A vision becomes true – 50 years of pipes made from High Density Polyethylene

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After the discovery of the low-pressure process for the production of polyethylene with the aid of Ziegler catalysts, only another year went by before pipes were being extruded in high-density polyethylene. In the same year, at the end of 1954, Hoechst had already begun to carry out long-term failure tests on HDPE pipes under internal hydrostatic pressure at temperatures of between 20 and 80 °C. Consistent continuation of the long term test has shown that even the first pipes to be made of HDPE more than exceeded the former design service life of 50 years. Through intensive material developments, the long term hydrostatic strength ascertained in long-term failure tests on HDPE pipes rated for 50 years' service meanwhile exceeds 10 MPa.

FROM LOW-PRESSURE PROCESS TO PIPE MATERIAL

In terms of pipe length, there are more plastic pipes installed worldwide per year than pipes made from any other materials[1]. Of all plastic materials, high-density polyethylene (HDPE) manifests the strongest growth. Since 2005, only 50 years after the first ever installation of HDPE pipes, polyethylene is – in terms of quantity – Germany's most important pipe manufacturing material [1]. The reasons for this success lie not only in the exemplary collaboration between researchers, pipe material manufacturers, processors and users but also in the incredibly short time span between the discovery of a new polymerization process and the initial practical application of pipes manufactured from polyethylene by means of this process.

It was in November 1953 that Prof. Dr. Karl Ziegler and his research staff first succeeded in polymerizing ethylene in the presence of organo-metallic catalyst blends under "mild" pressure and temperature conditions – the low-pressure process for the manufacture of high-density polyethylene had been discovered. It was for this discovery that Karl Ziegler, together with the Italian chemist Giulio Natta, was awarded the Nobel Prize for Chemistry in 1963.

Only a month after this discovery, the chemical industry, including Dr. Otto Horn, the head of research of Farbwerke Höchst, received initial HDPE samples for testing and evaluation (Fig. 1). The first pilot plant with a production capacity of 10 tonnes per month went into operation in Frankfurt as early as 1954. Ziegler reports that 200 tonnes had already been produced in 1955 [2]. By the end of 1955, the first regular production plant with a capacity of 200 tonnes per month was already in service.

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[1] Source: P.Townsend und Basell Marktforschung


MAX-PLANCK-INSTITUT FÜR KOHLENFORSCHUNG

Direktor: Professor Dr. Dr. e. h. Karl Ziegler

MÜLHEIM (RUHR), den 14.12.1953/Pi.
Kaiser-Wilhelm-Platz 1
Fernsprecher 40541 und 40542

Herrn
Dr.Otto Horn
i/Farbwerke Hoechst

Frankfurt/Main-Hoechst

Sehr geehrter Herr Dr. Horn !

Vielen Dank für Ihren Brief vom 11.XII.53 und die Mitteilung der Festigkeitswerte unseres Polyäthylens. Ich freue mich zwar, daß Sie, wie Sie mir am Telefon sagten, einen sehr günstigen Eindruck hatten, trotzdem [illegible] anschliessend regelmässig auf die einzelnen Komponenten hin aufgearbeitet haben. Unsere m-Xylol-Gehalte lagen zwischen 0 und 3 %.

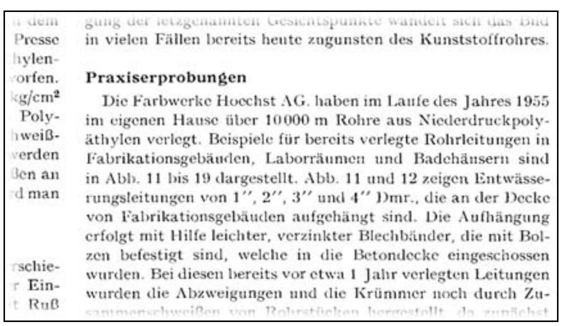
Mit freundlichen Grüßen
Ihr sehr ergebener

Karl Ziegler
(Prof.Dr.K.Ziegler)

Fig. 1. Prof. Ziegler writes letter thanking Dr. Horn, head of research of Farbwerke Hoechst, for carrying out strength tests on HDPE specimens

In 1958, the global production of HDPE amounted to 17,000 tonnes; by 1962, it had already reached 200,000 tonnes. Licences were granted worldwide, and by 1963 there were already 19 plants in Europe and a further 17 in the USA and Japan producing HDPE by the "Mülheim Process" on an industrial scale. Today the quantity of HDPE produced worldwide every year is estimated at 28 million tonnes (2005; all polymerizing processes), of which approximately 3.7 million tonnes are processed into pipes for outdoor applications[1].

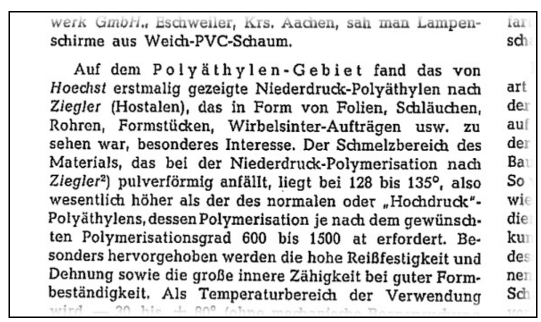
The first applications of HDPE pipes were the subject of a whole multitude of publications in the late 1950s. Plastic pipes were first used in those areas of application in which engineers and users looked back on many years' experience with such traditional pipe materials as steel, cast iron, stoneware and concrete. Consequently, it was first necessary to create the basic technical and/or scientific preconditions for trustful acceptance of the new materials. One of these publications contains a reference to the very first use of pressure pipes made from HDPE (Fig. 2) [3]. This new pipe material had already been introduced at the Hanover Fair at the beginning of May 1955 under the trade name of Hostalen (Fig. 3) [4].


in vielen Fällen bereits heute zugunsten des Kunststoffrohres.

Praxiserprobungen

Die Farbwerke Hoechst AG. haben im Laufe des Jahres 1955 im eigenen Hause über 10000 m Rohre aus Niederdruckpolyäthylen verlegt. Beispiele für bereits verlegte Rohrleitungen in Fabrikationsgebäuden, Laborräumen und Badehäusern sind in Abb. 11 bis 19 dargestellt. Abb. 11 und 12 zeigen Entwässerungsleitungen von 1″, 2″, 3″ und 4″ Dmr., die an der Decke von Fabrikationsgebäuden aufgehängt sind. Die Aufhängung erfolgt mit Hilfe leichter, verzinkter Blechbänder, die mit Bolzen befestigt sind, welche in die Betondecke eingeschossen wurden. Bei diesen bereits vor etwa 1 Jahr verlegten Leitungen wurden die Abzweigungen und die Krümmer noch durch Zu-

Fig. 2. Reference to the very first use of HDPE pipes [3]


werk GmbH., Eschweiler, Krs. Aachen, sah man Lampenschirme aus Weich-PVC-Schaum.

Auf dem Polyäthylen-Gebiet fand das von *Hoechst* erstmalig gezeigte Niederdruck-Polyäthylen nach *Ziegler* (Hostalen), das in Form von Folien, Schläuchen, Rohren, Formstücken, Wirbelsinter-Aufträgen usw. zu sehen war, besonderes Interesse. Der Schmelzbereich des Materials, das bei der Niederdruck-Polymerisation nach *Ziegler*[2]) pulverförmig anfällt, liegt bei 128 bis 135°, also wesentlich höher als der des normalen oder „Hochdruck"-Polyäthylens, dessen Polymerisation je nach dem gewünschten Polymerisationsgrad 600 bis 1500 at erfordert. Besonders hervorgehoben werden die hohe Reißfestigkeit und Dehnung sowie die große innere Zähigkeit bei guter Formbeständigkeit. Als Temperaturbereich der Verwendung

Fig. 3. Hoechst introduces the new low-pressure polyethylene at the Hanover Fair in 1955 under the trade name of Hostalen [4]

EXPECTED SERVICE LIFE OF HDPE PIPE SYSTEMS

From the very beginning, the development of HDPE pipe materials took into account the need of water supply companies for pipe systems having a minimum service life of 50 years. Creep tests on HDPE pipes were begun as early as the middle of 1954 (Fig. 4) [5]. Dr. Kurt Richard, the then Head of the Materials Department at Hoechst, adapted the method of predicting the service life of high-temperature steels to plastics [6], applying the Larson-Miller correlation to the stress characteristics of HDPE. This correlation states that the crack propagation rate in the creep test – like the speed of chemical processes – is a function of the activation energy. Based on an Arrhenius formula, the Larson-Miller correlation is expressed thus:

$$T \cdot (C + \lg t) = const$$

where T is the absolute temperature in K, t the crack propagation rate and C a constant. A C-value of 47.5 was obtained for the flat slope of the creep curve, while the subsequent, steeper slope of the creep curve was put at C = 22. In 1959, after only 4 ½ years of testing at 80 °C, extrapolation of the time-to-failure values already permitted a prediction of the service life of the tested pipes to be 50 years at 20 °C. Thus it was that Dr. Erwin Gaube, who was later to succeed Richard as Head of the Materials Department at Hoechst, could already make the following assertion in his dissertation of 1959: "...long-term behaviour can be reliably predicted for durations of up to 50 years" [7].

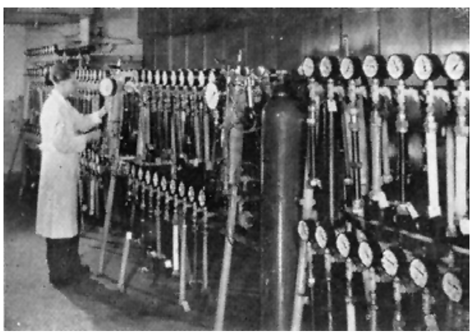


Fig. 4. Early creep tests in the Materials Department at Hoechst

Fig. 5. A "historical test bench" still in operation at R&M Industrieservice Höchst GmbH

Two pipe specimens installed in October 1956 are still undergoing creep tests at a temperature of 20 °C and under hoop stresses of 5 and 7.5 N/mm² respectively (Fig. 5). On 18th October 2006, the extrapolations of the pioneers of pipe life prediction will be finally and most impressively confirmed! The findings obtained in this concrete case of application are of fundamental importance, as the very same extrapolation method is used for predicting the service life of today's generation of HDPE pipe materials.

RESIDUAL SERVICE LIFE AFTER 41 YEARS' SERVICE

Of extreme interest in this context are not only the HDPE pipes tested in the laboratory but also the HDPE pipes used in actual practical application. The first of these pipes were installed by the German chemical company Hoechst AG, Frankfurt am Main, in 1955 – fifty years ago – on its own premises for the supply of water to laboratory and office buildings and for showers and general sanitary installations. Some of the buildings are still standing. One of them, built in 1961, fell into disuse in 2002 as a result of structural changes within the company. A number of pipes in *Hostalen* GM5010 (Fig. 6) were removed from a changing and shower room, in which there had always been an ambient temperature of around 23 °C, and examined. The pipe system had been used for the supply of fresh water to an entire floor of the building. It could be assumed that the pipes had provided a continuous flow of oxygen-saturated water for the duration of 41 years. Reliable information on the additives used at the time and old data sheets served as the basis for evaluating the present condition of the pipes (Table 1).

Fig. 6. Ceiling-mounted HDPE pipes in a changing and shower room built in 1961

Property		Specification Sheet 1966 for *Hostalen* GM5010	Pressure pipe, black 75 mm dia. SDR11		
			Inside	Middle	Outside
Density	g/cm³	0.955	–	0.9547	–
MFR 190/5	g/10 min	0.3	–	0.22	–
MFR 190/21.6	g/10 min	–	–	7.89	–
Reduced viscosity	dl/g	3.5	3.64	3.6	3.23
OIT at 200°C	min		2	25	28
DSC Tm_1	°C		130.28	130.01	129.98

Table 1. Comparison of data obtained in tests on the removed pipes with the data given in the original specification sheets

Data given in specification sheets cannot of course be a substitute for batch control data. Moreover, measuring and control technology has changed constantly since 1966. Nevertheless, measurements show that these pipes are still in a decidedly good condition after 41 years. Comparison of the viscosity numbers (these are a measure of the mean molecular weight) with those indicated for the original materials show that the pipes had not yet reached the limits of their thermal stability. Residual stabilizer content is indicated by the OIT (Oxygen Induction Time). On account of the type of stabilizer used, however, the (surprisingly high) OIT values obtained did not permit any conclusions as to the residual life expectancy of the pipes. As was expected, the inside walls of the pipes manifested a lower stabilizer content as a result of 41 years' elutriation. In Arrhenius tests performed on the pipes to establish their residual life expectancy, the firm of Hessel Ingenieurtechnik GmbH obtained a value corresponding to at least 27 years [8]. Thus the pipes would not only have attained the expected service life of 50 years but would also have clearly exceeded it.

DEVELOPMENT OF APPLICATIONS

The next logical step in the innovation chain was the development of manufacturing and joining technologies for pipes. The existing extruders were able to process these new materials of high molecular weight only to a limited extent. The problem was solved through close collaboration with machine manufacturers (Fig. 7). Suitable extrusion dies and downstream units were developed.

Fig. 7. Demonstration of pipe extrusion in the pilot plant of Farbwerke Hoechst, probably around the end of the 1950s

One of the decisive factors for the successful application of a pipe system is the technology used for joining the pipes. The advantages of fused, absolutely tight systems in HDPE were recognized and implemented very early on. References to the electro fusion of pipe sockets could be found as early as 1956 (Fig. 8) [3]. The hot plate butt fusion process was developed (Fig. 9).

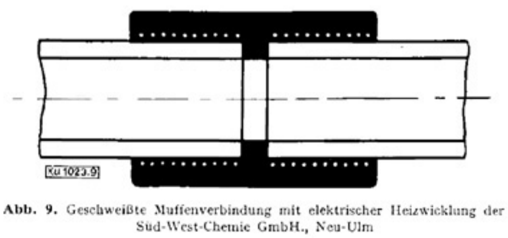


Fig. 8. Electrical fusion socket from Süd-West-Chemie (1956)

Fig. 9. Manual application of joining pressure in hot plate butt fusion process

Dr. Wilhelm Müller, the then Head of Pipe Application Technology at Farbwerke Hoechst, and Dr. Gaube carried out a multitude of experiments and tests in their respective research groups, the results all contributing to the successful introduction of HDPE pipe systems, many of them still being implemented today. They concerned

- the influence of processing conditions,
- internal stresses in pipes,
- establishing resistance to chemicals and determining resistance factors,
- hydraulic rating and determining coefficients of roughness,
- the long-term deformation of large-bore pipes underground and pipe statics,
- behaviour under impact and dynamic load,
- stability when subjected to denting and bending,
- gas permeability,
- determining wear rate when conveying solids,
- behaviour when exposed to flames,
- behaviour when exposed to alternating temperatures,
- weather resistance.

The results of these experiments and tests were readily shared with processors and users, a tradition which was then taken over by the subsequently involved raw material manufacturers and which is still very much alive today [9 to 18]. Meanwhile, a large "HDPE pipe community" has evolved. Supported by national and international conferences and publications, this community practices a very active exchange of information. A representative example is the PE100+ Association - www.pe100plus.net .

PROCESSORS TAKE TO THE NEW PIPE MATERIAL

By the mid-1950s, a great many companies had already recognized the potential of pipe systems manufactured from the newly developed HDPE. Some of these companies were already plastics processors (PVC, LDPE), while others saw the manufacture of HDPE pipes and sheets as an opportunity for a complete change of their production programme.

In 1956, for example, the first contacts took place between Dr. Müller of Hoechst and the then managing director of Carl Simon Söhne GmbH (now Simona AG), the recently deceased Dr. Wolfgang Bürkle. Attempts at manufacturing pressed HDPE sheets on existing leather presses were altogether successful and marked the beginning of the transformation of a leather processing company into a world market leader in plastic sheets, pipes and semi-finished products.

A similar change took place in 1958 when, with great entrepreneurial courage, Werner Strumann turned the then firm of Gröter (now egeplast Werner Strumann GmbH & Co. KG) from a jute processor into one of today's most innovative manufacturers of polyethylene tubes and pipes. In that same year, the company was already producing not only the famous Hula Hoops (Fig. 10) but also pressure pipes for drinking water supply systems.

Fig. 10. Production of Hula Hoops at Gröter in Emsdetten (by courtesy of egeplast)

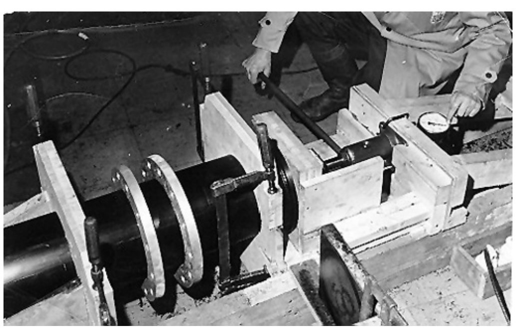
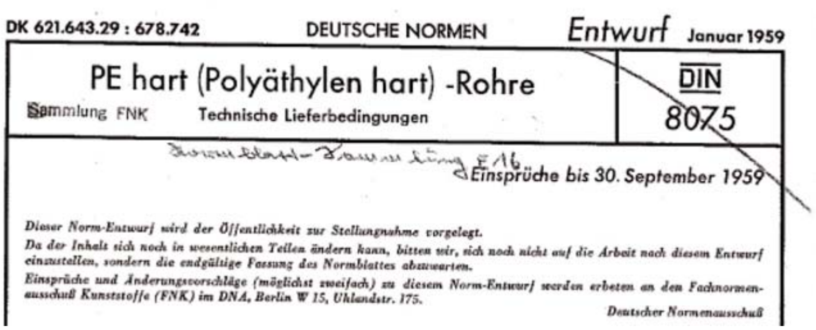


Fig. 11. Wooden jig used as a fusion aid for stub ends (by courtesy of KWH Pipe)

In the early 1950s, the former firm of Wiik & Höglund in Finland (now KWH Pipe) was already producing pipes in LDPE. When import restrictions were lifted in 1957, this company lost no time in adding the newly developed HDPE to its production range. It was precisely in the field of large-bore pipe extrusion that Wiik & Höglund accomplished pioneering groundwork. Other pioneers of the first hour were Omniplast in Ehringshausen, Dynamit Nobel in Troisdorf and Mannesmann in Hamburg – and this list makes no claim to being comprehensive.

STANDARDIZATION AND QUALITY ASSURANCE FURTHER STRENGTHEN CONFIDENCE

As HDPE pipe systems very quickly developed to become a widespread industrial commodity, the corresponding standards were not long in coming. A DIN study group was formed in 1957 with the collaboration of six German companies: Badische Anilin- und Soda-Fabrik, Chemische Werke Hüls, Dynamit-Aktiengesellschaft, Farbwerke Hoechst, Mannesmann and Rhein-Plastik-Rohr [19]. The result was the first draft of DIN Standard 8074/8075 – Rigid PE (rigid polyethylene) – Pipes, dimensions and technical conditions of delivery, which was published in January 1959 (Fig. 12).


DK 621.643.29 : 678.742 DEUTSCHE NORMEN Entwurf Januar 1959

PE hart (Polyäthylen hart) -Rohre

Sammlung FNK Technische Lieferbedingungen

DIN 8075

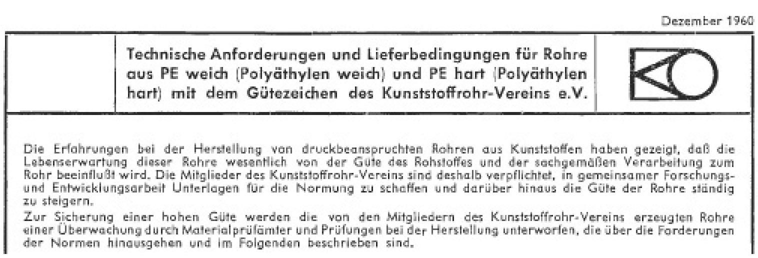
Einsprüche bis 30. September 1959

Dieser Norm-Entwurf wird der Öffentlichkeit zur Stellungnahme vorgelegt.
Da der Inhalt sich noch in wesentlichen Teilen ändern kann, bitten wir, sich noch nicht auf die Arbeit nach diesem Entwurf einzustellen, sondern die endgültige Fassung des Normblattes abzuwarten.
Einsprüche und Änderungsvorschläge (möglichst zweifach) zu diesem Norm-Entwurf werden erbeten an den Fachnormenausschuß Kunststoffe (FNK) im DNA, Berlin W 15, Uhlandstr. 175.

Deutscher Normenausschuß

Fig. 12: First drafts of DIN standard (by courtesy of the German Institute for Standardization)

In 1958, an ISO working group (ISO TC5/SC 6) organized a symposium in Amsterdam where experts from five European countries exchanged their experiences in the quality and production control of plastic pipes. Founded in Düsseldorf in July 1957, the Kunststoffrohr-Verein e.V. (now Kunststoffrohrverband e.V.) set itself the task of monitoring the quality of plastic pipes and already began to award quality marks in those early days. The first quality guidelines for HDPE pipes came out in December 1960 (Fig. 13) [20]. The method of determining melt viscosity (now MFR) was still governed by British and American standards. The minimum time-to-failure for rigid PE pipes subjected to a temperature of 80 °C and a hoop stress of 41 kgf/cm² was 48 hours.


Dezember 1960

Technische Anforderungen und Lieferbedingungen für Rohre aus PE weich (Polyäthylen weich) und PE hart (Polyäthylen hart) mit dem Gütezeichen des Kunststoffrohr-Vereins e.V.

Die Erfahrungen bei der Herstellung von druckbeanspruchten Rohren aus Kunststoffen haben gezeigt, daß die Lebenserwartung dieser Rohre wesentlich von der Güte des Rohstoffes und der sachgemäßen Verarbeitung zum Rohr beeinflußt wird. Die Mitglieder des Kunststoffrohr-Vereins sind deshalb verpflichtet, in gemeinsamer Forschungs- und Entwicklungsarbeit Unterlagen für die Normung zu schaffen und darüber hinaus die Güte der Rohre ständig zu steigern.
Zur Sicherung einer hohen Güte werden die von den Mitgliedern des Kunststoffrohr-Vereins erzeugten Rohre einer Überwachung durch Materialprüfämter und Prüfungen bei der Herstellung unterworfen, die über die Forderungen der Normen hinausgehen und im Folgenden beschrieben sind.

Fig. 13: Quality guideline issued by the Kunststoffrohr-Verein e.V. in 1960 (by courtesy of the KRV)

FURTHER DEVELOPMENT AND IMPROVEMENT OF HDPE PIPE MATERIALS

Ziegler's discovery marked the beginning of a long success story. It goes without saying that developments have not stood still, and certain chapters of this success story at least deserve brief mention. Although the basic chemistry of catalysts has changed very little during the last fifty years, it is precisely here that considerable progress has been made from the point of view of the plastics manufacturer. Catalyst yield has been increased many times over. One of the decisive quality criteria in the initial quality guidelines was the pH value, which indicated a possible residual catalyst content. This problem is now a thing of the past. Thanks to catalyst-specific research, the polymer structure of HDPE has been considerably improved. It manifests itself primarily in a balanced toughness/rigidity ratio.

Shortly after the introduction of Ziegler's HDPE pipe materials, PE pipe materials based on chromium catalysts had also been developed and were now marketable. As a result, PE materials of medium density (MDPE) and high density (HDPE) found themselves in competition with each other. MDPE materials generally manifest a larger number of side chains in their molecular structure than HDPE. Consequently, pipes manufactured from MDPE are tougher, but at the same time they have a lower rigidity, a property that is decisive for high axial strength and hoop strength.

A decisive breakthrough in polymerization came with the development of bimodal technology. The firm of Solvay Polyolefins was the first to introduce PE100 material around the end of the 1980s. This then necessitated a differentiation in the relevant standards between the three material classes PE63, PE80 and PE100. One spoke of materials of the 1st generation (PE63) and 2nd generation (PE80). With the introduction of the 3rd generation, characterized by the use of bimodal technology, it was possible to produce improved PE80 grades and, for the first time, PE100 grades (Fig. 14).

Further progress was achieved on the one hand through changes in co-monomer composition and, on the other, through the manufacture of multimodal pipe materials by means of a cascaded process (Advanced Cascade Process, ACP) using improved Ziegler catalysts [21]. HDPE produced with this technology features a microstructure of short-chain, highly crystalline areas (they are responsible for the necessary long-term strength of 10 MPa required for PE100 classification) and long-chain areas with selectively built-in branchings (they are responsible for the high resistance to stress cracking and rapid crack propagation. The service life extrapolated from the results of tests carried out on pipes manufactured from this high-performance HDPE is in the region of 100 years. The designation "4th generation" is already going the rounds.

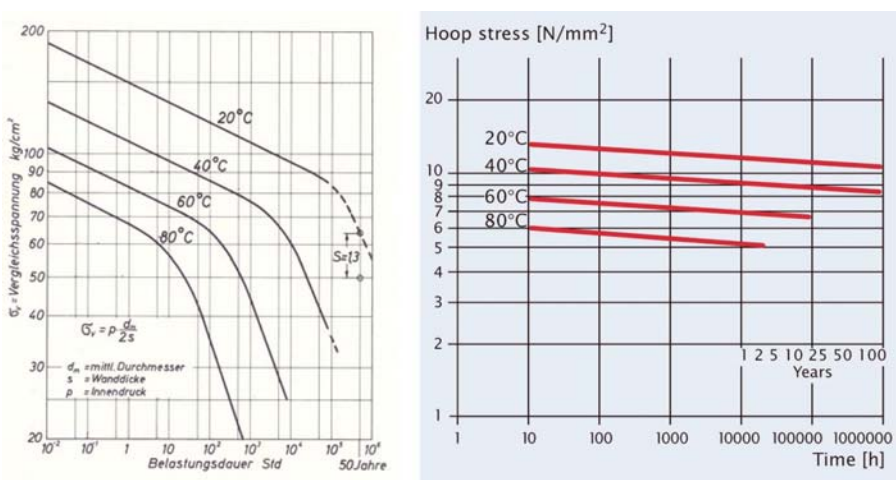


*Fig. 14. Creep curves for **Hostalen** GM5010 (left, 1st generation) and **Hostalen CRP** 100Black (right, 3rd generation)*

Definite progress has also been achieved in additive technology. The beginning of the 1980s saw the introduction of a new generation of antioxidants that were able to increase time-to-failure through heat ageing many times over. The processing stability of the materials has been increased. New types of carbon black have a lower tendency to absorb water. All the same, one must not ignore the fact that pipes manufactured from materials of the 1st generation are still in use today. Users report that old mains pipes – provided that they were laid properly in the first place – show no signs of adverse damage!

PIPES FOR DOMESTIC AND INDUSTRIAL WATER, SEWAGE AND GAS

By reason of their many advantages, HDPE pipes were very quickly accepted by the market. Domestic water supply systems were one of the first areas of application. A survey carried out in 1961 by the German Association of Gas and Water Engineers (Deutscher Verein von Gas- und Wasserfachmännern e.V.) was highly informative [22]: Of the 1085 water supply companies covered by the survey, 599 replied. These replies represented 43.7% of the total German water supply industry. 522 companies stated that they used plastic pipes (Table 2). Concerning the methods used for joining the pipes, 123 companies stated that they used metal connectors when laying HDPE pipes. Only one company was already using heated tool fusion technology.

Material	Pipe length km
Rigid PVC	836
Flexible PE	601
Rigid PE	674
Total	2.111

Table 2. Overview of the total length of plastic pipe (in kilometres) laid during the period from 1957 to 1960 according to the DVGW survey of 1961 [22]

Since 1960, HDPE pipes have been used to an ever increasing extent in the USA for gas supply systems. The first German gas supply company to follow suit – Main-Gas, Frankfurt – has been using HDPE pipes since 1971. Today, domestic water and gas supply systems would be unthinkable without HDPE pipes. For the renewal and extension of its waste water system, Farbwerke Hoechst began using HDPE pipes in 1962. By 1965, the total length of HDPE pipes laid was 35 km. Pipes with diameters of up to 1000 mm were extruded, while filament winding was used for pipes up to 1400 mm diameter [23]. The company's experiences with these pipes have been most positive [24].

Since these beginnings, hundreds of other HDPE pipe projects have been successfully implemented worldwide. Today, HDPE pipes can be extruded in diameters of up to 2 m, while the limit for spirally wound pipes is around 4 m. The latter are used not only for a multitude of industrial applications but also, for example, for sea discharge pipelines, storm water reservoirs and all kinds of protective conduits.

OUTLOOK

A total of 1.28 million tonnes of HDPE was processed into pipes for outdoor applications in Europe in 2005. These applications are broken down as follows:

- Drinking water 411,000 tonnes
- Gas 167,000 tonnes
- Sewage pressure pipes 44,000 tonnes
- Non-pressure sewage pipes 250,000 tonnes
- Cable conduit 139,000 tonnes
- Others 270,000 tonnes

A whole multitude of positive project reports on the use of HDPE pipes bears eloquent witness to the success of these systems. Although the earliest pipes have completely come up to expectations as regards their service life, the development of both materials and systems has certainly not reached its zenith, for raw material producers are committed to the constant improvement of their products, as the member companies of the PE100+ Association clearly demonstrate. Their endeavours are based on the recognition that HDPE is ideal for the manufacture of pipes and can be readily processed. Pipes manufactured from HDPE give reliable service and have high safety reserves – in short: they are indispensable when it comes to the low-cost, environmentally friendly manufacture of pipe systems capable of meeting the highest demands.

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