

Mechanical behaviour of machined polyethylene filaments subjected to aggressive chemical environments

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1. Introduction

Polyethylene tubes made out of high density resins (HDPE) are largely used in distribution and transmission of natural gas and water. The choice of polyethylene (PE) for manufacturing of gas and water distribution networks rises from the many technical and economic advantages presented by such polymeric materials. PE is a resistant and lightweight material, which facilitates the handling operations and implementation for above and underground applications. It has a good corrosion resistance; whatever the conditions on the ground might be and it makes it possible to avoid the extra costs due to passive and active protection coatings associated with steel pipes. Moreover, PE systems support efficiently the effects of the ground movements due to soil instabilities and high temperature gradients. Because of their good resistance to cracking, the PE pipes have a high reliability level for normal conditions of use. In such cases, their lifespan is estimated at more than 50 years on the basis of bursting tests used to build a regression curve correlating stress level and failure times [1]. Because the creep phenomenon of polymers can be important even at ambient temperature, the effect of deformation on polymers widely differs from that of structural metals as it is not only a function of the magnitude of the stress but also of exposure time. Lifetime management of underground pipelines is mandatory for safety and the use of HDPE tubes subjected to internal pressure, external loading and environmental stress cracking agents requires a reliability study in order to define the service limits and the optimal operating conditions. In service, the time-dependent phenomena especially creep, lead to significant strength reduction. In recent work, a reliability-based study of pipe lifetime model was carried out to propose a probabilistic methodology for lifetime model selection and to determine the pipe safety levels as well as the most important parameters for pipeline fracture toughness [2], reliability [3], impact failure and fatigue when transporting water, steam and hydrocarbons [4]. Moreover, the temperature and the application mode of the working loads have catastrophic consequences on the mechanical properties of the plastics compared to those of metals. Mechanical properties of HDPE tubular structures are the subject of several research works concerning various aspects,

such as the variation of physical and mechanical properties in relation to the molecular structure [5], the applied load patterns, the mechanisms of fracture and the effects of environments during service conditions [6]. The resistance of polymeric materials to chemical agents depends on the nature of polymer as well as additives processed during extrusion. The strongly oxidizing acids may chemically attack plastics, provoke fading and substantially degrade the mechanical properties. The organic liquids such as fuel oils, mineral oils and various organic solvents cause swellings, softening or finally the dissolution of polymer [7]. Another study treated the degradation of the surface active environment (Arkopal in de-ionized water) and it was found that failure times of PE pipes increased continuously with the age of the solutions. This is a good indication of how dangerous, could be the exposure to various chemicals. In addition, ranking of several PE pipe materials can be established based on the behaviour in aggressive environments [8].

The objective of this study is to present a new approach to investigate the mechanical heterogeneity through the wall thickness of a high resistance HDPE pipe using machined filaments. Also, the study is aimed at illustrating the interaction between HDPE and some chemical environments representing ground aggressiveness. Such type of tests and the obtained results are sought by PE manufacturers and users to understand the effects of aggressive environments and to find ways to protect buried gas and water network from environmental stress cracking (ESC).

2. Experimental approach

2.1. Material

The material used in this study was extruded by STPM CHIALI Co. of Sidi-Bel-Abbès (Algeria). It was donated in the form of tubes which are intended for the transport and the distribution of natural gas. The HDPE resin was obtained by addition polymerization, whereas the tubes were obtained by co-extrusion. Typical properties of such an HDPE-100 polymer are provided in Table 1 [9]. External diameter of the provided pipe (SDR=11) and wall thickness are 113 mm and 12 mm respectively.

Table 1
Some physical properties of HDPE-100

Property	Value
Density	0.95 to 0.98 g/cm ³
MFI	0.75 g/10 min
Young's modulus	0.55 to 1 GPa
Yield strength	20 to 30 MPa
Toughness	2 to 5 MPa m ^{1/2}
Vitreous transition temperature	300 K
Softening temperature	390 K
Thermal expansion coefficient	150 to 300 m K ⁻¹
Oxydation stability	≥ 20 min
Resistance to cracking in surface-active environment	≤ 15 mm/day

2.2. Specimen preparation

In order to measure the mechanical properties in every layer within the pipe, it is needed to prepare specimens with the following criteria: (1) the specimens should be directly extracted from the pipe so that to preserves the

build-in thermo-mechanical history, (2) they must obey a reproducible preparation methodology and (3) structural morphology disturbances should be kept to minimum through the reduction of contact stresses during the automatic machining operation. Several cutting conditions were tried with a machining program to obtain the most regular filament section for a through wall turning. A fitted wooden mandrel was fabricated to support internally the pipe and thus, the lathe spindle firmly held both the pipe and mandrel without damaging the clamped portion as shown in Fig. 1, a. To avoid any radial displacement of the revolving assembly, the mandrel was also supported by a headstock at the other extremity. Filament cutting was performed continuously in the radial direction (Fig. 1, b) with a right carbon steel tool at a low spinning speed of 45 rpm, a depth of cut of 4 mm and a feed rate of 0.5 mm/rev. A batch of 5 test specimens of dimensions 120x4x0.5 mm³ is depicted in Fig. 1, c. A microscopic examination permitted us to verify the uniformity of the filament edges and the rectangular sectional area [10].

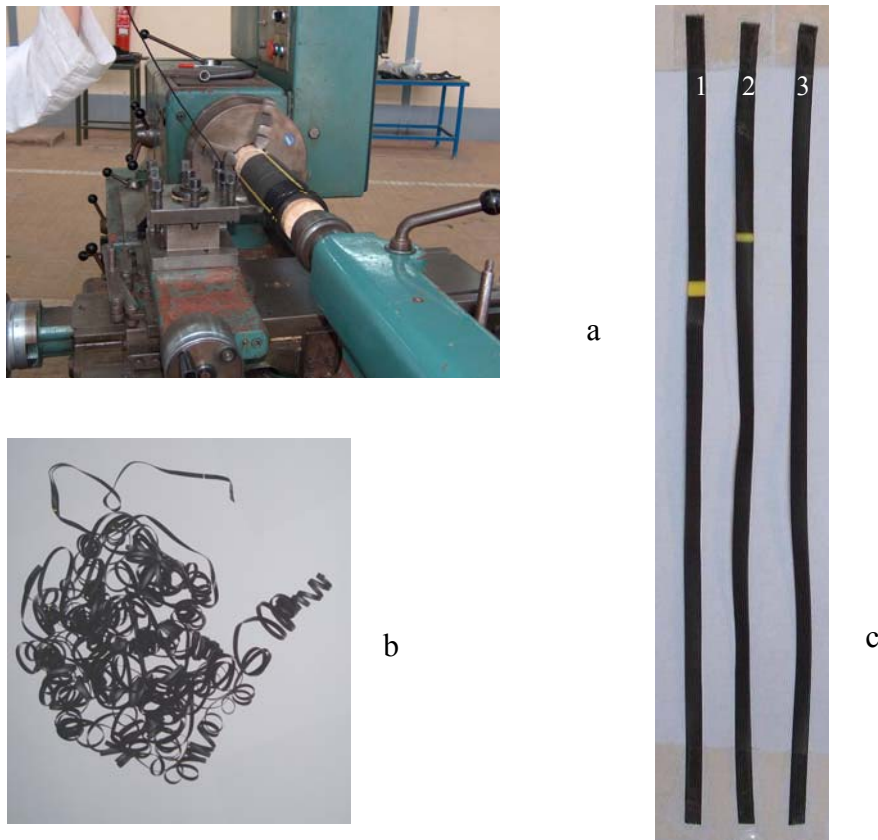


Fig. 1: a) machining of PE filament, b) prepared filament, and c) prepared set of specimens

2.3. Experimental procedure

The filament specimens as illustrated in Fig.1, c were subjected to monotonic tensile loads using a Zwicki 1120 universal testing machine (Ulm, Germany) especially designed for polymer characterization and equipped with a 2 kN load cell. The TestXpert® software (Technosid-Zwick, Annaba, Algeria) controlled the experimental output data and recorded the checked information in real time through an RS232 computer interface. A 1.66 mm/s testing speed was used and the setup was monitored with a com-

puter program that allowed carrying out all tests in exactly the same way on the basis of the general recommendations of ASTM D-638. To obtain final failure of the filament, the gauge length was reduced from 64 to 40 mm to accommodate the specimen geometry with the machine's maximum crosshead displacement [10].

The selected conditioning environments are the combination of various chemical agents which can be present underground or transported with the fluid in contact of the pipe inner surface. The environments are sulphuric acid (H₂SO₄ at 20%), Algerian crude oil and a (50-50%) mix-

ture of toluene–methanol, while air environment is used as a reference for comparison. The specimens were immersed in tightly closed containers in contact with a given liquid environment for a period of 15 months. More than 152 specimens exposed to various corrosive conditions and to ambient air were tested in this study.

3. Results and discussion

The results presented in this study concern HDPE exposed to ambient air and to environments impregnated with chemical solutions. Tensile tests are presented in the form of stress strain curves at constant speed as illustrated in Figs. 2 and 3. Globally, the curves kept the same overall configuration. Primarily, 3 distinct zones are observed: (I) elastic, (II) cold drawing and (III) plastic hardening. The thorough examination shows behaviours which vary from one environment to another what indicates that the interactions are different due to each type of aggressiveness [11]. These curves illustrate that HDPE has elastic-plastic behaviour like the majority of polymers and is drastically influenced by chemical environments.

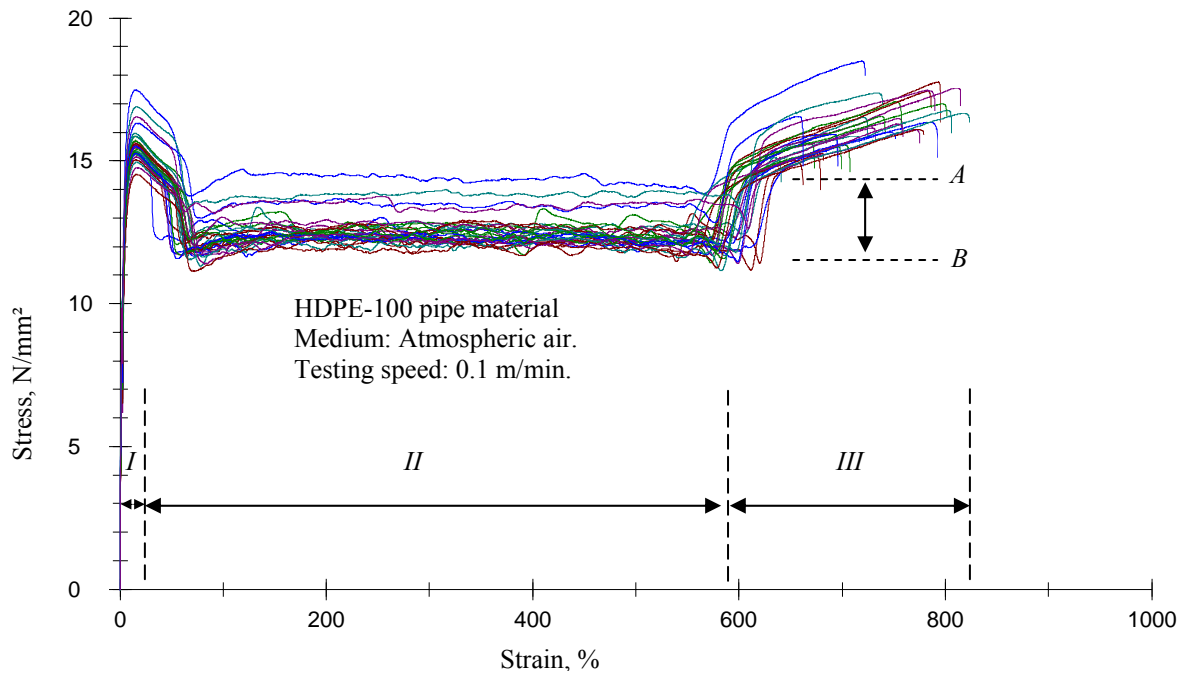


Fig. 2 Stress strain curves of HDPE resin (PE-100) in laboratory atmospheric air

3.2. Interaction with toluene-methanol

Fig. 3 presents the interaction of HDPE with a mixture of toluene-methanol which is known to be a good solvent of many materials. It is found that the yield stress is definitely smaller and the thresholds of matter flow (onset of drawing) occurred at slightly more important deformations compared to air results. The drawing phenomenon being located at around 11 MPa is appreciably shortened than that of air. With regard to the onset of plastic hardening, the slope is reduced; i.e. it becomes weaker indicating a direct effect of the mixture of both solvents on the polyethylene structural chains which are now completely oriented around 500% strain up to 900%. The strain at failure reached the 950% limit with less dispersion before rupture.

3.1. Stress strain behaviour in air

During and after machining, each HDPE filament is left to deform freely in order to avoid any further effects due to handling. This step is observed in the same way for all specimens and as a result, the inherent manufacturing residual stresses conferred a curly shape which is kept as it is until testing time (Fig. 1, b). Stress and deformation values were checked and they are similar to those obtained using standard test specimens. The spreading of the curves represented by zone II in Fig. 2 is a good indication on how much heterogeneity is contained in a polyethylene pipe wall since all the curves describe the whole pipe wall thickness (from outer to inner layer).

The mechanical behaviour of HDPE-100 under monotonous traction and which was exposed only to ambient air is depicted in Fig. 2. Three characteristic zones are observed: (I) elastic zone over less than 25% strain, (II) the zone of drawing propagation which extends roughly over 550% strain and (III) the zone of plastic over-drawing over approximately 250% of strain. Zone III is characterized by maximum material orientation and is terminated by tearing.

3.3. Interaction with crude oil

The interaction of HDPE pipe material with crude oil for a similar experimental set up shows substantial decreases in all mechanical properties relating to the state of stress. Indeed, yield stress, drawing stress threshold and average drawing stress are all smaller than those of the air case. The dispersion of the curves obtained for the whole thickness of the tube (from outer to inner surface) is rather important especially for zones (II) and (III) illustrated in Fig. 2. For instance, the average stress during the drawing phase is spread out between 9.5 and 12.5 MPa, whereas the nominal breaking stress reached approximately the value of 19 MPa. Zone III is laying roughly from 500% to 1500% strain and exhibits some dispersion compared to air and toluene-methanol mixture cases (Fig. 4).

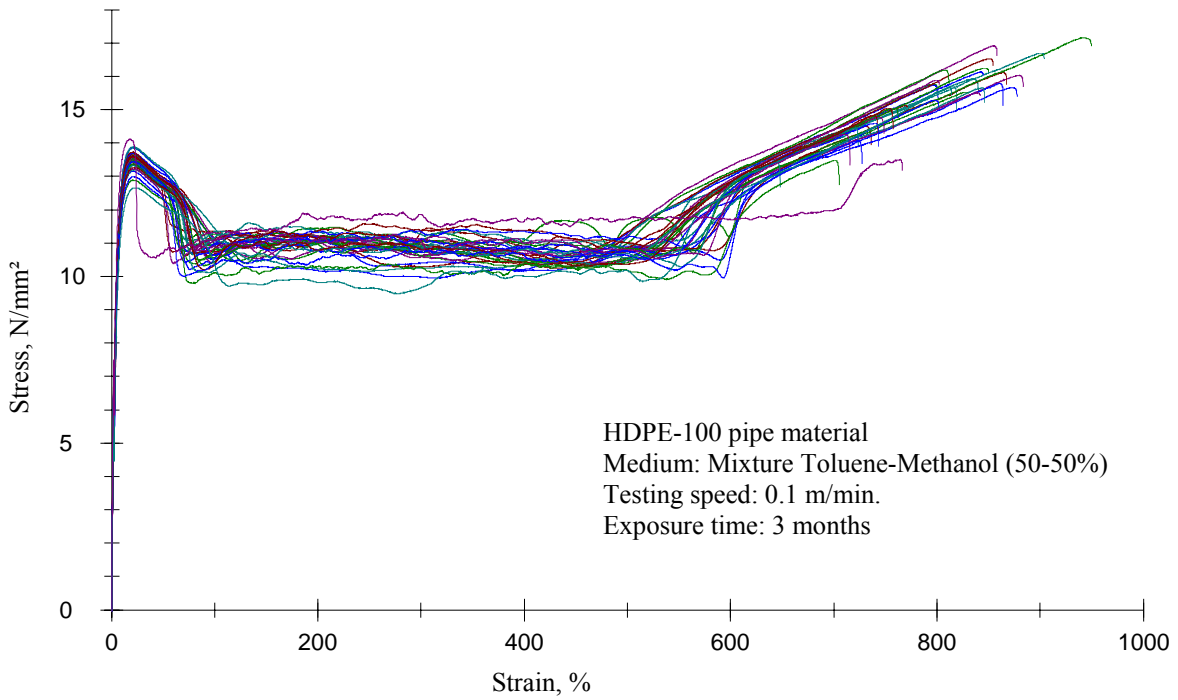


Fig. 3 Stress strain curves of HDPE resin (PE-100) in toluene-methanol solution

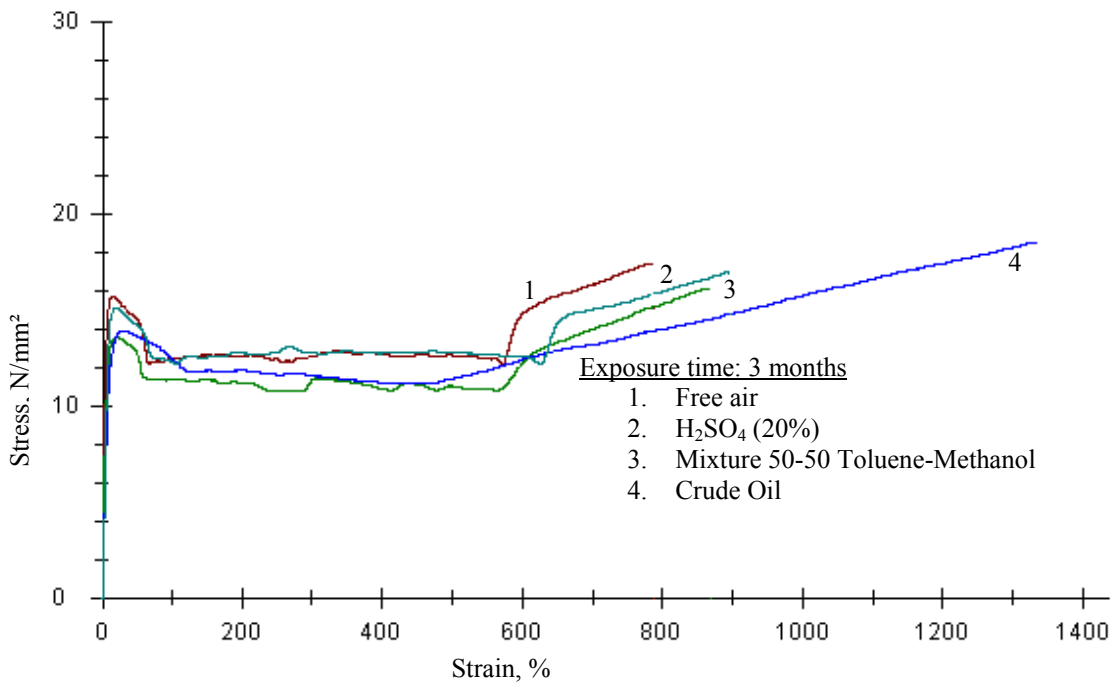


Fig. 4 Comparison of stress strain curves of HDPE resin PE-100 in testing environments

It is also noted a consequent contraction of the drawing zone (II) which lays over only approximately 400% of strain (while zone III occupies more than 1000% of strain). The interaction with crude oil is probably owing to the fact that certain oil fractions might be transported by the gas flow. Moreover, the hardening plastic slope is almost rolled out in this case and ensured a smooth continuity between zones II and III while for both air and toluene-methanol environments the transition is quite drastic.

3.4. Interaction with sulphuric acid

In contact with the H⁺ ions coming from sulphuric acid, the HDPE presents a behaviour contrasting those of

crude oil and toluene-methanol environments. The yield stress is comparable with that of the air if not slightly lower but the dispersion of the curves is important. It was found that the mean drawing stress is also similar to that of the air (~12.5 MPa) whereas its extent largely exceeds that of the reference case of air as shown in Fig. 4 for all considered environments. The zone of plastic hardening remains comparable but the strains at failure approaches approximately 1000%, which represents more than 200% compared to the air but it is much less than that of crude oil. Zone III is also characterized by a strong dispersion and the nominal stress at failure is relatively smaller (<16 MPa) compared to the other cases.

3.5. Mechanical properties evolution through pipe thickness

The change in Young's modulus is depicted in Fig. 5. It is observed that all environments influence negatively on the HDPE pipe as compared to air. The dimensionless thickness is the ratio (t_i/t_0) where (t_i) is the thickness at a given position and t_0 the initial one. The most important reduction is associated with crude oil as the change reached more than one third especially at the pipe outer and middle regions. The sulphuric acid and toluene-methanol solution and crude oil affected also the structure reducing E by 36%, 55% and 64% respectively. Through the pipe thickness, some fluctuations of E are pointed out but it seems that the lower values are basically at the outer layers in the following order:

$$E_{(Crude-Oil)} < E_{(Sulfuric-Acid)} < E_{(Toluene-Methanol)} < E_{(Air)}$$

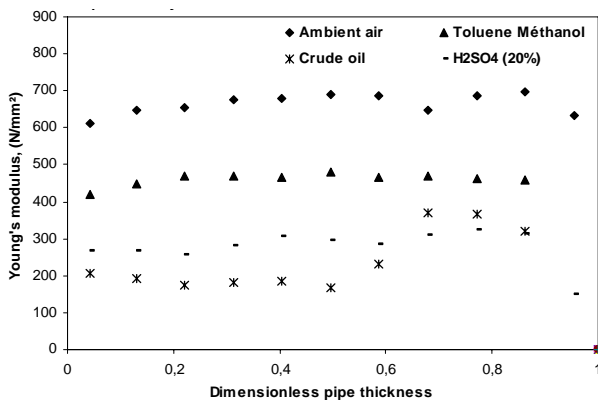


Fig. 5 Young's modulus as a function of pipe thickness

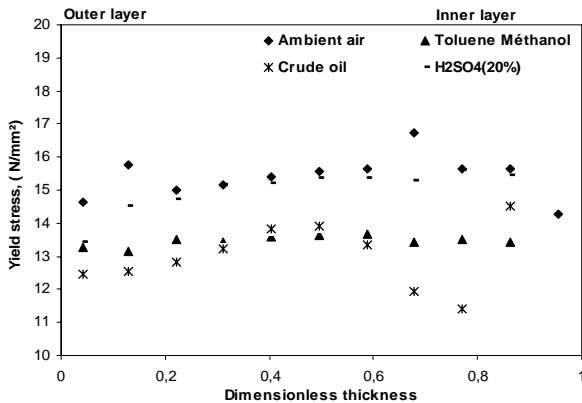


Fig. 6 Yield stress as a function of pipe thickness

For the yield stress values, the effect order is inverted as toluene-methanol solution is more aggressive and reduced the elastic limit by 14% as illustrated in Fig. 6. In addition, crude oil remains the most degrading environment especially at inner pipe layer which presents the most critical condition for natural gas distribution networks.

In fact, the inner pipe layers are under positive residual stresses and for any defect, crack propagation may initiate at any moment. In the case of the nominal drawing stress, definitely toluene-methanol solution is the most aggressive as it lowered the stress level by 16% (Fig. 7).

The inner layer shows heterogeneous effects dominated by crude oil whereas sulphuric acid is shown to have very minute effect since its curve is overlapping with

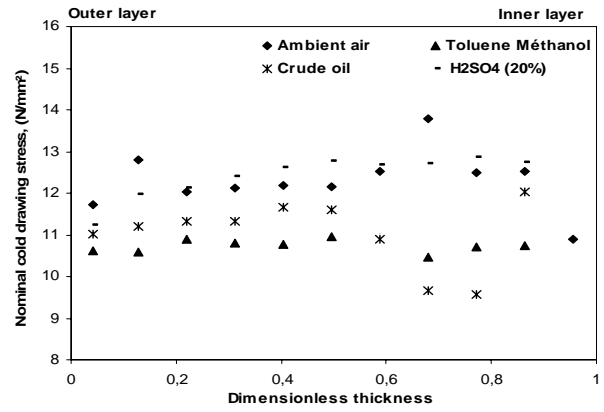


Fig. 7 Nominal cold drawing stress through pipe thickness

that of air. In terms of strain ϵ_f , Fig. 8 illustrates the effects of the environments on the failure strain. It is observed that crude oil has the most preponderant activity as it emerges from the lot with the strain at failure 200% higher. It is possible to advance the following order when comparing failure strains which is basically the opposite case for both E and σ_y

$$\epsilon_{f(Crude-Oil)} > \epsilon_{f(Toluene-Methanol)} > \epsilon_{f(Sulfuric-Acid)} > \epsilon_{f(Air)}$$

Table 2 gives a comparison of the values of E , $R_{p0.2}$ (elastic limit at 0.2%), σ_y (maximum yield stress), ϵ_y (yield strain), ϵ_{rupt} (strain at failure), σ_{CD} (mean drawing

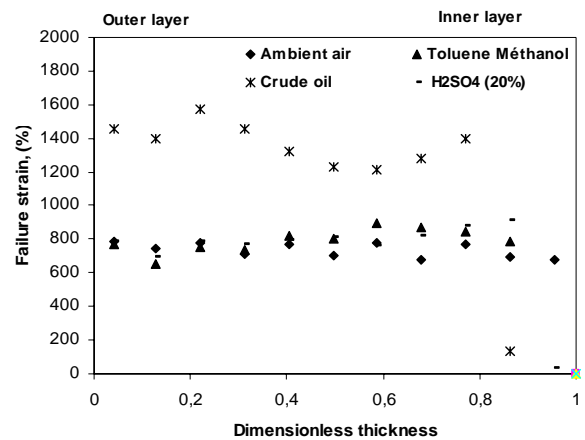


Fig. 8 Evolution of nominal failure strain through pipe thickness

stress or the stress plateau) and $\Delta\epsilon_{CD}$ (extent of drawing in terms of % strain) for the HDPE exposed to considered environments. The data for $\Delta\epsilon_{CD}$ is measured directly on stress stress curves and it represents the extent of the plateau of zone II where the stress is sensibly constant. It can be seen that in term of yield strain, crude oil is allowing the highest value while sulphuric acid exhibits the highest change in cold drawing strains. It is also noted that the measured plastic pipes mechanical properties are strongly influenced by the contact with the chemical agents and the swelling phenomenon plays another role as it degrades polymer chains.

The resistance of polymeric materials to chemical agents depends on the nature of polymer as well as additives especially anti-oxidants [12]. The results indicate that

there is an evolution of properties from the external layer towards the inner layer and this is probably due to crystallinity changes which increase in the same direction

[13, 14]. Crude oil and its derivatives should be kept away from HDPE pipes as long term mechanical properties are drastically degraded with increasing exposure time.

Table 2

Comparison of the main mechanical properties as a function of aggressive environments

Environment	E , N/mm ²	$R_{p0.2}$, N/mm ²	σ_y , N/mm ²	ε_y , %	ε_f , %	σ_{CD} , N/mm ²	$\Delta\varepsilon_{CD}$, %
Air	669.42	6.39	15.33	14.56	737.50	12.22	520.62
H ₂ SO ₄ (20%)	277.36	10.72	15.01	19.32	731.90	12.40	553.50
Toluene Methanol	461.16	4.29	13.45	19.15	791.74	10.74	452.18
Crude Oil	239.82	8.64	13.00	27.95	1245.44	11.02	452.90

4. Conclusions

This study allows drawing the following conclusions based on stress strain curves and the exposure to aggressive environments:

1. The technique of filament machining through an HDPE pipe thickness is found to be convenient for studying material heterogeneity and environmental effects on mechanical properties.

2. Crude oil and toluene-methanol environments are found to be very effective in degrading stress components while sulphuric acid basically affected strain ones.

3. These changes in mechanical properties are consequences of crystallinity distribution imposed by the manufacturing process which is extrusion.

4. Since transported natural gas may contain such species (crude oil traces and/or organic solvents); it becomes necessary to consider the effects of such chemical agents when designing gas networks.

5. In the same aspect, buried natural gas and water networks are inevitably in contact with underground aggressive environments, thus, considering appropriate safety factors is highly recommended.

Acknowledgements: The authors wish to thank the STPM-Chiali Company (Algeria) for providing the samples. They are also grateful to Pr N. Aouf, head of the Organic Chemistry Laboratory (LCOA), University of Annaba, for supplying chemical solutions and Pr L. Boulanouar for fruitful discussions. The cooperation of LMS of Guelma University is also well appreciated.

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AGRESYVIO CHEMINĖS APLINKOS VEIKIAMO POLIETILENINIO VAMZDŽIO MECHANINIŲ SAVYBIŲ KITIMAS

R e z i u m ė

Projektuojant skysčio transportavimo sistemas, svarbu tinkamai parinkti vamzdyno medžiagą. Gamtinės dujos ir geriamasis vanduo dažniausiai transportuojamas požeminiiais polietileninių (PE) vamzdynų tinklais. Yra žinoma, kad sąveikos tarp didelio tankio PE (DTPE) medžiagos ir ją veikiančios aplinkos atsiranda kritinių veiksnių susijusių su struktūros pokyčiais. Šių tyrimų tikslas yra nustatyti tam tikrų cheminių medžiagų įtaką po žeme klojamų PE vamzžių mechaninėms savybėms.

Tyrimų rezultatai remiasi įtempių diagramomis bei mechaninio PE atsparumo agresyviai aplinkai nagrinėjimu. Nustatyta, kad tirpikliai blogina mechanines vamzžių savybes, sumažėja pastebėtas sistemos konstrukcijos standumas. Organiniai tirpikliai pasižymi ypatingomis oksidacinėmis savybėmis, dėl kurių susilpnėja PE grandinės, o rūgštys, pavyzdžiui H_2SO_4 , palyginti su minėtais tirpikliais, mažiau turi įtakos vamzžių mechaninėms savybėms. Žalia nafta vidutiniškai veikia PE, nors yra žinoma, kad ji gadina daugelį polimerų. Naudojant tirpiklius, žalios naftos tamprumo modulis sumažėja net iki 64 %. Tai sukelia didelių problemų požeminiams PE vamzdynų tinklams. Projektuojant konstrukcijas, kai PE sąveikauja su agresyvia aplinka, skaičiavimo lygtys turi būti koreguojamos atsižvelgiant į ilgalaikes apkrovas.

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MECHANICAL BEHAVIOUR OF MACHINED POLYETHYLENE FILAMENTS SUBJECTED TO AGGRESSIVE CHEMICAL ENVIRONMENTS

S u m m a r y

During the construction process of fluid handling structures, material selection for pipe applications is an important step. Both natural gas and drinkable water are mainly transported in underground polyethylene (PE) pipe networks. It is known that the interaction between materials like HDPE (high density PE) and its service environment represents critical factors of the influence on the structure behaviour for short and long terms. The goal of this study is to establish the effect of some chemical agents on the mechanical properties of PE tubes for underground use. The results are discussed basing on the stress strain curves and according to the mechanical resistance to aggressive environments. It was found that the environments consisting of solvents show degradation of mechanical

properties of the tubes and a structural weakening of the rigidity of the system is observed. Organic solvents have significant oxidizing capacity which weakens polyethylene chains, whereas acids such as H_2SO_4 have less influences on the mechanical properties compared to organic solvents. In the case of crude oil, the results show moderate effect although it is known to attack many polymers. In the case of solvents, the elasticity modulus is reduced up to 64% for crude oil which represents a serious problem for the underground polyethylene networks. Design equations of such structure should take into account serious adjustments of long term loads when polyethylene is exposed to aggressive environment.

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ИЗМЕНЕНИЕ МЕХАНИЧЕСКИХ СВОЙСТВ ПОЛИЭТИЛЕНОВОЙ ТРУБЫ ПРИ ВОЗДЕЙСТВИИ АГРЕССИВНОЙ ХИМИЧЕСКОЙ СРЕДЫ

Р е з ю м е

При проектировании систем для транспортирования жидкости, важную роль играет подбор материала для трубопровода. Искусственный газ и питьевая вода, в основном, транспортируются при помощи подземных полиэтиленовых (ПЭ) сетей трубопроводов. Известно, что взаимодействие ПЭ материала большой плотности (БППЭ) с его окружающей средой, влияет на появление критических факторов, связанных с структурными изменениями материала. Цель исследований – установление влияния определенных химических материалов на механические свойства ПЭ труб находящихся под землей. Результаты исследований обоснованы на изучении диаграмм напряжений - деформаций и определении механического сопротивления ПЭ, находящегося в агрессивной среде. Установлено, что растворители ухудшают механические свойства труб, уменьшая упругость конструкций системы. Растворители на органической основе отличаются специфическими оксидационными свойствами, которые уменьшает взаимосвязь между цепями ПЭ, с другой стороны кислоты, например H_2SO_4 , оказывает меньше влияния на механические свойства труб по сравнению с упомянутыми растворителями. Сырые нефтепродукты в среднем влияют на ПЭ, хотя известно, что они портят множество полимеров. Модуль упругости сырой нефти под воздействием растворителей уменьшается до 64 %, что оказывает серьезные проблемы на подземные ПЭ сети. При проектировании конструкций, когда ПЭ взаимодействует с агрессивной средой, уравнение расчетов должны быть скорректированы с учетом долговременных нагрузок.

Received December 01, 2008
Accepted May 06, 2009

DOI: 10.5755/j02.mech.15233