JANA

KNOWLEDGE ● VALUES ● SUCCESS

TECHNICAL REPORT

FORECASTING THE LONG-TERM STRENGTH OF THERMOPLASTIC PIPING MATERIALS – A REVIEW OF THE HISTORY, CURRENT STATE OF THE ART AND NEW ADVANCES

Confidential

FORECASTING THE LONG-TERM STRENGTH OF THERMOPLASTIC PIPING MATERIALS – A REVIEW OF THE HISTORY, CURRENT STATE OF THE ART AND NEW ADVANCES

S. Chung, J.D. Kim, M. Toro, P. Vibien and K. Oliphant
Jana Laboratories Inc., 280B Industrial Pkwy S., Aurora, ON Canada

ABSTRACT

Plastic piping materials have enjoyed a long and successful history. One of the key factors in their acceptance and successful application has been the development and refinement of methodologies for forecasting the long-term strength of these materials. In the development of these methodologies, three distinct failure regimes have typically been distinguished. These three regimes are typically referred to as Stage I, Stage II and Stage III, representing Ductile-Mechanical, Brittle-Mechanical and Brittle-Oxidative failure modes, respectively. A review of the history of the development and methodologies used for characterizing Stage I and Stage II performance is provided. With material evolution, the projected service lifetimes of plastic piping materials continue to increase. This has led to an increasing interest in characterization of the Stage III brittle-oxidative resistance of these materials to define the ultimate lifetime. The mechanisms associated with brittle failure of polyolefin piping materials are examined. The competing mechanisms of oxidative and mechanical crack initiation and propagation are found to control the ultimate mechanism of failure. Four different possible failure modes, in this regime, are identified and examples of the failure modes observed in laboratory testing are provided. Identification of the different failure modes and methodologies for forecasting and validating pipe oxidative performance are discussed.

DEVELOPMENT OF PIPE PRESSURE RATING METHODOLOGIES

Plastic piping materials have enjoyed a long and successful history. Since the introduction of the first commercial thermoplastic pipe in the 1940’s, plastic pipe usage and applications have continued to expand. Certainly the inherent characteristics of the materials themselves; such as resistance to corrosion, ductility, ease of handling and installation, etc., have played a large part in this success. Another key factor, however, in their acceptance and successful application has been the development and refinement of methodologies for forecasting the long-term strength of these materials (1). These methodologies enabled the transformation of plastic piping into an engineering material – a material with predictable and reproducible properties.

In the development of methodologies for forecasting long-term strength of plastic piping materials, three distinct failure regimes have typically been distinguished. These three regimes are typically referred to as Stage I, Stage II and Stage III, representing Ductile-

Mechanical, Brittle-Mechanical and Brittle-Oxidative failure modes, respectively. Stage I failures typically occur at higher stresses and shorter times. For some materials there is a potential for transition to Stage II failures at longer times. If a material does not fail by Stage I or Stage II failure in service, Stage III brittle-oxidative performance is believed to represent the ultimate lifetime of the piping material.

Characterization of Stage I performance is used to define a plastic pipe materials pressure rating or mechanical strength. In North America, the methodology defined in ASTM D2837, Standard Test Method for Obtaining Hydrostatic Design Basis for Thermoplastic Pipe Materials (20), has been successfully applied for over thirty years. A similar methodology, ISO 9080, Plastics Piping and Ducting Systems – Determination of the Long-Term Hydrostatic Strength of Thermoplastics Materials in Pipe Form by Extrapolation (5) has also been successfully applied in Europe and, more recently, in North America.

The development of the ASTM D2837 pressure rating methodology (1), was initiated in 1958 with the establishment of the 'Working Stress Committee' of the Thermoplastics Pipe Division of the Society of the Plastics Industry. This committee has since evolved into the Hydrostatic Stress Board (HSB) of the Plastics Pipe Institute (PPI). In 1961 the first tentative protocol for pressure rating thermoplastic pipes was published which eventually became ASTM D2837, first published in 1969. The primary four components remain to this day: 1. Adoption of a 'thin-wall' equation relating internal pressure to hoop stress; 2. A uniform test method for developing creep rupture data; 3. A uniform mathematical extrapolation procedure for analyzing the creep-rupture data and estimating a material's long-term strength; and 4. Adoption of a uniform design factor. Early in the development of this methodology the group considered the graphical approach then used in Europe (1), the forerunner to ISO 9080. This method was based on creep-rupture studies showing that creep-rupture data obtained on a pipe made from a certain grade of PE resin exhibited a sharp downturn (a 'knee') at some point in time and that the time which the downturn occurred was greatly accelerated by temperature (2). Because of preference for a mathematical approach and no indication of any down-turning tendency in the North American data analyzed at that time, this methodology was dropped from consideration. Early in the implementation of the ASTM D2837 methodology used to forecast long-term strength based on Stage I performance, however, reports appeared of pipes that were failing prematurely by brittle-like slits even though the pipes were subjected to operating pressures that were significantly below the projected safe value (based on characterization of Stage I performance). The recognition of these Stage II type failures led to the examination in the early 1980's of concepts for validating the assumptions of linearity of extrapolation inherent in the ASTM D2837 methodology. This lead to a number of additional requirements for pressure rating polyethylene pipe materials and the eventual adoption of a Rate Process based procedure for validation by PPI in 1985 and the eventual inclusion of the requirement for validation of the long-term strength projection of PE piping materials into ASTM D2837 in 1988. Since this time, a number of additional allowable validation methodologies have been added to ASTM D2837 and PPI TR-3 (3).

In the 1970's a small study group was formed within ISO/TC138 to examine the then European graphical approach, which acknowledged 'knees', and the North American statistical approach, which did not (4). The net result, after much work, was ISO TR 9080 employing the Standard

Extrapolation Method (SEM). With continued refinements, the SEM now provides a statistically based calculation methodology that accounts for knees explicitly within the methodology (5).

ACCELERATED VALIDATION METHODOLOGIES

With material evolution and the expansion of plastic pipe into ever more demanding applications, a practical problem has been introduced in that material characterization and qualification has become increasingly time consuming given the higher end use temperatures, material testing temperature limitations and extrapolation limits. This has led to efforts to develop alternative, more accelerated, validation methodologies. The most common of these methodologies, which have traditionally focused on characterization of Stage II performance, are summarized below.

Notched Pipe Test

The Notched Pipe Test (ISO 13479, ASTM F1474) provides an acceleration over standard elevated temperature hydrostatic pressure testing of pipe through external axial notching of the pipe at four points around the circumference. The test is proscribed in terms of a minimum performance requirement in a number of ISO and European standards. Its development as been reported previously (6). An acceleration of approximately ten fold is typically observed relative to standard hydrostatic pressure testing. For some current generation materials, however, test times can still exceed one year. The typical slow crack growth (SCG) failure mechanism observed in pipe testing appears to be replicated. While the methodology generally correlates with relative resistance to slow crack growth, it has not been explicitly related to forecasting long-term pipe performance.

PENT

The PENT test (ASTM F1473, ISO 16241), developed by Dr. Norman Brown, involves testing of a single edged notched specimen under constant load at elevated temperature. The test is proscribed in terms of a minimum performance requirement in North American standards. An acceleration of approximately fifty fold is typically observed relative to standard hydrostatic pressure testing. For some current generation materials test times in excess of 4000 hours can still, however, be observed. The typical SCG failure mechanism observed in pipe testing appears to be replicated. While the methodology generally correlates with relative resistance to slow crack growth, it has not been explicitly related to forecasting long-term pipe performance.

Full Notch Creep Test (FNCT)

The Full Notch Creep Test (FNCT) involves testing of a square bar section with central coplanar notches on each face under static load at elevated temperature in a surfactant. The methodology, little used in North America, is gaining in use in Europe and is being developed as an ISO standard (ISO/DIS 16770.2). The acceleration is typically on the order of 100 fold relative to standard hydrostatic pressure testing. The highly constrained crack tip geometry can generate SCG failures in materials where notch blunting is observed to occur in less severe tests. The relative acceleration of the test methodology also appears to vary widely between materials. The typical SCG failure mechanism observed in pipe testing appears to be replicated, although differences in the fibril structure of fracture surfaces associated with the effect of the surfactant on the SCG process are typically observed (8). While the methodology generally correlates with relative resistance to slow crack growth, it has not been explicitly related to forecasting long-term pipe performance.

Cone Test

The Cone Test (ISO 13480), developed by Gaz de France, involves monitoring crack initiation and propagation in a notched pipe sample into which a cone is inserted to strain the material. Testing is conducted at elevated temperature in a surfactant. The method can provide relatively quick results for some materials. For highly stress crack resistant materials, however, crack propagation may not occur. The typical SCG failure mechanism observed in pipe testing appears to be replicated, although differences in the fibril structure of fracture surfaces associated with the effect of the surfactant on the SCG process are typically observed (8). The methodology has not been explicitly related to forecasting long-term pipe performance.

Cyclic Stress Testing

A number of methodologies have been applied to accelerate SCG through the cyclic application of stress. The acceleration relative to standard hydrostatic pressure testing can be on the order of 500 fold. The typical SCG failure mechanism observed in pipe testing appears to be replicated. The methodology has also not been explicitly related to forecasting long-term pipe performance. The typical SCG failure mechanism observed in pipe testing appears to be replicated.

Natural Draw Ratio

The Natural Draw Ratio, or the percentage of elongation at the end of the plateau of plastic deformation at the onset of ‘strain hardening’, has been related by a number of authors to SCG resistance. While a number of issues remain to be resolved to enable practical use of this methodology it provides interesting opportunities for very quick characterization of slow crack growth performance.

While significant progress has been made in developing accelerated methodologies for characterizing relative slow crack growth, efforts continue to correlate more accelerated test methodologies with actual performance and the standard validation methodologies. Much work remains to be done to develop accelerated methodologies that provide meaningful characterizations of material slow crack resistance in reasonable time frames that can be explicitly related to end use performance. This has been necessitated by the excellent resistance to slow crack growth of current generation materials.

NEW ADVANCES IN PRESSURE PIPE RATING METHODOLOGIES

With current generation plastic pipe materials and the current methodologies for characterization of the Stage I and Stage II regimes, service lifetimes, dependant upon the application, in excess of 100 years (9) are often referred to. This, and the expansion of plastic pipe into ever more demanding applications, has resulted in a growing focus on the characterization of Stage III brittle oxidative performance of plastic piping materials. An ASTM

Standard, ASTM F2023, Test Method for Evaluating the Oxidative Resistance of Crosslinked Polyethylene (PEX) Tubing and Systems to Hot Chlorinated Water (10) has recently been developed and used as the basis for establishing minimum oxidative resistance for PEX pipe materials for potable water applications as a requirement in the product standard (ASTM F876, Standard Specification for Crosslinked Polyethylene (PEX) Tubing (11)). A similar test methodology for polyethylene (PE) piping materials has also been developed (ASTM F2263, Standard Test Method for Evaluating the Oxidative Resistance of Polyethylene (PE) Pipe to Chlorinated Water (12)).

An examination of the mechanisms associated with Stage III brittle-oxidative degradation of polyolefin materials is provided below. The competing mechanisms of oxidative and mechanical crack initiation and propagation are found to control ultimate failure. The different possible failure modes are examined and examples of those failure modes observed in laboratory testing are provided. Testing was conducted on commercially available polyolefin piping materials. Standard hydrostatic pressure testing was conducted in accordance with ASTM D1598 (13). Chlorine resistance testing was conducted in general conformance with the ASTM F2023 (10) or ASTM F2263 (12) methodologies.

Characterization of Failure Modes

In accelerated performance testing of polyolefin piping materials to characterize Stage III behavior, several possible failure modes have been identified. These failure modes depend on the time-scale of the competing oxidative and mechanical degradation mechanisms in crack initiation and propagation. It is important to identify and distinguish between these different failure modes to ensure that the methodologies being applied to forecast long-term strength are valid for the failure mode in question and so that different failure modes are not inadvertently grouped together in the same analysis.

For crack initiation, initiation can be either Mechanical or Oxidative. Mechanical Crack Initiation is the typical initiation associated with Stage II performance testing. This initiation process is typically regarded as defect driven with the initiation time dependent on the specific stress state, the temperature and the material properties (14). For Oxidative Crack Initiation, the initiation time is a function of the oxidative environment, the temperature, the stress and the material. Crack initiation occurs due to oxidative degradation of the inner surface leading to embrittlement (15).

Similarly, for crack propagation, propagation can be either Mechanical or Oxidative. Again, Mechanical Crack Propagation is the typical propagation associated with Stage II performance testing. Crack propagation occurs due to a mechanical breakdown of the fibrils spanning the process zone in the crack front (14). For Oxidative Crack Propagation, it appears that oxidative breakdown of the fibrils spanning the process zone in the crack front occurs.

Based on the specific test conditions and material properties there are, therefore, four possible overall failure modes:

Mode 1: Mechanical Initiation - Mechanical Propagation
Mode 2: Mechanical Initiation - Oxidative Propagation
Mode 3: Oxidative Initiation - Mechanical Propagation
Mode 4: Oxidative Initiation – Oxidative Propagation

Mode 1 is the typical failure mode associated with Stage II failures. Mode 2 behavior is potentially observed in some standard testing to characterize Stage II performance. Mode 3 failures appear to be a possible failure mode. This mode of failure has not however typically been differentiated in standard testing and field failure analysis. Mode 4 is the typical failure mode associated with Stage III failures. In characterizing oxidative performance of piping materials Mode 4 behavior is typically of primary interest. While the distinction has been made between modes in this paper to facilitate discussion, in reality there may be a continuum of failure modes progressing from pure mechanical to pure oxidative.

Observed Failure Modes

As discussed previously, it is important to identify and distinguish between the different possible failure mechanisms. To that end, examples of the different failure modes observed in laboratory testing are examined below.

Mode 1: Mechanical Initiation –Mechanical Propagation

A typical fracture surface for a Mode I or Stage II type failure is shown in Figure 1A. The failure was generated in a polyolefin material in standard hydrostatic pressure testing with water internal/water external at 80 ^{o}C. The failure is typical of Mode I failures in that failure occurred at a single point and that the inner pipe surface remained ductile (i.e. no oxidative degradation) as evidenced by reverse bend testing (i.e. no crack or crazing of the inner pipe surface). The initiation point is shown in Figure 1B and 1C. Initiation occurred at an inclusion approximately 15 µm in diameter. As shown by EDX analysis in Figure 2, the inclusion composition is carbon based. The classic microfibril structure of the fracture surface is clearly shown in Figure 1D. This failure typifies the classic Mechanical Initiation – Mechanical Propagation failure mode.

Mode 2: Mechanical Initiation - Oxidative Propagation

Mode 2 failures have not been clearly identified in experimental testing to date. There is evidence reported in the literature, however, of oxidative effects observed in Stage II testing (18). Current research projects at Jana Laboratories are attempting to generate and characterize Mode 2 type failures.

Mode 3: Oxidative Initiation - Mechanical Propagation

Figure 3 shows a plot of log (failure time) versus log (stress) for a polyolefin pipe tested in two different oxidative environments. The Mode I failure lines were generated by standard hydrostatic pressure testing according to ASTM D1598 (15) with water internal/air external at 80 ^{o}C. The Mode III failure line was generated by testing in general accordance with ASTM F2263 (12), which involves testing with oxidatively aggressive flowing internal water/air external at 80 ^{o}C.

The Mode I failures were as described above and shown in Figure 1. There was typically a single crack initiation-propagation point. There was also no evidence of inner surface oxidation and the fracture surfaces exhibited the typical microfibril structure seen in Figure 1.

The Mode 3 failures, on simple visual examination, appear similar to Mode I type failures. A comparison of typical Mode 1 and Mode 3 failures is provided in Figure 4. For the Mode 3 failures, there was typically a single crack initiation point and there was typically little or no visual evidence of inner surface oxidation. There was no evidence of bulk oxidation on examination by Fourier Transform Infrared (FTIR). The loss of inner surface ductility is clearly evident, however, on reverse bend testing or tensile elongation of the inner pipe surface (crazing and microcracking). The shift in initiation time is also clearly visible in Figure 3. At the test conditions, the failure curve is shifted a minimum of one log decade.

For the Mode 3 failures, SEM examination of the crack surface reveals the same microfibril structure observed for the Mode I failures, indicating Mechanical Crack Propagation. Crack initiation is, however, at the inner surface as opposed to near the inner surface and typically at a defect for Mode I failures. The slope of the Mode 1 and Mode 3 failure curves are similar, with the Mode 3 failure curve being somewhat steeper. This may be an indication that there is some oxidative component to the crack propagation or may simply reflect a different stress dependence of the oxidative and mechanical initiation mechanisms.

Mode 4: Oxidative Initiation – Oxidative Propagation

A typical Mode 4 type failure is shown in Figure 5. The sample is a polyolefin material tested in accordance with ASTM F2023, which involves testing with oxidatively aggressive flowing internal water/air external.

As previously reported (15,16,17), the failure mechanism appears to be: 1. Oxidation of the inner surface, 2. Once sufficient oxidation and degradation of the inner wall occurs a combination of degradation induced and applied stresses on the inner pipe surface causes micro-cracks to form in the degraded inner layer, 3. The crack density and crack length increase with exposure time. The cracks propagate through the wall of the pipe material, 4. The cracks begin to coalesce to form larger cracks and 5. Ultimately a brittle slit or pin hole failure is observed when a crack propagates through the entire wall thickness.

The distinguishing features of a typical Mode 4 type failure (as shown in Figure 5) are a typically highly degraded inner surface with microcracking and multiple crack initiation points. The crack surface is also typically highly degraded and does not typically show the classic microfibril structure of Mode 1 and Mode 3 type failures.

Modeling Test Data

Mode 1 Mechanical – Mechanical (or Stage II) type failures have been successfully modeled using elevated temperature testing and the Rate Process Model (19) for over two decades. The results of this approach have also been correlated with field performance of plastic piping materials (19).

Mode 2 Mechanical – Oxidative type failures have not typically been differentiated in analysis, although there are reports of an oxidative component in standard Stage II type testing (18). The applicability of current modeling approaches for Mode I type failures applied to Mode 2 type failures should be examined.

Mode 3 type failures have also not typically been differentiated. Given that the initiation is chemically driven and the propagation appears to be predominantly mechanically driven, caution needs to be applied to utilizing the current accepted methodology of the Rate Process Model to this failure mode. Further work needs to be conducted to either validate the application of the current failure models or to develop alternate analysis methodologies.

Mode 4 (or Stage III) type failures have been successfully modeled using either the Rate Process Model or Arrhenius type extrapolations (15,16,17). More recently this modeling approach has formed the basis for establishing minimum performance requirements for specific plastic piping materials (10,11).

The competing mechanisms of mechanical and oxidative crack initiation and propagation are found to control the ultimate mechanism of failure. The different possible failure modes were identified and examples of those observed in the laboratory were provided. Modeling of test data for Mode 1 and Mode 4 type failures has been successfully achieved using the Rate Process Model. For Mode 2 and Mode 3 type failures, further work needs to be conducted to either validate the application of the current models or to develop alternative analysis methodologies.

CONCLUSIONS

Plastic piping materials have enjoyed a long and successful history. Part of this success has been the development and refinement of methodologies for forecasting the long-term strength of these materials. The large advancements in material performance continue to drive advancement in methodologies for characterizing and validating performance. With the excellent performance of current generation materials and the expansion of plastic piping materials into ever more demanding applications the need for evolution in test methodologies has never been greater. Currently the focus is on the development of more accelerated methodologies to characterize Stage II performance and methodologies to characterize Stage III performance. As material performance and characterization methodologies continue to evolve so will the opportunities for successful application of plastic piping materials.

ACKNOWLEDGEMENTS

Jana Laboratories gratefully acknowledges the financial support of the Industrial Research Assistance Program (IRAP) and the assistance of Dr. Chudnovsky of the University of Illinois at Chicago for assistance in image analysis.

REFERENCES

1. K. Oliphant, S. Mruk,, *Forecasting the Long-Term Strength of Thermoplastic Piping Materials- A Review of the History, Current Status and Proposed Refinements to the Generally Accepted Knowledge,* presented at the American Gas Association Symposium, San Francisco, 2002.
2. Richard, K., Diedrich G., Gaube E., *Demands on High Quality Pipes for Potable Water Supply Made of Ziegler Polyethylene,* paper presented at a meeting of the division of

Paint, Plastics and Painting Ink Chemistry, American Chemical Society, September 1957.
3. PPI TR-3, *HDB/PDB/SDB/MRS Policies*, Plastics Pipe Institute, USA, 2004.
4. P. Vanspeybroeck, *SEM, The New Approach for the Classification and Designation of Thermoplastics Materials*, Plastics Pipes XI, Munich, Germany, September 2001.
5. ISO 9080, *Plastics piping and ducting systems – Determination of the long-term hydrostatic strength of thermoplastics materials in pipe form by extrapolation*, 2003.
6. W.J. Allwood and S.H. Beech, *Proc. 13th Int. Plastic Fuel Gas Pipe Symposium*, San Antonio, Texas, 2003.
7. S. Beech, C.R. Ferguson and E.Q. Clutton, *Mechanisms of Slow Crack Growth in PE Pipe Grades*, Plastics Pipes XI, Munich, Germany, September 2001.
8. E. Laurent, *Comprehensive Evaluation of the Long-Term Mechanical Properties of PE100 Resins Meeting the Requirements of Modern Installation Techniques*, Plastics Pipes XI, Munich, Germany, September 2001.
9. Nicke Bergman, *Polyethylene pipes – a 100 year service life?*, Plastics Pipes X, Goteborg, Sweden, 1999.
10. ASTM F2023, *Standard Test Method for Evaluating the Oxidation Resistance of Crosslinked Polyethylene (PEX) Tubing and Systems to Hot Chlorinated Water*, 2000.
11. ASTM F876, *Standard Specification for Crosslinked Polyethylene (PEX) Tubing*, ASTM International, USA, 2000.
12. ASTM F2263, *Standard Test Method for Evaluating the Oxidative Resistance of Polyethylene (PE) Pipe to Chlorinated Water,* in press.
13. ASTM D1598, *Standard Test Method for Time-to-Failure of Plastic Pipe Under Constant Internal Pressure,* Vol. 8.04, 2003.
14. E. Laurent, *Comprehensive Evaluation of the Long-Term Mechanical Properties of PE 100 Resins Meeting the Requirements of Modern Installation Techniques*, Plastics Pipes XI, Munich, Germany, 2001.
15. K. Oliphant, J. Couch, P. Vibien, A. Chudnovsky, B. Zhang, W. Zhou, *Chlorine Resistance Testing of Cross-linked Polyethylene Piping Materials*, Society of Plastics Engineers Annual Technical Conference (ANTEC), Dallas, USA, 2001.
16. K. Oliphant, J. Couch, P.Vibien, A. Chudnovsky, B.Zhang, W.Zhou, *Assessing Material Performance in Chlorinated Potable Water Applications*, Plastic Pipes XI, Munich, Germany, 2001.
17. S. Chung, J. Couch, J.D. Kim, K. Oliphant and P. Vibien, *Environmental Factors in Performance Forecasting of Plastic Piping Materials*, Society of Plastics Engineers Annual Technical Conference (ANTEC), 2003.
18. R. W. Lang, G. Pinter, A. Stern, *Applicability and Limitations of Current Life-Time Prediction Models for Thermoplastics Pipes Under Internal Pressure*, Plastics Pipes XI, Munich Germany, 2001.
19. Palermo, Gene, Dr., and Jana Laboratories, *Correlating Aldyl "A" and Century PE Pipe Rate Process Method Projections with Actual Field Performance*, PPXII, Milan, Italy, 2004.
20. ASTM D2837, *Standard Test Method for Obtaining Hydrostatic Design Basis for Thermoplastic Pipe Materials or Pressure Design Basis for Thermoplastic Pipe Products,* 2002.

Figure 1: Mode 1 Fracture Surface: 1A: Fracture Point, 1B: Initiation Point, 1C: Initiation Point Detail, 1D: Detail of Microfibril Crack Surface

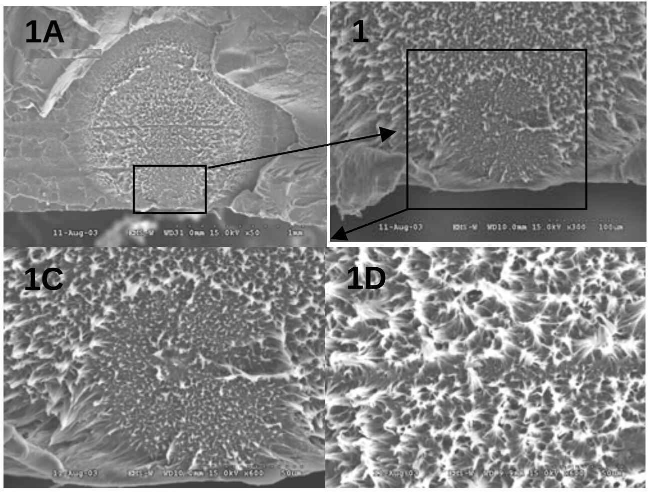


Figure 2: SEM EDX Analysis of Crack Initiation Point Inclusion

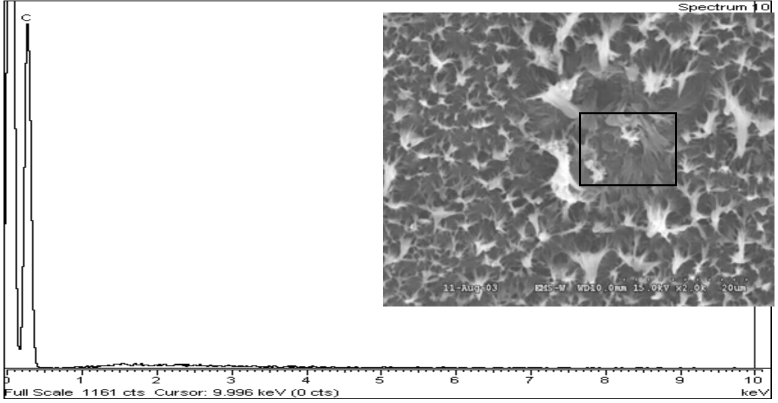


Figure 3: Comparison of Mode 1 and Mode 3 Failure Times

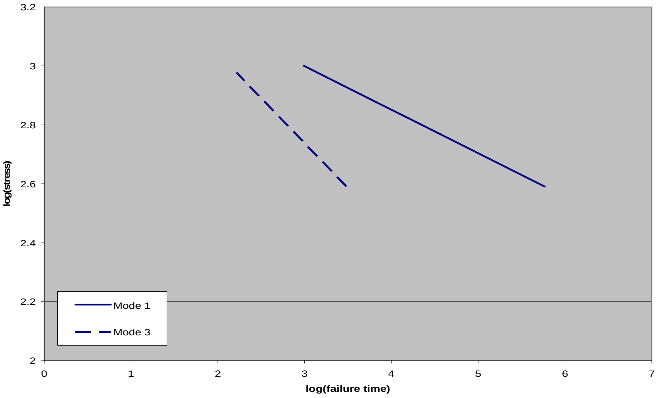


Figure 4: Visual Comparison of Mode 1 and Mode 3 Failures

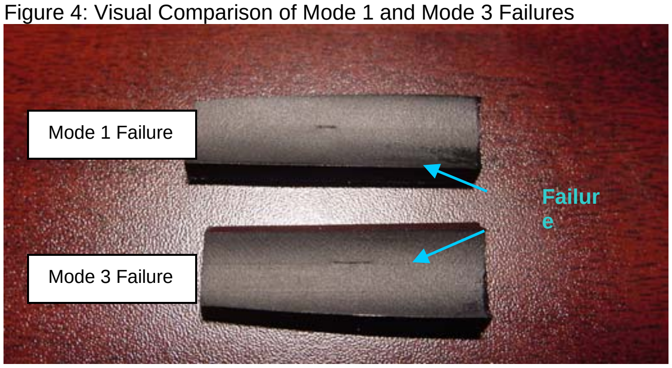
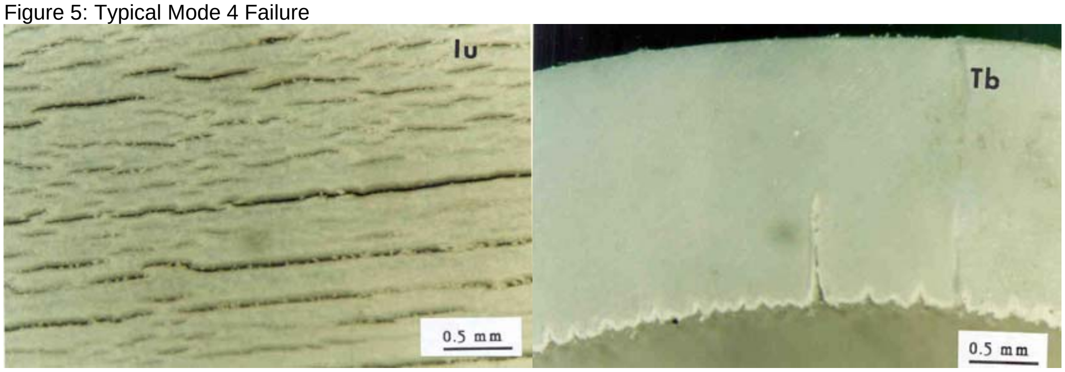


Figure 5: Typical Mode 4 Failure



JANA

JANA LABORATORIES INC.
280B Industrial Parkway South
Aurora, Ontario L4G 3T9
Canada
Ph: 905-726-8550
Fax: 905-726-8609
www.janalab.com