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SUMMARY: ISO 21809-1 as published in 2012 will set the international requirements for extruded 3-layer designs of polyethylene (PE) and polypropylene (PP) coatings of oil and gas transmission pipelines. Some areas of application such as gas and water distribution are not covered by the international standard. These will be specified at national level by revising and keeping DIN 30670 (PE coatings) and DIN 30678 (PP coatings) as residual standards. A preliminary version of ISO 21809-1 was published in 2010. Since the practical limits set by this standard will be the basis of future contracts and agreements, they should not go uncommented, the main reasons being the material properties of polyethylene and polypropylene.

INTRODUCTION

Steel pipes have been coated with polyethylene and polypropylene for decades. At national German level, these corrosion protection systems are defined by DIN 30670 and DIN 30678 [1] [2]. First drafts of DIN 30670 go back as far as the 70ies of the last century. DIN 30678 for polypropylene coatings did not emerge until 1992, i.e. not until first steps were taken towards establishing the requirements for polyethylene and polypropylene coatings at an international scale. ISO 21809-1 will now conclusively terminate all international efforts at harmonising these terms of delivery for oil and gas transmission pipelines [3].

DIN 30670 and DIN 30678 not only specify the requirements and tests but also outline the limits of application of these coatings. The scope of these standards is restricted to underground and underwater pipelines. DIN 30670 describes two designs of polyethylene coatings. Maximum operating temperatures are limited to 50 °C (type N) and 70 °C (type S). With regard to polypropylene coatings, DIN 30678 specifies a maximum operating temperature of 90 °C. In order to assess the material's sensitivity to low temperatures, DIN 30678 requires impact testing at 0 °C.

FIGURE 1 AND FIGURE 2: **PP coating damage discovered on a buried pipe and pipes on stock**

TABLE 1: **Areas of application of different 3-layer coatings according to ISO 21809-1 in dependence on the coating thickness and the pipe weight [3]**

Coating thickness class	1	2	3
Ground characteristic	Onshore: sandy ground	Clay w/o filler material	Stony/rocky ground or offshore
Total coating thickness LDPE top coat	1.8–3.2 mm	2.1–3.8 mm	2.6–4.7 mm
Total coating thickness MDPE/HDPE top coat	1.3–2.5 mm	1.8–3.3 mm	2.3–4.2 mm
Total coating thickness PP top coat	1.3–2.5 mm	1.7–3.0 mm	2.1–3.8 mm

TABLE 2: **Operating temperatures of 3-layer coatings to ISO 21809-1 [3]**

Top layer	LDPE	MDPE/HDPE	PP
Operating temperatures	−20 °C to +60 °C	−40 °C to +80 °C	−20 °C to +110 °C

The directives and regulations concerned with the planning and construction of gas and water mains pipelines contain further requirements on the bedding material. They demand that the pipes are generally bedded in material without any stones.

ISO 21809-1 expands these areas of application. In the case of greater coating thicknesses, for example, it also permits bedding in stony or rocky ground because it includes a verification of the material's environmental stress-cracking resistance by means of the so-called Bell test to ASTM D 1693 [4] (**Tab. 1**). The so-called Bell test checks for a material's crack resistance by applying a surface-active agent. As a material testing method, it thus compares to the FNCT (Full Notch Creep Test) which has become the more commonly known methodology. Its results are used to show that plastic materials are suitable for use under the critical installation conditions of alternative laying techniques, cf. PAS 1075 for example [5].

ISO 21809-1 now also includes practical restrictions for low-temperature applications. In the case of polyethylene, a type of coating comparable to type N of DIN 30670 is specified for temperatures between -20 °C and +60 °C and another type comparable to type S is specified for temperatures between -40 °C and +80 °C. According to the standard, polypropylene coatings can be operated between -20 °C and 110 °C (**Tab. 2**). There are no comments regarding the shorter useful life at higher operating temperatures as are included in DIN 30678.

Practical experience shows that these limitations need to be more carefully assessed with regard to the material properties and the fracture mechanical interrelations of temperature and aging in particular.

HOW DOES POLYPROPYLENE AND POLYETHYLENE RESPOND TO LOW TEMPERATURES?

Polypropylene coatings are known to fail under low ambient temperatures. **Figures 1 and 2** show examples of such a case of damage. The pipeline was buried by means of horizontal directional drilling (HDD). The pulled end of the pipe extended beyond the hole and showed clear signs of flaking after a period of cold weather at -10 °C to -15 °C (Figure 1). Even some pipes on stock revealed first cracks (Figure 2). When exposed to a hammer stroke at -4 °C, undamaged pipes on stock also showed this cracking. When the 3-layer PP coating was examined, it was found to meet all quality requirements. The data sheets of the special type of polypropylene used in this case specified a glass transition temperature of -40 °C. Standard impact testing to DIN 30678 at -20 °C did not lead to any coating failure. Taking account of the operating temperature of -20 °C to +110 °C specified by ISO 21809-1, another method of testing was required in order to realistically assess the area of application of such coatings.

If applied to a material become brittle due to low temperatures, the aforementioned impact test to DIN 30678 should have revealed major flaking or cracking such as was found on pipes on stock at -4 °C. At a closer look, the impact test array to DIN 30670 or DIN 30678 is not really suitable for such investigations. The test impact is applied with a drop weight exhibiting a hemispherical head. According to the standard, the diameter of the hemisphere is 25 mm. Since the head of the drop weight is hemispherical, the coating material is pushed towards the edges of the test area as the weight hits the test object. The ensuing bulge generates compressive stress in the area that cracks should have formed in (**Figure 3**).

A flat area of impact would avoid this effect (**Figure 4**). The planar head of the drop weight used in this modified impact test has a diameter of 21 mm. A 10 kg weight was

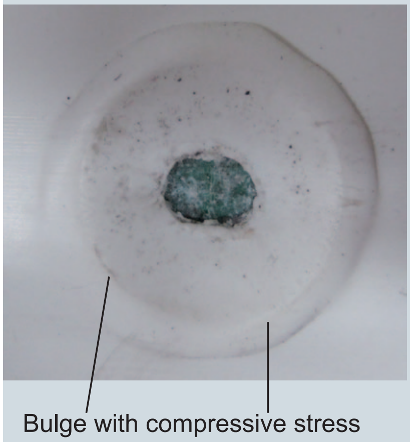


FIGURE 3: **Coating after conventional impact test**

FIGURE 4: **Drop weight used for modified impact testing**

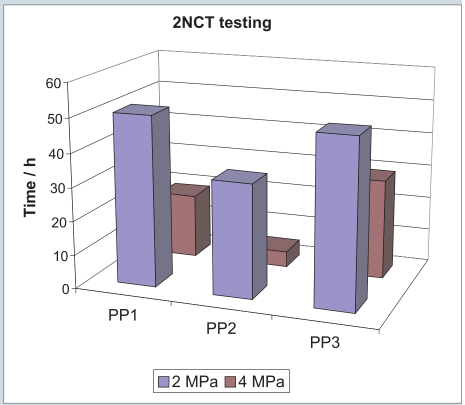
TABLE 3: **Minimum temperatures without cracking or flaking**

	LDPE	HDPE	PP1	PP2	PP3	Dual layer FBE
3-layer	−27 °C	−40 °C	+6 °C	+4 °C	0 °C	
2-layer						+15 °C
1-layer			−12 °C	−20 °C	0 °C	
Sheet	−33 °C	< −50 °C	−10 °C	−15 °C	−5 °C	

dropped onto the surface from a height of 1.0 m. The impact energy thus computes as 98.1 J. In order to find a minimum temperature at which no cracks emerge, the test is repeated at different temperatures.

At a first step, the significance of the test method was verified by examining pipe segments of the same dimension (DN 200) with a 3-layer coating consisting of an epoxy resin primer, adhesive layer and a polyolefin top layer (polyethylene, PE or polypropylene, PP). Tested 3-layer polyolefin coatings were between 2.4 mm and 3.0 mm thick. Comparative testing was performed on a 2-layer epoxy resin powder coating (dual layer FBE). Whereas the height of fall remained unchanged, the weight was reduced to 5 kg because the epoxy resin layer is just 770 µm thick.

FIGURE 5: **Results of comparing 2NCT tests**



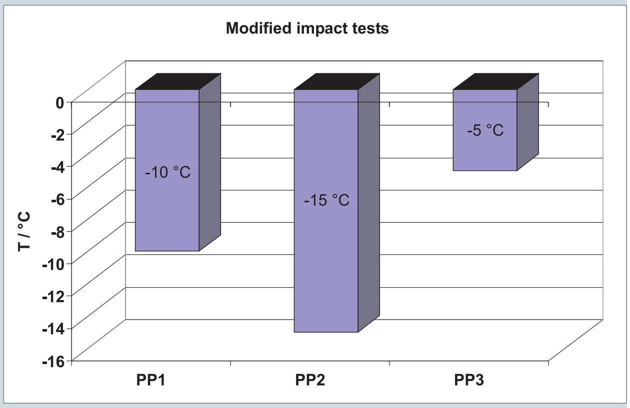


FIGURE 6: **Results of modified impact tests on extruded sheets**

The results yielded by the different polyolefins (**Tab. 3**) correlate well with practical observations. The brittle points of LDPE and HDPE are below -27 °C and -40 °C respectively. ISO 21809-1 specifies bottom application limits of -20 °C (LDPE) and -40 °C (HDPE) so far correctly and corresponds to the experience of decades of applications. The brittle point of a 3-layer PP coating system is below +6 °C. These results again correspond to practical observations. For PP coating systems an application range down to -20°C in ISO 21809-1 can't be realized. The comparative test of the epoxy resin system reveals a much greater susceptibility to mechanical loads which is a well-established fact from the handling of pipes with this kind of coating.

The temperature found in this test can thus be regarded as the lowest recommended operating temperature of the coating material. Below this temperature, cracking due to low-temperature embrittlement or flaking of the coating cannot be excluded.

Apart from 3-layer systems, the test was also carried out on 1-layer PP coatings, where the PP layer was extruded directly on the steel surface and hence showed no adhesion on the steel. These 1-layer PP coatings were specially prepared for comparison and are not used in the field. Every examined case yielded lower brittle points which leads to the assumption that the 3-layer design greatly contributes to this cracking behaviour. The results of the extruded 1-layer pipe samples more or less coincide with the results of parallel examinations on extruded sheets. Irrespective of the PP make, none of the 3-layer polypropylene systems under test achieved the -20 °C limit specified in terms of delivery like ISO 21809-1.

Since the technical product data sheets only provide some information about the fracture mechanical properties, a second step of testing tried to provide a qualitative assessment of the differences between the different types of polypropylene by means of the FNCT (test with a circumferential notch) and the 2NCT (test with with notches on either side). FNCT or Bell test results are normally shown on the technical data sheets which turns them into one of the criteria of selecting the right material for a given application.

Testing failed to find a correlation between the brittle points as assessed with modified impact testing and the results of the 2NCT. 2NCT test times were compared with the minimum temperatures used for the modified impact tests and found not to produce any cracking in the extruded sheets. The PP2 material yielded the worst in class 2NCT results. In the modified impact tests it did not start to crack until temperatures were below -15 °C. According to the modified impact test methodology, the other materials with significantly better 2NCT results have higher brittle points, i.e. below -5 °C and below -10 °C (**Figures 5 and 6**). Considering the fact that FNCT and 2NCT testing is performed at high temperatures, neither the change in these fracture mechanical properties can be measured nor a qualitative appraisal of the materials be provided.

MECHANISTIC ANALYSIS OF CRACK FORMATION

Investigations on the low-temperature embrittlement of polypropylene included mechanical tests helping to understand the causes. The tensile tests were carried out at different temperatures and confirmed interrelations found earlier in aged and, thus, embrittled polyethylene coatings [6]. **Figures 7 and 8** illustrate (i) a comparison of tensile tests carried out on polyethylene coatings become brittle with age and non-aged material and (ii) the tensile tests carried out on polypropylene at room temperature and around the brittle points observed. Both samples not only show an obvious reduction in the elongation at break but also an unfavourable ratio of yield stress and tensile stress at break.

The only difference is the fact that the tensile stress at break of aged material is reduced compared to the yield stress, whereas, in the case of low-temperature embrittlement, the yield stress increases compared to the tensile stress at break. For aged polyethylene the yield stress stays on the level of the unaged material. In both cases yield stress and tensile stress at break reach an unfavourable constellation leading to a significant decrease of the elongation at break. Main requisite for crack initiation is that the yield strain or the yield stress is overrun. This critical point can be overrun in the whole material, e.g. due to tensions at temperature changes, or only in small areas, e.g. due to point loads and point support.

Under mechanical aspects, cracking therefore has identical causes, which is confirmed by comparing the fracture patterns of these failure modes (**Figures 9 and 10**). In both cases, cracks on the pipe form in the same way.

Practical experience provides facts excellently documenting this interconnection with regard to cracks forming when UV light damages a polyethylene coating. **Figure 11** illustrates the mechanical investigations performed on a section where the material was peeled off from the entire circumference of the pipe. The damaged section is inevitably restricted to the side exposed to the sunlight and can be recognized in Figure 11 by the colour change from yellow to colourless. The underside remains undamaged. In the damaged sections discolouring was not only observed on the surface of the PE-layer, at least 1/3 of the layer thickness was discoloured.

The yield stress remains at about 155 N to 160 N in both the damaged and undamaged sections and only varied due to different coating thicknesses. In the damaged sections tensile stress at break and elongation at break have much lower values than in the undamaged sections. This is to show again that the tensile stress at break decreases as UV damaging increases. As the tensile stress at break decreases, the ratio of tensile stress at break and yield stress becomes more unfavourable and the elongation at break reaches its limit. These results gained from practical applications again confirm the interconnectedness of yield stress and tensile stress at break and its impact on the material's strainability and, thus, its flexibility.

Practically speaking, this also implies that ageing and low temperature embrittlement needs to be assessed together. Assuming that ageing causes the tensile stress at break to

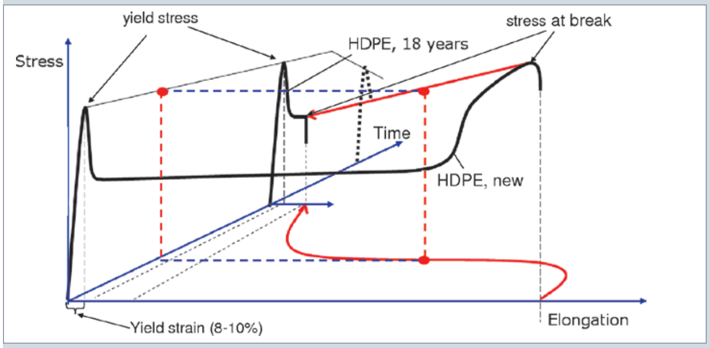


FIGURE 7: **Comparison of tensile tests: Aging of HDPE**

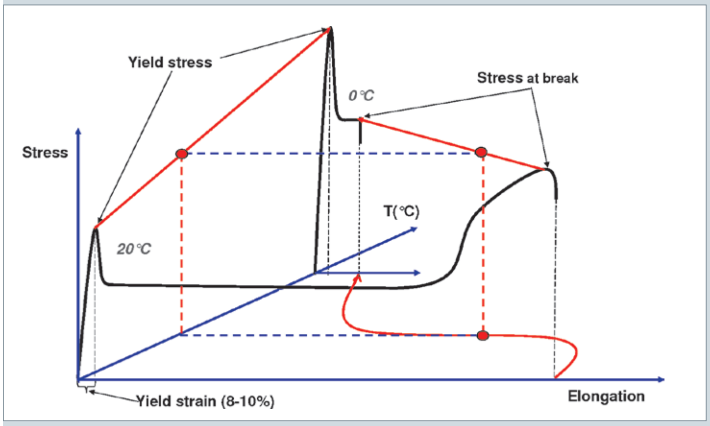


FIGURE 8: **Comparison of tensile Test: Low temperature embrittlement of PP**

FIGURE 9: **Cracking pattern as produced by aging of the material**

FIGURE 10: **Cracking pattern as produced by low-temperature embrittlement**

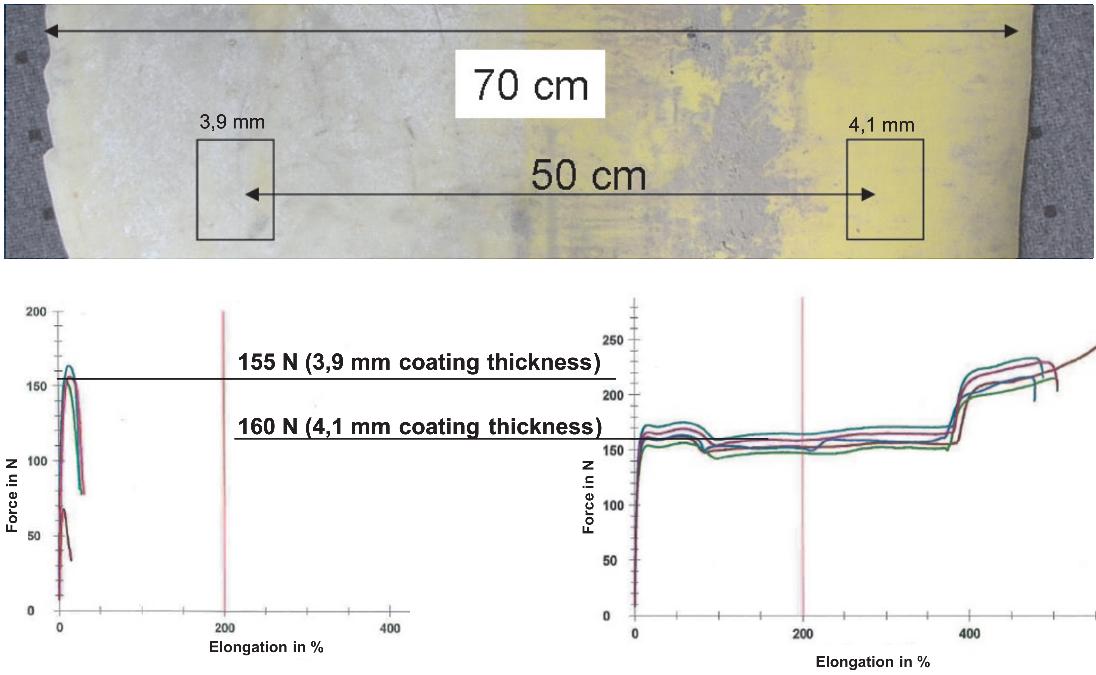


FIGURE 11: **Analysis of a steel pipe coating damaged by UV light – tensile tests**

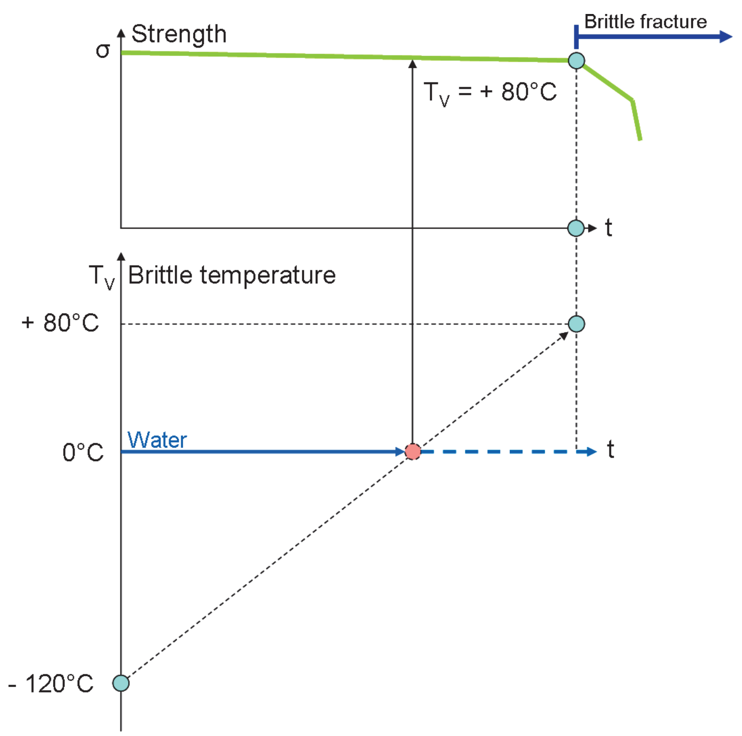


FIGURE 12: **Brittling behaviour explained by way of an internal pressure creep rupture test**

move closer to the yield stress, dropping temperatures will cause the yield stress to increase such that the brittle point is reached earlier. As temperatures drop, an aged material will thus be more susceptible to fracturing than a new material. For polyolefin based coatings in general only little information about creep-rupture behaviour is available. However, for polyolefin based pipes the creep-rupture behaviour is well known and should be transferable to polyolefin based coatings (**Figure 12**).

Internal pressure creep rupture testing of PE pipes has revealed that the second branch of brittle fracture should come to the fore when an extrapolation to 20 °C results in a theoretical serviceability of at least 50 years. This must take into account that, at this point in time and at a test temperature of +80 °C, the brittle point (T_v) has also reached a level of +80 °C.

However, the brittle point of new material is far below 0 °C. If the glass transition temperature is assumed to be the starting point under the mainly quiescent load of an internal pressure test, said starting point is as low as -120 °C. Whereas there are hardly any changes to the material strength (yield stress) in this test, the brittle point shifts considerably if the starting and end points are taken into account. The actual course the brittle point takes along the logarithmic time line currently remains unknown. This is why the course is shown as a dotted line in Figure 12.

For brittle behaviour in practice the intersection with the minimum operating temperature would be relevant. The minimum operating temperature to be considered depends on the climatic conditions and on how deeply the pipes are buried. Since water pipes for example are normally laid in a frost-proof manner, the analysis assumed the minimum operating temperature to be 0 °C. In this case the intersection with the 0 °C line, the material reacts brittle to external influences while at +80 °C the material would still be flexible and material strength seemed to be on a level as at the start.

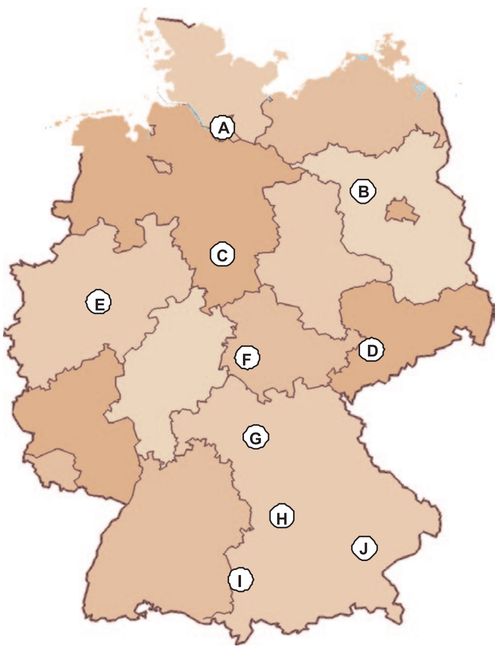
Comparing climate data with damage rates should be able to show whether this interdependency is assumed correctly. In this context it has to be recognized that damage of PE coatings are discovered most likely by cathodic protection measurements or when pipes are dug out later. The time of damage and also the climate condition at that time is not available. Evidence should be easier to find for polyolefin pipes where leakage occurs immediately when the material becomes brittle and, thus, more susceptible to mechanical loads such as point loads, point support, deformation etc. as main reasons for damage in pipeline service.

The comparison was based on the claims statistic published by DVGW (German Technical and Scientific Association for Gas and Water) [7] and the climate data published by DWD (German Weather Service) (**Figure 13**). How cold a winter was is judged by counting the number of ice-cold days. An ice-cold day defines as a day during which the temperature never rises above 0 °C. A large number of ice-cold days normally occurs under stable high-pressure winter weather. Under such weather conditions, the frost will penetrate the ground more deeply. The figure shows the locations of the chosen meteorological stations. Extreme locations such high mountains (Brocken in the Harz mountains or Zugspitze) or exposed coastal regions (Kap Arkona) were intentionally left out. The ice-cold days on record between 1997 and 2004 were averaged and the resulting curve combined with the damage rates published by DVGW for the same period.

Correlating the climate data with the DVGW's claims statistics clearly supports the assumed causal interdependency of aging and low-temperature embrittlement, even if the relevant curve is slightly tilted and if the 1999 damage rate is slightly biased. Embrittlement caused by aging is detected by polyolefins becoming more susceptible to low temperatures. In the case of external influences such as point support, point loads, deformation etc., it is also the cause of damages.

This finding is of high relevance for the fact previously established in the context of low-temperature embrittlement, i.e. that using tests utilising surface-active agents such as FNCT, Bell test etc. provide neither quantifying nor qualifying results with regard to a material's fracture resistance

Weather stations		1997	1998	1999	2000	2001	2002	2003	2004
A	Hamburg	16	11	4	6	16	16	14	8
B	Neuruppin	17	18	8	9	17	23	18	12
C	Seesen	14	20	14	7	16	17	20	10
D	Chemnitz	24	36	22	10	24	25	26	25
E	Essen	14	7	5	1	6	5	13	5
F	Meiningen	28	40	26	15	25	29	31	26
G	Ebrach	21	21	17	10	14	22	19	19
H	Donauwörth	27	30	26	12	17	18	25	20
I	Memmingen	22	32	23		26	22	35	33
J	Straubing	30	38	32	19	25	27	31	36
		213	253	177	89	186	204	232	194
		1997	1998	1999	2000	2001	2002	2003	2004
		21	25	18	10	19	20	23	19



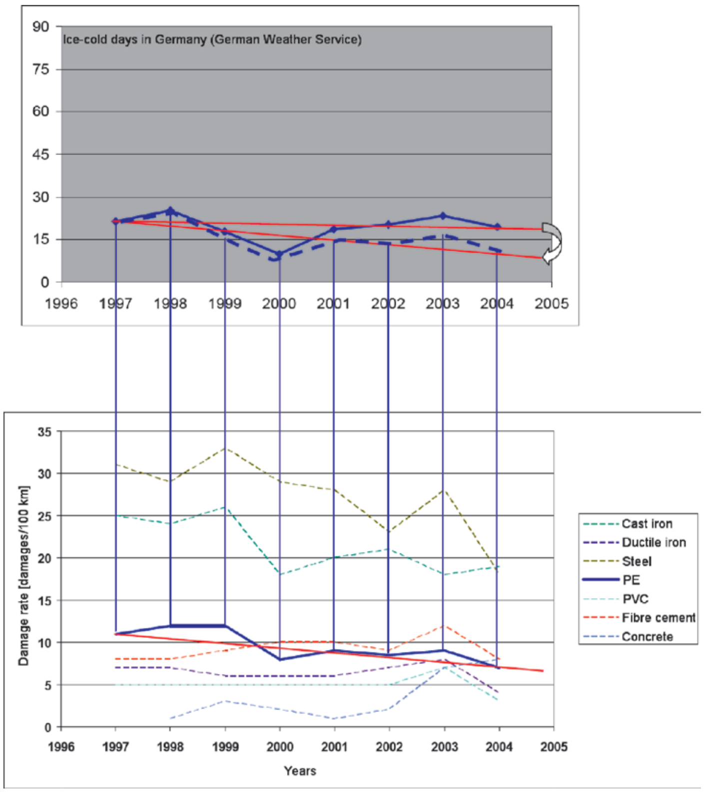


FIGURE 13: **Comparison of climate data and the DVGW's claims statistics of PE pipe damages [7]**

under point loads, point support, deformation etc. because these tests are carried out under high temperatures and fail to include the changes in fracture mechanical properties occurring at low temperatures in both new and aged materials. Earlier publications already acknowledged this general issue [6] [8] [9].

CONCLUSIONS

The results have already indicated the answer to the question asked in the headline of this article. According to our current knowledge and in the light of publically available information about the relevant material properties, the areas of application ascertained in technical specifications such as ISO 21809-1 can not be realized completely. Both the low-temperature properties of polypropylene coatings and the scope stated for the bedding of pipes with polyethylene and polypropylene coatings would inevitably cause problems in practical applications. Since coated steel pipes are permanently monitored in conjunction with cathodic protection, such failure modes would always be detected soon. Ensuing discussions about hidden faults may be avoided by commenting and explaining actual limits and restrictions in advance.

Investigations show that the type of PP normally used for pipeline coating does not support temperatures down to -20 °C as described in ISO 21809-1. The results suggest to preliminarily restricting handling and installation of 3-layer PP coated pipes to temperatures of 0 °C. In a general sense, this restriction is covered by DIN 30678 pointing to the basic need for an impact resistance test at this temperature. Whereas the last version of ISO 21809-1 will not include this aspect, it was possible to make readers aware of the susceptibility to low temperatures by adding a footnote concerning the handling and installation of pipes.

Further investigations of the damaging mechanisms were able to show that crack initiation due to aging and low-temperature is all in all attributable to the same causes. This is backed up by:

1. Identical mechanical interdependencies
2. Identical failure modes
3. Comparing climate data with the DVGW's claims statistics

Due to the changes in fracture mechanisms, data provided by tests with surface-active agents does not support conclusions as to the susceptibility to low-temperature embrittlement. The same inevitably applies to embrittlement caused by aging which should actually be regarded as premature low-temperature embrittlement. In no case do tests using surface-active agents justify the range of application of non-conventional pipe installations without a sand bed as specified by ISO 21809-1 for greater coating thicknesses. If pipes are installed in beds without stones according to previous global requirements, even a fully embrittled PE coating on a steel pipe will continue to provide a sufficient barrier against corrosion. Documented evidence of the relevant practical experience is available [10].

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