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POLYAMIDE 12
FOR HIGH PRESSURE GAS INSTALLATIONS

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Abstract

PA 12 is a high performance polymer with outstanding mechanical properties and excellent chemical stability. It is the preferred material in many demanding applications, e. g. in the automotive industry for fuel lines of passenger cars or for air brake tubings in trucks. Compared with polyethylene (HDPE) it has a higher mechanical and impact strength and melting temperature. That is why it can withstand higher pressures and temperatures. It also has a much lower gas permeability and is easy to be processed in extrusion and injection molding.

For more than 20 years PA 12 have been evaluated as a pipe material for gas distribution and for more than 10 years PA 12 pipes have been used in Australia, Poland and Chile for low pressure installations. In recent years PA 12 is being investigated for operation pressures above 10 bar. Since 2002 a working group within the ISO/TC 138 is elaborating standards for PA 12 gas installation systems with operation pressures up to 20 bar. In USA the ASTM F17 committee is working on the standardization of PA 12 for gas installation and the Gas Technology Institute (GTI) has set up a comprehensive test program for a feasibility study of PA 12 gas pipes with diameters up to 6" (168 mm OD, SDR-11). This program is sponsored by 15 major gas utilities in USA and will last 27 months including some test installations in various states which are scheduled for the beginning of 2006.

Degussa AG, Marl, Germany, as one of the four manufacturers of PA 12 and E.ON Ruhrgas are cooperating on a first test installation of a 60 m pipe system (110 mm OD, SDR-11) in the technical center of E.ON Ruhrgas in Dorsten, Germany. The system with two butt fusion, two electro-fusion joints, and electro-fusion end caps was checked 72 hours at 36 bars (3.6 MPa, 520 psig) and is operating now for 2 years at 24 bar (2.4 MPa, 347 psig). In the oral presentation a short video will be presented demonstrating processing of the pipe and installation on the E.ON Ruhrgas Technical Center.

The authors will give an introduction to the excellent technical performance of PA 12 and demonstrate the superior cost position of high pressure PA 12 gas pipes with respect to steel. PA 12 is technically and economically the material of choice for gas distribution at 10 - 20 bar (1 -2 MPa, 145 - 290 psig).

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1. PA 12 – A HIGH PERFORMANCE THERMOPLASTIC MATERIAL

Due to the specific carbon-amide group in the polymer chain polyamides (PA) have strong inter-molecular actions which induce high mechanical strength, high melting temperatures and chemical stability. The "long chain" PA 12 with 11 carbon atoms between carbon-amide groups has the lowest water absorption of all commercial available PA and represents the best compromise in thermal and mechanical properties. That is why PA 12 is the material of choice for some challenging applications relied on already for decades, e. g. in the automotive industry for fuel lines of passenger cars or for airbrake tubings of trucks.

Compared with medium and high density polyethylene (MDPE, HDPE) in use for low pressure gas supply, "long chain" polyamides like PA 12 provide "naturally" superior performance due to their described chemical structure (table 1). Besides PA 12 only PA 11 is commercially available with almost identical properties. PA 11 supplied by one manufacturer is based on a planted feed stock, castor bean, while PA 12 is synthesized from butadiene, a crude oil by-product, in a complicated multi-step process. Degussa is the only company of the four PA 12 suppliers who is fully back-integrated to butadiene.

Property	**PA 12**	**HDPE**	**MDPE**
Melting temperature (°C)	178	130	126
Tensile strength at yield (MPa) Tensile elongation at break (%) Flexural Modulus (MPa)	45 >200 >1200	20 – 23 >800 950	17 – 19 >800 700
Charpy impact strength (kJ/m^2)	No break!	30	20
Hardness, Shore D	74	63	58
Permeability (23 °C, mm^3/bar/day) Methane Hydrogen *(data for PA 12 gas pipe grade pending)*	< 0.005 < 0.01	0.7 0.24	

Table 1: PA12 - PE basic properties

2. PA 12 - A MATERIAL OF CHOICE FOR HIGHER PRESSURE GAS SUPPLY

There are always two major concerns about thermoplastic materials used in gas supply: the creep rupture process and occurrence of a fatal crack. Due to the intermolecular forces in PA 12 the hydrostatic strength of PA 12 pipes is much higher than for HDPE, even for the optimized grade PE 100. Long term hydrostatic strength investigations according to ISO or ASTM standards have proven that in a 50 years extrapolation PA 12 is able to operate at pressures up to 20 bar using a safety factor of 2 for gas, in the ASTM society a coefficient of 0.4 (table 2). In contrast to PE PA 12 is naturally resistant against stress cracking in general and slow crack growth and passes easily all the relevant tests originally created for this weakness of PE.

For the time being resistance against rapid crack propagation (RCP) at low temperatures is a matter of concern and discussion in the gas utilities. In the ASTM territory RCP is under consideration. RCP data have to be provided but they are not limiting the listings for pipe materials from the Plastic Pipe Institute for use in the USA. In the European standards and also adopted in the ISO standard the maximum operation pressure (MOP) for a pipe material is limited from two sides, the hydrostatic strength of the material and the MOP derived from a RCP test at 0 °C. For bigger pipes with thicker walls the MOP was limited from the RCP test results. A simplified laboratory test (S4-test, ISO 13477) using a shorter pipe sample was used and a correlation formula to a full scale tests on a pipe of “indefinite” length which was formerly developed for PE pipes (ISO 13478).

Recently this formula was determined for PA 12 and PA 11 from independent certificated test institutes. Using these PA specific correlations it could be proven, that for PA 11/12 the MOP calculated from S4-test data, even with the arbitrary safety factor of 1.5 is no more the limiting factor for the MOP. This is investigated so far for pipes with sizes up to 168 mm SDR-11. (Remark: On the transmission date of this paper-preprint, Jan. 31st, 2006, the correlation formulas were under re-confirmation and not published, final formulas will be presented on the conference).

In USA MOP for PA 11 and PA 12 pipes were derived from the so called Hydrostatic Design Base (HDB) determined according to ASTM D2837-02 : “Standard Test Method for Obtaining Hydrostatic Design Basis for Thermoplastic Pipe Materials or Pressure Design Basis for Thermoplastic Pipe Products“. The procedure for evaluating the hydrostatic strength limits for a thermoplastic pipe material is quite similar to the ISO 9080 “Plastics Piping and Ducting Systems - Determination of the Long-term Hydrostatic Strength of Thermoplastics Materials in Pipe Form by Extrapolation”. With a (safety) design factor of 0.4 (corresponding with a design coefficient of 2.5 in the ISO) for PA 12 the following MOP for pipes with SDR-11 (Standard Dimension Ratio = outer diameter/wall thickness =11) were calculated (Table 2).

MOP in bar (SDR-11 pipes)	**23 °C**	**60 °C**	**80 °C**
MDPE	4	4	0
HDPE	10	7	0
PA 12	>16	>10	>8

Table 2: Maximal Operation Pressures for PA 12 and PE based gas pipes

These figures demonstrate the superior performance of PA 12 over PE at ambient temperatures and the safety reserve at elevated temperatures, where PE is falling out of service.

3. TECHNICAL POSTIONING OF PA 12 BETWEEN PE AND STEEL

Steel has a long history in gas installation and it is still the only option for real high pressure gas transportation at 50 bar or higher. Reinforced plastic pipe constructions might be feasible to serve systems possibly with pressures up to 45 bar (Lit. 1).
Welding of steel is a well established technology and weld quality control procedures are in place. Also with respect to third party damages steel has naturally a high stability. On the other side steel pipes are rigid, heavy and corrosive, which makes them principally not attractive for installations.

In Europe and USA the gas distribution networks are operating at pressures of up to 25 bar, with a big share around 16 bar. Up to now only steel is approved for gas distribution. PE100 as the latest development of High Density Polyethylene (HDPE) received the approval for operating pressures up to 10 bar however this comprises only a small share of distribution networks. As described before PA 12 pipe systems are technically able to carry gas at pressures up to 20 bar at ambient temperatures and withstand elevated temperatures up to 80 °C, even in a longer term. This would fit very well to serve distribution networks running at a pressure of 16 bar, provided PA 12 installations are compatible with steel in terms cost.

4. ECONOMICAL POSITIONING OF PA 12 BETWEEN PE AND STEEL

For a realistic gas network cost analysis the systems based on different pipe materials have to be analyzed on a comparable base and including all the costs over the lifetime. From the utilities point of view networks with the same gas transportation capacity have to be compared. Degussa had asked a gas engineering consulting bureau in Germany to work out a cost analysis and comparison for HDPE (PE100), for steel with and without active cathodic corrosion protection and for PA 12. For all materials systems carrying 3000 Nm^3 per hour are compared. PE100 pipe operating at 10 bar the maximal approved pressure, PA 12 and steel running at 20 bar. For the named capacity the pipe dimensions have to be chosen adequately: Steel D100, PA 12 D110 and PE100 D140 mm, both plastic pipes SDR-11. There is a series of contributions to the overall installation and operation costs. In fig. 1 they are listed and presented for the three systems in comparison. The specific costs are taken from the actual situation in Germany.

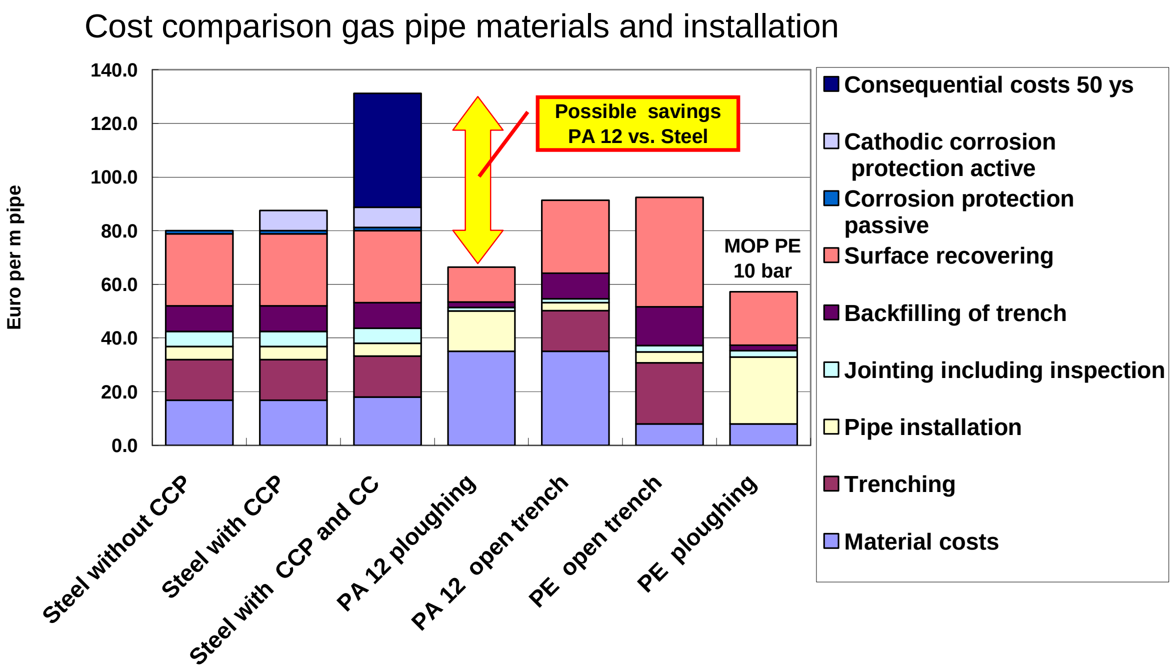

Fig. 1: Cost comparison Steel vs. PA 12 and HDPE, calculated for same gas transportation capacity of 3000 Nm^3

Material prices play of course a role. The used steel price is a taken from a long term rather stable situation some years ago. Actually it is significantly higher due to shortages on the world market. For PE100 the actual average market price was introduced in the calculation. For the new developed special PA 12 gas pipe compound a price of 10 Euro per kg was fixed for this calculation.

The diagram shows clearly the cost driving and saving factors for the systems. Installation of steel pipes is only possible using straight pipes of 12 m maximum length. They are laid down and welded in an open trench. The corrosion protection is a very cost effective component in the steel system calculation and in spite of all corrosion protection efforts over the life time rather high maintenance costs have to be carried for the steel networks. These “consequential cost” figures were picked up from statistics published from the German gas utility society.

For plastic pipes there are three main cost advantages: No corrosion protection, supply of coiled pipe up to 400 m in one piece and in optimal field preconditions installation is done in a more or less continues process ploughing a trench inserting the pipe in the trench and refilling the trench, all done simultaneously with specialized machines. Thus up to 400 m of pipe can be buried in line in a rather short term. Especially in remote areas with easy, non-rocky soils this is a highly efficient and cost saving procedure.

While PE gas pipe has to be buried in a sand bed for PA 12 it is expected that this is not necessary because of its high resistance to point loads applied from stones in the ground. Special approvals for sand-free bedding are ongoing. This opens all options for the ploughing technology and cost savings.

To make it clear, principally PA 12 is not competing with PE. It is the material of choice for serving the 16 bar networks, where PE is out of discussion. Only if other superior properties of PA 12 are requested PA 12 might interfere in the PE gas supply business.

5. FIELD EXPERIENCE WITH PA 12/11 IN GAS INSTALLATIONS

Polyamides have already a longer field experience for rehabilitation and new lines. Already in the 1980s PA 11 was introduced in the gas supply networks in Australia. There are some 10,000 km of low pressure installations. PA 12/11 was chosen because of its good stability against the aggressive synthetic gas in use. In the 1990s also PA 12 from one PA 12 supplier was approved in Australia and is in use now for many years. The pipes were partly joined using PA solvents for solution welding. The lines are doing their job without any reported failures. PA was also approved for single low pressure installations in Poland and Chile.

In USA PA 11 has been promoted for higher pressure gas installations since the 1990s. Seven test installations with 51 mm (2”) SDR-11 were installed in different climatic zones of the USA and in different grades of soils operating up to 10 bar. The fist test installation was buried in 1997.

6. STANDARDIZATION PROCEDURES ON PA 12/11 FOR GAS SUPPLY

The international recognition of PA 12/11 for gas is in progress in all developed regions of the world. National standards were issued already in the 1980s in Australia. The ASTM society has been working on the standards since the later 1990s. The ASTM F17 committee has listed PA 11 (PA 12 is about to follow in 2006) in Annex A5 of ASTM D2513 “Standard Specifications for Thermoplastic Gas Pressure Pipe, Tubing, and Fittings”. An ISO working group within the ISO/TC 138 SC4 started in 2002 with participants from USA, France, Switzerland, Germany, Japan and Australia including representatives from all PA 12/11 manufactures to issue the full set of regulations for gas systems made of PA 12 and PA 11 in ISO 22621 “Plastics piping systems for the supply of gaseous fuels for maximum operation pressure up to and including 2 MPa (20 bar) – Polyamide (PA)”. “Part 1: General” and “Part 2: Pipes” of are now in the DIS status. In Germany the DVGW (Deutscher Verband fuer das Gas- und Wasserfach) has started with a working group on PA for gas and on the European platform GERG was recently attracted to investigate PA 12/11 for higher pressure gas supply.

7. New VESTAMID® GRADE FROM Degussa AG, PA 12 DESIGNED FOR BIG PIPES AND HIGH PRESSURE GAS DISTRIBUTION

Controlling molecular weight and intermolecular interacting forces is a Degussa core competence to design PA 12 extrusion grades with optimized processing and performance properties. Degussa PA 12 is sold under the registered trade mark VESTAMID® L (L for laurolactam, the monomer for PA 12). When Degussa decided to join the activities of the PA 12/11 manufactures for the high pressure gas pipes application a new high molecular PA 12 grade for extruding pipes with bigger sizes was designed.

For various test series at gas institutes and for test installations pipes with dimensions up to 168 mm (6”) SDR-11 have been manufactured so far using this new grade at two leading pipe extrusion companies without any problems. According to the extrusion teams the VESTAMID gas pipe grade

can be handled and processed like the polyolefine gas pipe materials. Only the different shrinkage behavior of the PA 12 has to be taken into account in the settings at the calibration.
It turned out in the gas pipe specific tests that this VESTAMID® grade provides an optimized performance with respect to processibility, hydrostatic strength and cold impact properties (RCP).

8. Short and long-term performance tests on VESTAMID® gas pipes

According to ISO 22621 “Plastics piping systems for the supply of gaseous fuels for maximum operation pressure up to and including 2 MPa (20 bar) – Polyamide (PA)” there is a series of requirements for the materials (PA 12/11 compounds) and for manufactured pipes to be fulfilled.
Some of them can be tested in a short-term other need a longer distinct time to accomplish.

Characteristics	Requirements	Test parameter	Test method	VESTAMID Gas Pipe Grade
Hydrostatic strength at 20 ℃ at 80 ℃	No failures in test period No failures in test period	Hoop Stress: 19.0 MPa/1000 h 11.5 MPa/165 h	ISO 1167	Passed Passed
Resistance to Slow Crack Growth (Notch Test)	No failures in test period	80 ℃ 2 MPa/20 bar 500 h	ISO 13479	Passed
Resistance to Rapid Crack Propagation (new correlation p_c full scale / S4 Test for PA)	pc >= 1.5 MOP with $p_c = 7.8\ p_{c\ ,\ S4} + 6.8$	0 ℃	ISO 13479	For 168 mm SDR-11 MOP = **24 bar !**

Table 3: Selected test results according to ISO standards on VESTAMID® gas pipe samples collected in preparation for the approval projects in Europe

9. Joining and flow control of VESTAMID® gas pipes

In gas installations every joint, transition, tee, and other is a critical point. Welding of steel pipes and the quality control of welds are well established. For plastics pipes procedures and tools for butt fusion of pipes are standardized and training of technicians is a requirement of the gas utilities. For low pressure systems mainly served today by MDPE and HDPE pipes mechanical fittings made of PA 11 or PA 12 are in use because of their higher strength, however only for installations in the ASTM territory USA. Injection molded fittings with inserted heating wires for electro-fusion actually have the highest respect in the gas society because of their conceptional high reliability. Standardized electro-fusion parameters are simply read in by bar codes in power generators offer more safety and failure prevention.

Friatec AG, one of the world market leaders for electro-fusion fittings, has developed various fittings, end caps and tapping valves with the Degussa VESTAMID® grade for gas pipes. Standard electro-fusion automats have been used to perform installations.

Butt fusion with standard equipment and slightly modified temperature programs have been carried out numerously with PA 12. Although water absorption of up to 1.5 % (saturation) is possible there is no inferior effect in fusion strength observable. Squeeze off and re-rounding tests of PA 12 gas pipes were successfully passed in the feasibility study reported below.

10. Feasibility study of PA 12 for high pressure gas supply in USA at GTI

Since the later 1990s PA 11 has been investigated in the USA for higher pressure gas distribution and is under observation in a series of test installations with operating pressures up to 10 bar (125 PSI).

Sponsored by 15 big gas utilities in the USA Gas Technology Institute (GTI) in Des Plaines/IL invited in 2004 all four PA 12 manufacturers to participate in a feasibility study for high pressure gas supply. The motivation was to broaden the choice of suppliers for high performance PA, not only to be restricted to a single manufacture of PA 11.

Degussa joints the program and delivered 2" and 6" SDR-11 pipe samples manufactured from the new designed high molecular PA 12 for big pipe extrusion. GTI had set up a project plan for 27 months including comprehensive short term and long term tests relevant to the targeted application and finally several test installations in various states of the USA.

Characteristics	**Test Parameter**	**Test Method**	**Requirement**	**VESTAMID Gas Pipe Grade**
Burst behavior	Quick burst pressure within 60-70 sec 23 °C (74 F)	ASTM D2513/ D1599	>3900 psi > 27 MPa	6899 psi 47.9 MPa
Flexural modulus	3 point bend 23 °C (74 F)	ASTM D2513/ D790	1000 MPa	1470 MPa
Apparent tensile strength	Split ring specimen 0 °C (32 F)	ASTM D2513/ D2290	> 3900 psi > 27 MPa	7086 psi 48.9 MPa
Chemical resistance to mineral oil, toluene (15%), methanol, TDM (5%)	Change of strength Change of mass	ASTM D2513/ D543	Defined limits	all passed

Table 4: Short term test results from a feasibility study for high pressure VESTAMID® gas pipes according to ASTM standards in USA at GTI/Des Plaines, IL

Characteristics	**Test Parameter**	**Test Method**	**Requirement**	**VESTAMID Gas Pipe Grade**
Hydrostatic Design Base 23°C, 60°C, 80°C	Longterm Hydrostatic Strength	ASTM D 2513 / D2837 / D1598	2500 or 3150 psi 17.4 or 21.7 MPa at 23 °C	After 2000 h of 10,000 h on track for min. 2500 psi
Slow Crack Growth Notch 20 %	No failure in test period	ASTM D2513 / ISO 13479	> 877 h at 1848 psi/12.8 MPa	> 2000 h
PENT Test	80 °C, 2.4 MPa	ASTM F1743	> 100 h	> 10 00 h
Rapid Crack Propagation	RCP at 0 °C	ISO 13477 new correlation for S4- to full scale test for PA	Crack arrest	168 mm (2") SDR-11 MOP = **24 bar !**

Table 5: Long term test results from a feasibility study for high pressure VESTAMID® gas pipes according to ASTM standards in USA at GTI/Des Plaines, IL

All tests have been passed successfully, the determination of the Hydrostatic Design Base for the listing of the VESTAMID gas pipe grade at Plastic Pipe Institute (PPI) in USA is still ongoing. Intermediate data extrapolated to 50 years performance indicate a listing of minimum 2500 psi (enough for MOP of > 17 bar including a design factor of 0.4).

11. DEGUSSA COOPERATION WITH E.ON Ruhrgas IN GERMANY ON EVALUATING PA 12 FOR HIGH PRESSURE GAS Supply

Degussa and E.ON Ruhrgas decided in 2005 to set up together a high pressure test installation on the E.ON Ruhrgas Technical Center site in Dorsten, Germany. Degussa had to deliver 60 m of 110 mm SDR-11 VESTAMID® pipe on a coil (!) and some straight pipes for assembling a system including two butt fusion and two electro-fusion joints. Degussa also had to provide electro-fusion end caps.

For the extrusion and coiling of the requested pipe Degussa choose the company Egeplast in Greven, Germany. Although having PA 12 the first time on their production line Egeplast was able to extrude the pipe within the tolerances and with excellent appearance without any problem. The online coiling on a 2.5 m diameter drum was running smooth without cranking the pipe on the coil at all (a movie of the extrusion and coiling will be shown on the conference).

Fig. 2: Coil of 60 m VESTAMID gas pipe D110, SDR-11

For the development of electro-fusion fittings and end-caps Degussa cooperated with company Friatec AG in Mannheim, one of world leading companies in electro-fusion fittings. Using the VESTAMID® gas pipe material they manufactured the required components for the test installation in a perfect manner.

Fig. 3: VESTAMID electrofusion fittings

Before the test installation on the E.ON Ruhrgas site a burst pressure test was carried out with a 3 m test pipe including a butt fusion and an electro-fusion joint and electro-fusion end caps. In a short term burst pressure test the system bursts at 93 bar (!) in a ductile crack in the tube region, while the fittings withstand the loads unaffected (fig. 4).

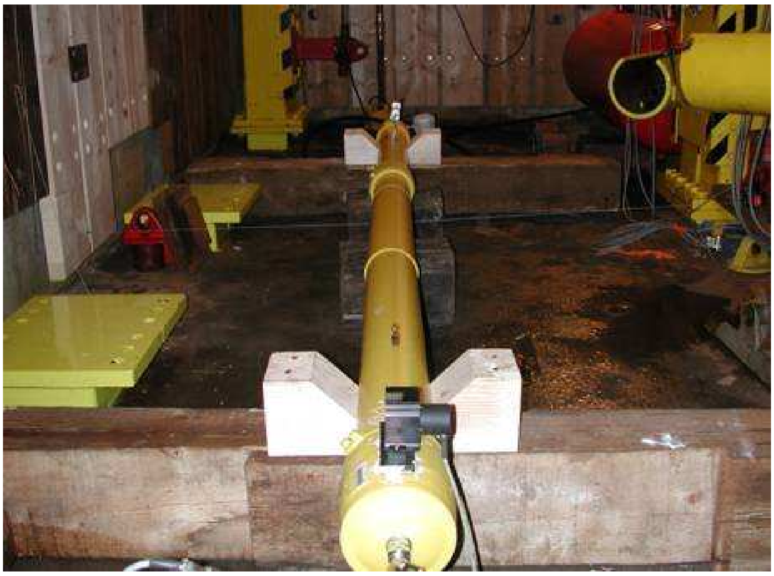

Fig. 4: VESTAMID gas pipe D110, SDR-11, with joints, quick burst test,

Fig. 5: VESTAMID gas pipe D110 mm, SDR-11, unrolled for test installation

For the test installation the coiled tube was unrolled in the field without any mechanical stretching tool. Butt fusion was carried out with standard equipment and slightly adjusted temperature profiles. For the electro-fusion fittings standard power generators from Friatec AG were used. The adjusted fusion conditions were read from a bar code adhered to the fittings. The system with one butt fusion and one electro fusion joint and two electro-fused end caps was installed and sealed in a 500 mm steel pipe. For 72 hours 36 bar (!) natural gas was applied for checking the tightness of the system. Then the pressure was lowered to 24 bar. This pressure will be applied for 2 years and the installation is supervised continuously with respect to constant pressure, leakages and gas permeation.

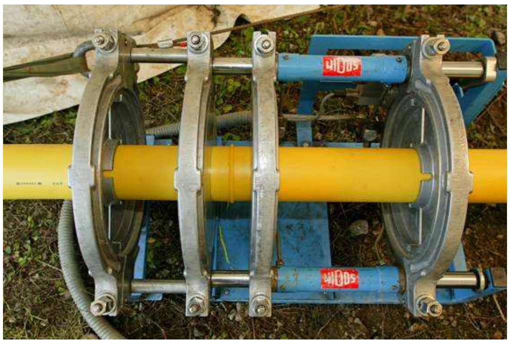

Fig. 6: Butt fusion joint

Fig.7: electro-fused end cap with gas connection

Final remark:

After finishing the current feasibility studies and approval processes with convincing results the gas communities all over the world should consider PA 12 for their distribution networks. Degussa is searching for more cooperation partners to develop more and more confidence in this outstanding material.

References

1: Wessing, W., Grass, K., Kanet, J., and Capdevielle, J-P., (2004), “Novel PE gas supply system for a maximum operation pressure of 16 bar”, conference book, Gasresearch Conference 2004, Vancouver).