

New requirements and testing for the certification process of PE100-RC pipes

For the summary of benefits of the new PE100-RC for the stakeholders, including timeline of standardisation, we refer to the paper "More benefits with Crack-resistant PE100-RC pipes" which is available here.

EN and ISO pressure pipe standards include PE100-RC

Summary: Improving the slow crack growth (SCG) performance/resistance of PE100 pressure pipe materials has been the key focus area of development, leading to improved robustness of PE pipes supporting modern installation practice to further grow the value versus traditional materials like ductile iron and steel pipes, especially related to its longevity. Under the name PE100-RC (resistance to crack) in Europe and PE100-HSCR (high stress crack resistance) in other parts of the world, those materials have already been in use for more than 15 years but mainly based on local standards, individual end user specifications or certification schemes like the PAS1075 [1] (Public Available Specification published by DIN (Deutsches Institut für Normung e.V.) in 2009 and withdrawn in Feb 2020). However, the revision of the application standards for water (EN 12201 [2] and ISO 4427 [3]) and for gas (EN 1555 [4] and ISO 4437 [5]) now considers suitable and standardised transparent test methods for the PE100-RC designation.

INTRODUCTION

Today modern polyethylene materials used for the manufacture of piping systems for water and gas supply are solving the corrosion issues of steel and ductile iron pipes. Since the 1960s, these materials have been developed and improved in terms of performance, pressure rating, and above all durability and resistance to slow crack growth (SCG) and rapid crack propagation (RCP).

Whilst initial improvements increased the material's hydrostatic pressure resistance (PE32 < PE40 < PE50 < PE 63 < PE 80 < PE 100), considerable progress has been made during the last 15 years by enhancing the resistance to SCG of PE100, consequently resulting into PE 100-RC (RC: **R**aised **C**rack resistance, see EN 1555). The main technical benefit of PE100-RC is a more robust pipe so that scratches and notches caused during the transport and installation phase of the pipe will not lead to crack propagation over time through the pipe wall and will not lead to a premature failure of the pipe system. Products made of PE 100-RC materials therefore exhibit significant advantages, especially for the non-conventional installation methods. As a result the durability of the installed pipe system is increased. For decades PE80 and PE100 materials have been used for so called No-Dig, trenchless installation methods like horizontal directional drilling (HDD), mole ploughing and rehabilitation methods like close-fit relining or pipe bursting (see examples in EN ISO 21225, EN ISO 11295) [6],[7],[8]. Especially for such rough installation techniques with unpredictable stress and installation situation, PE100-RC offers significant further safety margin.

The standard installation guidance in many markets is the use of sand-bedding around the PE pipe to avoid any damage caused by stones in the ground around the pipe which could cause point loads which may initiate slow crack growth and premature pipe failure.

Network owners are interested in sand-less installations by using the excavation soil as a back fill. That saves costs, time for the installation and often causes less of a disturbance for the proximity to the installation site or the traffic passing nearby. Nowadays in view of the sustainability comparison of different installation techniques and materials, a further strong argument is the reduced CO_2 emission of those new installation methods.

Besides the existing requirements valid for PE80 and PE100 materials in the PE application standard series of EN 1555, EN 12201, ISO 4427 and ISO 4437, a new separate material designation 'PE100-RC' was defined with a set of higher SCG requirements compared to normal PE100 for the material, for the pipe, the fitting and valve components given in Part 1 to Part 5 as appropriate.

Of importance is the focus on the use of international published, recognized and suitable ISO test standards for the definition of the SCG level of resin property and/or the pipe and fitting property. The old PAS 1075 published by the German DIN was withdrawn in February 2020 and there will not be any ISO alternative test method developed for example for the point load test as no working correlation to other methods and a repeatability could be developed.

Notched Pipe Test (NPT), ISO 13479 [9]

The NPT has probably been the most well-known pipe test to simulate outer scratches on polymer pipes for more than 40 years. This test was of specific importance at the beginning of the development of polymer resins for pipe application in the 1970s when such damage could still lead to early failures due to the low SCG performance. With modern polymerization technology and the use of comonomers and branching of the polymer chains, failure times in the classical NPT now exceed 1 year for a PE100-RC.



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Picture 1: Pipe specimen prepared with 4x notches for the NPT or ANPT test

Faster methods are required for the development of new resins, for PVT (process verification test) and AT (audit test) purposes. The new ANPT (Accelerated Notched Pipe Test) method has been in use for about 10 years and just uses a 2% Nonylphenol solution (Arkopal N-100) at the outside of the pipe where the detergent can directly act on the notched zone, the crazing zone. Austria's certification scheme makes use of this test with good experience and tested many different PE100-RC resins over the years. The acceleration factor for PE100-RC resins with Arkopal is >30 that means in a comfortable time of >300 hours it is possible to check the same performance as in a >8760 hours

(=1year) test in water.[10] [21] The revised ISO 13479 standard covers a separate Annex for the accelerated method and the use of alternative detergents. Due to the cessation of production of Arkopal an alternative detergent, Lauramine oxide (Dethyton PL) is under evaluation and a new threshold condition will be defined for the required failure time. The ANPT can then help to define further correlations to existing long-term but also to short-term SCG test methods as further described in this paper.

In the DVGW study [11], 110 mm SDR11 pipes were used to develop new SCG methods and identical pipes were then used to also perform the ANPT in Dehyton in a second step (supported by the PE100+Association[12]) so that a further method could be added to the study and to the new EN/ISO standards afterwards.

Strain Hardening Test (SHT), ISO 18488 [13]

There are many SCG methods existing for the evaluation of PE resins like the ESCR (Environmental Stress Cracking) test ASTM D1693 [14] or the PENT (Pennsylvania Edge Notch Tensile) test ASTM F1473 [15], but also here the modern bimodal PE100-RC materials would have very long and inefficient testing times. The tests are therefore no longer affordable, are not suitable for development and not suitable as a batch release test for a resin manufacturer.

The SHT is a relatively new, but easy way to measure SCG behavior of a material in reasonable time and without the use of any detergents. A very small amount of material is compression molded to a 300 μ m plaque and tensile test pieces are taken from the plaque with a punch out device. A simple tensile test is performed at 80 °C.



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Picture 2: Tensile testing machine with 80 °C heated chamber for SHT



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Picture 3: Punched out tensile test specimen ~300 ym for Strain Hardening Test (SHT)

The results are very reliable with a very low inter-laboratory scatter and the results are available within a few days, regardless of the PE grade, so suitable as a Batch Release Test (BRT). The SHT is usually performed on resin material, but an end user can order samples taken directly from pipes, sheets or fittings to check if indeed a PE100-RC material was used for the delivered pipe and fitting. The samples are ground and the regrind is compression molded again to a plaque. ISO 18488 describes in detail the required steps of preparation, testing and determination of the SHT which is taken directly from the tensile test curves.

Cracked Round Bar Test (CRB), ISO 18489 [16]

Test bars are usually machined as 14 mm round cylinders from compression molded plaques or machined directly from thicker pipes or sheets and then a defined razor blade notch is attached in the middle of the specimen. The notched cylinder is dynamically loaded with a tensile force. The test is performed at room temperature and no detergents are required to accelerate the SCG propagation from the notch to the middle of the specimen.



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Picture 4: Tensile testing machine equipped to perform CRB test



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Picture 5: 14mm round CRB specimen with applied notch in the middle

CRB results are commonly available within several days, considering the crystallization time and thickness of the sample and depending on the polymer design. Such a characterization of the polymer in combination with other SCG methods like the SHT or the ANPT adds to the picture, especially in the development phase of a polymer.

Full Notch Creep Test (FNCT), ISO 16770 [17]

The FNCT normally uses squared 10 x 10 mm thick specimens, either compression molded or taken from pipes. The plaques are notched in the middle with a razor blade according to a defined method and depth. In a constant tensile load test the specimens are exposed to an aqueous medium containing a stress cracking detergent like Arkopal N-100 and a defined temperature of 80 °C and a failure time of >1 year has been experienced for PE100-RC.



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Picture 6: FNCT test specimen prepared with notch before testing

The fracture surface area is examined to define if a ductile or a brittle failure has occurred over time. The ligament area measured afterwards defines the applied real stress after the test.

An AFNCT test condition has been developed in the DVGW project [11] for rough beddable pipes and here the detergent is Lauramine oxide (Dehyton PL). Test temperature is 90 °C. The required failure time is minimum >550 hours at a reference stress of 4 MPa. Brittle failure structure surface must be achieved. An alternative 5 MPa stress can be applied for stiffer materials if a brittle failure picture still occurs. Then the required failure time is >300 hours. Both conditions are equal to a 1-year FNCT testing for PE100-RC materials in Arkopal.

Is the Point Load Test as in PAS1075 still needed?

The Point Load Test (PLT) [1] developed by Hessel was designed to simulate a stone pressing onto the exterior wall of a polymer pressure pipe. PE100-RC materials sustain >8760 hours PLT testing time even under the accelerated conditions of 80 °C and 2% Arkopal N-100 condition inside the pipe and hoop stress of 4 N/mm², due to its high SCG resistance design. As the testing time is far too long for a practical regular use and for development work, an accelerated 'PLT+' test method was introduced by Hessel but the exact conditions and testing media were not published and are not known. The ISO standardisation group TC138 SC5 WG20 aimed at a standardised PLT and PLT+ method with the support of enhanced detergents and higher temperatures. The work was supported by the German DVGW study and supported by a broader number of companies and associations. However, the repeatability and acceleration could not be defined and no correlations are proven by the three involved test institutes: SKZ [18] Germany, KIWA [19] The Netherlands and TGM [20] Austria. Therefore it was decided, that the PLT test method or an accelerated version PLT+ cannot be part of the revision of the EN and ISO PE pressure pipe standards and the draft work ISO/DIS 22102 has been stopped.

However, that is maybe also not needed, as Hessel and other labs have shown so many further correlations of PLT to other existing SCG methods like the FNCT (ISO16770) with an accelerated

version (AFNCT) supported by the DVGW study or the NPT (Notch Pipe Test ISO 13479) and its accelerated version called ANPT. Based on former Plastic Pipes Conference papers, there should not just be one requirement to distinguish between a PE100 and a PE100-RC but a set of defined tests to pass for the resin manufacturer and selected ones for the pipe and fitting manufacturer as polymer designs might be different. This will create confidence for the network owner of the gas and water system, even without the specific PLT test as we always measure some kind of SCG in a correlated method!

Implementation of the requirements for PE100-RC into EN and ISO pressure pipe standards

With the revision of the EN 1555 and EN 12201 series, the integration of PE100-RC as a separate material designation in the standard has been done already. All requirements of PE100 are also valid for the PE100-RC, but PE100-RC is demonstrating higher SCG resistance. Where more than 500 hours are required for the standard PE100 resin and pipe in the NPT, the PE100-RC has to pass the accelerated test method ANPT with >300 h in Arkopal at 80 °C which corresponds to a normal NPT testing of greater than 1 year at 80 °C in water. The SHT, the CRB and the AFNCT requirements are added to the different parts of the standard and can be performed as resin or pipe test.

The informative Annex of the EN 1555 and EN 12201 shows a summary of the comparison between a normal PE100 and the future PE100-RC test requirements in the pressure pipe standards. There have been numerous publications [23] [24] by test institutes and during the Plastics Pipes Conferences during recent years to show the correlations between those different test methods. Of importance is that each defined PE100-RC resin grade has to fulfil all 4 test methods, SHT, CRB, ANPT and the ANFCT, so is not trimmed to one property only.

The implementation of PE100-RC in the EN and ISO pressure pipe standard as of 2021 was an important step to enable the gas and water industry and further industrial applications to make full use of the PE100-RC material and pipe performance and to have a publicly available, transparent requirement, testing and certification base. This will enable end users to utilize the benefits of the interesting alternative installation methods like No-Dig, sand-less and various relining methods. The PE100-RC pipe, fitting and valve manufacturers are looking forward to a further growing market potential by substitution of traditional materials.

PE100, typical performance	Expected performance PE100-RC	Test method for PE100-RC in EN + ISO
NPT Notched Pipe Test	NPT Notched Pipe Test ≥ 8760	ANPT (Accelerated in 2% solution
≥ 500 h, water, 80 °C	h, water, 80 °C	Arkopal N100, 80 °C) ≥ 300 h ¹ or
		alternative detergent conditions
FNCT ≥ 300 h, 80 °C, 2%	FNCT > 8760h, 80 °C, 2%	AFNCT (Accelerated in 2% solution
solution Arkopal N100	solution Arkopal N100	Lauramine oxide), 90 °C)
		> 550 h at 4 MPa stress ¹ or
		> 300 h at 5 MPa stress ¹
		(brittle fracture surface required)
Strain Hardening SHT <gp></gp>	Strain Hardening SHT <gp> ≥</gp>	Strain Hardening SHT <gp> ≥ 53</gp>
≥ 40 MPa ¹	53 MPa¹for resin and ≥ 50	MPa¹ for resin and ≥ 50 MPa¹ for
	MPa ¹ for pipe & fittings	pipe & fittings

	CRB Crack Round Bar <u>></u> 0,9	CRB Crack Round Bar <u>></u> 1,5 x	CRB Crack Round Bar > 1,5 x 10 ⁶
	x 10 ⁶ cycles ¹	10 ⁶ cycles ¹	cycles ¹
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¹ Derived from DVGW study (Determining limits and minimum requirements for materials and pipes for the rough-beddable pipes made from PE100-RC. 2018) [11]

Table: Table of Annex A of EN 1555-1 and Annex C of EN 12201-1

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