

Evaluation Standard for Internal Pressure Sheath Polymers for High Temperature Flexible Pipes

API TECHNICAL REPORT 17TR1
FIRST EDITION, MARCH 2003



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Upstream Segment

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FOREWORD

This technical report is under the jurisdiction of the API Subcommittee on Subsea Production Systems. This document has been produced as part of a joint industry project "Evaluation of Polymers for High Temperature Flexible Pipes".

This JIP involved a materials test programme, and finite element analyses of both global and local flexible pipe cross sections. Prototype testing of a high temperature flexible pipe was not performed under this JIP; consequently, the material properties discussed in this document are those which the steering committee considered are/will be important and relevant to the application. The JIP has been technically and financially supported by an international consortium of oil companies, flexible pipe manufacturers, polymer manufacturers, regulatory authorities and contractors.

A methodology is described for the development of a test plan for the evaluation of a polymer for high temperature service. Also defined are a set of evaluation criteria by which a material must be assessed for qualification. The standard does not seek to rigidly define acceptance and rejection criteria. These should be specified by the user, giving consideration to a particular flexible pipe application. This standard aims to give the user guidance on the selection of these criteria.

The evaluation methodology recommends performing a set of preliminary evaluation tests on polymers in situations where little or no information is available to indicate their likely performance. This is a low cost approach to which allows key materials properties to be screened. A *preliminary evaluation* is not required where there is sufficient confidence in, and information about, a materials suitability to proceed directly to a more comprehensive testing programme. In this instance, or where a material has passed the preliminary evaluation programme, a *full evaluation* programme is undertaken. This involves longer term more extensive testing to identify performance characteristics of the material. The full evaluation stage is compulsory for the qualification of materials for service according to this document.

Material properties which are relevant to the evaluation of polymers for high temperature flexible pipe service application are described. A test procedure is presented for each of these properties, making reference to existing standards where applicable. A protocol covering test procedural matters relating to the overall evaluation test programme is also provided.

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1 SCOPE

1.1 GENERAL

1.1.1 This technical report defines the methodology and test procedures necessary for the evaluation of polymeric materials suitable for use as the internal pressure sheath of an unbonded flexible pipes in high temperature applications. It describes the processes by which the critical material properties, both static and dynamic, can be measured and evaluated against relevant performance criteria.

1.1.2 This document relates primarily to the properties necessary for an internal pressure sheath required for oil and gas production. These are most relevant to high temperature applications. Only thermoplastic materials are considered for the internal pressure sheath. Elastomeric materials, which are used in bonded flexible pipes, are not considered in this document.

1.1.3 This document has the following format:

- Section 1 **Scope**
- Section 2 **Referenced Documents**
- Section 3 **Definitions and Acronyms**
- Section 4 **Service Application**—Background to selection of material tests used in the evaluation programme
- Section 5 **Evaluation Overview**—The overall methodology employed in the evaluation of a candidate material
- Section 6 **Evaluation Test Programme**—Presents, in detail, each of the material tests which form the material evaluation test programme
- Section 7 **Material Evaluation**—Presents the criteria against which material test results should be compared and gives guidance on the interpretation of results.
- Section 8 **Bibliography**

1.1.4 The following two applications of the Evaluation Standard are considered:

- a. the evaluation of a candidate polymer for HT flexible pipe service,
- b. the evaluation of a candidate polymer to determine its generic performance envelope/limits.

1.2 SIGNIFICANCE

1.2.1 This Evaluation Standard provides a procedure for determining whether candidate polymeric materials have the property levels necessary for successful use as the internal pressure sheath of an unbonded flexible pipe in high temperature high pressure applications.

1.2.2 For the purposes of this document, high temperatures are defined as those between 130 and 200°C. High pressure is considered to be at least 34.5 MPa (5000 psi).

1.2.3 This Evaluation Standard also provides a means for comparing the performance attributes of several potential pressure sheath materials for high temperature applications.

1.3 CURRENT STANDARDS

1.3.1 The current standards which relate to the evaluation of polymers for internal pressure sheath applications are:

- i) API Specification 17J, "Specification for Unbonded Flexible Pipe," First Edition [1],
- ii) API Recommended Practice 17B "Recommended Practice for Flexible Pipe," Second Edition [2].

1.3.2 These documents address all aspects of unbonded flexible pipe technology relevant to current levels of high temperature service; that is, to 130°C.

1.4 UNITS

Système International (SI) units are used in this evaluation standard. Imperial units may be given in brackets after the SI units.

1.5 SAFETY

The procedures described in this evaluation standard include materials tests requiring the use of high temperature and high pressure conditions, often with hostile chemicals. It is the responsibility of individuals or organisations using the standard to ensure that all appropriate safety procedures are implemented to prevent injuries to personnel and/or damage to equipment or facilities.

2. REFERENCED STANDARDS

- API Spec 17J Specification for Unbonded Flexible Pipe
- API RP 17B Recommended Practice for Flexible Pipe
- ASTM D256 Test Methods for Impact Resistance of Plastics and Electrical Insulating Materials
- ASTM D638 Test Method for Tensile Properties of Plastics
- ASTM D2990 Test Methods for Tensile, Compressive, and Flexural Creep and Creep Rupture of Plastics
- ASTM E831 Test Method for Linear Thermal Expansion of Solid Materials by Thermo-mechanical Analysis
- ESIS draft protocol "A Testing Protocol for Conducting J-Crack Growth Resistance Curve Tests on Plastics," Hale, G., May 1994.

3 DEFINITIONS AND ABBREVIATIONS

3.1 DEFINITIONS

For the purposes of this standard, the following definitions apply:

3.1.1 Flexible Pipe: An assembly of a pipe body and end fittings. The pipe body comprises a composite of layered materials (polymeric and metallic) that form a pressure containing conduit. The pipe structure allows large deflections without a significant increase in bending stresses. The term “pipe” is used in this document as a generic term for unbonded flexible pipe.

3.1.2 Conventional Flexible Pipe: An unbonded flexible pipe which is suitable for internal fluid temperatures of up to 130°C.

3.1.3 High Temperature Flexible Pipe: An unbonded flexible pipe which is suitable for internal fluid temperatures of between 130 and 200°C.

3.1.4 Internal Pressure Sheath: The innermost contiguous polymeric layer of an unbonded flexible pipe, designed to contain transported fluids and ensure pipe integrity.

3.1.5 Candidate Polymer: A polymeric material which is proposed for use as the internal pressure sheath of an unbonded flexible pipe and is being submitted for evaluation testing.

3.1.6 End Fitting: A mechanical device which forms the transition between the flexible pipe body and the connector. The different pipe layers are terminated in the end fitting in such a way as to transfer the load between the flexible pipe and the connector.

3.1.7 Test Temperature, Test Pressure: The temperature (T) and pressure (P) at which a test is to be run for the purposes of evaluating a candidate polymer. The choice of test temperature/pressure will depend only on the performance requirements for the proposed application.

3.1.8 Test Result: The outcome of a materials test. This may be in the form of a physical quantity (e.g., Young’s modulus) or a subjective rating (e.g., blister damage).

3.1.9 Maximum Property Temperature (MPT): The maximum temperature for which a specified property meets the performance criterion; for example, if a candidate polymer swells 50% in oil at 165°C, but only 1% at 160°C, its MPT (for compatibility with the oil) is 160°C, assuming that 1% volume swell has been defined as acceptable for the application. See Figure 1.

3.1.10 Maximum Service Temperature (MST): The maximum temperature for which all measured

properties meet the specified performance criteria. In practice, there is little point in testing above the rapid gas decompression (RGD) MPT (other than when conducting accelerated ageing based on another property). If the MPT value of another property is lower then it will give the MST. For example, if a candidate polymer has the following MPTs – RGD (190°C), tensile (>190°C), crack growth fatigue (165°C), liquid compatibility in three different fluids (180°C, >190 °C, 170°C) – then the MST for the material is defined by the lowest MPT, that is, 165°C. See Figure 2. It is implicit that MST is significantly below candidate polymer melting point.

3.1.11 Maximum Pipe Service Temperature (MPST): The maximum temperature for which all measured properties meet performance criteria specific to a particular flexible pipe design. For example, the analysis of a particular design might indicate that the allowable strain in a material with a MST of 180°C is exceeded under certain bending conditions at 175°C but not at 170°C—hence MPST must be set to 170°C. Note that the determination of MPST is beyond the scope of this evaluation standard; it is included here for completeness. See Figure 1.

3.1.12 Evaluation Criterion: A predetermined criterion against which test results are compared in order to facilitate the evaluation of a candidate polymer.

3.1.13 Performance Criterion: A performance criterion specifies an acceptable range or limit for a measurable material property, within which a candidate polymer must lie if it is to have any chance of functioning successfully in the high temperature flexible pipe application.

3.1.14 Extrusion: For flexible pipe application, extrusion is the preferred term to describe the deformation (“flow”) of the pressure sheath into armour gaps as a result of applied pressure. Extrusion (of a semi-crystalline polymer) has three components: *elastic extrusion*, the instantaneous response to the applied stress, *plastic extrusion* (irrecoverable deformation of the polymer) and time-dependent *creep extrusion*. For completeness, the definition of creep is also provided. *NB Internal pressure sheaths are normally produced by a manufacturing process also termed extrusion, involving polymer melt flow. This is only referred to herein by the term “extruded pipe” or the like.*

3.1.15 Creep: The increase in strain of a viscoelastic material as the result of a constant applied stress. The response of an amorphous polymeric material to the stress commences with an instantaneous elongation, followed by creep (rapid at first, then slowing to a constant rate). The viscoelastic term creep has both elastic and viscous components. When the stress is removed, the creep deformation is not completely reversible; the non-recoverable portion being due to viscous flow.

3.2 ABBREVIATIONS

The following acronyms are used in this document:

- API:** American Petroleum Institute
- ASTM:** American Society for Testing and Materials
- ESIS:** European Structural Integrity Society
- HP:** High Pressure [34.5 MPa (5000 psi) for development work]
- HT:** High Temperature

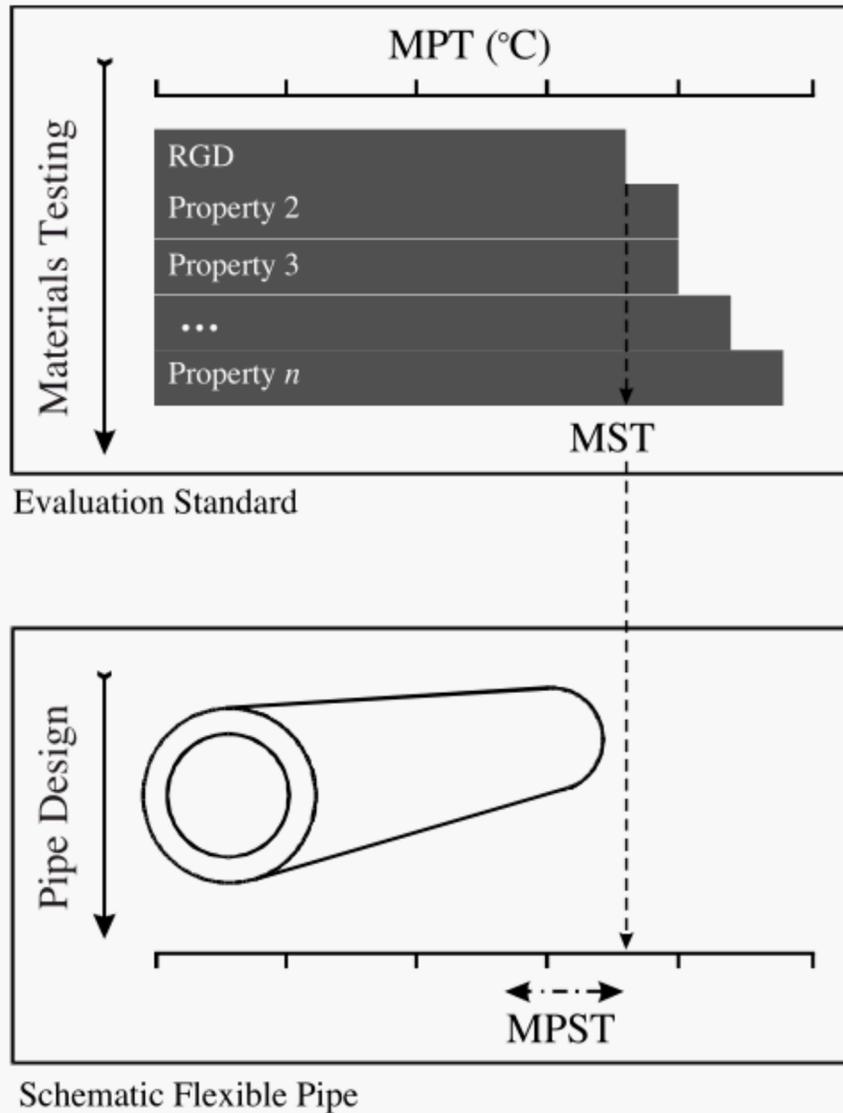


Figure 1—Relationship between MPT, MST, and MPST

4 SERVICE APPLICATION

4.1 OVERVIEW

This section relates potential failure modes of an internal pressure sheath for high temperature service to their underlying failure drivers and critical material properties. These properties form the basis of the material evaluation test programme as defined in Section 6.

4.2 POTENTIAL FAILURE MODES AND RELATED MATERIAL PROPERTIES

4.2.1 Table 1 lists potential failure modes of an internal pressure sheath relevant to high temperature service. Each mode, and associated failure mechanism, has a number of failure drivers which relate to key material properties.

4.2.2 Table 2 presents a list of the service conditions that are critical to the definition of the material tests carried out in the material evaluation test programme. These conditions provide the basis for test parameters such as temperature, pressure, etc. Annexe A presents a more detailed review of these service conditions, including analytical considerations and typical values.

Table 2 Critical Service Conditions

Mechanical Load Limits	Physical/Chemical Conditions
Static Bending	Maximum Internal Temperature
Dynamic Bending	Minimum Internal Temperature
End-fitting Crimping	Temperature Cycling
Internal Pressure	Transported Fluid Composition
Decompression Cycling	

4.2.3 The materials tests which comprise the evaluation programme are defined such that they will measure each of the material properties identified in Table 1; these are summarised in Table 3 and are applicable across the service temperature range.

Table 3 Critical Material Properties

Polymer Material Property
RGD resistance
Stress-strain curve
Extrusion/creep characteristics
Stress relaxation rate
Fracture toughness
Crack growth resistance
Coefficients of permeation, diffusion, solubility at HP
Liquid compatibility
Coefficient of Thermal Expansion*

Note: These properties relate to those properties measured during the material evaluation test programme, ref. Table 4, except *.

4.2.4 The failure modes listed are based on current technology and experience of conventional flexible pipes which has been expanded to encompass high temperature applications.

Table 1—High Temperature Pressure Sheath—Potential Failure Modes¹

Cause of Failure	Potential Failure Mechanisms	Failure Drivers and Critical Levels		Related Material Properties Relevant to this standard	Comments
		Drivers ²	Critical level		
Extrusion	Local stiffening of pipe where armour grooves fill. Local thinning of sheath leading to possible crack/hole in sheath ³ .	Temperature Pressure Sheath wall thickness Sheath mechanical props Local armour geometry Liquid absorption Pipe curvature Chemical attack	High High Low Low Large g/r ⁴ High Large High	Extrusion/creep resistance, Stress-strain relation, Fracture toughness, Crack growth resistance, Liquid compatibility	In dynamic situations, high local stresses may cause crack initiation. Depending on the stress direction, the crack may propagate radially, causing sheath failure, or across the base of an extruded rib, causing it to break off. Sheath materials which resist stiffening (via chemical means) will extrude more than those which do not.
Wear/abrasion	Local thinning of sheath leading to crack/hole in sheath ³ . Local reduction in bending stiffness of pipe.	Fatigue Pipe layer motions Temperature Sheath wall thickness Sheath mechanical props Liquid absorption Chemical attack	Low High slip High Low Low High High	Fracture toughness, Crack growth resistance, Liquid compatibility	Mainly the result of dynamic service; complete abrasion of sheath extremely unlikely, hence not a common concern with the internal pressure sheath layer.
Chemical ageing	Stiffening/cracking, especially of inner sheath wall, leading to sheath leakage ³ . Embrittlement may increase pipe bend stiffness. Possible eventual molecular weight loss (extreme).	Chemical attack Liquid absorption Temperature Pressure (for gases) Sheath wall thickness Sheath morphology Pipe curvature Fatigue	High High High High Low Low crystal. Large Low	Fracture toughness, Crack growth resistance, Liquid compatibility, HP permeation,	Ageing tends to embrittle sheath polymers, making dynamic service likely to promote localised sheath surface fracturing. Pressure is relevant only if the gas chemically attacks the sheath polymer. High molecular weight/crystallinity helps resist attack.
Rapid gas decompression (RGD)	Blistering, splitting, cracking, micro-fracturing; Volume increase likely, unless sheath polymer is plasticized.	Temperature Pressure Decompression rate Sheath wall thickness Gas concentration Gas concentration profile Diffusion coefficient Sheath constraint Sheath mechanical props Local armour geometry Extrusion	High High High High High Saturated Low None Low Large g/r ⁴ High	RGD resistance, Modulus, Fracture toughness, Diffusion coefficient, Gas concentration, Extrusion/creep resistance	The amount of dissolved gas in the sheath is important; for some candidate sheath materials extruded ribs are more susceptible to RGD damage than bulk polymer, hence the desirability of a low extrusion/creep rate; adequate mechanical properties essential. Some types of bulk damage may be acceptable for service.
Dimensional change	Shrinkage, loss of sealing at end-fitting leading to pull-out of sheath and subsequent leakage into the annulus ³ .	Temperature Liquid absorption Sheath constraint Extrusion	Large change High Low High	Thermal expansion coefficient, Dimensional stability Extrusion/creep resistance Liquid compatibility	Could have change for thermal reasons; shrinkage due to leaching (e.g. of plasticizer); swelling due to liquid uptake; processing stresses often exist in extruded pipes; extrusion in service will alter sheath shape.
Permeation (of hostile chemicals)	If permeant is chemically hostile to the sheath polymer, degradation will result. Gas pressure build-up in annulus unless vented. Tensile armour corrosion with some gases.	Temperature Pressure (for gases) Sheath wall thickness Permeation coefficient Local armour geometry Chemical attack Sheath constraint Extrusion	High High Low High Large g/r ⁴ High Low High	Permeation coefficient, Liquid compatibility, Extrusion/creep resistance	Certain chemical species may not degrade the sheath but attack the surrounding metal layers once they have permeated the sheath polymer; in extreme cases, gas build-up could cause cover damage.
Stress relaxation	Loss of sealing force at end fitting leading to pull-out of sheath and subsequent leakage into the annulus ³ .	Stress relaxation rate Extrusion Temperature Dimensional stability Liquid absorption Chemical attack End fitting design	High Low High Low High High High Poor	Stress relaxation, Liquid compatibility, Extrusion/creep resistance, Dimensional stability	Swelling and/or chemical ageing may affect stress relaxation rate. Liquids may not have access to end-fitting region due to the metal flange. Stress relaxation can affect main pipe body during storage/service.

- Notes: 1. These potential failure modes are based on the assumption of existing flexible pipe cross-section configurations.
2. Not every failure driver on its own is sufficient to bring about particular types of sheath failure; in other words, drivers can interact. For instance, high temperature alone is insufficient to allow permeation of a hostile gas through the sheath. However, when high temperature is combined with high gas pressure, a thin sheath wall, a high permeation coefficient, low chemical resistance (i.e., the gas does not interact chemically with the sheath polymer, but may be hostile to the armour layers), small armour gaps (i.e., the sheath does not deform and present, locally, an increased travel path for the gas) and a low level of constraint (meaning the gas can permeate widely into the annulus), then the conditions for permeation of hostile gas through the sheath have been “optimised”.
3. Once the internal pressure sheath of a flexible pipe has ruptured, a flexible pipe is considered to be at the end of its life.
4. g/r is the pressure armour gap-to-fillet radius ratio.

5 EVALUATION METHODOLOGY

5.1 GENERAL

This section describes the methodology employed for the evaluation of polymeric materials for high temperature flexible pipe applications. This methodology involves the evaluation of a *candidate polymer material* against a set of *performance criteria*, which are identified from application-specific service conditions.

5.2 APPROACH

An overview of the evaluation methodology is presented in Figure 2.

5.2.1 Candidate Materials

A *candidate polymer* is initially identified as being suitable for use as an internal pressure sheath on the basis of available property information. The relevant material characteristics have been categorised in API Spec 17J [1] as follows:

- c. Mechanical
- d. Physical/thermal
- e. HP permeation characteristics
- f. Compatibility and ageing

The cost, availability and processability of candidate polymers are also relevant in determining their suitability for flexible pipe service.

5.2.2 Service Application

5.2.2.1 The *service application* for the pipe will be characterised by the internal temperature and pressure, transported fluids and the mechanical loading environment.

5.2.2.2 The service application can consist of:

- a) the service loads specific to a particular pipe application, or
- b) a set of envelopes which may relate to predicted service conditions.

5.2.2.3 The parameters that will be used during the evaluation test programme will be defined from the service conditions that the pipe is expected to experience

during its service life. Where no specific application has been identified, a generic service application for the flexible pipe may be developed based on a global design base case or field experience and projected service conditions.

5.2.3 Performance Criteria

5.2.3.1 In order to assess the performance of a candidate polymer in relation to the required service application, it is necessary to identify a set of performance requirements or *performance criteria* which should define the material properties required of the candidate polymer. Performance criteria are defined in Section 7 of this document.

5.2.4 Evaluation Test Programme

5.2.4.1 Depending on the quality of available validated test data, and the service application under consideration, a decision has to be made as to whether a low cost *preliminary evaluation test programme* is required as a precursor to the *full evaluation test programme*. The full evaluation programme will typically involve more extensive longer-term testing. Further guidance on preliminary and full evaluation test programmes is presented in Section 6.

5.2.4.2 With agreement between relevant parties, the information used for preliminary evaluation of a candidate polymer does not have to originate from test methods described in this standard. However, such data may not be used as a substitute for validated test data in the full evaluation test programme.

5.2.5 Material Evaluation

The final step in the evaluation process is the comparison of test results with the performance criteria. This will indicate whether the candidate material is suitable for the proposed pipe application. Even if the material fails at this stage it is not automatically precluded from future pipe service: design modifications to the pipe cross-section and/or a reduced qualification envelope are situations which may justify its future consideration. The material evaluation process is described in more detail in Section 7.

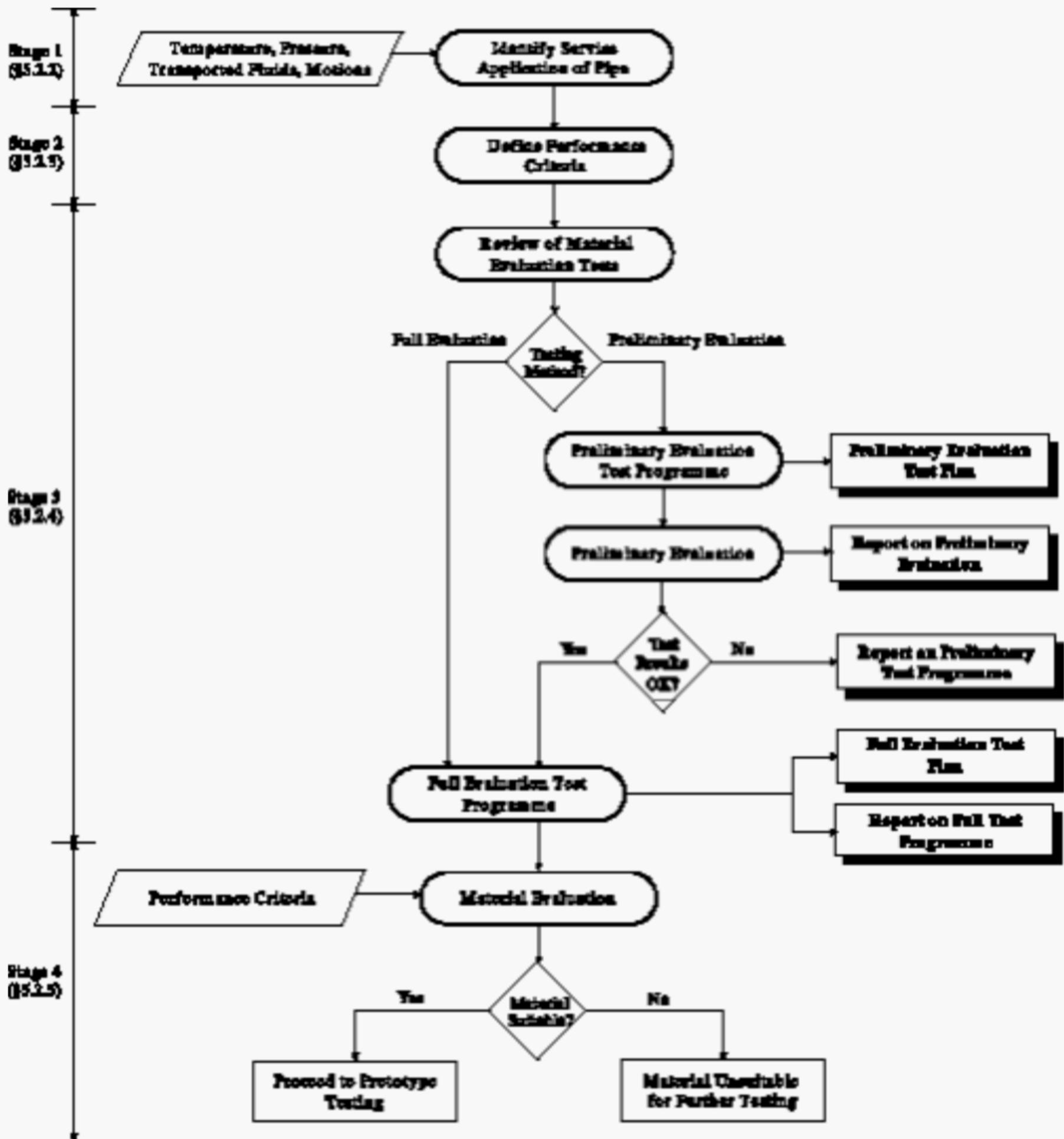


Figure 2—Overall Material Evaluation Methodology.

6 EVALUATION TEST PROGRAMMES

6.1 OVERVIEW

The evaluation of a candidate polymer may be performed on two levels; *preliminary evaluation* and *full evaluation*. Annexe B contains the general test protocols for the evaluation test programme. Mechanical, time-dependent and physico-chemical test methods which are applicable to this standard are fully described in Annexes C and D; standard test procedures are referred to where relevant.

6.2 TEST METHODS

6.2.1 Evaluation of material performance will involve using appropriate tests to quantify material properties and performance-related characteristics which are relevant to the application. This involves the definition of a set of material tests and the identification of the critical properties to be measured. The test methods required for materials evaluation include both published international standard tests and other relevant performance-related tests.

6.2.2 Table 4 lists the test methods which are used in the preliminary and full stages of evaluation. For all tests, the measured properties on which performance is evaluated are also presented. Pertinent information about each test is presented briefly in the sections below.

6.2.3 Testing over a range of temperatures requires that sample temperature be equal to test temperature. Accordingly, the temperature sensor should be located as close as possible to the testpiece failure site; see Annexe B.

6.3 PRELIMINARY EVALUATION

6.3.1 Preliminary evaluation (“screening”) tests are optional and are typically low cost tests of short duration. Properly selected preliminary evaluation tests provide screening to highlight, at an early stage, any serious deficiencies in the performance of a candidate polymer; failure may immediately exclude the material from further consideration.

6.3.2 A preliminary evaluation on its own is not sufficient to qualify a candidate material for service—it must be followed by a successful full evaluation if it is to be qualified. However, screening can save time and costs by removing non-contenders at an early stage.

6.3.3 The approach to preliminary evaluation of candidate polymers is shown schematically in Figure 3. However, if information is already to hand (e.g., from technical literature of polymer supplier) it can be used. Otherwise appropriate tests from Table 4 are employed. The approach involves the following stages:

a) Review and Identification

Review of available literature data and identification of tests required to provide missing property information. Hence test selection will be based on available material data, particular performance requirements for the application (and their respective priority ratings) and cost. The emphasis will be on performing tests which are likely to highlight any gross inadequacies in material performance. The tests in Table 4 are of nominally equal importance but in reality some are overriding (e.g., RGD resistance); hence the value of RGD as a screening test.

b) Definition of Test Parameters

These will depend on the transported fluids, operating temperatures and pressures, etc.

6.4 FULL EVALUATION

6.4.1 Full evaluation of a candidate polymer involves application of the set of materials tests identified as essential for obtaining critical property information. Some of these tests are longer term (e.g., accelerated chemical ageing). The purpose of the full evaluation test programme is to comprehensively assess the suitability of a candidate polymer for the high temperature flexible pipe application. The information necessary to determine whether or not a material has the characteristics necessary to justify manufacture and testing of a prototype flexible pipe will come from the evaluation of these test results. The tests required to assess performance are listed in Table 4 and the overall approach shown in Figure 4.

6.4.2 The emphasis in full evaluation testing is on realism; that is, as far as possible, tests are tailored to the pipe application. One important requirement is that all testpieces originate from extruded pipe; samples processed in other ways (e.g., injection moulded) are unacceptable for full evaluation testing. As in the preliminary testing stage, test parameters need to be defined.

6.5 TENSILE TESTING

6.5.1 Tensile testing is a basic means of obtaining material data such as the full stress-strain curve, Young's modulus, tensile (ultimate) strength at break and percent elongation (strain) at break for design criteria for use in Global and Local analyses, and for assessing ageing-induced changes. Tensile tests should be carried out according to ASTM D638M (or ISO equivalent), modified if necessary as described in Annexe C.

6.5.2 It is imperative that the influence of temperature on mechanical property levels be established by appropriate testing. The temperature range covered should be defined by the extremes likely to be experienced during service; these include the expected maximum, normal operating and shutdown temperature conditions for the pipe. The range of test temperatures used to develop this standard included —10, 4, 23, 120, 150, 175 and 200°C. There is little point in conducting tests on unaged testpieces above RGD MPT (see section 6.12). Tensile tests should be carried out on both unaged and aged testpieces; see section 6.13 for details of ageing conditions.

6.5.3 Reductions in ambient temperature modulus of up to 60% (hydrocarbon oil) for equilibrium swollen candidate polymers have been measured during the associated JIP. Values at high temperatures (above oil boiling point) have to be proportionated to overcome the problem of rapid liquid evaporation during direct high temperature testing of swollen testpieces.

6.6 FRACTURE TOUGHNESS TESTING

The basis of this test (Annexe C) is given in a draft ESIS protocol [4]. It provides a measure of fracture toughness (J) by relating the total work done in separating a compact tension testpiece by a pre-selected amount, to the depth of the crack which results. Other tests at the same temperature on replicate testpieces at different J values should follow. This procedure can be repeated at other temperatures; testing should cover the flexible pipe service temperature range, and include aged samples. Note that, according to the test standard, a slower loading rate (e.g., 0.1 mm/min) must be used if a material exhibits unstable crack growth.

6.7 FATIGUE TESTING

6.7.1 This test extends the fracture toughness concept by providing stress/strain cycles on the same testpiece type to illustrate the fatigue behaviour of thermoplastic materials in reasonably realistic strain modes. The purpose of fatigue measurements is to ascertain whether the crack growth rate of a candidate polymer lies above or below a critical value. With this test, a single testpiece can be used to cover a range of J values.

6.7.2 Video microscopy is used to determine crack growth rates at a series of J (fracture toughness) values: for testpieces machined from extruded pipe, a choice of initial notch orientation exists—axial or circumferential. (Circumferential cracking has been observed in current flexible pipe service). Plots of crack growth rate versus J are then developed for comparisons between different materials. To aid comparisons, crack growth resistance has been defined (empirically) as the value of J at a crack growth rate of 10 nm/cycle.

6.7.3 Testing should cover the flexible pipe service temperature range, and include aged samples. Additional test information is provided in Annexe C. Experience during development of this standard has found fatigue testing to be very expensive due to the nature of the equipment required (servohydraulic machine) and time required to perform the testing.

6.8 STRESS RELAXATION TESTING

6.8.1 This property applies mainly to flexible pipe end fittings, as in the pipe body hoop stresses arising from fluid pressure override stress relaxation—extrusion becomes an issue in such circumstances, as outlined in Section 6.9, although local effects of pipe bending (during storage, service) might have associated stress relaxation on occasions.

6.8.2 Standard tests may be employed. The method used during the development of this standard measures stress relaxation in compression using a dedicated spherical indenter; the latter has a radius which is representative of the crimping arrangement used in the flexible pipe end fittings. This method is described in detail in Annexe C.

6.8.3 A linearity of applied stress with log time is the norm for viscoelastic materials, providing that no chemical (air) ageing occurs during the test.

6.8.4 Testing should be carried out across the full temperature range applicable (see Section 6.5.2 for an example series) and be conducted on both unaged and aged samples (see Section 6.13 for ageing conditions). It has been found in the associated JIP (see Foreword) that stress relaxation rates of the candidate polymers lie within a narrow range, at temperatures up to RGD MPT.

6.9 EXTRUSION/CREEP TESTING

6.9.1 The objective here is to characterise the “flow” of a candidate polymer into the grooves in the pressure armour, which surrounds the internal pressure sheath, at realistic temperatures and pressures. A dedicated high temperature extrusion cell, designed to accommodate polymeric testpieces machined from extruded pipe, and to simulate a variety of armour configurations, was developed to achieve this during the preparation of this standard.

6.9.2 Extrusion is defined, for the purposes of this test, as the change in deformation (strain) of sheath polymer with time due to a constant applied pressure (stress); see section 3.1.13. The response of a viscoelastic material to an applied stress commences with an instantaneous elongation, followed by time-dependent creep (rapid at first, then slowing to a constant rate) and, over the longer term, viscous flow. When the stress is removed, the initial elastic response is immediately

regained as is the deformation due to creep, eventually; any non-recoverable deformation is the result of viscous flow.

6.9.3 In the dedicated extrusion cell, the testpiece inner surface (equivalent to the sheath inner surface) is subjected to a constant elevated pressure at test temperature by means of an inert liquid. The testpiece is supported on its outer surface by a simulated pressure armour insert and the extrusion of material into the armour grooves is monitored as a function of time. Extrusion/creep characteristics depend upon a number of factors: polymer modulus, testpiece thickness, armour geometry, temperature and pressure (stress). The test cell and its operation are described more fully in Annexe C.

6.9.4 For the candidate polymers studied in the development of this standard, strains generated locally within the extruded regions can exceed the global strain limit of 7.5%. Whether this is cause to reject a candidate polymer will depend on other factors, notably, whether HP gas service is envisaged (see section 6.12).

6.9.5 A candidate material is considered to fail the extrusion/creep test if the testpiece (which is realistically formed and of representative thickness, see Annexe B) undergoes a greater than 30% reduction in thickness over its service life, estimated by extrapolation of the creep curve. This condition is clearly dependent upon armour gap geometry and sheath thickness. The wider and deeper the armour gap, the more likely that the pressure sheath will thin unacceptably.

6.10 DIMENSIONAL STABILITY TESTS

6.10.1 Dimensional stability tests are recommended for preliminary evaluation because the consequences at the end fitting attachment points of a deficiency in this characteristic can be serious. Refer to Annexe D for full details of the test procedure to be employed.

6.10.2 The significant test variables are temperature and time. Dimensional stability tests are usually conducted in a vacuum oven. For preliminary evaluation purposes, a test temperature at or close to the MST should be used; if this is not known, a temperature of 150°C should be employed. Changes in testpiece mass, volume and dimensions are recorded at intervals over the test period, which may be several weeks. Under normal circumstances, significant changes would only be expected in those candidate materials which are plasticized.

6.11 HP GAS PERMEATION TESTING

6.11.1 See Annexe E for background theory. The permeability of the pressure sheath to relevant gases at high pressure (34.5 MPa (5000 psi) and preferably service temperatures should be established by appropriate testing. A dedicated HP gas permeation cell, in which polymeric testpieces machined from extruded pipe can be accommodated, was employed during the preparation of

this standard. Methane (CH₄) is the preferred permeant. The testpiece must be suitably sealed to prevent gas entering the polymer by any route other than the inner curved (service) surface. The build-up of permeated gas on the other surface of the sample is monitored and, once steady-state conditions have been established for a reasonable period, the test can be stopped and appropriate computations undertaken to obtain relevant coefficients; permeation (Q), diffusion (D) and solubility (s). These effectively give rates for unit cubes of polymer and hence can be used for comparative purposes.

6.11.2 The variable parameters for the HP gas permeation tests are temperature and pressure. A typical test sequence involves making measurements at three (high) temperatures in order to develop Arrhenius plots for each pressure for the coefficients of diffusion and permeation (Annexe E); extrapolation to service temperature should then be possible if tests can only be performed at lower temperatures (NB if appropriate, care should be taken to ensure that Arrhenius-type linearity applies across the glass transition temperature (T_g) of the polymer). HP permeation tests should be performed on both unaged and aged testpieces (see Section 6.13 for details of ageing conditions). Additional detail is provided in Annexe E.

6.11.3 No pass/fail criteria are specified for this test. If the gas permeation rate is high for a particular candidate material, pipe design considerations will dictate whether the material is allowable as a pressure sheath. The barriers to gas permeation presented by the carcass and pressure armour layers of a typical unbonded pipe should be considered when assessing performance. In the absence of these barriers, or if a proportionation factor describing their influence is known, permeation rate through the pipe sheath can be estimated once Q is known (see Annexe E).

6.12 RGD TESTING

6.12.1 RGD resistance is a critical property for situations in which the flexible pipe transports/contains high pressure gas but one which, in screening tests, it may not be possible to simulate service conditions. Since unconstrained testpieces are invariably more badly damaged by RGD events than constrained samples, it is crucial not to reject materials out of hand based on (apparently) poor preliminary test results. The gas mixture employed should reflect service conditions as far as possible, but the carbon dioxide (CO₂) content is of some importance; carbon dioxide provides a more searching test of RGD resistance than methane as it is invariably the more soluble of the two in polymeric materials. Hence it is acceptable to use natural gas (primarily CH₄ with about 1% CO₂) for preliminary RGD tests.

6.12.2 In full evaluation tests, the level of CO₂ present in the test gas mixture should reflect the intended service environment: if no figure is available, a 90/10 CH₄/CO₂ mixture is recommended. Moreover, the full evaluation test should enable the testpiece to be realistically constrained; that is, its position between the carcass and pressure armour layers in the flexible pipe should be simulated. For the purposes of developing this standard, dedicated RGD pressure vessels were designed and manufactured; these are similar to the extrusion/creep cell described in Annexe C.

6.12.3 RGD test conditions should be applied consistently throughout any test programme. Example conditions include 34.5 MPa (5000 psi) test pressure, 150–200°C temperature range, 1000 psi/minute decompression rate and a cooling/degas period of 2 hours. The soak period should be sufficient to saturate the sample; estimates can be made from methane diffusion coefficients (see section 6.11).

6.12.4 When high gas pressure is applied to the testpiece, deformation (extrusion/creep) into the supporting armour layer occurs. Gas then diffuses through the polymer in the normal way; the gas can escape the material only out of an armour groove—elsewhere the polymer becomes saturated, the worst case scenario. It has been found that the “ribs” of candidate sheath polymer which extrude into armour gaps are significantly less resistant to RGD damage than the bulk polymer; this is the result of changes in local polymer morphology due to extrusion/creep, and other internal stress concentrations. Hence, smooth (no grooves) armour inserts can be employed in order to decouple the influence of extrusion from bulk polymer RGD resistance; in essence these inserts mimic the service situation in which the sheath is wrapped to prevent extrusion occurring.

6.12.5 RGD test strategy requires that unaged samples be employed to determine values of single and multi-cycle RGD MPT; the multi-cycle value cannot exceed the single cycle level. The same approach must then be taken with chemically aged testpieces. The final material RGD MPT will be that resulting from the multi-cycle testing of chemically aged samples; a maximum of 20 cycles should be undertaken and changes in testpiece density and appearance monitored. For the current candidate polymers, this RGD MPT defines MST; *this may not always be the case*. Additional test information is provided in Annexe D.

6.12.6 For the candidate polymers investigated during the development of this standard, RGD MPT is the limit above which bulk decompression damage other than micro-fracturing occurs in both unaged and chemically aged testpieces after multiple RGD test cycles (from a saturated state); in practice, aged samples are likely to define MPT.

6.12.7 For multi-cycle RGD tests undertaken at temperatures up to RGD MPT, density and testpiece

appearance should be recorded after each cycle; if density remains approximately constant for cycles after the initial RGD event, this is a potential indication of good performance. For the materials used to develop this standard, bulk “cloudiness” (due to micro-fracturing) was characteristic of the initial decompression event, resulting in a volume increase; testpiece volume (and by implication internal fracture damage) was found to be stable below RGD MPT during subsequent cycles. Above RGD MPT, blistering/cracking of the material is always unacceptable for service.

6.13 LIQUID COMPATIBILITY AND AGEING

6.13.1 There are several factors to be considered when determining liquid compatibility of a candidate polymer, not the least of which is the variety of liquids which may contact the pressure sheath during flexible pipe service. For preliminary evaluation purposes, short term exposure tests are suitable. These involve exposing specimens to relevant liquids (e.g., hydrocarbon oil, methanol, sea water) at elevated temperature and vapour pressure for a fixed time period (usually sufficient for equilibrium mass uptake to be established, as judged by experience). The minimum test temperature employed in the development of this standard was 150°C. Changes in testpiece mass and volume can be recorded at intervals over the test exposure period and any alteration in testpiece appearance noted; see Annexe D for further details.

6.13.2 During full material evaluation, the diffusion coefficient for the relevant liquid (or representative liquid mixtures) should be determined. This involves establishing a record of liquid mass uptake as a function of square root of time; from the resulting plot, D can be computed (Annexe E). Ideally, D should refer to the service (curved pipe) surface. However, sheath diffusion properties may be anisotropic, a consequence of molecular orientation arising from the extrusion process.

Hence the influence of exposed testpiece edges on D should be established and, where relevant, discounted. This is not a trivial task and involves preventing, by whatever means, fluid from entering the testpiece via exposed edges; see Annexe D.

6.13.3 The full evaluation should also determine the influence of equilibrium levels of liquids on relevant mechanical properties (e.g., modulus). This requires that tensile testpieces be exposed for a period sufficient to attain equilibrium swelling.

6.13.4 Any flexible pipe pressure sheath in service is aged to some degree; time-dependent changes and chemical ageing processes proceed inexorably. A fully aged pipe sheath could be defined as one having ended its useful life. By definition, this pipe will possess properties just above

the limits specified in the failure criteria. Hence, with accelerated chemical ageing at elevated temperatures, aged samples are obtained after times which change the properties to their "fail" levels.

6.13.5 Accelerated chemical ageing involves exposing testpieces to "high" concentrations of aggressive (but realistic) oilfield chemicals at high temperatures and pressures for different periods of time. One option, used during development of this standard, is the multiphase "cocktail" ageing environment (see Table 4). In this, the testpieces are located in a suitable hydrocarbon phase, which is sandwiched between an aqueous phase (sea water)

and a gaseous phase (a CH₄/CO₂/H₂S mixture, where H₂S is hydrogen sulphide). A chemical (e.g., ethylene diamine) is added to simulate the presence of aggressive corrosion inhibitors and the whole system is maintained at high temperature (150–200°C) and pressure (34.5 MPa (5000 psi) for long periods (3–9 months).

6.13.6 Appropriate safety procedures must be implemented when working with HPHT sour systems. Testpieces retrieved from ageing pressure vessels are subjected to all full evaluation test procedures, except liquid compatibility (see Table 4), to determine the effect of ageing on critical material properties.

Table 4—Materials tests and measured properties

TEST	MEASURED PROPERTIES	Preliminary/Full Test	Section
Tensile	a. Stress/strain relationship b. Ultimate strength c. Elongation at break d. Young's modulus e. Secant modulus (3% strain)	Preliminary ¹ and Full	6.5
Fracture toughness	a. Fracture toughness	Full	6.6
Crack growth fatigue	a. Crack growth resistance	Full	6.7
Stress relaxation	a. Stress relaxation rate b. Residual stress level (after 1 hour)	Full	6.8
Extrusion/creep ²	a. Extrusion/creep characteristics b. Influence of T, P and armour geometry	Full	6.9
Dimensional stability	a. Dimensional change b. Mass/volume change	Preliminary	6.10
High pressure gas permeation	a. Permeation coefficient (Q) b. Diffusion coefficient (D) c. Solubility coefficient(s), leading to concentration	Full	6.11
Rapid gas decompression	a. RGD resistance, leading to MPT b. Density as function of cycle number c. Solubility coefficient(s)	Preliminary ³ and Full	6.12
Liquid compatibility ⁴	a. Mass/volume change (at equilibrium)	Preliminary	6.13
	a. Mass changes (to equilibrium) b. Diffusion coefficient c. Tensile properties at equilibrium volume swell	Full	
Chemical ageing ⁵	a. Assessment of physical deterioration b. Tensile (see above) c. Fracture toughness (see above) d. Crack growth fatigue (see above) e. Stress relaxation (see above) f. Extrusion/creep (see above) g. HP gas permeation (see above) h. RGD resistance (see above)	Full	6.13

- Notes:
- For preliminary evaluation, appropriate supplier data are acceptable.
 - The measurement of polymeric sheath deformation into armour gaps under realistic conditions.
 - Preliminary RGD test conditions are less severe than full evaluation, and do not require testpieces to be constrained.
 - In relevant liquids; for example, sea water, methanol, hydrocarbon oil.
 - In a representative environment—this might be a multiphase "cocktail"—at high temperature and pressure; the cocktail constitutes—hydrocarbon oil phase (55/30/15 volume% heptane/cyclohexane/toluene), aqueous phase (sea water, including ethylene diamine at 1% total liquids (oil + water)), gas phase (89/10/1 CH₄/CO₂/H₂S). Testpieces are located in the hydrocarbon phase during exposure.

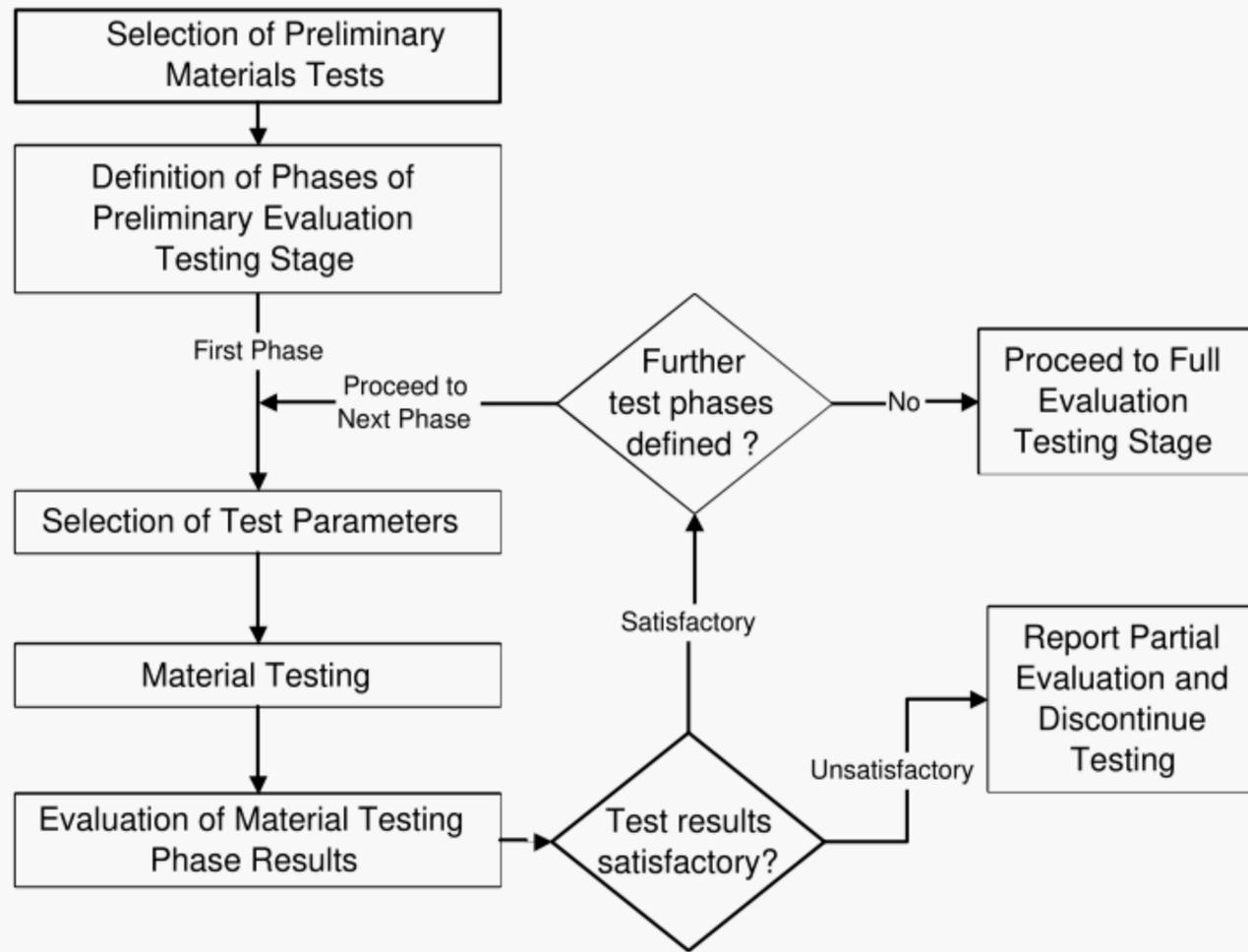


Figure 3—Preliminary Evaluation Stage.

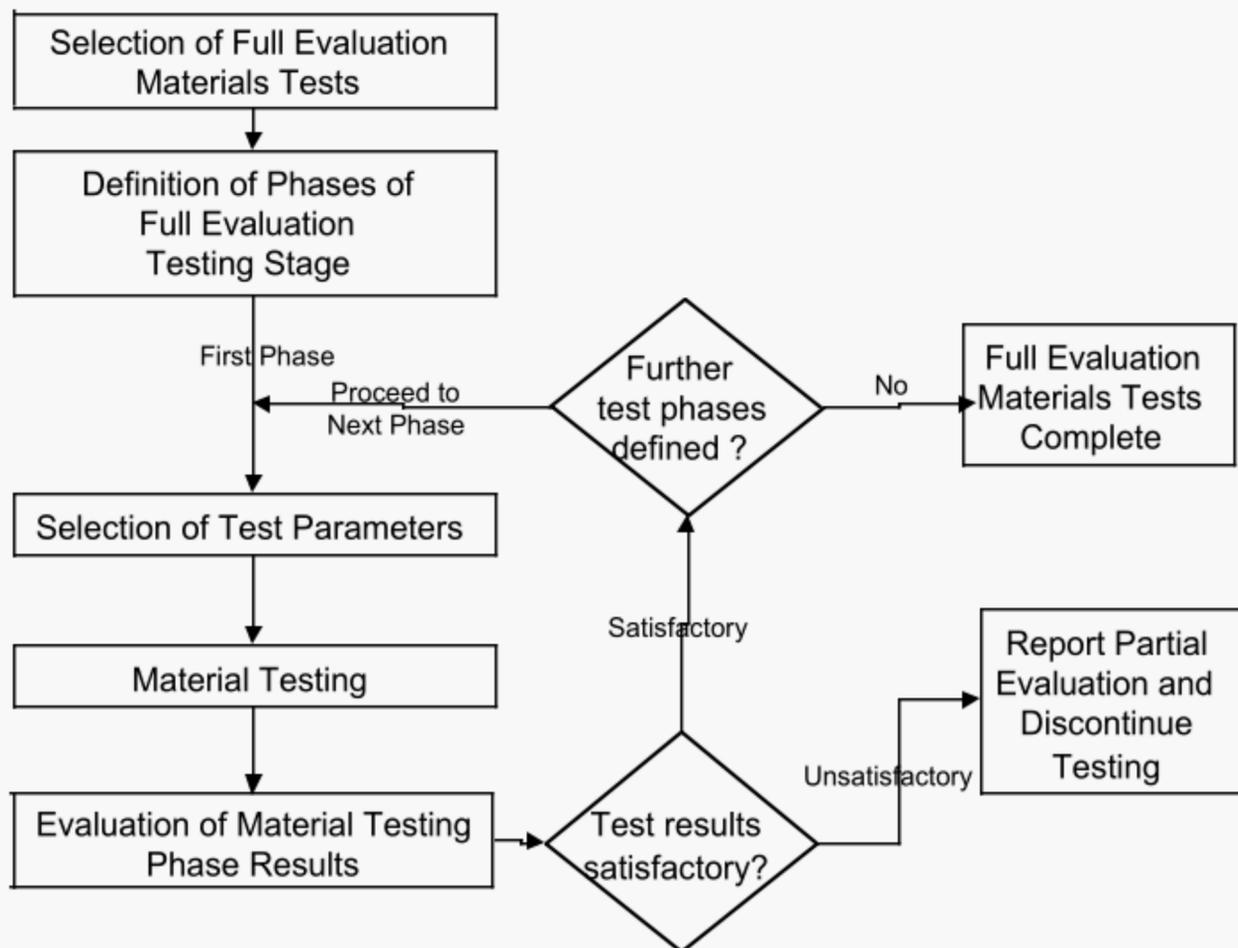


Figure 4—Full Evaluation Stage.

7 MATERIAL EVALUATION

7.1 GENERAL

7.1.1 Results of the full material tests must be evaluated against the performance criteria for the application. This section discusses the following topics:

- i) definition of performance criteria,
- ii) interpretation of material test results (Table 5).

7.1.2 A performance criterion specifies an acceptable range or limit for a measurable material property, within which a candidate polymer must lie if it is to have any chance of functioning successfully in the high temperature flexible pipe application. Exceeding a performance criterion may lead to the onset of pipe failure.

7.1.3 This section outlines the definition and ranking system of performance criteria which should enable an unambiguous interpretation of material test programme results. Performance criteria for an internal pressure sheath material are presented in this section under the following headings:

- g. Mechanical Requirements
- h. HP Permeation & RGD Requirements
- i. Liquid Compatibility and Ageing Requirements
- j. End Fitting Issues

7.2 PERFORMANCE CRITERIA

7.2.1 Mechanical Requirements

Performance criteria relating to the mechanical performance of the candidate polymer are presented as Criteria 1 to 5 in Table 6. A conservative estimate of the reduction in thickness of the internal pressure sheath (Performance Criterion 3), used during the associated JIP, is defined as the maximum deformation of the inner wall of the sheath expressed as a percentage of the original wall thickness, i.e. $(\Delta t/t) \times 100$ as shown in Figure 5. Assuming the candidate material is incompressible, the reduction in thickness can be roughly approximated from the level of polymer extrusion into the armour gap, measured during the Extrusion test, using the following:

$$\% \text{ Reduction in Thickness} = \frac{\text{Extrusion}}{0.024 \times \text{thickness}} = \sqrt{\frac{\text{gap}}{(0.35 \times \text{thickness}) - 0.6}}$$

7.2.2 HP Permeation, RGD resistance, Liquid Compatibility and Chemical Ageing Requirements

Performance criteria relating to fluid compatibility, high pressure permeation, RGD resistance and chemical ageing of the candidate polymer are presented as Criteria 6 to 9 in Table 6.

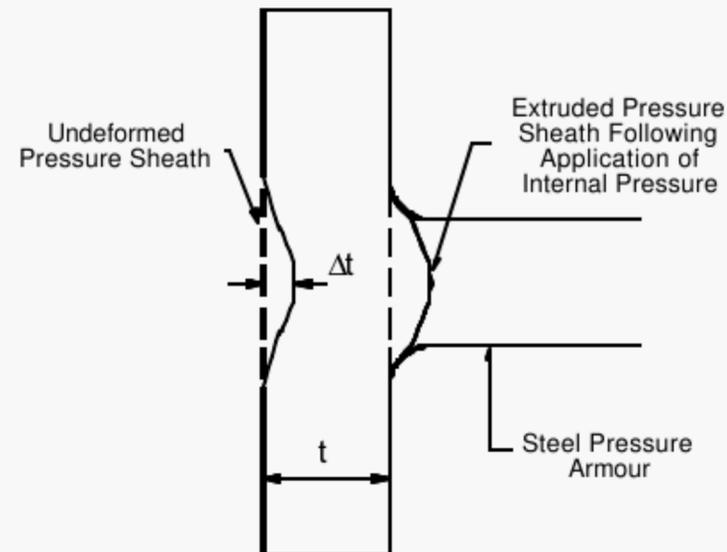


Figure 5—Conservative Estimate of Reduction in Thickness of Internal Pressure Sheath.

7.2.3 End-fitting Issues:

The performance criterion relating to the end fitting requirements of the candidate material is listed as Criterion 10 in Table 6. The crimping of the internal pressure sheath at the end fitting results in the imposition of a local compressive strain. The stress relaxation properties of the candidate material must be such that the required grip of the sealing ring on the pressure sheath is maintained over the design life of the pipe. A value of 20-30% of the sheath layer thickness may be considered representative of the indentation used in current end fitting designs. The strength and strain capacity required by the candidate polymer to achieve a successful end fitting design will depend on:

- i) the strains experienced in service, as determined from the base case design process using the appropriate stiffness properties,
- ii) the local strains experienced where the material has extruded into the pressure armour gaps,

iii) the degree of crimping used at the sealing rings of end fittings.

Reference should be made to API RP 17B for guidance on the determination of criteria for the specification of minimum allowable elongation at break and ultimate strength for a candidate polymer at the end of its service life.

7.3 INTERPRETATION OF RESULTS

7.3.1 Each performance criterion should be ranked in accordance with its relative importance. Table 5 presents recommendations for specifying two classes of performance criterion importance.

Table 5 Performance Criterion Classification

Class	Description of importance
I	Criterion is intrinsic and considered essential. The candidate polymer must fulfil criterion requirements for the duration of the flexible pipe service life.
II	Criterion is essential with current design features, but may be altered by future modification (improvement) of pipe and/or end fitting design.

Table 6 includes representative classifications for each performance criterion.

7.3.2 For the purpose of evaluating the performance of a candidate polymer against the criteria defined in this section, it should be possible to categorise the material under one of the following headings.

- a) *Suitable for Use in HT Flexible Pipe*—Candidate polymers which pass all Class I and Class II performance criteria.
- b) *Potentially Suitable for Future Use*—Candidate polymers which pass all Class I performance criteria and fail any Class II criterion.
- c) *Unsuitable for Use in HT Flexible Pipe*—Candidate polymers which fail any Class I performance criterion.

7.3.3 Annexe F presents a comparison of the properties of the polymeric materials currently used as the pressure sheath in unbonded flexible pipes; plasticized PVDF (Solef 1015/0078, Solvay), PA 11 (Rilsan BESNO P4-TL, Elf Atochem) and plasticized PVDF (Kynar 50HDCP900, Elf Atochem).

Table 6—Performance Criteria For High Temperature Candidate Polymers

No.	Performance Criterion ¹	Associated Test ²	Class	Pass/Fail Requirement ³⁻⁵	Comment
1	Young's Modulus	Tensile	I	It should be possible to achieve a successful global configuration design such that the allowable strain of the material is not exceeded. The stiffness of the candidate material, in both the unaged and aged condition, over the full range of service temperatures should be taken into account. ⁶	Finite element analyses of a global riser configuration for a base case severe environment have shown that materials with Young's Moduli between 10–4000 MPa, and sheath thicknesses of 2–20 mm, can achieve configuration designs with maximum global tensile strains of 2–3%.
2	Tensile Strength (Elongation At Break)	Tensile	I	The minimum level for ultimate strength ⁷ and strain at service temperature ⁵ at the end of flexible pipe service life should be: <ul style="list-style-type: none"> Elongation at break greater than 1.5 times the maximum global and local strain levels.⁸ Aged strain should never fall below service strain level.⁸ 	Accelerated laboratory ageing tests (see criterion 9) may be required to estimate the deterioration in candidate polymer mechanical properties.
3	Extrusion/creep Resistance	Extrusion/creep	II	The reduction in the thickness ⁹ of the pressure sheath due to extrusion of the polymer into gaps in the pressure armour should not exceed 30% by the end of the service life of the pipe; the effect of instantaneous elastic (or elastic/plastic) extrusion and longer term creep extrusion should be taken into account. ⁸ Extrusion of sheath polymer should be limited such that it does not result in rapid gas decompression failure or unacceptable increase in the global stiffness of the pipe. ⁶	The measurement of sheath deformation into appropriate armour gap configurations under realistic conditions is essential. Relevant variables include pressure, temperature, modulus, thickness, armour geometry (gap width, depth, radius of edge curvature).
4	Fracture Toughness	Fracture Toughness	I	Extent of crack growth at $J = 5 \text{ kJ/m}^2$ should be 0.3 mm or less and at $J = 10 \text{ kJ/m}^2$ should be 0.6 mm or less. ¹⁰	Basic property to quantify resistance to failure by cracking. May indicate fatigue behaviour.
5	Crack Growth Resistance	Crack Growth Fatigue	I	The crack resistance, that is, the fracture energy, J , of the unaged polymer at 10 nm/cycle at 100°C and should be greater than 3 kJ/m ² . ¹⁰	This limit might not apply at service temperature extremes, or after chemical ageing. Crack growth resistance might be described well enough by fracture toughness.
6	High Pressure Permeation	HP Gas Permeation	II	The venting arrangement at the end-fitting should be capable of removing gas at a rate greater than that at which the gas enters the annulus by permeating the pressure sheath. ⁸	At service temperatures and pressures, a low permeation coefficient would be desirable (but a balance might be necessary with RGD requirements).
7	Blistering Resistance	Rapid Gas Decompression	I	The polymer should be resistant to rapid gas decompression events such that, after 20 decompression cycles, no blistering and/or slitting should be observed at a magnification of $\times 20$. ^{6,8} A constant testpiece density after the initial RGD cycle is one indicator of material stability. ¹¹	The candidate polymer must be constrained realistically during testing. A high value of diffusion coefficient would be desirable, as would a low concentration of gas dissolved in the candidate polymer at equilibrium saturation.
8	Liquid Resistance	Liquid Compatibility	II	Candidate polymer volume swell should not exceed 5% (for constrained testpieces) over a period of 30 days at MST in relevant fluids; for example, seawater, methanol, (model) oil. ⁶ The polymer should not exhibit evidence of Environmental Stress Cracking or other degradation in representative fluids. ⁸	Any change in mechanical properties at equilibrium volume swell should comply with performance criteria 1 and 2. There are indications from the industry that this limit could be slightly greater.
9	Ageing Resistance	Chemical Ageing	I	After chemical and physico-chemical ageing at service temperature over its service life or after accelerated laboratory ageing tests the candidate polymer material should comply with all other criteria in this table. ⁶	Reduction in tensile properties should not result in non-compliance with performance criteria 1 and 2. Specimen distortion may preclude testing, for some tests. Swollen samples; see Section 6.5.3
10	Stress Relaxation	Stress Relaxation	II	An indicative guide for plasticised PVDF is that the stress relaxation rate in the polymer should not exceed 13% per decade at 23°C. ^{10,12}	This property should be the subject of detailed investigations as part of an end-fitting qualification programme.

- Notes: 1. The factors which influence a performance criterion are primarily the loads which contribute to the failure of the pipe as a result of non-compliance with that requirement. The criteria originate from the full evaluation testing programme (Table 4); it is implicit that candidate materials have passed the preliminary evaluation stage.
2. Candidate polymer testpieces should originate from actual sheath or, if unavailable, a model extruded pipe.
3. The candidate polymer should be capable of meeting each performance criterion at all service temperatures unless otherwise stated; see note 5.
4. The candidate polymer should be capable of meeting each performance criterion in the aged and unaged condition unless otherwise stated.
5. By mutual consent, details of the requirements for performance criteria might be changed to suit a particular service situation, providing technical justification is agreed between supplier and customer.
6. Pass/Fail requirement has been derived from analysis and/or testing of the four candidate polymers considered in this JIP
7. A criterion for minimum tensile strength at the end of flexible pipe service life seen from this JIP is 5 MPa
8. Pass/Fail requirement derived from comparison with conventional flexible pipe materials
9. The reduction in thickness of the internal pressure sheath is defined in the script under Section 7.2.1.
10. Pass/Fail requirement based on plasticized PVDF (Coflon).
11. In situations where bulk micro-fracturing is evident it is advisable to undertake further testing to establish if the material still satisfies fundamental performance criteria. For the candidate polymers tested, essentially no density change occurs after the initial decompression cycle; hence, it is thought that tensile properties would follow a similar trend.
12. See Section 6.8.4.

8. BIBLIOGRAPHY

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- [3] Campion, R, "Model Test Oils based on Solubility Parameters for Artificial Ageing of Polymers," Presented at Polymer Testing '96 (RAPRA, Shrewsbury, Shropshire, UK), September 1996.
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ANNEXE A – MECHANICAL CONSIDERATIONS

A.1 MECHANICAL SERVICE CONDITIONS

Mechanical service loads on flexible pipe, whether static and/or dynamic, can arise from the sources listed in Table A.1.

Table A.1—Summary of Pipe Load Sources.

Load Source	Application Type
Handling/storage/installation	Static/Dynamic
Burial/Trenching	Static/Dynamic
Self weight/buoyancy	Static/Dynamic
Internal fluid pressure	Static/Dynamic
Hydrodynamic forces (current) on pipe	Static/Dynamic
Hydrodynamic forces (wave zone) on pipe	Dynamic
Movements of supports	Dynamic
Vessel or buoy/float motions	Dynamic

Mechanical loads are transmitted to the flexible pipe cross section as tensile loads, compressive loads, bending loads, internal/external pressure (radial and axial) and torsional loads, any of which may fluctuate. Mechanical loads can be broadly divided into two categories as follows; *global* loads refers to loads due to global motions of the pipe body, and *local* loads which relate to the loads on the pipe cross section at a detailed level.

Global Loads

Failure of the internal pressure sheath of a flexible pipe due to global mechanical loading is caused primarily by bending loads; these can be either static or dynamic. Flexible risers and jumpers which connect wellhead platforms and floating support vessels will experience dynamic loading. The requirements for static applications such as seabed flowlines will be less demanding. Refer to API RP 17B [2] for further details on static/dynamic and riser/flowline/jumper applications.

The contiguous polymeric layers of an unbonded flexible pipe will experience strains due to pipe bending. The maximum allowable strain in the pressure sheath layer should not exceed the strain at which *an increase in strain occurs without an associated increase in stress*. This condition should apply to all material states (e.g., aged) across the full service temperature range.

The maximum level of axial strain in the internal pressure sheath of a pipe body is geometrically related to the bending radius as follows:

$$R = \frac{r_{outer}}{\epsilon_{max}}$$

where R is the global bending radius of the flexible pipe, r_{outer} is the outer radius of the internal pressure sheath and ϵ_{max} is the maximum strain in the internal pressure sheath. The minimum bend radius (MBR) is a design requirement which is generally verified using global finite element analysis techniques [1]. More detailed analysis is used to determine the MBR at a bend stiffener or other connection point.

Local Loads

Mechanical loads also affect the stresses and strains of the internal pressure sheath at a local scale. The critical loading parameters are internal fluid pressure and bending loads. High local strains can occur where the polymer layer is forced against the gaps in the surrounding armour layer by the internal pipe pressure. In such circumstances all polymeric materials initially experience rapid elastic/plastic extrusion into the gap, followed by slower time-dependent creep extrusion. In other words, a steady-state extrusion situation is reached quickly, with the rate of creep dependent upon material properties, pressure, temperature and gap geometry (see Section 6.9). The majority of this deformation is the result of large plastic strains over small localised regions of the material near the corners of the pressure armour wire cross-section. The properties of the internal pressure sheath polymer must be sufficient to withstand these deformations and maintain the integrity of the pressure sheath throughout the service life of the pipe.

Bending loads contribute to the local stress and strain conditions through their affect on the width of the gap between neighbouring armour wires. Dynamic bending causes cyclic strain variations in the gap size, thereby influencing movement of the polymer into the gap. This also gives rise to the possibility of crack initiation at the gap; the notch sensitivity of the sheath material becomes relevant here. Should this become a critical issue, it may be possible to design internal sheath with a sacrificial layer to prevent crack initiation, by preventing extrusion.

Analytical methods to assess the local deformations of the sheath into the pressure armour gaps are based on finite element analyses, calibrated with the results from realistic tests.

Extrusion of the sheath polymer into the gaps in the pressure armour wires could, in extreme cases, fill the available groove space entirely; this might alter global pipe stiffness. Also, wear between the internal pressure sheath and the adjacent metallic layers (normally the carcass and the pressure armour) could be caused by the relative movement of these layers in bending. Another possible source of wear is sand from the production stream. However, wear and abrasion of the pressure sheath are not expected to be significant factors in service.

A.2 THERMAL LOADS

Polymers to be used as internal pressure sheath materials must be capable of performance at maximum and minimum design temperatures. The upper service temperature limit for new sheath polymers is projected to be between 150 and 200°C. In service, temperature cycling can occur during shutdown or other periodic procedures such as hot oil flushing. It has been estimated that for continuous operation of an FPSO development in a water depth of 380 metres over a 25-year lifetime, an estimated total of 1095 temperature cycles can be expected; see Table A.2 for a breakdown.

Table A.2—Typical Estimated Temperature Cycles and Variations over 25 Years Continuous Service.

Temperature Variation	No. of Cycles	Operating hours at Lower Temperature
T _{HIGH} to -20°C	1	2
T _{HIGH} to -5°C	117	1004
T _{HIGH} to 20°C	300	2400
T _{HIGH} to 35°C	677	1354

The insulating properties of the internal pressure sheath can be an important design factor if it is necessary to maintain the transported fluid above a certain temperature to ensure adequate flow properties. An internal pressure sheath with low thermal conductivity may eliminate the need for additional insulating layers in the pipe cross section and reduce the temperature to which the other layers are exposed. On the other hand, a material with low thermal conductivity will increase pipe insulation requirements and expose surrounding layers to higher temperatures.

A high coefficient of thermal expansion will mean that considerable stresses can be induced with in polymeric layer.

Temperature cycling of the polymer may lead to significant changes in the rate of stress relaxation—this is a particular concern for the design of the end fitting of the pipe.

A.3 PERMEATION

The major influences on the permeation characteristics of the polymeric pressure sheath are:

- characteristics of the transported fluid
- characteristics of the polymer
- gas pressure (minimum/maximum and rate of change); vapour pressure for liquid
- temperature (minimum/maximum and duration)
- pressure sheath thickness
- masking effect of pressure armour.

Permeation of hostile fluids through the internal pressure sheath can give result in corrosion of the metallic armour wires in the pipe annulus, leading to the failure of the pipe. The rate of permeation will depend on the properties of the fluid, conditions within the pipe and the nature of the sheath polymer (see Annexe E). The range of transported fluids to which a polymer sheath would typically be exposed are summarised in Annexe G.

The design objective when considering the permeability of a material is to select a polymer with a low permeability to the pipe bore gases in order to minimise the quantities of aggressive gases (e.g., H₂S and CO₂) which permeate to the pipe annulus, providing the associated permeation and diffusion requirements for RGD resistance are also met.

If outer sheath failure is an issue then total permeated gas quantity is relevant; both the carcass and pressure armour layers act as barriers to permeation. Methane is the major constituent of oilfield gases.

The first part of the permeation process involves dissolution of the permeant in the surface layers of the contacting polymer. The solubility coefficient (s), when multiplied by applied gas pressure, gives a concentration term (see Annexe E). Hence, for a gas, the dissolving process is dependent on its partial pressure; for liquids the process is essentially pressure-independent. The fluid then penetrates further into the polymer over time through a diffusion process. The rate of diffusion is largely independent of pressure, but may be reduced at high pressures, due to compaction. These hydrostatic effects are seen in the *permeation* of gases through polymer sheaths. The final step in the permeation process occurs when diffusing molecules reach the other side of the sample and evaporate: evaporation stage where the fluid evaporates from the side of the polymer away from the contacting fluid: a liquid uptake test is equivalent to permeation without the evaporation stage. Permeation is a physico-chemical process, not a pure chemical one (unless there is some chemical involvement if the permeant reacts with

the polymer), as is the related phenomenon of absorption which can lead to mechanical weakening of the polymer.

Rapid Gas Decompression

Rapid gas decompression is a term used to describe sudden depressurisation of the pipe bore after service has been well established. Gas which is present within the internal pressure sheath expands and seeks to exit the materials by diffusion. When this is not possible, fracturing/blistering can result, threatening sheath integrity. The susceptibility of a semi-crystalline polymer to this mode of failure is related to its stiffness (modulus), fracture toughness, morphology, constraint, diffusion coefficient and gas solubility. For instance, all other factors being equal, decompression damage is more likely to be observed in a material with a low diffusion coefficient than a high value of D , with the reverse applying for concentration.

A.4 LIQUID COMPATIBILITY AND AGEING

The degree to which a semi-crystalline polymer (such as PE or PVDF) will absorb quantities of a low viscosity liquid is influenced by the solubility of the liquid in the polymer. The Hildebrand solubility parameter (δ) is an important material parameter in determining which polymers are likely to resist physico-chemical swelling and/or chemical attack in specified liquids. This parameter is an inherent property of both liquid and polymer. A liquid will not enter a polymer to an appreciable degree if their solubility parameters are significantly different. However, if their solubility parameters are similar, a stronger drive for liquid ingress exists and swelling is likely. This can be combated if the polymer structure is sufficiently organised (i.e., highly crystalline in a stable way). If the structural features in the polymer are not sufficiently robust or widespread, the liquid will enter the amorphous region of a (chemically) compatible polymer and swell it. This is a physico-chemical ageing effect which invariably weakens the material mechanically. An engineer considers this system to be incompatible.

If the liquid is chemically hostile, degradative chemical reactions are likely to occur. In extreme cases this can result in the total disintegration of the polymer, but effects such as crosslink formation, eventually leading to embrittlement, are more likely in the early stages. Among the other factors which can affect the swelling of thermoplastics in a liquid, are its glass transition temperature T_g , any filler content and the viscosity of the liquid.

For a mixture of n liquids, the following equation has been proposed to calculate an overall solubility parameter [3]. This is an extension of an established equation in which $n = 2$.

$$\delta = \phi_1\delta_1 + \phi_2\delta_2 + \phi_3\delta_3 + \dots + \phi_n\delta_n$$

here ϕ_i are ϕ_i the volume fractions of the liquids present. One consequence of this relationship is that a liquid mixture will swell a polymer to a much greater degree than any of its individual components if δ of the mixture lies closer to δ of the polymer. This approach can be used to specify a model hydrocarbon oil which is representative of production fluid from the point of view of both solubility parameter and the aliphatic:naphthenic:aromatic component ratio [3].

If possible the Hildebrand solubility parameter δ of a candidate polymer should not be close to those of the expected range of transported liquids to avoid the risk of swelling (and weakening) of the internal pressure sheath.

The kinetics of liquid absorption again involve diffusion aspects, see Annexe E.

The major loading issues relating to the compatibility and ageing of a polymeric material in service are similar to those involved with permeation. These are:

- characteristics of the transported fluid
- pressure (minimum/maximum and rate of change)
- temperature (minimum/maximum and duration).

If the transported fluid chemically attacks the sheath, embrittlement leading to pipe sheath failure can result. The range of transported fluids to which a polymer sheath would typically be exposed are summarised in Annexe G.

An important factor in determining the compatibility of a polymer with a given fluid is the influence of the chemical environment on the materials ageing characteristics. Ageing can occur by chemical, physico-chemical and physical means. The latter two are, in theory, reversible but this is not usually applicable in flexible pipe service.

Physical Ageing

One example of a physical ageing process is that of a thermoplastic which, when rapidly cooled to below its glass transition temperature, attempts to reach thermodynamic equilibrium while shrinks and being constrained by its glassy state—tiny modulus changes might be noted. Other examples are creep and stress relaxation.

Chemical Ageing And Embrittlement

Chemical ageing can occur rapidly at the surface when a hostile species contacts a polymer. Due to the severity of typical flexible pipe applications, the ageing characteristics of the sheath material is a critical performance issue. The ageing process is generally characterised by detrimental changes in mechanical (e.g., a reduction in stiffness or ductility) and/or physical properties (e.g., a change in molecular weight). If the

sheath polymer is amenable to molecular weight characterisation, valuable insight into degradation mechanisms and their effect of mechanical properties may be gained. If physico-chemical swelling occurs, the chemical attack can then continue within the bulk.

Ageing test criteria should be based on required physical and/or mechanical properties at the end of pipe service life. Important issues in the determination of ageing characteristics are the effect of plasticizers and deplasticization, the influence of environmental and load conditions, water cut, pH level, fluid absorption and dimensional changes.

Ageing can have a profound effect on the failure properties of a material. The ageing criteria given in draft API RP 17B [2] for recommended properties of existing internal pressure sheaths at the end of service life, at a test temperature of 20°C, are as follows:

- HDPE: Tensile Strength: Min. 15 MPa.
Elongation at Yield: Min. 7.7%
- XLPE: Tensile Strength: Min. 15 MPa.
Elongation at Yield: Min. 7.7%
- PA-11: Tensile Strength: Min. 20 MPa.
Elongation at Yield: Min. 30%
Elongation at Break: Min. 50%
- PVDF: Tensile Strength: Min. 25 MPa.
Elongation at Yield: Min. 7%

A.5 END-FITTING ISSUES

Flexible pipes are terminated with end fittings, which transfer the loads from the individual layers to the attached connector. The adequate design of the termination and sealing of the internal pressure sheath layer is critical to the integrity of the pipe. Some flexible riser designs have more than one pressure sheath layer; the role of the outer layer(s) being to provide sacrificial protection against micro-crack initiation during dynamic service, as the material extrudes into available carcass and pressure armour grooves.

The internal pressure sheath is terminated by crimping with a serrated sealing ring, whose function is to ensure fluid and pressure containment. The mechanical properties, particularly the stress relaxation characteristics, of the internal pressure sheath material will determine the level of compressive strain necessary to achieve a secure and lasting grip at the end-fitting over the service life of the pipe.

The magnitude of this crimping force is dictated by the specified indentation, which is normally defined as a certain percentage of sheath thickness (e.g., 20%). This represents an imposed compressive strain, which may not exceed the yield strain of the material, where the yield

strain is considered relevant. Local stress relaxation of the pressure sheath material may mean that part of the deformation associated with the original strain becomes permanent set. Sealing stresses are thus reduced. For flexible pipe attachments of this kind, the level of compressive strain necessary to securely grip the internal pressure sheath must be determined.

Table A.3 summarises the potential defects for internal pressure sheaths at the end fitting and the associated material properties and service conditions.

Table A.3—Loading Conditions and Material Properties associated with End Fitting Failure Modes

Defect	Relevant Material Properties	Associated Service Conditions
Sheath Pull-out	Creep Resistance † Stress Relaxation † Coefficient of Thermal Expansion †	Temperature Number and Range of Temperature Cycles Internal Pressure
Loss of Seal in End Fitting	Creep Resistance † Stress Relaxation † Coefficient of Thermal Expansion †	Temperature Number and Range of Temperature Cycles. Internal Pressure

† At maximum and minimum temperatures, following exposure to fluid environment and thermal conditions for service life duration.

For some dynamic applications, turret mounted risers are surrounded by still air. During shutdown, these end fittings will cool at a slower rate than if immersed in seawater. This will have an effect on the temperature cycles experienced by the internal pressure sheath material in this area.

A heavy carcass may transmit vertical loads to the internal pressure sheath, which contributes to the pull-out load. When the pipe bore is pressurised, the weight of the layers and the carcass is supported by friction with the pressure armour layer. However, in the case of riser depressurisation, this friction will be insignificant, and the carcass and pressure sheath weight will be supported only at the sealing ring.

The contributing factors to internal pressure sheath pull-out include axial forces and temperature cycling. The clamping force of the seal ring on the riser with multiple sheath layers was reduced by stress relaxation in the layer polymer (plasticized PVDF), followed by slipping of adjacent layers. The primary factors which contribute to this failure mode are [1]:

- a. Loss of plasticizer from internal pressure sheath due to chemical exposure causing dimensional change (reduction) of the sheath layers.

- b. Compression of the sheath polymer in the region of the sealing ring may contribute to loss of plasticizer (by squeezing).
- c. Because the thermal coefficient of expansion for the sheath material is higher than that of the tensile layers (which govern the expansion/contraction of the pipe), the sheath transmits tension to the end fitting during thermal cycling and retracts from the sealing ring at the low point of cycling.
- d. Creep and stress relaxation in the sheath material reduces the clamping force at the end fitting, rapidly at first but then less so.
- e. The multiple sheath layer design contributes to the pull-out since only the outer layers are gripped by the sealing ring and carcass, leaving the central layer(s) to slide.
- f. An understanding of how sheath material properties vary over service life is critical.
- g. The number and range of temperature cycles affects the susceptibility of risers to this failure mode.
- h. Differential cool-down rates of the end fitting and the main pipe body during thermal cycling are also important factors.

Note: These will be strongly dependent on whether the end fitting is immersed in seawater or exposed to air.

An optimum end-fitting design would minimise the possibility of failure through the use of sheath materials which can be used in a single layer construction, or can be extruded without processing aids (e.g., plasticizer). Resistance to the various contributing factors associated with this failure mode will be significant performance requirements for candidate high temperature polymers.

A.6 MANUFACTURING REQUIREMENTS

The manufacturing process currently used to produce a flexible pipe internal pressure sheaths requires that the polymer layer (or layers) be extruded onto the carcass. For practical reasons, it would be a beneficial if candidate materials be processable using existing extrusion equipment.

Manufacture of internal pressure sheaths will involve extrusion of pipe layers that may be exceed a kilometre in length. If a modified or special extrusion process is required for a particular candidate material, it must be capable of production at this scale in order to offer a realistic possibility for flexible pipe manufacture. Internal pressure sheaths range in thickness from 2–20 mm.

API RP 17B provides guidelines on manufacturing tolerances for the extrusion of internal pressure sheaths [2]. The minimum allowable thickness should be based on the requirements of API Spec 17J [1]. Thickness selection shall take into consideration the extrusion/creep characteristics of the candidate and strains due to pipe bending as well as axial, torsional, and radial deformation.

The rate at which a candidate material can be extruded to form the internal pressure sheath layer has obvious manufacturing cost implications. The flow properties of a material under processing conditions affect its suitability for extrusion. The flow properties of a polymer are often quoted in terms of a melt flow index at a certain temperature. The melt flow index for PVDF at 230°C is quoted in Annexe F.

The material properties and extrusion conditions must be matched to ensure that any additive (e.g., plasticizer) is not leached out during processing.

The heating requirements for the extruder will depend on the processing temperature of the polymer. For high temperature candidate materials, processing temperatures will be significantly higher than for existing internal pressure sheath polymers.

A.7 TABULAR SUMMARY

Table A.4 provides a summary of the service loads likely to be experienced by high temperature flexible pipe.

Table A.4—Summary Of Typical Loads

Load/condition	Units	Benchmark Value	HT Pipe Range
Service Life	Years	25	5 – 30
Internal Pressure	bar (psi)	350 (5000)	0 – 700 (0 – 10000)
Static Bending	% Strain	3.5	0 – 7.7
Dynamic Bending	% Strain	± 1.0	± 2.0
Maximum Temperature	°C	150	120 – 200
Minimum Temperature	°C	0	-35 – 0
No. of Temperature Cycles (entire service life)	-	1000	250 – 2000
Chemical Environment	-	Refer to Annexe G	
Local Strains due to Extrusion of Sheath into Metallic Layer Gaps	% Strain	250	100 – 300
Global Strains due to Bending of Pipe	% Strain		0 – 7
Local Strains due to End Fitting Sealing Ring Clamp	% Strain	20 – 30	20 – 30
Vertical Pull-out Stresses due to Carcass and Internal Pressure Sheath Weight at End Fitting Sealing Ring	MPa	4.6	3 – 8

Pressure units: 350 bar ≈ 34.5 MPa ≈ 5000 psi

ANNEXE B—TEST PROTOCOLS

B.1 SAMPLES

When a test is conducted using standard procedures, the test specimens should be prepared according to that standard. Non-standard specimen sizes are acceptable for evaluation only if agreed between client and supplier. For test procedures described in this standard, the specimen sizes given in Annexes F and G should be used.

For preliminary evaluation samples may be cut or machined from extruded material, or may be moulded, provided that this fact is fully recorded. For full evaluation testing, it is essential that all test specimens originate from extruded pipe processed in as realistic as way as possible for the application.

For the test procedures specified in this document, the tolerance on test specimen dimensions should be $\pm 1\%$, and should be measured using a micrometer with accuracy to within 0.02 mm.

B.2 TEST EQUIPMENT

Equipment requirements for each materials test are described in Annexes C and D. All test equipment and instrumentation should be calibrated on a regular basis (a minimum of once per year). Current certification/calibration certificates for all test equipment should be readily accessible.

B.3 TEST PROCEDURES

In cases where pressure or temperature is required to be constant, stabilisation may be considered to have occurred under the under the following circumstances:

Pressure: Pressure variation within one hour is within $\pm 1\%$ of the test pressure.

Temperature: Sample temperature variation within one hour is within $\pm 2.5^{\circ}\text{C}$ of the test temperature. This temperature should apply to the potential failure region; hence the recording temperature should be appropriately located.

B.4 DOCUMENTATION

The test report should contain the following details:

- a) sample main dimensions, type, source and test equipment used.
- b) raw test data and material parameters calculated from the raw test data.
- c) descriptions of material failure modes including severity ratings where applicable.
- d) recommendation on suitability of material for service in specified conditions.

B.5 AVAILABILITY OF RESULTS

Results and records should be maintained so that they may be accepted in lieu of replicate testing for similar applications.

B.6 VALIDITY OF RESULTS

If the grade of polymer is changed, or if extrusion process variables are modified, tests must be performed to demonstrate that critical material properties still fulfil the material acceptance criteria. Evaluation tests on a material for a particular application may be considered to be valid for the same polymer in a less severe application. It must be shown that none of the performance requirements for the new application are more stringent than those for the original application. Where only some of the performance requirements have been increased, a reduced set of full evaluation tests, focusing on the increased requirements, will be acceptable.

ANNEXE C—MECHANICAL TESTS

The mechanical property tests required by this standard are described in the following sub-sections. All testpieces must originate from extruded pipe.

C.1 TENSILE

Tensile testing is a basic means of obtaining material data such as the full stress-strain curve, Young's modulus, tensile (ultimate) strength at break and percent elongation (strain) at break for design criteria for use in Global and Local analyses. Tensile tests are to be carried out according to ASTM D638M (or equivalent ISO standard) over the appropriate temperature range.

C.1.1 Apparatus

As described in ASTM D638M, the test apparatus is to consist of a testing machine and an extension indicator. The machine should allow testing at a constant strain rate. In addition, a temperature controlled autoclave or oven which fits onto the test machine is required for this application. Temperatures must be accurate within $\pm 2^{\circ}\text{C}$.

The test specimen and grips (shaped to accommodate testpiece curvature) will be contained within the oven chamber and connected to the test machine via steel connecting rods which enter the oven through purpose-built holes at the top and bottom. These rods may contain ceramic inserts to thermally isolate the oven from the rest of the test machine.

ASTM D638M describes three specimen types. Type M-II test specimens are recommended but any size complying with existing standards or agreed between suppliers and client may be employed. ASTM D638M recommends that at least five specimens be tested for isotropic materials for each set of test conditions; for pragmatic reasons, only three replicates were employed in the development of this standard. For extruded pipes with wall thicknesses relevant to the application (6–8 mm), testpieces machined axially lengthwise only are appropriate.

C.1.2 Description of Test Method

The recommended rate of crosshead displacement is based on the strain rates expected in flexible pipe dynamic service. Any testing speed within the range 0.5-50 mm/min can be used but, for expediency, the 50 mm/min rate is preferred.

C.1.3 Measurements

Polymeric materials exhibit highly non-linear overall stress-strain behaviour; initial linear regions apply across the low tensile strain range (0-7.5%) which is relevant to flexible pipe pressure sheath service. Hence the stress-strain plots and Young's and secant moduli should be measured for the full temperature range applicable and mean values quoted.

C.2 FRACTURE TOUGHNESS

Long established methods of quantifying the suitability of a polymer for service in high duty engineering applications involve as a basis two assessments—tensile related properties (see C.1) and the resistance to crack growth in a single event. For elastomers the latter is tear strength; fracture toughness is the equivalent for thermoplastics.

The basis of this test is given in a draft ESIS protocol [4]. It provides a measure of fracture toughness by relating the total work done, in displacing the compact tension testpiece by a pre-selected amount, to the depth of the crack which results.

C.2.1 Apparatus

A testing machine with displacement control and a load indicator is required. In addition, a temperature controlled autoclave or oven which fits onto the test machine is required when testing above ambient temperatures. Temperatures must be accurate to within $\pm 2^{\circ}\text{C}$.

For realism, samples should be machined from extruded pipe. Hence, although the testpiece is a curved variation (Figure C.1) of the compact tension specimen described in the draft ESIS document, the other requirements of the standard are followed, projected dimensions being used. This testpiece design functions broadly under plane strain conditions, which are seen as applying generally to flexible pipes in service. Two holes are provided for clamping purposes; dimension W is normally from the line joining the centre of these holes to the unnotched edge of the testpiece. The specimen will be nominally 6–8 mm thick (B).

Any bending force contributions arising from straightening of curved specimens should be considered if thought necessary.

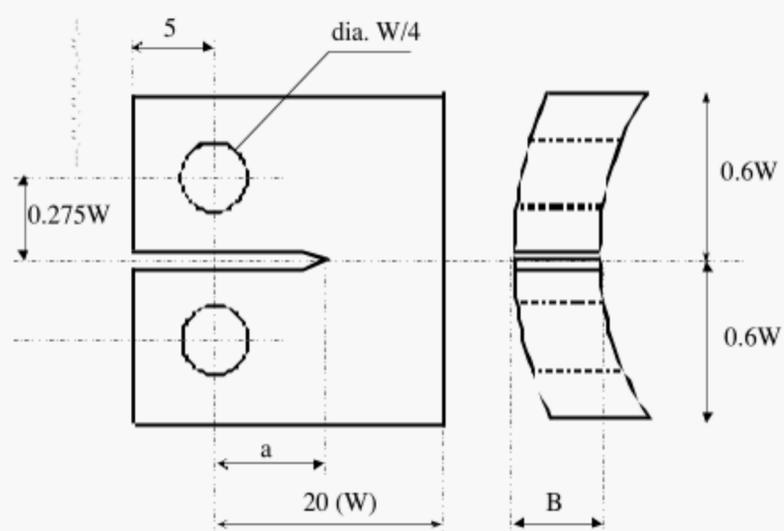


Figure C.1—Projected and side views of compact tension testpiece machined from pipe, with notch in axial direction; all dimensions in mm; B = thickness.

The specimen should be notched using either a broach or a single point fly cutter so that the tip radius $\rho \leq 20 \mu\text{m}$. To make the crack tip as sharp as possible, specimens are pre-cracked by tapping a razor blade into the root of the machined notch. The pre-crack length a_0 should satisfy the requirement:

$$0.55 \leq \left(\frac{a_0}{W} \right) \leq 0.65$$

Initial pre-crack lengths (a_0) for all the specimens should not vary by more than $0.05W$.

C.2.2 Description of Test Method

The testpiece is separated by a defined amount on a universal test machine and the amount of crack growth subsequently measured after breaking open the sample to reveal the crack surface. A useful form in which to present data is as a plot of fracture toughness (resistance) versus crack growth, constructed using the results from several separate tests in which different levels of deflection are employed. Fracture toughness, J , is calculated from an appropriate equation for the compact tension testpiece: The fracture toughness J is defined as:

$$J = \frac{\eta U}{B_N (W - a_0)}$$

where term $\eta = 2 + 0.522 (1 - a_0/W)$ for compact tension, a_0 is the initial, pre-crack, length, W is the testpiece width, U is the stored energy under the loading curve and B_N is the testpiece thickness.

C.2.3 Measurements

Three testpieces are measured by applying a different, appropriate, strain to each so that plots of crack growth

versus J can be established. Fracture toughness is then quoted for either zero crack growth or a small crack growth (e.g., 0.2 mm).

C.3 FATIGUE CRACK GROWTH

This test extends the fracture toughness concept by providing stress/strain cycles on the same testpiece type to illustrate the fatigue behaviour of thermoplastic materials in reasonably realistic strain modes. Any comments on force contributions in section C.2.1 apply.

C.3.1 Apparatus

The testpiece is as described in Section C.2.1. The test machine must be capable of cyclically (or dynamically) loading the testpiece; servohydraulic machines are the most suitable. In addition to this, a temperature controlled autoclave or oven which fits onto the test machine is required. Temperatures must be accurate to within $\pm 2^\circ\text{C}$. Heat-resistant lamps for illuminating the sample whilst it is being cycled at elevated temperature are located within the oven, which has a glass viewing port. Video microscopy is used to track crack growth.

C.3.2 Description of Test Method

The testpiece is subjected to small amplitude cyclic loading, representative of fatigue loading conditions. The load and frequency conditions used in this test will be a function of the stress-strain characteristics of the material. When significant crack growth has occurred, a video print-out is obtained from which accurate measurements are made. Testing is then continued at a higher amplitude, which increases U and thus J .

C.3.3 Measurements

Video microscopy is used to determine crack growth rates as accurately as possible. J is calculated as before. A particular J value is employed until a discernible crack growth rate is measured; J is then altered and the crack growth rate again determined, and so on. Plots of crack growth rate versus J are then developed for comparisons between different materials. To aid comparisons, the "crack resistance" value of J (kJ/m^2) at a crack growth rate of 10 nm/cycle is used.

C.4 STRESS RELAXATION

This test mainly applies to the end-fitting region of a flexible pipe, where the pressure sheath is secured and sealed by a crimping ring; stress retention is critical here. The test measures the rate of stress relaxation of a polymer in compression or tension.

C.4.1 Apparatus

A rounded indenter, mounted on a universal test machine, is used to impose a fixed level of compressive strain on the material under test (see Fig. C.2). The radius of the indenter should be chosen to be representative of indentation pattern of end fitting sealing rings on internal pressure sheaths. A tip radius of 3 mm is currently considered representative. A temperature controlled oven which fits onto the test machine is also required. Temperatures must be accurate to within $\pm 2^{\circ}\text{C}$.

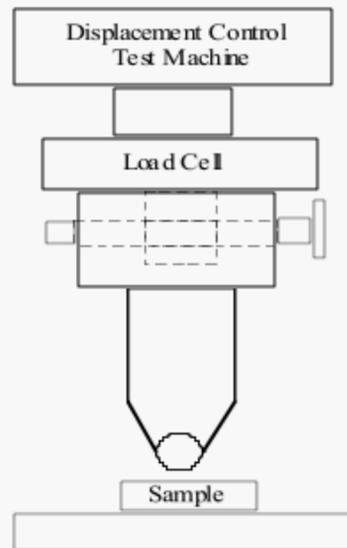


Figure C.2—Stress Relaxation Indenter

A projected 25 mm square testpiece, cut from extruded pipe with a realistic wall thickness, will be sufficient to eliminate edge effects during the test.

Data acquisition will be required for force response versus time for the duration of the test.

C.4.2 Description of Test Method

An indentation of 20% (typically) of specimen thickness is imposed using the rounded indenter. The compressive indentation is typically imposed over a period of approximately 15 seconds. Data from the first five minutes of the test, when the response is usually non-linear, is ignored. The end of this period is taken as the origin for recording a meaningful relaxation response.

C.4.3 Measurements

The relaxation of the applied compressive stress is plotted as a function of time. Since this relationship after an initial settling down period of a few minutes is frequently linear/logarithmic a test period of one hour is normal; if the observed stress relaxation behaviour follows some other function of time, a longer test may be required to establish the trend.

C.5 EXTRUSION/CREEP

Under the pressure of contained fluids during service the pressure sheath is forced against the surrounding metallic pressure armour. Current pressure armour designs comprises flat wires which when interlocked remain

flexible enough to accommodate pipe bending motions; hence the gap between adjacent armour windings is variable. As the pressure sheath is a viscoelastic material it is capable of deforming under the influence of heat and pressure and “flowing” (extruding/creeping) into armour gaps.

CONSTANT STRESS

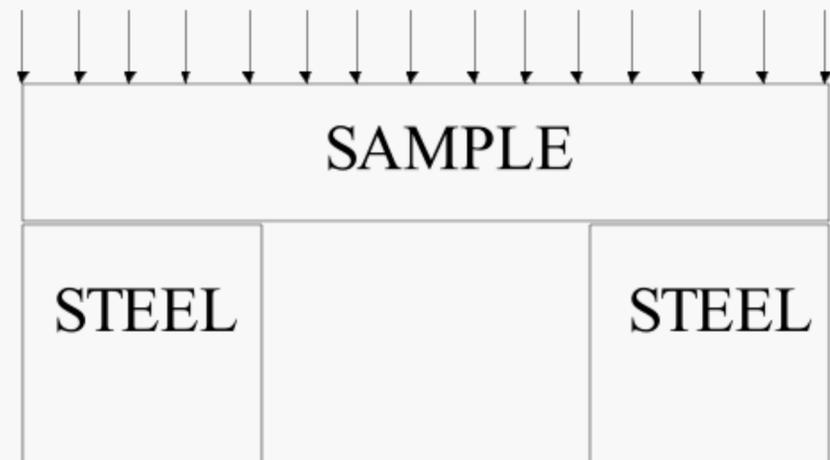


Figure C.3—Approach to Extrusion/creep Testing

C.5.1 Apparatus

For this Standard, a dedicated HP extrusion/creep cell was designed and manufactured to aid in methodology development. The principle of the cell is shown in Figure C.3: a constant pressure is applied to the polymer testpiece, which is located above a realistic simulation of an armour groove. The simulation insert is located, suitably sealed, in a pressure vessel which has an opening in one end to enable extrusion measurements to be made. As the polymer extrudes/creeps into the gap at a particular combination of temperature and pressure, its progress is tracked by means of a displacement transducer. Equipment enabling a constant pressure to be maintained on the inner testpiece surface as it deforms is required; the surface is uniformly pressurized using an inert liquid. Temperature control is achieved using a band heater with appropriate feedback controls; internal cell pressure and temperature should be monitored continuously.

C.5.2 Description of Test Method

The testpiece is allowed to equilibrate at test temperature in the dedicated cell before being pressurized. Once test pressure has been reached (within 2 minutes), the response of the polymer is monitored using the displacement probe. A test duration of 24–30 hours is usually sufficient to establish material extrusion/creep performance. When the pressure is removed at the end of the test, the elastic response of the polymer can be recorded if the displacement transducer is spring-loaded.

C.5.3 Measurements

Test output is a plot of polymer displacement (into the armour groove) plotted as a function of log time. The materials studied in the development of this standard all displayed classic responses to the applied pressure: an

instantaneous large elastic response (elastic/plastic extrusion) was followed by time-dependent behaviour (creep extrusion) which overall was less significant in terms of groove fill.

ANNEXE D—PHYSICAL/CHEMICAL TESTS

D.1 DIMENSIONAL STABILITY

This test assesses the dimensional changes that a material experiences at elevated temperatures over long periods of time. Dimensional changes could be the result of stress relief or loss of volatile constituents. The latter include trapped moisture, low molecular weight species and additives such as plasticizers (used to improve the processability of some materials).

D.1.1 Apparatus

A vacuum oven, calipers and weighing apparatus are required. Oven temperature must be controllable to within $\pm 2^\circ\text{C}$. The suggested specimen size is 50 x 50 (minimum) x 6 mm. Alternatively, specimen dimensions may be selected based on application parameters.

D.1.2 Description of Test Method

Specimen mass and dimensions are recorded. Testpieces are then placed in the vacuum oven and heated to test temperature for the required period. Following testing, the samples are cooled to ambient temperature and re-weighed and re-measured and changes in mass, volume and dimensions recorded.

D.1.3 Measurements

Changes in mass, volume and dimensions for particular combinations of test temperature and exposure time are recorded.

D.2 COEFFICIENT OF THERMAL EXPANSION

The Coefficient of Thermal Expansion of the polymer material should be determined using the standard test method ASTM E831. Refer to this standard for all testing information.

D.3 HP GAS PERMEATION

D.3.1 Apparatus

A test assembly with the following features is required:

- A chamber which can be pressurised to high pressure by a gas supply. This will be separated from a low pressure chamber by a suitably sealed polymer testpiece. Facilities for monitoring the pressures in the high and low pressure chambers over time are required.
- Heating (insulated band heater) facilities and temperature monitoring and control of the permeation cell to within $\pm 2^\circ\text{C}$.

Test times are governed by diffusion characteristics of candidate polymers. Testpieces used during development are discs of 34 mm diameter, machined from extruded pipe.

D.3.2 Description of Test Method

The test specimen is installed in the HP permeation cell. The cell is then heated to test temperature and allowed to equilibrate before applying test pressure to the sample. As a minimum, the following test conditions shall apply:

- High Pressure (P_1) Pipe design pressure. (A value of 34.5 MPa (5000 psi) may be used if pipe design pressure is not known.)
- Temperature (T) Service, or three lower temperatures for Arrhenius extrapolation to service (see Annex C).

The increase of low pressure with time may be converted to the rate of gas permeation (cm^3/hr at STP), assuming the gas to be ideal, by :

$$\frac{dq}{dt} = \left(\frac{\partial p_2}{\partial t} \right) \left(\frac{273 V_{LP}}{T_{LP}} \right)$$

where V_{LP} and T_{LP} are the volume and temperature (ambient) of the low pressure chamber and P_2 is in atmospheres. The permeation coefficient Q can be obtained from the gradient, at steady state conditions, of the permeated gas pressure versus time plot using:

$$\left(\frac{q}{t} \right) = QA \left(\frac{P_1 - P_2}{h} \right)$$

where h is the specimen thickness and P_1 refers to the pressure in the high pressure chamber. The initial transient stage of the test can be used to provide an estimate of the diffusion coefficient D :

$$D = \frac{h^2}{6\tau}$$

where τ is the time where the extrapolated steady state line intersects the time axis. The solubility coefficient s is then determined from:

$$Q = Ds$$

The concentration c of a single gas absorbed into the polymer surface may be calculated from:

$$c = sP_1$$

D.3.3 Measurements

The measured criteria are the Permeation Coefficient (Q), the Diffusion Coefficient (D), the Solubility Coefficient (s) and the concentration (c) applying at the high pressure face. For a material contacting an essentially impermeable layer (e.g., the pressure armour), c will be eventually nearly achieved throughout.

In the development work for this standard the high sealing forces necessary to prevent leakage of the HP permeation cell resulted in the deformation of candidate polymer testpieces during cell assembly. The distortion had the effect of increasing the pathlength for permeating gas. Accordingly, a “normalization” procedure, in which the deformation of individual testpieces was taken into account when calculating Q and D, was developed and applied.

D.4 RAPID GAS DECOMPRESSION

RGD resistance is a critical property for all service fluids with a HP gas phase, and possibly the most important in that other properties become irrelevant in service terms if RGD events cause unacceptable damage to the sheath polymer. The extent of damage increases with increasing temperature: hence candidate polymer MPT is the first factor governing all other tests when establishing an MST.

D.4.1 Apparatus

For the development of this standard dedicated high pressure RGD cells, instrumented to monitor and control temperature and pressure, were manufactured. The design incorporates means to constrain the testpiece between simulations of carcass and pressure armour. Test gas should be representative of service (e.g., 90/10 CH₄/CO₂ mixture) with test pressure of 34.5 MPa (5000 psi); the decompression rate is 6.9 MPa (1000 psi) per minute. Soak times, which can be estimated from methane diffusion coefficients (see section G.3), should be long enough to saturate the testpiece. Soak periods of 18–24 hours were typical for the candidate polymers investigated.

D.4.2 Description of Test Method

The broad strategy requires that unaged samples be employed to determine values of single and multi-cycle RGD MPT; the multi-cycle value cannot exceed the single cycle level. The same approach must then be taken with chemically aged testpieces. The final material RGD MPT will be that resulting from the multi-cycle testing of chemically aged samples. For the candidate polymers used to develop this standard, RGD MPT defines MST; this may not always be the case.

Samples should be decompressed from the saturated state, the worst-case situation. Inter-cycle cooling conditions

should be consistent. One convenient way to track performance is to monitor testpiece density (and appearance) during multi-cycle tests. After testing, samples should be sectioned and inspected for damage.

These RGD procedures extend the requirements of API Spec 17J.

For multi-cycle RGD tests undertaken at temperatures up to RGD MPT, density and testpiece appearance should be recorded after each cycle. The effect of decompression damage on mechanical properties should be established by appropriate testing (e.g., tensile).

D.5 LIQUID COMPATIBILITY

Pressure sheath polymers should be compatible with contacting fluids relevant to service. However, all polymers interact with liquid to some degree, usually weakening them. Such effects must be quantified. Accordingly, several tests are recommended for the purpose of determining the compatibility of the sheath polymer with representative transported liquids. Liquid compatibility also includes chemical ageing. Accelerated ageing methods are required if estimates of service life (based on particular properties; e.g., modulus) are to be made.

D.5.1 Apparatus

For all liquid compatibility tests, suitable exposure vessels should be employed; if test temperature exceeds liquid boiling point, or if a high pressure exposure is called for, a pressure vessel is required. The temperature in the cell must be controllable to within $\pm 2^{\circ}\text{C}$. A weighing balance is essential.

For the development of this standard, the following liquid mixtures were employed to assess compatibility: sea water, methanol, hydrocarbon oil, acid mixture. The hydrocarbon oil may be optimised for a particular oilfield using a method described elsewhere [3]. A generic oil might have the following composition: 50% heptane, 30% cyclohexane, 13% toluene, 7% 1-propanol, by volume.

D.5.2 Description of Test Method

Testpieces are exposed to a liquid at test temperature and pressure, for a period sufficient to allow equilibrium uptake to be established. If tensile properties are required, the testpieces should be tensile bars. If the uptake curve is to be obtained, regular measurements of mass uptake need to be performed. Morphological anisotropy in extruded pipe is a possibility that should be accommodated when measuring diffusion coefficients; an edge blocking technique has been developed and shown to work well for

oil uptake; this is not necessary for methanol and sea water.

The options for accelerated chemical ageing are numerous.

D.5.3 Measurement

To determine the effect of equilibrium mass uptake of a liquid on material mechanical properties, tensile testpieces are required; if tensile testing at elevated temperature, allowance should be made for possible loss of absorbed liquid by evaporation before/during the test. Changes in

Whatever the conditions employed, appropriate safety procedures should be put in place; for example, if H₂S is specified as part of the ageing environment, equipment for the safe handling of this gas is essential.

testpiece mass, volume and appearance should be recorded. Mass uptake plots are typically established using small testpieces (e.g., 34 mm diameter discs machined from extruded pipe); a diffusion coefficient can be calculated from such plots.

ANNEXE E—TRANSPORTED FLUIDS PERMEATION MECHANISM

When a liquid comes into contact with a polymer surface, it dissolves rapidly up to a concentration c_0 . For hydrocarbons, the movement of the liquid into the material bulk is then controlled by diffusion, which is governed by Fick's law. Diffusion-related phenomena such as swelling increase with the square of polymer layer thickness. This is usually only discernible for liquids, although high pressure gases can swell polymers after an initial hydrostatically-induced compaction. Both the dissolution or diffusion processes are essentially independent of pressure.

To determine the diffusion coefficient D (discussed below) for a liquid, absorption tests involving weighings are used. Then

$$\frac{m_t}{m_\infty} = \left(\frac{2}{h'} \right) \left(\frac{Dt}{\pi} \right)^{1/2}$$

where m_t represents mass uptake at time t , m_∞ represents mass uptake at equilibrium swelling and $2h'$ is sample thickness. Eventually D becomes concentration-dependent as, after a while, liquid that has already entered the polymer can contribute to the rate at which fresh liquid diffuses. One simple and convenient method of obtaining a representative value of diffusion coefficient is to measure D_{av} , an "average" D at the point of 50% mass uptake, so that:

$$0.5 = \left(\frac{2}{h'} \right) \left(\frac{D_{av} t}{\pi} \right)^{1/2}$$

(Note that thickness is expressed as $2h'$ in this equation, but will be denoted as h in later discussions.

$$h = 2h'$$

D_{av} is most conveniently derived from plots of m versus t , reading off t_{av} at 50% uptake).

A miscible liquid mixture is treated herein as if it were a single species—a representative D is obtained as above. Gas also enters a polymer by a process of solution (in the polymer surface) followed by diffusion. While the amount of gas dissolved depends on its pressure, the rate of diffusion is essentially pressure-independent.

Gas permeation is dependent upon pressure, temperature, and the nature of the gas and polymer involved. Gas permeation involves two steps. The gas is dissolved in the surface of the polymer according to Henry's Law:

$$c = sP$$

where c is concentration, P is the applied pressure and the proportionality term s is the solubility coefficient. In the event of a gas mixture, the concentration of any constituent gas in the surface of the polymer may be determined by using its partial pressure in the above expression.

Diffusion of gas through polymeric materials is governed by Fick's law, an integrated version of which is given for a sheet membrane sample as:

$$\left(\frac{1}{A} \right) \left(\frac{q}{A} \right) = D(c_1 - c_2) \left(\frac{1}{h} \right)$$

where q is gas volume diffused in time t , D is the diffusion coefficient, A is the surface area available for permeation and h is thickness. The initial and final concentrations are denoted c_1 and c_2 respectively.

The permeation coefficient is simply the product of the coefficients of diffusion and solubility:

$$Q = Ds$$

The equation for gas permeation through a sheet membrane is therefore as follows:

$$\left(\frac{q}{t} \right) = QA(P_1 - P_2) \left(\frac{1}{h} \right)$$

For a hollow cylinder (applying to this application), the equation is:

$$\left(\frac{q}{t} \right) = \frac{2\pi LQ(P_1 - P_2)}{\ln(r_2/r_1)}$$

where L is the length of the pipe, r_1 is the inner radius and r_2 is the outer radius, and the other terms are as before.

Annulus environment predictions in terms of the presence of CH_4 , H_2S , CO_2 and H_2O should take gas permeation (likely to be reduced by the contacting armour layer) into consideration, as well as venting flow rate, reduction of annulus volume due to water condensation and consumption of CO_2 in corrosion reactions.

All of the coefficients Q , D and s (collectively termed C) are described by Arrhenius-type relationships with temperature as follows:

$$C = C_0 \exp(-E_a/RT)$$

where C_0 , E_a and R are all constants. Hence plots of $\log C$ versus reciprocal temperature are linear and suitable for extrapolations.

ANNEXE F—TYPICAL MATERIAL PROPERTIES OF CURRENTLY USED INTERNAL PRESSURE SHEATH MATERIALS OR CONVENTIONAL FLEXIBLE PIPES

F.1 INTRODUCTION

This Annex summarizes the properties of the polymer materials currently used for internal pressure sheaths in flexible pipe applications. The material property data

shown in Table F.1 have been obtained from supplier data sheets and technical publications, as listed in the table footnotes.

Table F.1—Current Material Properties.

Property	Test / Standard	Plasticized PVDF Solvay (Solef 1015/0078)	PA-11 Elf Atochem (Rilsan BESN P40 TL)	Plasticized PVDF Elf Atochem (KYNAR 50HDCP900)
Tensile Modulus	ASTM D 638 ⁷	1025 MPa (23°C) ¹ (Rate = 1 mm/min) 785 MPa (23°C) ⁴ 190 MPa (110°C) ⁴ (Rate not given)	335 MPa (23°C) ² 173 MPa (60°C) ² 166 MPa (80°C) ² 160 MPa (100°C) ² 49 MPa (120°C) ³	919 MPa (20°C) 510 MPa (60°C) 339 MPa (90°C) 162 MPa (120°C)
Flexural Modulus (Tangent)	ASTM D 790 ⁷		320 MPa (23°C) ²	
Yield Strength	ASTM D 638 ⁷	9.9 MPa (120°C) ¹ 18.5 MPa (80°C) ¹ 38.5 MPa (23°C) ¹ 36 MPa (23°C) ⁴ 11 MPa (110°C) ⁴	No Yield (23°C) ² 27.5 MPa (20°C) ³ 9.8 MPa (120°C) ³	31.4 MPa (20°C) 20.8 MPa (60°C) 13.9 MPa (90°C) 8.2 MPa (120°C)
Elongation at Yield	ASTM D 638 ⁷	34.4% (120°C) ¹ 24.8% (80°C) ¹ 15.8% (23°C) ¹ 22% (23°C) ⁴ 28% (110°C) ⁴	No Yield (23°C) ²	20% (20°C) 24% (60°C) 29% (90°C) 38% (120°C)
Ultimate Strength	ASTM D 638 ⁷	18.4 MPa (120°C) ¹ 20.3 MPa (80°C) ¹ 32.3 MPa (23°C) ¹ 23 MPa (23°C) ⁴ 25 MPa (110°C) ⁴	65 MPa (23°C) ²	28.8 MPa (20°C) 30.5 MPa (60°C) 29.5 MPa (90°C) 28.4 MPa (120°C)
Elongation at Break	ASTM D 638 ⁷	352.8% (120°C) ¹ 204.8% (80°C) ¹ 79.1% (23°C) ¹ 63% (23°C) ⁴ 420% (110°C) ⁴	360% (23°C) ²	360% (20°C) 373% (60°C) 407% (90°C) 460% (120°C)
Impact Strength (Izod) (Notched)	ASTM D 256 ⁷	775 J/m (23°C) ¹ (Partial Break)	No Break (23°C) ²	
Impact Strength (Izod) (Unnotched)	ASTM D 256 ⁷		No Break (23°C) ²	
Hardness (Rockwell "R")	ASTM D 785 ⁷		78 ²	
Fracture Toughness (J _{0.2})	ESIS Protocol ⁵	9.0 kJ/m ² (23°C) ⁴ 2.7 kJ/m ² (70°C) ⁴		
Heat Deflection Temperature	ASTM D 648 ⁷ [ISO 75]	133°C (0.45 MPa) ¹ 55°C (1.82 MPa) ¹	[130°C (0.46 MPa)] ⁶ [45°C (1.85 MPa)] ⁶	
Melting Point (°C)	-	170°C	178–184 °C ⁶	
Melting Flow Index	ASTM D 1238 ⁷	1.2 (230 °C, 5 kg) ¹		

- Notes
1. Plasticised PVDF Homopolymer (Grade 1015/0078), Solvay Technical Data Sheets.
 2. Rilsan Polyamide Resins, Elf Atochem.
 3. Makino, Y., Ishii, K., Yamaguchi, T., Goto, Y., "Design of Flexible Pipe for High Temperature Fluid," OTC 6727, Offshore Technology Conference, Houston, May 1991. [N.B Unsure what grade of PA11 is used]
 4. Stevenson, A., Campion, R., "Thermoplastic Performance Properties for Flexible Pipes and Umbilicals," Proceedings of the Second European Conference on Flexible Pipes, Umbilicals and Marine Cables—Structural Mechanics and Testing, MARINFLEX 95, November 1995, London, U.K.
 5. A Testing Protocol for Conducting J-Crack Growth Resistance Curve Tests on Plastics, May 1994, ESIS.
 6. Rilsan B Technical Guide, Elf Atochem.
 7. Or ISO equivalent.

ANNEXE G—TRANSPORTED FLUID DATA

G.1 INTRODUCTION

This Annexe presents data on transported fluids for high temperature flexible pipes that has been obtained from participants and other sources.

Table G.1 presents the information on the service

conditions and the constituents of the production fluids which will normally be transported in the flexible pipe. Table G.2. presents information on additives to the production stream and special operating procedures which are relevant to the internal pressure sheath service conditions.

Table G.1 Service Fluid Properties

Quantity	Units	Max.	Min.	Comments
Fluid Pressure	(bar)	700	207	Projected design pressure requirements.
Partial Pressure H ₂ S	(bar)	1.5	traces	Operating pressures, based on operator experience.
Partial Pressure CO ₂	(bar)	64	1	Operating pressures, based on operator experience.
Partial Pressure CH ₄	(bar)	400	50	Operating pressures, based on operator experience.
Water Cut	(% volume)	90	0	Based on operator experience.
pH of Aqueous Phase	-	6.5	3.5	Based on operator experience.
Titrated Acid Number	(mg.KOH/g)	4.0		Estimated value.
Asphaltenes Content	(% weight)	0.06		Estimated value.
Salt (NaCl) Content	(g/l)	200–300	25–30	Based on operator experience.
Sulphur	(% weight)	0.42		Estimated value.
Chlorides Content	(g/l)	57	22	Based on operator experience.
Gas - Oil Ratio	(m ³ /m ³)	1754	227	Based on operator experience.

Pressure units: 700 bar ≈ 70 MPa ≈ 10000 psi

Table G.2 Injection Fluid Properties

Treatment / Function	Treatment Method / Frequency	Fluid	Comment
Stimulation Treatment	Injected via a service line. Typical duration 1–2 days, at intervals of three years.	HCl (28% or 15%) HCl + HF HCl + CH ₃ COOH (plus corrosion inhibitors)	–
Scale Inhibitor	Continuously injected into production stream.	Acrylate polymers or phosphates/phosphonates in aqueous solution, with ether and alcohol additions for increased solubility. Generally acidic	–
Hydrate Inhibitor	Continuously injected into production stream or injected neat during shutdown.	Methanol, glycol or ethanol	Typical quantities of 10–100 ppm
Hot Oil Flushing	Injected topsides. Typical duration 24 hours, once per month.	Hot Oil	High temperatures.
Corrosion Inhibitor	Continuously injected into production stream.	Ethoxylated and quaternary amines in aqueous glycols or alcohols.	–

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