degraded by photooxygenation, which led to the fracture of some large molecular chains on the sample surfaces and the formation of white particles and fragments. Because of the UV irradiation and contact with air, the rough surface expanded, which further facilitated oxidative degradation of the sample surfaces, and resulted in the development of deeper and broader gullies and denser holes. However, when the aging time was extended, the surface gradually formed thicker oxide films. Because the oxidation reaction rate decreased, the aging rate slowed down. Therefore, the surface-microscopic morphology aging of the samples gradually tended to be stable.

![Figure 3. Surface microscopic morphology of PE80 at different aging times](image)

3.2. Mechanical performance result analysis
The tensile strength decay curve of the samples was plotted (Fig. 4). The curve indicated that two stages of the tensile strength variation of the pipe existed: rapid aging stage (0–840 h) and aging platform stage (840–1344 h). During the rapid aging stage, the tensile strength of the samples rapidly decreased from 23.5 to 19.9 MPa, which represented a decrease of 15.3%. During the aging platform stage, the change in the comprehensive evaluation index of the aging performance was minimal, which showed that the aging rate of the PE80 pipes tended to be gentle, and the general performance of the pipe was relatively stable. This behavior occurred because in the rapid aging stage, the PE molecular chain disintegrated under UV irradiation on the sample surface, and the intermolecular force decreased, which reduced the
tensile strength. However, after the aging platform stage was reached, it was difficult for UV radiation to radiate through the samples, and the PE chain disintegrated slowly, which gradually tended to stabilize the mechanical properties.

3.3. Thermal stability result analysis
During the aging process, the material was influenced by heat, oxygen, and other factors, which decreased the thermal stability. Typically, the thermal stability of polymer materials is usually measured using the OIT of the material. The longer the OIT, the better the antioxidant capacity.

The relationship between the OIT of the sample with the aging time was determined (Fig. 5). It was found that during the rapid aging stage, the OIT decreased from 97 to 77 min, which showed a decrease rate of 20.6%. However, during the aging platform stage, the variation in the OIT tended to be stable. This stability was because, in the rapid aging stage, the surface groups of the material were stimulated using UV rays, which accelerated the material oxidation reaction and generated numerous free radicals, and consequently, weakened the material antioxidant capacity and reduced the thermal stability. During the aging platform stage, the material aging layer was completely oxidized, UV rays could not penetrate the material interior, generated free radicals were significantly reduced, oxidation reaction was insignificant, the antioxidant capacity of the material changed slightly, and the thermal stability performance was steady.

3.4. Crystallinity result analysis
Crystallinity refers to the proportion of the crystallization area in the polymer, which reflects the regularity of the internal structure of the material. The more significant the crystallinity, the lower the impact strength and ductility of the material.

The relationship between the crystallinity of the sample and the aging time was determined (Fig. 6). It was observed that during the early stage of aging, crystallinity appeared to increase considerably. This trend occurred because as PE80 is a polymer, its molecules were closely interconnected and exerted strong intermolecular forces. Hence, large molecular chains fractured into small molecular chains during the aging process, and secondary crystallization of the noncrystalline region easily occurred, which improved the crystallinity of the samples. However, during the late aging stage, oxygen oxidized the samples in the noncrystalline region through the gullies, and the generated oxygen-containing functional groups disrupted the molecular structure, which disturbed the material crystallinity. Therefore, based on the overall accelerated-aging cycle, crystallinity initially increased rapidly, and then, decreased slowly.

Figure 4. Tensile strength of PE80 at different aging times

Figure 5. OIT of PE80 at different aging times
3.5. Melting characteristics result analysis

The MFR refers to the mass of the sample strip that flows out from the mold within 10 min under specific temperature and pressure conditions. To some extent, the MFR indicates the degree of difficulty of resin melt flow, and the flow rate speed reflects the size and distribution of the overall molecular weight of the polymer material.

The trend of the MFR at different aging times changed slightly (Fig. 7), and the experimental value remained approximately 0.8 g/10 min. This trend occurred because, during the aging process, oxidation mainly occurred on the exposed surface of the material, and the aging layer was relatively thin, which had a slight effect on the overall molecular weight of the material.

4. Comprehensive evaluation method of integrity

4.1. Standardization of indicators

The test results for the variation in aging time are presented in Table 1. \( X_1, X_2, X_3, \) and \( X_4 \) in Table 1 denote the tensile strength, OIT, crystallinity, and MFR, respectively, corresponding to different aging times.

| Aging time /h | \( X_1 \)/MPa | \( X_2 \)/min | \( X_3 \)/% | \( X_4 \)/(g/10 min) |
|---------------|---------------|---------------|-------------|---------------------|
| 0             | 23.50         | 97            | 59.50       | 0.81                |
| 168           | 22.61         | 95            | 68.67       | 0.80                |
| 336           | 21.97         | 91            | 68.60       | 0.79                |
| 504           | 21.36         | 87            | 66.60       | 0.81                |
| 672           | 20.58         | 82            | 65.60       | 0.81                |
| 840           | 19.90         | 77            | 65.60       | 0.80                |
| 1008          | 19.73         | 77            | 64.90       | 0.80                |
| 1176          | 19.59         | 76            | 65.30       | 0.81                |
| 1344          | 19.64         | 77            | 64.10       | 0.80                |

The standardization method is widely used for the nondimensional processing of data[19]. In this method, each variable is subtracted from the average value, then the difference value is divided by the variable, and finally, the standard deviation is calculated. Thus, the effects of the dimensions and number of stages are eliminated. The data after performing standardization is represented using \( X_i' \) (i = 1, 2, 3, 4), as shown in Table 2.
Table 2. Standardized test data of PE80 gas pipeline samples on performance index

| Aging time /h | $X'_1$ | $X'_2$ | $X'_3$ | $X'_4$ |
|---------------|--------|--------|--------|--------|
| 0             | 1.842  | 1.603  | -2.312 | 1.000  |
| 168           | 1.190  | 1.350  | 1.263  | -0.500 |
| 336           | 0.721  | 0.844  | 1.236  | -2.000 |
| 504           | 0.274  | 0.337  | 0.456  | 1.000  |
| 672           | -0.298 | -0.295 | 0.066  | -0.500 |
| 840           | -0.797 | -0.928 | 0.066  | -0.500 |
| 1008          | -0.921 | -0.928 | -0.207 | -0.500 |
| 1176          | -1.024 | -1.055 | -0.051 | 1.000  |
| 1344          | -0.987 | -0.928 | -0.519 | -0.500 |

4.2. Determination of index weight

Based on the influence degree of different indicators on the entire performance of the PE pipe, the weight of each aging performance index was determined using the AHP. The tensile strength, OIT, crystallinity, and MFR were each compared in pairs to obtain a judgment matrix using 1–9 and its reciprocal scale method for quantitative processing [6].

The most significant index for analyzing the integrity of the PE pipe is the mechanical property. Therefore, the index weight of the tensile strength was ranked first. As a resin material, the pipe was more sensitive to temperature variations, and hence, the OIT was ranked second. The crystallinity reflected the regularity of the internal structure of the material, and so it was ranked third. Previous analysis showed that no significant increase in the MFR occurred during the aging process, which demonstrated that the MFR had a minimal effect on the pipe. Therefore, the MFR was ranked fourth. From the above analysis, the judgment matrix D was established as follows.

$$ D = \begin{bmatrix} 1 & 3 & 5 & 7 \\ 1/3 & 1 & 3 & 5 \\ 1/5 & 1/3 & 1 & 3 \\ 1/7 & 1/5 & 1/3 & 1 \end{bmatrix} $$

The eigenvector of the judgment was selected as the weight vector of each index, and the matrix D was solved using the square root method. The calculation steps are as follows[7]:

1. Multiplying the elements in each row of matrix D and taking the nth root of the result, we obtain $w_i$.

   $$ w_i = \sqrt[\text{nth root of the result}] {D_{ij}} $$

2. The normalization of $w_i$, the weight of each indicator $m_i$, is obtained using the following equation.

   $$ m_i = \frac{w_i}{\sum_{i=1}^{n} w_i} $$

The weight vector of the four indicators were calculated as $M = [0.5638, 0.2634, 0.1178, 0.0550]^T$. The consistency of the judgment matrix was tested[7], and the calculation equations used were as follows:

$$ \lambda_{\text{max}} = \frac{1}{n} \sum_{j=1}^{n} (D \cdot M)_j $$

$$ I_C = \frac{\lambda_{\text{max}} - n}{n - 1} $$
\[ R_C = \frac{I_C}{I_R} \] (5)

where \( \lambda_{max} \) is the largest characteristic root of matrix D; IC is the consistency index value; RC is the consistency ratio; IR is the random consistency index.

If the consistency ratio RC is less than 0.1, the consistency is considered adequate. Otherwise, the value needs to be revised again. The values of the random consistency index IR are listed in Table 3.

| n  | IR  | n  | IR  |
|----|-----|----|-----|
| 1  | 0   | 6  | 1.24|
| 2  | 0   | 7  | 1.32|
| 3  | 0.58| 8  | 1.41|
| 4  | 0.9 | 9  | 1.45|
| 5  | 1.12| 10 | 1.49|

The calculated value of the highest characteristic root of matrix D was 4.1187, and the consistency index value IC was 0.03957. As \( n = 4 \), the value of the random consistency index IR was set to 0.9, and the consistency ratio RC was obtained as 0.044 < 0.1. This RC value showed that matrix D passed the consistency test, and the weight distribution is realistic.

4.3. Comprehensive evaluation of integrity

The comprehensive evaluation index for the integrity of PE gas pipelines at different aging times was obtained based on the individual performance indexes and their respective weights in the dimensionless treatment.

\[ Z = \sum_{i=1}^{4} X_i m_i \] (6)

In Equation (6), Z is the integrity of the comprehensive evaluation index. Z is used to integrate the data for different performance indexes. Moreover, its application helps in overcoming the limitations of itemized evaluation of the single-performance index and shows the variations in the pipeline integrity more comprehensively.

The curve of the PE80 gas pipeline integrity attenuation was plotted using Equation (6), and the graph is depicted in Fig. 8. The integrity comprehensive evaluation index of the PE gas pipeline under the photo-oxygen aging conditions gradually decreased with the increase in aging time, and finally tended to be stable. During the rapid aging stage, the Z-value slowed down rapidly, and its overall performance slowed down rapidly. During the aging platform stage, the Z-value changed slightly, which showed that the aging rate of the pipeline tended to be flat, and the overall performance was relatively stable. The trend of comprehensive performance attenuation curve of integrity was consistent with the variation trends of the main evaluation performance indexes (tensile strength and OIT). The curve showed that the integrity comprehensive evaluation index could reflect the variation in pipeline integrity more comprehensively.
5. Conclusion

(1) The samples experienced two different stages of aging: rapid aging stage and aging platform stage. During the accelerated-aging process, deep gullies and holes were formed on the surface of the samples detected through microscopic analysis. The tensile strength and OIT initially decreased and later tended to stabilize. The crystallinity of the samples increased and subsequently decreased, whereas the MFR remained almost unchanged.

(2) During the rapid aging stage, the comprehensive evaluation index for the aging performance decreased rapidly. However, the comprehensive evaluation index for aging performance almost remained unchanged during the aging platform stage.

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