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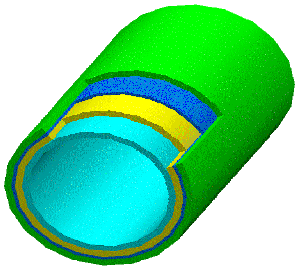

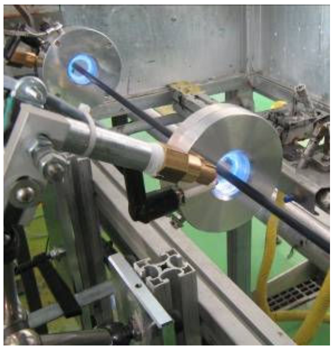

Plastic pipe extrusion process improvement

Master's Thesis in Automotive Engineering

FLORIAN MORICE

Department of Applied Mechanics
Division of Vehicle Engineering and Autonomous Systems
CHALMERS UNIVERSITY OF TECHNOLOGY
Göteborg, Sweden 2011

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Cover:
left side: five-layer extrusion head, Rivoli, Italy
right side, top: PA6/EVOH/ADH/PA12 multilayer structure
right side, bottom: flaming treatment unit

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Abstract

This thesis is the introductive part of a study on extrusion process improvement where extruded products are multilayer smooth thermoplastic pipes. The study focuses specially on PA9T/PA12 and PA6/EVOH/ADH/PA12 structures used for fuel transfer applications.

The first reason for working on process improvement is to reduce production cost which depends on process reliability and line speed in the case of extrusion. A long-term study focussed on pipe dimensional properties will be carried out in 2012 to improve process reliability. The first purpose of this thesis is to prepare this study. Another reason is when some essential characteristics of the product do not meet customer specification requirements. For plastic pipes, elongation at break is an important characteristic. On new projects in development, minimum value required for pipe elongation at break has been increased and is not reached with current process configuration. The purpose of this thesis is also to improve this characteristic.

Polymer main characteristics in relation with pipe properties and extrusion process are presented as a prerequisite of this study. Then, an analysis of the mechanisms occurring for each operation of the process allows identifying the main factors that influence pipe essential characteristics then identifying extrusion line parameters which allow controlling these factors. Impact of these parameters on pipe elongation at break is studied later in this thesis.

Beforehand, pipe monitored essential characteristics and their respective controls are identified. A first study is carried out to improve current dimensional controls in anticipation of the study on process reliability improvement. Thus a concept of cutting machine and a program to automate measures is presented but their development is out of the scope of this thesis. A second study is carried out to improve tensile test reliability before working on pipe elongation at break improvement. New test configuration allows reducing significantly the quantity of test results rejected.

Finally a design of experiment is carried out on PA9T/PA12 and PA6/EVOH/ADH/PA12 structures to improve pipe elongation at break. Target value of elongation at break is 200%. Best results are obtained for extrusion line speeds lower than current ones which would impact process cost. Moreover, even with optimized parameters, some pipes are statistically below 200% of elongation at break that is not acceptable. A solution is been to equip extrusion line with a flaming treatment that allows achieving target value of 200% and in the meantime, improving process performance by increasing line speed.

Contents

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Preface

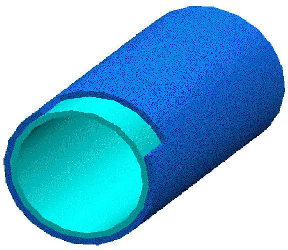
This thesis is the introductive part of a study on extrusion process improvement where extruded products are multilayer smooth thermoplastic pipes. This study was performed from September 2010 to February 2011 in collaboration with Hutchinson company which core business is polymer processing. Department involved belongs to the division “low pressure fluid transfer system” specialized in plastic pipe production for automotive sector. Technical centre is located at Rivoli, Italy whereas principal production plant is located at Zywiec, Poland. Tests required for this study have been carried out in both sites. The thesis has been supervised by Dr. Katia Rossi, responsible for structure development and Eng. Luciano Bertero, technical director.

First, I would like to thank Mr Ronconi, Vice President of Hutchinson Company, that gave me the possibility to carry out this thesis. I would like to thank also Dr. Katia Rossi, Eng. Luciano Bertero and all the team, especially Daria Cereghino, Romain Jeanson, Salvatore Rizzuto, Luca Barberis and Claudio Ramaro for their help during this study. Lastly, I would like to thank my parents and my friends for their support during this difficult period.

Rivoli, September 2011
Florian MORICE

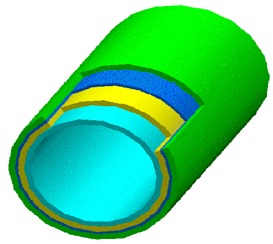
1. Introduction

This thesis is the introductive part of a study on extrusion process improvement where extruded products are multilayer smooth thermoplastic pipes. The study focuses specially on PA9T/PA12 and PA6/EVOH/ADH/PA12 structures used for fuel transfer applications. These structures are illustrated Figure 1 and presented more in details in Appendix 1.



Layer	Material	Thickness
Inner	PA9T	0,2 mm
2	PA12	0,8 mm

a. PA9T/PA12 structure

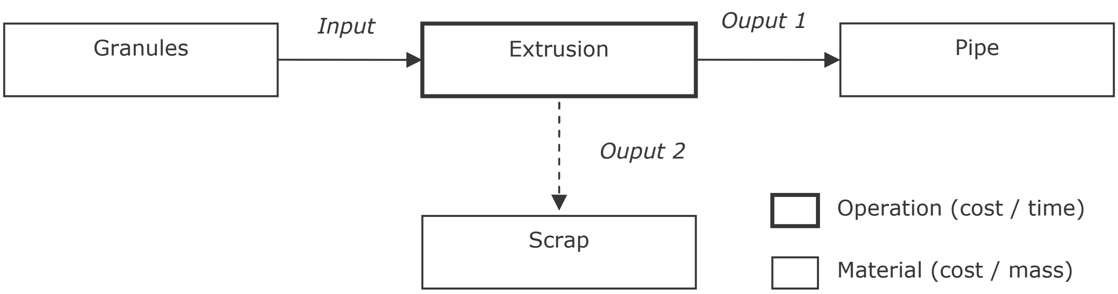


Layer	Material	Thickness
Inner	PA6	0,45 mm
2	EVOH	0,15 mm
3	Bonding	0,1 mm
4	PA12	0,3 mm

b. PA6/EVOH/ADH/PA12 structure

Figure 1 – Example of multilayer structures

The first purpose of working on process improvement is to reduce production cost. Extrusion cost depends on process reliability (scrap mass) and line speed (production time) as it can be deduced from the model Figure 2. Set-up cost has been voluntarily excluded from this model as set-up cost reduction is out of the scope of this study.



Where:

Extrusion cost $$\cos t_{extrusion} = \cos t_{operator} + \cos t_{machine}$$

Production cost $$\cos t_{production} = \cos t_{pipe} + \cos t_{scrap} + \cos t_{extrusion}$$

Process efficiency $$\eta = \frac{\cos t_{pipe}}{\cos t_{production}}$$

Figure 2 – Extrusion process cost model

A long-term study focussed on pipe dimensional properties will be carried out in 2012 to improve extrusion process reliability. The first purpose of this thesis is to prepare this study.

Another reason for working on process improvement is when some essential characteristics of the product do not meet customer specification requirements. For plastic pipes, elongation at break is an important characteristic. On new projects in development, minimum value required for pipe elongation at break has been increased and is not reached with current process configuration. The purpose of this thesis is also to improve this characteristic.

Thesis structure has been built according to six-sigma method as presented Figure 3. This method was created and implemented by Japanese people many years ago. Nowadays it is still considered as a reference for people working on continuous improvement.

Defining

1. Polymer characteristics:
- Identifying polymer main characteristics in relation with pipe properties and extrusion

2. Extrusion mechanisms:
- Understanding the mechanisms occurring for each operation of the process
- Identifying the main factors that influence pipe essential characteristics
- Identifying extrusion line parameters which allow controlling previous factors

Measuring

3. Extrusion performance monitoring:
- Identifying pipe monitored essential characteristics
- Identifying control method and instrument for each characteristic monitored
- Improving current controls: tensile test, dimensional controls

Analyzing

4. Extrusion performance improvement: results and discussion:
- Carrying out two designs of experiment to improve pipe elongation at break:
  - optimizing current process
  - adding a flaming treatment
- Analyzing the results

Improving

- Implementing a flaming treatment on extrusion line: currently in progress

Controlling

Figure 3 – Six sigma method applied to the thesis

First chapter of this report presents polymer main characteristics in relation with pipe properties and extrusion process. Second chapter describes the mechanisms occurring for each operation of the process and identifies the main factors that influence pipe essential characteristics then extrusion line parameters which allow controlling these factors. Third chapter identifies pipe monitored characteristics and presents control method and instrument for each characteristic monitored. This chapter includes also a study to improve current dimensional controls in anticipation of the study on process reliability improvement and a study to improve tensile test reliability before working on pipe elongation at break improvement. Lastly, fourth chapter presents and discusses the results obtained from the designs of experiment carried out to improve pipe elongation at break. Explanations provided in this report try to refer to basic physical concepts to be easily understandable.

2. Polymer characteristics

Before working on plastic pipe extrusion process and as a prerequisite of this study, it is necessary identifying polymer main characteristics in relation with pipe properties and extrusion process. They are presented in this chapter.

2.1. Chemical composition

Plastic pipes for automotive application are essentially composed of semi-crystalline polyamides. For instance, PA9T/PA12 and PA6/EVOH/ADH/PA12 structures contain both a layer of polyamide 12 that is the most widely used polyamide. Its chemical formula is presented Figure 4.

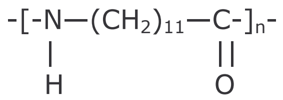


Figure 4 – Chemical formula of polyamide 12

A particularity of polyamides is the presence of hydrogen bonds between macromolecular chains at amide group location. As presented Figure 5, these electrostatic interactions occur between:

- *N-H* group: highly electropositive
- *C=O* group: highly electronegative

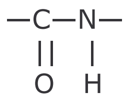


Figure 5 – Chemical formula of amide group

These bonds are very energetic because dipolar moments are high. It gives to polyamides better characteristics in terms of mechanical and chemical resistance than that of other polymers like polyethylene or polypropylene.

2.2. Water absorption

Polyamides are formed by poly-condensation of an amino-acid group. Poly-condensation is a balance reaction between polymerisation that releases water molecules and hydrolysis by incorporation of water molecules between macromolecular chains: the shorter the chain, the higher the sensibility to hydrolysis. For instance, quantity of water absorbs by polyamide 12 in humid environment is lower than polyamide 6 whose carbon chain is shorter. This property is important because hydrolysis reaction damages partially material cohesion increasing molecule mobility. As a consequence, mechanical characteristics can be significantly lowered. Water absorption of different polyamide is presented Figure 6.

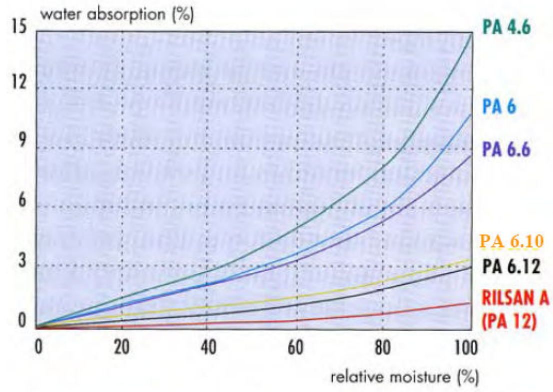


Figure 6 – Water absorption according to ambient air relative moisture of different polyamides [CARI05]

Pipe mechanical characteristics are sensible to air moisture content. Thus when controlling pipe mechanical characteristics, air moisture content in test room should be controlled to ensure reproducibility of the results for different atmospheric conditions outside.

High moisture content in plastic pellets would result in water degassing inside the extruder that would perturb process operating conditions then degrade final product mechanical characteristics. Thus pellets must be dried before being processed.

2.3. Thermal conductivity

Thermal conductivity defines the ability to conduct heat. Polymers are often considered to be thermal insulators because of their low thermal conductivity. Therefore heating or cooling the material inside the extruder is a slow process.

2.4. Thermal stability

Polymers can degrade when exposed to high temperatures: the higher the temperature, the faster the degradation. Thus the two main factors involved in degradation mechanism are:

- temperature level
- time of exposure

Degradation mechanism inside extrusion machine can result in:

- low viscosity of plastic melt
- inclusions of degraded material in the pipe: dimensional, mechanical, visual

Thermal stability of polymers can be improved by adding thermal stabilizers.

2.5. Glass transition temperature and melting point

Glass transition temperature corresponds to a change of molecule conformation that affects polymer mechanical properties. Polymers are softer above this temperature than below. Melting point is an important characteristic of semi-crystalline materials. It corresponds to crystal melting temperature. Polymers behave like fluids above this temperature and like solids below. Glass transition temperature and melting point of different polymers are given Table 1.

Table 1 – Glass transition temperature and melting point of different polymers

Material	Glass transition temperature [°C]	Melting point [°C]
PA6	45	195
PA12	50	165
PA9T	140	255
EVOH	65	175

2.6. Viscous properties at melted state

Viscosity of a fluid defines its resistance to flow uniformly: the higher the viscosity, the higher the resistance. It depends on fluid molecule entanglement: the lower the entanglement, the lower the viscosity. Thus viscosity is influenced by fluid molecular weight: the higher the weight, the higher the viscosity. A fluid which flow is not uniform is called viscous fluid. Polymers behave like viscous fluids at melted state. Viscosities of different fluids are compared Table 2.

Table 2 – Comparison between viscosities of different fluids (order of magnitude)

Fluid	Viscosity [Pa.s]
Air	10^{-5}
Water	10^{-3}
Oil	10^{-1}
Plastic melts	10^{2} to 10^{6}

2.6.1. Sensibility to temperature

Molecule mobility depends on fluid temperature: the higher the temperature, the higher the mobility. Moreover viscosity depends on molecule mobility: the higher the mobility, the lower the entanglement, the lower the viscosity. Therefore viscosity depends on fluid temperature: the higher the temperature, the lower the viscosity. For semi-crystalline polymers, viscosity varies from 2 to 3 % per degree centigrade.

2.6.2. Sensibility to shear rate

Pseudo-plasticity of a fluid defines viscosity sensibility to shearing. It depends on the capacity of fluid entangled molecules to rearrange when exposed to shearing. The rearrangement increases with increasing shear rate: the larger the rearrangement, the lower the entanglement, the lower the viscosity. A fluid which viscosity depends on flow shear rate is called non-Newtonian or non-linear fluid. Polymers which are composed of long entangled molecules behave like non-linear fluids at melted state. Non-linear behaviour can be characterized by plotting the power law curve of the material as shown Figure 7.

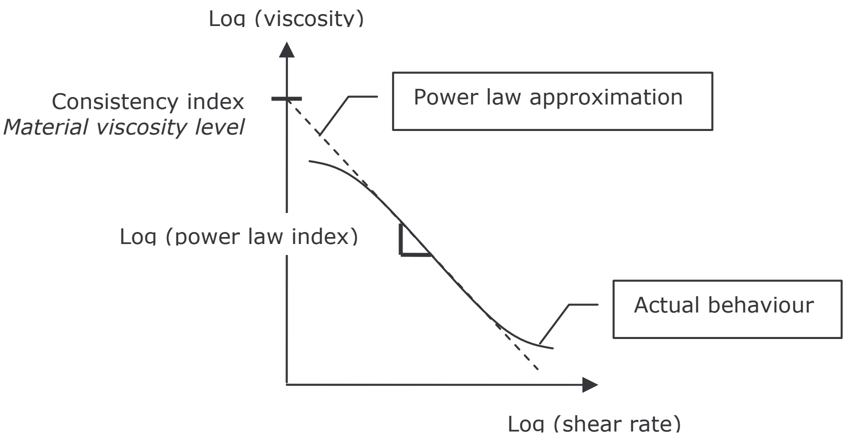


Figure 7 – Power law curve of a polymer [RAUW10]

Considering two elements of a viscous flow, shear rate is the ratio between their velocity difference and their normal distance as illustrated Figure 8. It corresponds also to velocity profile slope also called velocity gradient. Thus it depends on:

- Flow velocity: the higher the velocity, the higher the shear rate
- flow channel width: the smaller the channel, the higher the shear rate

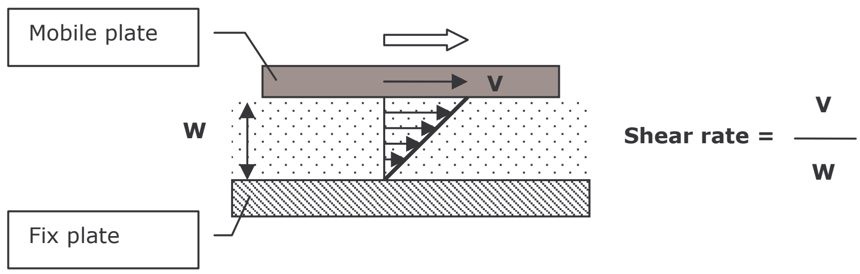


Figure 8 – Fluid shearing between two plates

Effect of shear rate on a rectangular fluid element is illustrated Figure 9: the higher the shear rate, the higher the shear strain and the higher the shear stress at given viscosity.

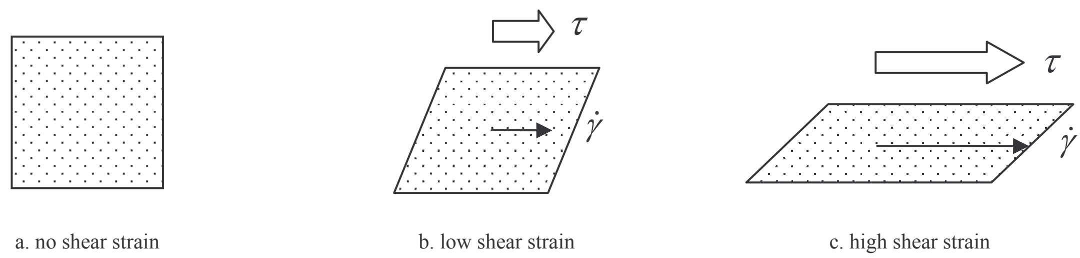


Figure 9 – Effect of shear rate on a rectangular fluid element

Shear stress is given by the following formula:

$$\tau = \eta(T,\dot{\gamma}).\dot{\gamma}$$

With:

τ	Shear stress
$\eta(T,\dot{\gamma})$	Material viscosity
$\dot{\gamma}$	Shear rate
T	Material temperature

High shear stress inside extrusion machine prevents material accumulation in dead zones: the higher the stress, the higher the removal. This is important:

- To avoid material degradation (see 2.4.)
- To ensure efficient cleaning of the machine (during cleaning phase)

2.6.3. Viscous heat generation

A viscous material generates heat when sheared. This mechanism is called viscous heat generation. Heat amount is calculated from the product of viscosity and shear rate squared:

$$H_{viscous} = \eta(T, \dot{\gamma}).\dot{\gamma}^2$$

With:

$H_{viscous}$	Viscous heat
$\eta(T, \dot{\gamma})$	Material viscosity
$\dot{\gamma}$	Shear rate
T	Material temperature

2.7. Specific volume variation between melted and solid states

Specific volume defines a volume per unit of mass. It depends on material molecule organization and mobility. Thus it is influenced by:

- pressure: the lower the pressure, the higher the specific volume
- temperature: the lower the temperature, the lower the specific volume

Specific volume of semi-crystalline materials decreases during cooling process as illustrated Figure 10.

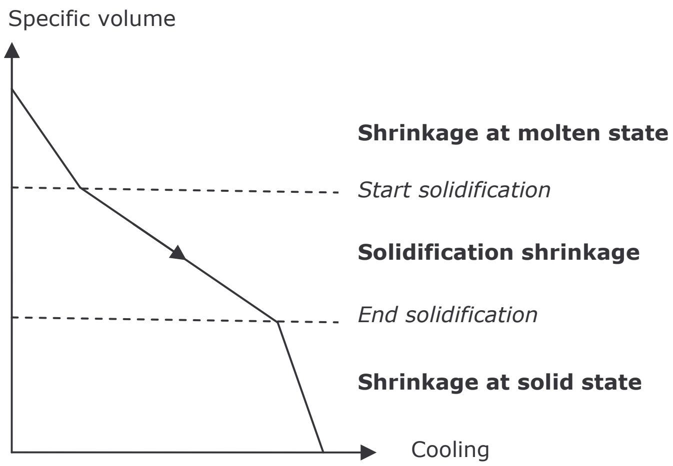


Figure 10 – Semi-crystalline material shrinkage during cooling process

Two phenomena related to specific volume variation can be observed during extrusion process:

- pipe swelling during plastic melt discharge from the die (released pressure)
- pipe shrinkage during cooling process inside sizing sleeve (cooling temperature)

2.8. Mechanical properties at solid state

Two types of organisation can be identified when looking at the molecular structure of semi-crystalline materials at solid state as illustrated Figure 11:

- amorphous phase: random and irregular structure
- crystalline phase: highly regular structure composed of crystals

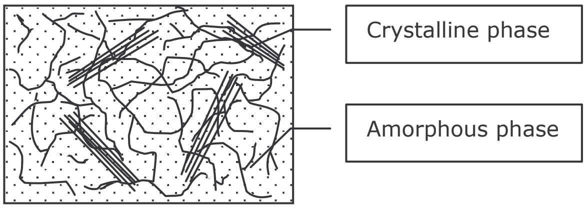


Figure 11 – Molecular structure of semi-crystalline materials

Two factors related to the molecular structure influence material mechanical properties:

- Crystal rate (below 25%)
- Molecular orientation

When crystal rate increases, material internal energy decreases and therefore mechanical energy required for displacing molecules increases. When material molecules are pre-orientated, stretching capacity of molecular chains is reduced in orientation direction and mechanical energy required increases also.

A break is caused by propagation of a crack through the material. A crack initiation is typically a defect into the material. This crack propagates only if the load applied to the material exceeds a limit that depends on defect location, size and orientation in the material.

When mechanical energy required for displacing molecules increases, the load applied to the material increases at given elongation. Therefore crack propagation probability increases and elongation at break decreases fatally. As a consequence, material elongation at break decreases with increasing crystal rate and molecular orientation.

Thus pipe mechanical properties, especially elongation at break, depend on:

- Material crystal rate: the higher the crystal rate, the lower the elongation at break
- Material molecular orientation: the higher the molecular orientation, the lower the elongation at break
- Surface state: the higher the quantity and size of the defects, the lower the elongation at break

2.9. Conclusion

Before working on plastic pipe extrusion process, it is necessary identifying polymer main characteristics in relation with pipe properties and extrusion process.

Plastic pipes for automotive application are essentially composed of semi-crystalline polyamides. An important characteristic of semi-crystalline materials is their melting point that corresponds to crystal melting temperature. Polymers behave like viscous fluids above this temperature and like solids below.

2.9.1. **Flow properties at melted state**

Plastic melt viscosity and temperature inside extrusion machine are closely linked. They are important factors when working on extrusion as it will be explained next chapter.

Plastic melt viscosity depends on:

- shear rate: the higher the shear rate, the lower the viscosity
- temperature: the higher the temperature, the lower the viscosity

Plastic melt temperature depends on:

- viscous heat generation:
  - shear rate (predominant factor): the higher the shear rate, the higher the heat generation
  - plastic melt viscosity: the higher the viscosity, the higher the heat generation
- conductive heat transfer: limited by polymer low thermal conductivity

2.9.2. **Specific volume variation between melted and solid states**

Specific volume of semi-crystalline materials, which defines a volume per unit of mass, decreases during cooling process.

2.9.3. **Mechanical properties at solid state**

Pipe mechanical properties, especially elongation at break, depend on:

- Material integrity: no inclusions of non melted or degraded material in the pipe
- Material crystal rate: the higher the crystal rate, the lower the elongation at break
- Material molecular orientation: the higher the molecular orientation, the lower the elongation at break
- Pipe surface state: the higher the quantity and size of the defects, the lower the elongation at break

When controlling these characteristics, room air moisture content should be controlled to ensure reproducibility of the results for different atmospheric conditions outside.

3. Extrusion mechanisms

Hutchinson has several extrusion lines which differ by the number of layers that can be extruded simultaneously:

- monolayer line: monolayer pipe
- three layer line: till three layer pipe
- five-layer line: till five layer pipe

This study focuses specially on the five-layer line presented Figure 12 on which are produced PA9T/PA12 and PA6/EVOH/ADH/PA12 structures.

a. extrusion head + sizing system   b. cooling bath   c. caterpillar + cutting unit

Figure 12 – Five-layer extrusion line, Rivoli, Italy

Components of a monolayer line are presented Figure 13. On a five-layer line, the single extruder is substituted by five extruders connected to a head with inside a multi-manifold that combines material flows just before forming tools. Head and multi-manifold are presented Figure 14. Process operations are also specified Figure 13.

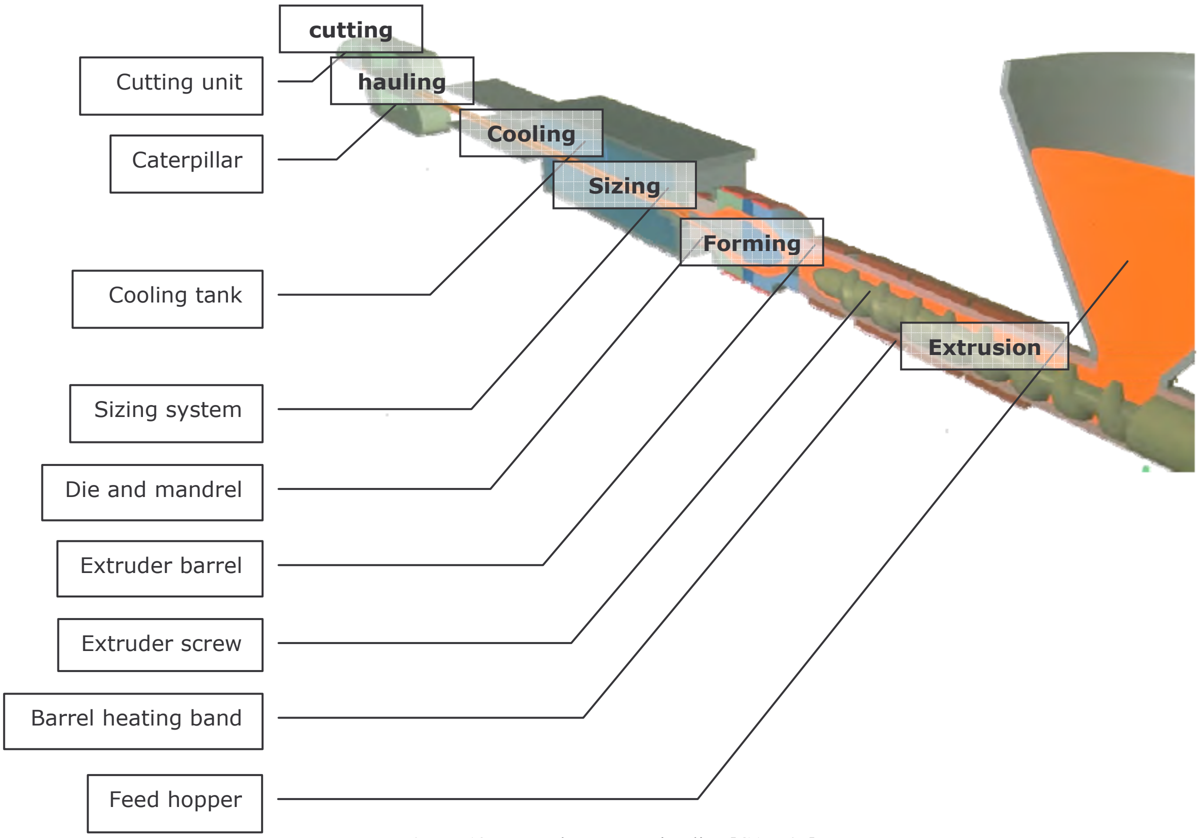


Figure 13 – Monolayer extrusion line [CARI05]

a. multi-manifold components

b. back view of the head (multi-manifold removed)

Figure 14 – Five-layer extrusion head, Rivoli, Italy

Before working on extrusion process improvement, it is necessary identifying, for each operation of the process, the main factors that influence pipe essential characteristics then identifying extrusion line parameters which allow controlling these factors. Beforehand, it requires understanding the mechanisms occurring for each operation of the process. All these aspects are presented in this chapter.

3.1. Extrusion

The main function of an extruder is to generate sufficient pressure to force the material through the die. Thus an extruder works like a pump. In the case of plastic pipe extruders, pumping action is created by the rotation of the screw as shown Figure 15 screw where motor load increases with increasing melt pressure in PA12 and PA9T extruders.

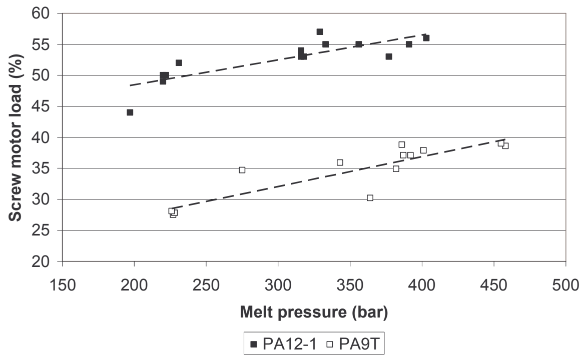


Figure 15 – Influence of melt pressure in the extruder on screw motor load

The screw is the principal component of an extruder. Its rotation speed determines extruder output and material shear rate inside the extruder. As the extruder is fed by a rotating screw, extrusion process is continuous by definition and instabilities are very often caused by irregular material feeding. Therefore feeding regularity is an important factor to control.

The profile of a screw dedicated to polyamides extrusion is presented Figure 16 where functional and operating zones have been identified.

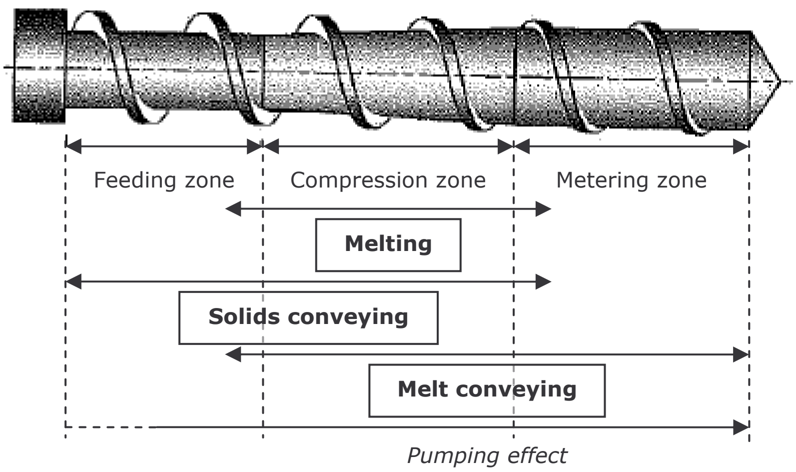


Figure 16 – Screw geometry and functional zones [MIAN09]

Functional zones are geometrically defined whereas operating zones are not determined and are interdependent. In fact, melting process starts where material temperature first exceeds melting point and ends when the last granule is melted whatever the zone of the screw. However if end of this process is postponed until the end of metering zone, there could be some inclusions of non melted material in extruded pipe. Moreover if beginning of this process is anticipated until feeding zone, feeding process could be disrupted affecting feeding regularity. Therefore melting rate in compression zone of the screw is an important factor to control.

In a five-layer extrusion machine, when materials from the different extruders are combined, flow properties should be compatible to limit distortion phenomenon at pipe layer interface. Therefore plastic melt viscosity at the exit of extrusion machine is an important factor to control.

3.1.1. **Solids conveying**

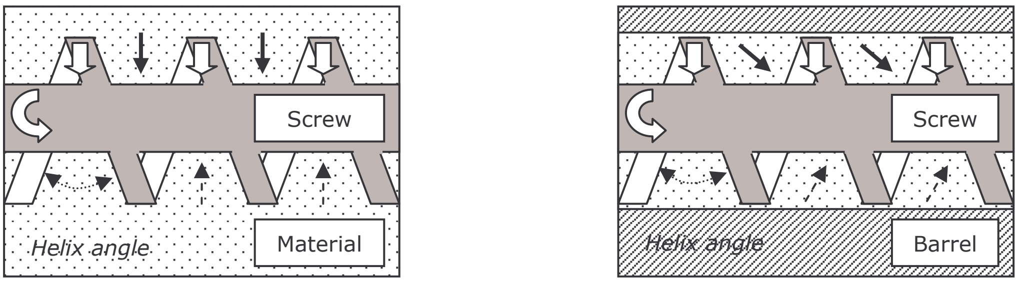
Solids conveying process starts in the feed hopper and ends when the last granule is melted.

3.1.1.1 Gravity induced conveying

Conveying mechanism in the feed hopper is called gravity induced conveying because it is generated by plastic pellet weight. Hopper is designed to ensure a steady flow of material. A vibrating device can be added for high flow resistance pellets.

3.1.1.2 Drag induced conveying

Conveying mechanism along screw channel is called drag induced conveying as it results from friction forces acting on the material. This mechanism determines machine output. It is illustrated Figure 17.



a. excluding barrel interaction: radial trajectory

b. including barrel interaction: spiral trajectory

Figure 17 – Material movement along screw channel

This mechanism is generated by the combined effect of barrel friction forces acting on the material that limits its rotation and screw helical flight rotation that pushes the material forward: the lower the material rotation, the greater the conveying. In fact, considering only screw interaction, the material would not have any axial movement as it would rotate together with the screw around the shaft. An analogy can be made with a nut free of rotating around a threaded shaft.

From previous analysis, feeding regularity in feeding zone of the screw depends on:

- material characteristics:
    - pellet flow resistance
- screw design:
    - helix angle: the larger the angle, the lower the friction on the screw
- machine parameters:
    - extruder barrel temperature: the lower the temperature, the higher the friction on the barrel

3.1.2. **Melting**

Melting process starts when material temperature first exceeds melting point and ends when the last granule is melted.

3.1.2.1 Contiguous solids melting

In single screw extruders like Hutchinson ones, melting mechanism is called contiguous solids melting. This mechanism is illustrated Figure 18.

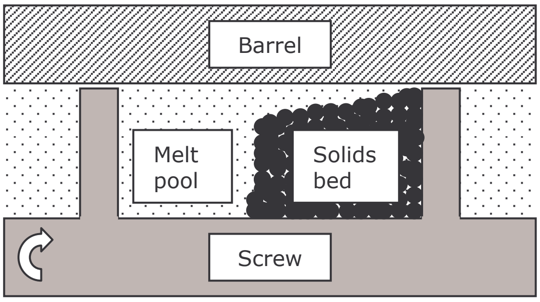


Figure 18 – Melting mechanism in single screw extruders

While moving along screw channel, pellets are compacted, heated up and melted at the contact of barrel wall which creates a melted film. Then melting process is principally located at the interface between solids bed and melted film.

Conductive heat transfer from barrel wall to solids bed is limited by polymer low thermal conductivity (see 2.3.): the thinner the melted film, the higher the transfer. Thus viscous heat generation is the main source of heat inside the extruder as illustrated Figure 19 and it depends on shear rate in the film (see 2.6.3.). Therefore melting rate depends on shear rate in the film: the higher the shear rate, the higher the melting rate. However shear rate depends on film thickness (see 2.6.2.): the thinner the film, the higher the shear rate. Therefore melting rate depends also on film thickness: the thinner the film, the higher the melting rate. Film thickness is primarily determined by flight clearance: the smaller the clearance, the thinner the film. Thus melting rate decreases irreparably when screw or barrel get worn.

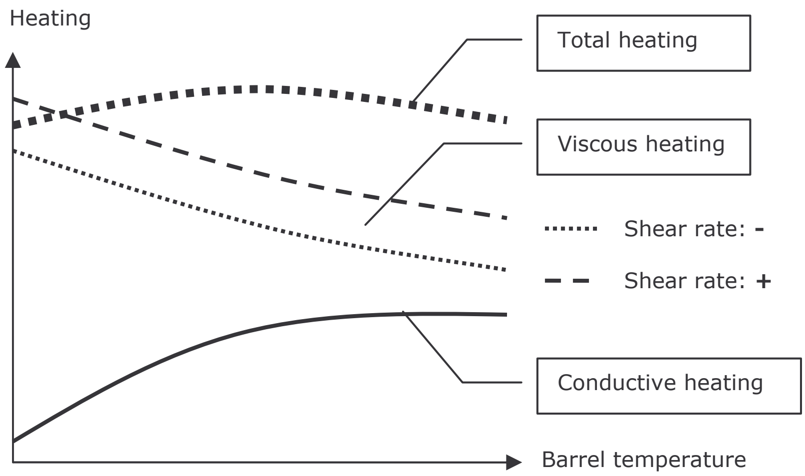


Figure 19 – Effect of barrel temperature on viscous and conductive heating

Increasing barrel temperature increases conductive heat transfer from barrel wall to solids bed. It increases also melted film temperature and as a consequence, reduces material viscosity then viscous heat generation. Thus, over a given barrel temperature, total heating decreases and melting rate decreases.

From previous analysis, melting rate in compression zone of the screw depends mainly on:

- material characteristics:
  - melting point
  - viscous properties: power law curve of the material
- screw design:
  - helix angle: the lower the angle, the higher the shear rate, the higher the melting rate
  - compression ratio: the higher the ratio, the higher the shear rate, the higher the melting rate
- machine parameters:
  - screw speed: the higher the speed, the higher the shear rate, the higher the melting rate
  - barrel temperature: depending on temperature level

3.1.2.2 Drag induced melt removal

Newly melted material is continuously dragged away to the melt pool by the combined effect of barrel friction forces acting on plastic melt and screw helical flight rotation. This mechanism called drag induced melt removal limits melted film expansion and as a consequence, avoids melting rate decreasing during melting process (see 3.1.2.1).

Hutchinson extruder screw is composed of two threads in feeding and melting zones as presented Figure 20:

- one principal thread that conveys the material
- one variable barrier thread that separates physically solid from molten material

Figure 20 – Hutchinson extruder screw

Melt channel width increases as quantity of melt material increases and quantity of solid material decreases. End of the solids channel is closed. Working principle is illustrated Figure 21.

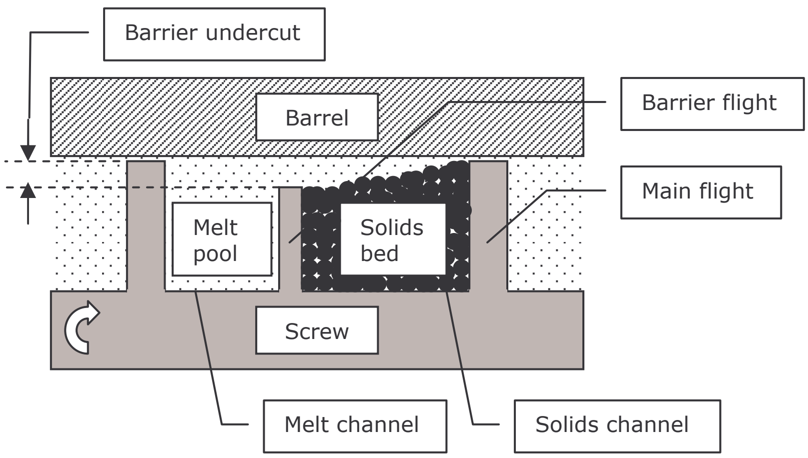


Figure 21 – Melting mechanism in Hutchinson extruder

The advantage of this configuration is that probability that none melted particles be at the end of melting zone is reduced. In fact, while particles are accumulated at the end of solids channel, pressure increases in the channel that accelerates melting mechanism. Therefore process operating window is larger with this type of screw than with a standard one.

3.1.3. **Melt conveying**

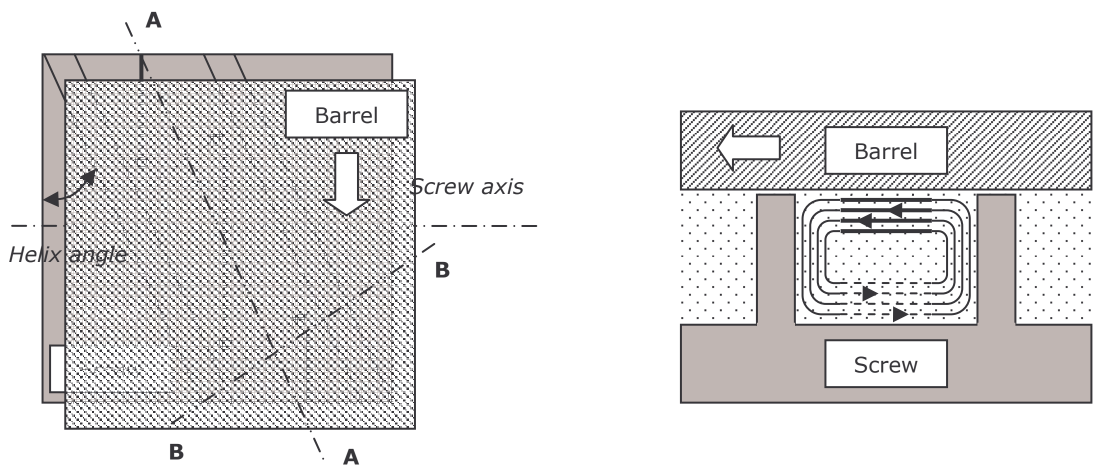
Melt conveying process starts as soon as melting process starts. Thus both solids conveying and melt conveying occur in the compression zone of the screw with quantity of solid material decreasing while quantity of melted material increases gradually. Melt conveying mechanism is viscous drag and it is fundamentally identical to solid conveying. It is generated by viscous forces acting on barrel surface and limited by viscous forces acting on screw surface (see 3.1.1.2).

Plastic melt viscosity depends on shear rate inside extrusion machine and plastic melt temperature (see 2.9.1.). Influence of barrel temperature on plastic melt temperature is limited by polymer low thermal conductivity (see 2.3.). Thus, for instance, plastic melt temperature decreases when screw speed decreases. In fact, shear rate decreases when screw speed decreases and, although residence time of the material inside the machine increases, viscous heat generation decreases more than conductive heat transfer increases.

From previous analysis, plastic melt viscosity in metering zone of the screw depend mainly on:

- material characteristics:
  - viscous properties: power law curve of the material
- screw design in metering zone:
  - helix angle: the lower the angle, the higher the shear rate, the lower the viscosity
  - compression ratio: the higher the ratio, the higher the shear rate, the lower the viscosity
- machine parameters:
  - screw speed: the higher the speed, the higher the shear rate, the lower the viscosity
  - barrel temperature: influence limited on plastic melt temperature and viscosity

Plastic melt flow relative to the screw can be illustrated by unrolling screw and barrel and considering them to be respectively a straight trough and a flat plate as presented Figure 22. Barrel movement direction makes an angle with screw channel that corresponds to screw flight helix angle.



a. unrolled view b. view section B-B

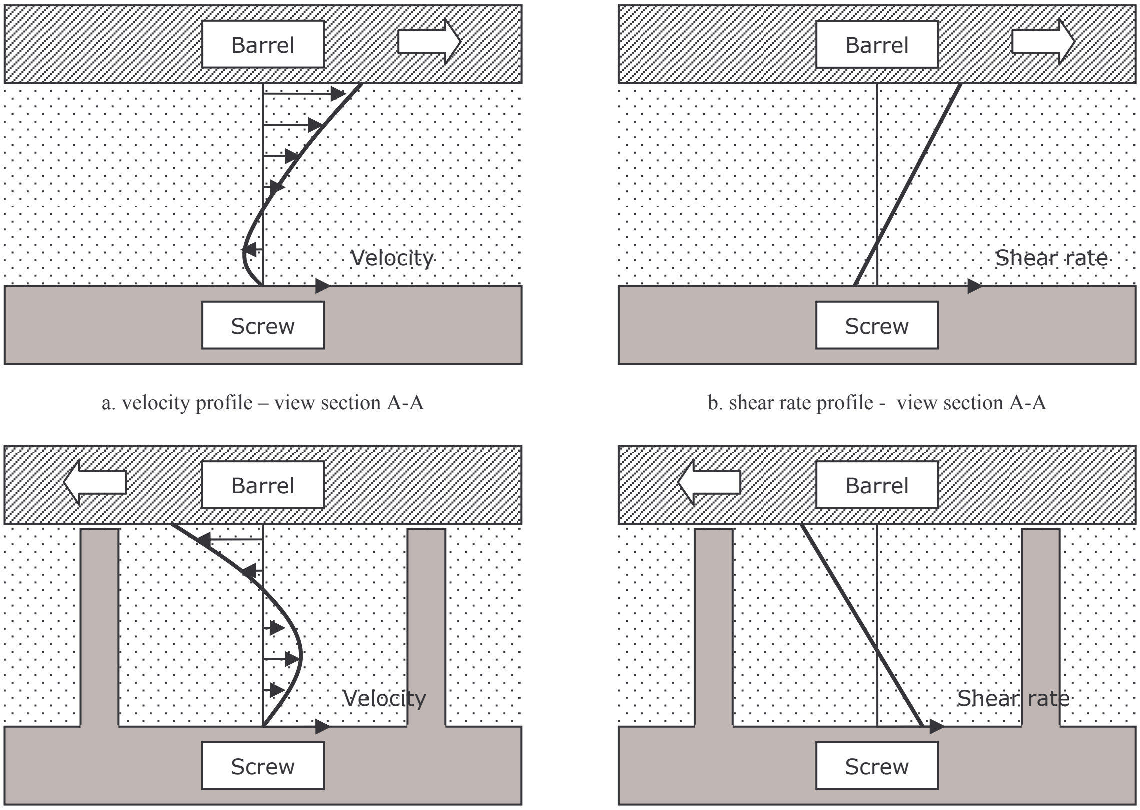
Figure 22 – Plastic melt element path in screw channel

Considering an element from the upper region of the channel:

- It travels following barrel movement until the pushing side of the flight.
- It moves downward until the bottom of the channel.
- It travels across the channel until the trailing side of the flight.
- It moves upward until the top of the channel

This pattern is repeated numerous times along screw channel. Therefore an element follows a helical path. Flow close to barrel wall is induced by viscous drag whereas flow across screw channel is generated by the negative pressure gradient between pushing side and trailing side of the flight.

Figure 23 presents velocity and shear rate profiles in down-channel (section A-A) and cross-channel (section B-B) of the screw.



a. velocity profile – view section A-A

b. shear rate profile - view section A-A

c. velocity profile – view section B-B

d. shear rate profile - view section B-B

Figure 23 – Velocity and shear rate profiles in screw channel

Pressure gradient in screw channel is positive as the screw works like a pump (see 3.1.). Therefore there is a backflow also called recirculation at the bottom of down-channel as shown Figure 23 a. which intensity increases with increasing melt pressure.

Mass flow balance inside the machine is given by the following formula:

$$Q_{screw} = Q_{output} + Q_{recirculation}$$

With:

Q_{screw} Mass flow provided by the screw

Q_{output} Mass flow provided by the machine

$Q_{recirculation}$ Mass flow recirculation inside the extruder

When recirculation intensity increases, plastic melt average residence time inside the machine increases and temperature increases either. Therefore average melt temperature increases when melt pressure increases.

3.1.4. **Mixing**

Shear rate in screw channel is not uniform as shown Figure 23. Thus viscous heating is not uniform either and melt temperature in screw channel is likely to be heterogeneous because of polymer low thermal conductivity (see 2.3.).

If melt temperature is heterogeneous in die channel, plastic melt flow will be heterogeneous either. It will result in a distorted pipe. Therefore it is necessary homogenising the material before it reaches the die by providing an intensive mixing during melt conveying.

Mixing intensity is deduced from shear strain that is the product of shear rate and time of exposure to shearing:

$$\gamma = \dot{\gamma} \times t_{\dot{\gamma}}$$

With:

γ Shear strain

$\dot{\gamma}$ Shear rate

$t_{\dot{\gamma}}$ Time of exposure to shearing

Thus mixing distribution in screw channel depends on:

- shear rate distribution
- residence time distribution:
    - flow path
    - velocity profile

Part of mixing mechanism occurs in screw channel. Shear distribution is presented Figure 23. Residence time is deduced from flow path and velocity profile presented respectively Figure 22 and 23. It is long close to barrel and screw walls and short further away from the walls as illustrated Figure 24.

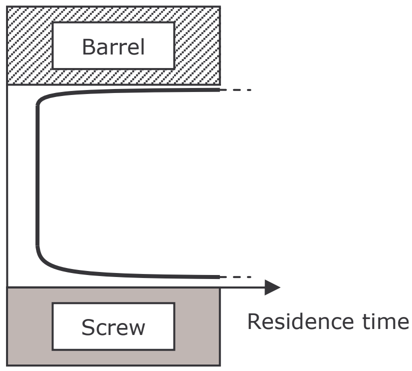


Figure 24 - Plastic melt element residence time in screw channel

Thus total cross-channel shear strain is approximately the sum of shear strain in lower and upper regions of the channel. Total down-channel shear strain is deduced the same way. Total shear strain is the sum of total cross-channel shear strain and total down-channel shear strain. Therefore it depends also strongly on element location in channel depth as illustrated Figure 25.

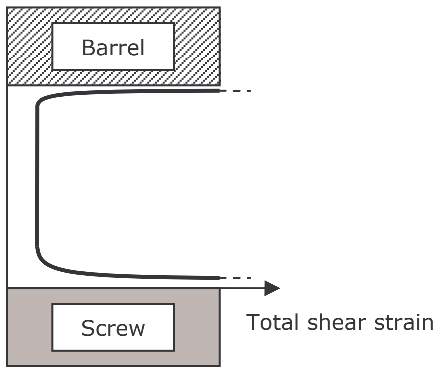


Figure 25 – Total shear strain in screw channel

Elements close to barrel and screw walls experience a high level of shear strain whereas elements further away from the walls experience a low level. Thus mixing mechanism in screw channel is not uniform and plastic melt temperature could be not fully homogeneous at the exit of extrusion machine. However mixing mechanism can be improved by adding mixing sections along material channel inside the machine. In Hutchinson five-layer machine, these sections are included in the design of multi-manifold component presented Figure 14 a.

3.2. Forming

Forming operation gives extruded product shape to plastic melt. Forming tools are located at the exit of extrusion machine. Pipe products are formed with annular die and annular mandrel. Examples of annular die and annular mandrel are presented Figure 26.

a. die b. mandrel

Figure 26 – Extruded pipe forming tools

Flow channel is designed so that flowing material achieves a uniform velocity across die exit. It can be divided in three zones as illustrated Figure 27:

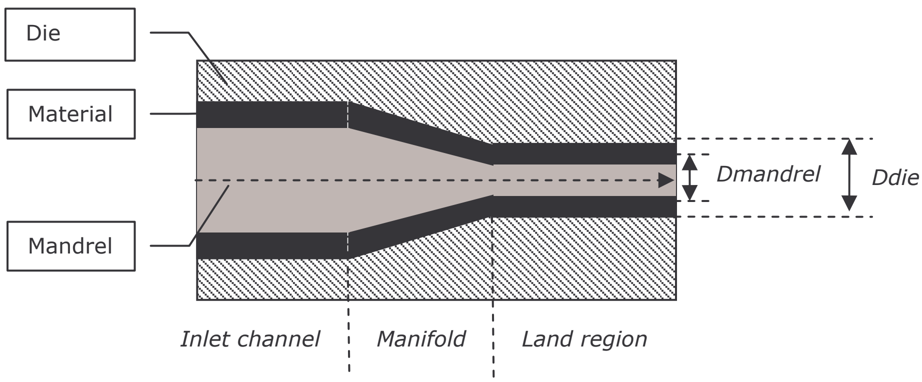


Figure 27 – Die flow channel zones

Two factors can characterize extruded pipe at the exit of extrusion machine. These factors are important because they influence sizing operation conditions:

- Diameter ratio
- Thickness ratio

Their formula is given below:

$$D_{ratio} = \frac{D_{die}}{D_{outer\_pipe}}$$

$$T_{ratio} = \frac{D_{die} - D_{mandrel}}{D_{outer\_pipe} - D_{inner\_pipe}}$$

With:

D_{die} Extruded pipe outer diameter (= Die diameter)

$D_{mandrel}$ Extruded pipe inner diameter (= Mandrel diameter)

D_{outer_pipe} Final pipe outer diameter

D_{inner_pipe} Final pipe inner diameter

Melt pressure inside the machine is strongly influence by forming tool dimensions as illustrated Figure 28 since it depends on pressure discharge along die channel given by the following formula:

$$\Delta P = K.\eta_{apparent}.Q \qquad \textit{(Poiseuille law)}$$

With:

ΔP	Pressure discharge
K	Geometrical and dimensional factor
$\eta_{apparent}$	Material apparent viscosity
Q	Material flow

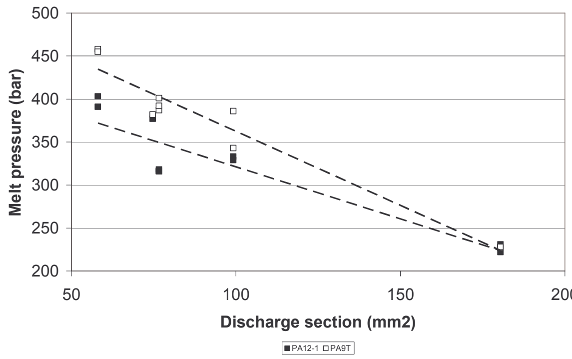


Figure 28 – Influence of forming tool dimensions on melt pressure inside the extruder

Material viscosity close to tooling wall increases when tooling temperature decreases. Thus material apparent viscosity increases and pressure discharge at given material flow increases either. Therefore melt pressure inside the machine increases. However average melt temperature increases when melt pressure increases (see 3.1.3.). Moreover plastic melt viscosity decreases when plastic melt temperature increases (see 2.9.1.). Therefore plastic melt viscosity can be decreased by decreasing forming tool temperatures.

From previous analysis, plastic melt viscosity at the exit of extrusion machine depends also on:

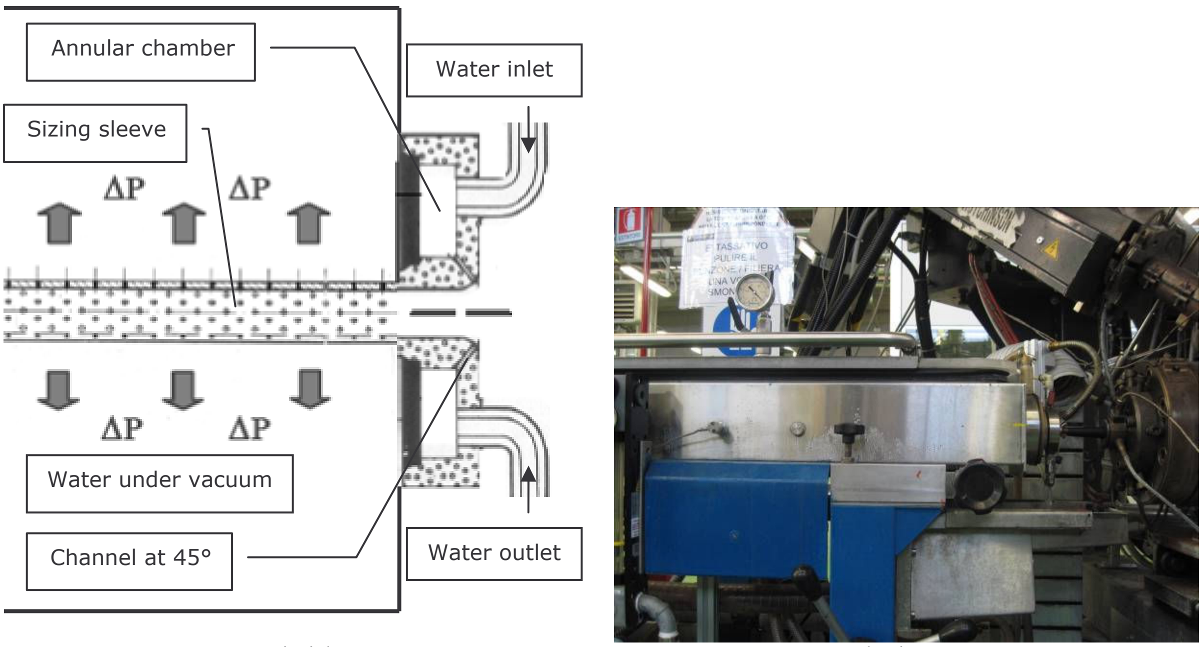
- forming tool dimensions: the smaller the discharge section, the lower the viscosity
- forming tool temperature: the lower the tool temperatures, the lower the viscosity

3.3. Sizing and cooling

Sizing operation fixes extruded pipe final dimensions, especially external diameter and thickness.

Extruded pipe needs to be supported otherwise it would collapse under its own weight. It means that after sizing operation, pipe surface should be stiff enough to not be damaged by guiding devices. Therefore it has to be cooled down rapidly and in this condition, air cooling is not sufficient and water cooling is necessary.

Hutchinson vacuum-sizing system is presented Figure 29.



a. principle b. picture

Figure 29 – Hutchinson sizing system

In this system, the pipe is sized from outside while passing through the sizing sleeve located inside the vacuum tank. The vacuum tank is filled with cooling water. Vacuum level is typically between 60 and 300 mbar. Water lubrication is required to reduce friction at the interface between pipe and sleeve. This water is provided by the annular chamber located at the entrance of the sizing sleeve. Water flow is typically between 40 and 80 L/h.

An example of sizing sleeve is presented Figure 30. Sizing sleeve is made of high thermal conductivity material such as copper, bronze or brass. It has a cylindrical geometry like the pipe but its internal diameter is higher than final pipe external diameter, typically between 1.07 and 1.1 times, to take into consideration material shrinkage during solidification (see 2.9.2.). Its minimum length depends mainly on line speed: the higher the speed, the longer the sleeve. Holes drilled along the sleeve allow vacuum drawing the material on sleeve inner wall and fix pipe external diameter: the higher the vacuum, the larger the external diameter at given sizing sleeve. Therefore vacuum is an important factor to control.

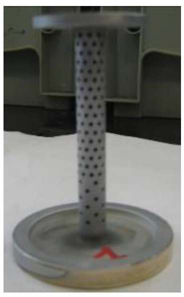


Figure 30 – Hutchinson sizing sleeve

Three major phenomena are involved in sizing process These phenomena are important because they affect pipe mechanical properties:

- heterogeneous cooling rate: residual stress in pipe material
- draw down and violent cooling rate: crystal rate and molecular orientation of pipe material
- friction: surface state of the pipe, draw down

3.3.1. Heterogeneous cooling rate

While entering into the sizing sleeve, the pipe is violently cooled down at the contact of annular chamber water. This violent cooling creates a high thermal gradient in the external layer of the pipe due to polymer low thermal conductivity (see 2.3.). This gradient depends initially on water temperature. Then it decreases steadily along cooling bath as heat from the core is conduced outside the pipe and dissipated in the water.

Heterogeneous cooling rate in pipe external layer generates residual stress in the material due to the variation of material specific volume during solidification process (see 2.9.2.). This mechanism is illustrated Figure 31. This residual stress affects pipe mechanical properties, especially pipe elongation at break.

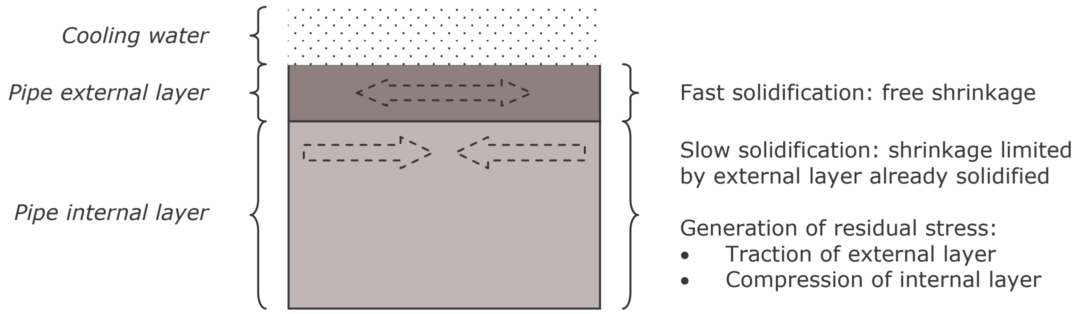


Figure 31 – Residual stress generation mechanism

3.3.2. Draw down and violent cooling rate

Pipe thickness depends on pipe section and pipe external diameter determined by vacuum level (see 3.3.). Pipe section depends on pipe draw down which is the ratio between extrusion line speed and plastic melt speed inside the die given by screw rotation speed. Therefore pipe thickness is determined by pipe draw down and vacuum level. Pipe draw down ratio (DDR) is given by the following formula:

$$DDR = \frac{V_{line}}{V_{melt}} \approx \frac{D_{die}^2 - D_{mandrel}^2}{D_{outer\_pipe}^2 - D_{inner\_pipe}^2} \qquad \textit{(Mass flow conservation)}$$

Draw down mechanism occurs both between die and sizing sleeve and inside sizing sleeve as illustrated Figure 32. Therefore previous formula can also be written:

$$DDR = \frac{V_{line}}{V_{melt}} = \frac{V_{line}}{V_{sleeve}} \times \frac{V_{sleeve}}{V_{melt}} = DDR_{sleeve} \times DDR_{air}$$

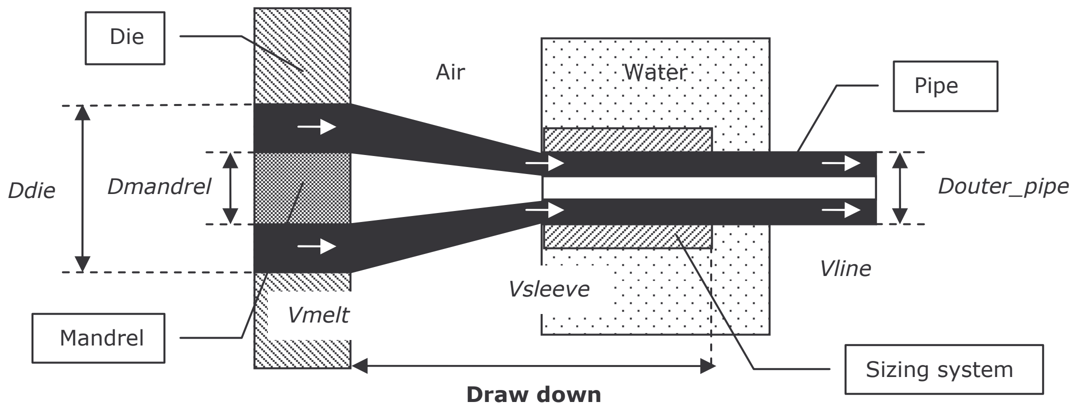


Figure 32 – Schematic of sizing operation

Pipe draw down inside sizing sleeve increases with increasing friction as illustrated Figure 33. Friction forces are concentrated at the beginning of the sleeve because of pipe shrinkage during cooling process (see 2.9.2.).

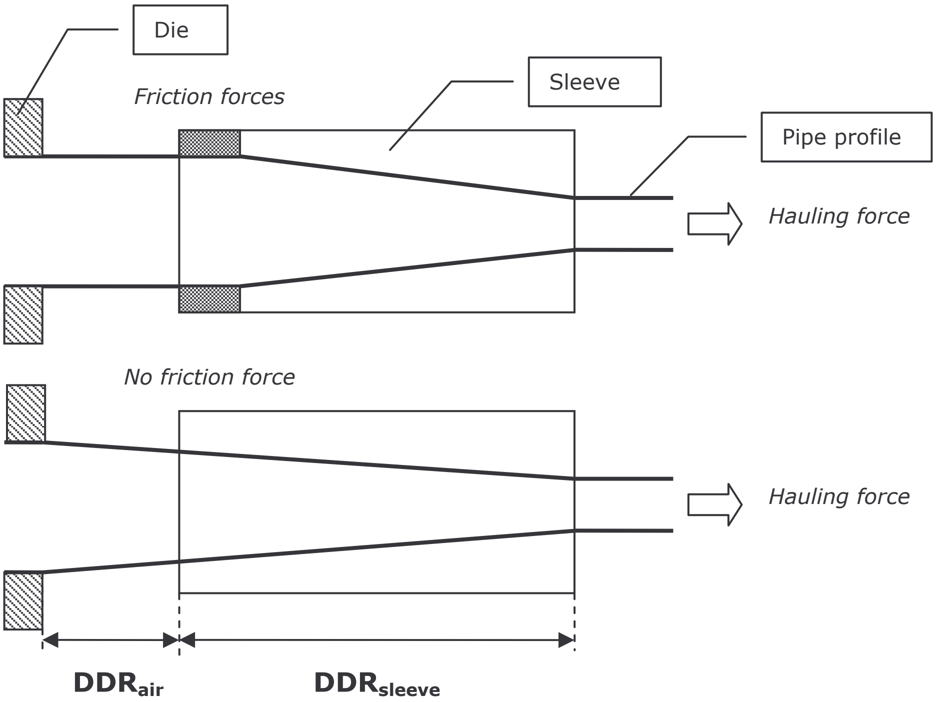


Figure 33 – Influence of friction forces inside sizing sleeve on draw down distribution

Friction forces inside sizing sleeve counteract caterpillar hauling force. Thus when friction forces increase, pipe hauling force upstream the sleeve decreases. Therefore air draw down decreases and draw down inside the sleeve increases in the meantime.

3.3.2.1 Between die and sizing system

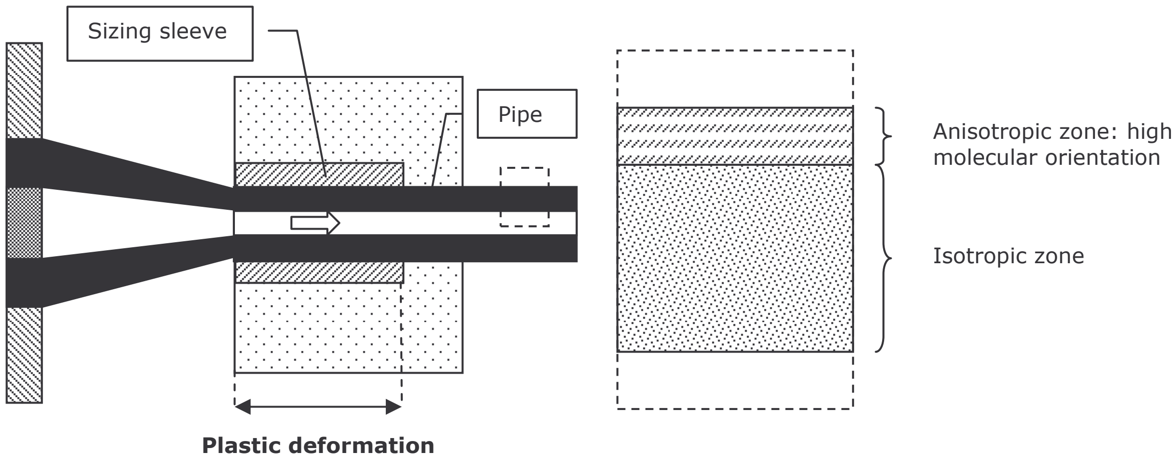
Between die and sizing system, the material is not solidified yet. As a result, draw down does not affect the process as long as no cracks appear in the flow. These cracks appear if plastic melt viscosity is too high or not homogeneous (inclusions of non melted or degraded material). If viscosity is too low, melted pipe will collapse before reaching the sleeve and it will not be possible to size it.

3.3.2.2 Inside sizing sleeve

Inside the sleeve, draw down mechanism occurs while solidification process is in progress. In fact, solidification starts at the contact of annular chamber water and continues along the sleeve. The combined effect of draw down and violent cooling rate generates a high thermo-mechanical load on the pipe that results in a plastic deformation of the material being solidified. This phenomenon is limited to pipe external layer since solidification is firstly limited to pipe external layer (see 3.3.1.).

Previous studies confirm this analysis. In fact, the crystalline structure of a pipe has been analysed after sizing operation and two singularities have been noticed in the external layer (about 200 µm) which reveal the presence of plastic deformation mechanism:

- high crystal rate whereas cooling is violent
- high anisotropy whereas internal layer is isotropic: illustrated Figure 34



a. sizing operation b. pipe molecular structure

Figure 34 – Influence of sizing operation on pipe molecular structure

Violent cooling results usually in low crystal rate because crystallisation kinetic is too slow. As a consequence, plastic deformation has accelerated and orientated crystallisation by providing mechanical energy.

Pipe mechanical properties, especially pipe elongation at break, depend on material crystal rate and molecular orientation (see 2.9.3.). Therefore they depend on draw down and cooling inside sizing sleeve: the higher the thermo-mechanical load, the lower the elongation at break. Effect of thermo-mechanical load on pipe elongation is illustrated Figure 35.

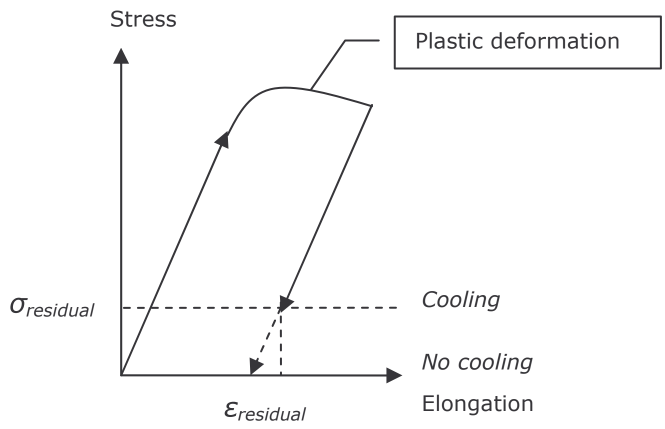


Figure 35 – Effect of thermo-mechanical load on pipe elongation

From previous analysis, draw down conditions during sizing operation depends on:

- plastic melt viscosity at the exit of extrusion machine
- forming tool dimensions: the higher the dimensions, the higher the draw down
- water temperature: the lower the temperature, the more violent the cooling rate while drawing down
- friction: the higher the friction, the higher the draw down inside the sleeve

3.3.3. **Friction**

During sizing operation, extruded pipe is forced through the sizing sleeve. Interaction between pipe and sleeve generates friction forces. When friction forces are high, some defects can appear on pipe surface, especially if there is adhesion between pipe and sleeve. These defects are potential crack initiations during pipe elongation.

Friction force intensity along the sleeve depends on:

- extrusion line speed: the higher the speed, the higher the friction
- water lubrication: the higher the lubrication, the lower the friction
    - vacuum: the higher the vacuum, the lower the lubrication
    - sleeve internal diameter: the larger the diameter, the lower the vacuum
- sleeve surface state: the better the surface state, the lower the friction
- pipe material shrinkage: friction forces concentrated at the beginning of the sleeve

3.3.3.1 Extrusion line speed

A previous study carried out with a specific sizing system equipped with a stress gauge [CARI05] shows that friction increases when extrusion line speed increases. In fact, pipe and sleeve interaction through the water film generates a viscous friction, typically speed dependant.

3.3.3.2 Water lubrication

Using the same equipment, previous study demonstrates also that friction increases when vacuum increases as shown Figure 36.

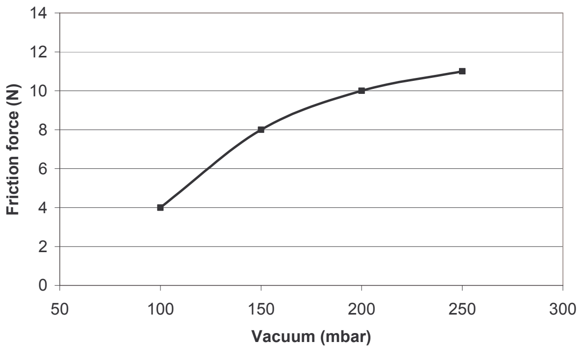


Figure 36 – Influence of vacuum level on friction [CARI05]

In fact, while travelling along the sleeve, lubricating water film is dissipated through the holes by vacuum effect: the higher the vacuum, the shorter and the thinner the lubrication.

3.3.3.3 Sleeve surface state

Sleeve surface state influences two mechanisms that occur at the interface between pipe and sleeve:

- capillary water flow: the higher the roughness, the higher the lubrication
- friction: the higher the roughness, the higher the friction

A study could be performed to quantify the effect of sleeve roughness on both mechanisms. Such a study is out of the scope of this thesis. For some applications, Hutchinson sleeves are coated with chromium to get a smooth surface that is easier to control in production.

3.4. Conclusion

Before working on extrusion process improvement, it is necessary identifying, for each operation of the process, main factors that influence pipe essential characteristics, then identifying extrusion line parameters which allow controlling these factors.

3.4.1. **Extrusion factors**

Process factors that influence pipe essential characteristics during extrusion operation are the following:

- Feeding regularity
- Melting rate
- Melt viscosity

Feeding regularity determines extrusion machine output stability. Melting rate is adjusted so that melting process occurs exclusively in the compression zone of the screw. If end this process is postpone, there could be some inclusions of non melted material in extruded pipe. If beginning of this process is anticipated, solids conveying process could be disrupted affecting feeding regularity. Plastic melt viscosity is adjusted to have compatible flow properties when materials from the different extruders are combined to limit distortion phenomenon at pipe layer interface. Plastic melt viscosity should be also compatible with sizing operation. If viscosity is too high or not homogeneous, some cracks could appear while melted pipe is drawn down. If viscosity is too low, melted pipe will collapse before reaching the sleeve and it will not be possible to size it.

These factors depend on:

- material characteristics
- screw design
- operating conditions (machine parameters)

The screw is the principal component of an extruder. It is originally designed for a unique combination machine-material. Therefore its design is optimized according to the characteristics of the material as presented Table 3.

Table 3 – Influence of material characteristics on optimal screw design

Screw design (effect) / Characteristics (+)	Helix angle	Compression ratio
Pellet flow resistance	+	
Sensibility to shearing	-	+

In practice, the same screw is used for several materials when it is possible. However operating conditions may have to be adapted to keep process factors matching with pipe required characteristics. Machine parameters that can be adjusted on extrusion line are presented Table 4 with their respective influence on process factors.

Table 4 – Effect of increasing extrusion line parameters on process factors

Factors (effect) / Parameters (+)	Feeding regularity	Melting rate	Melt viscosity
Screw speed		+	-
Barrel temperature in feeding zone	-		
Barrel temperature in compression zone		+ / -	
Barrel temperature in metering zone			=
Head temperature			=
Forming tool dimensions			+
Forming tool temperature			+

Feeding regularity depends on solids conveying mechanism, especially friction of extruder barrel on the pellets, which is influenced by barrel temperature in feeding zone. Melting rate and plastic melt viscosity depend on shear rate inside the extruder determined by screw speed. Melting rate depends also on barrel temperature in compression zone: over a given temperature, it decreases with increasing barrel temperature because viscous heating in the melted film decreases more than conductive heating increases. Plastic melt viscosity depends also on forming tool dimensions and temperature that influence melt pressure inside extrusion machine then melt temperature because of recirculation phenomenon. Influence of barrel temperature in metering zone and head temperature on plastic melt viscosity is limited by polymer low thermal conductivity.

Impact of each parameter on process factors and pipe essential characteristics will be studied in detail during the study on process reliability improvement that will be carried out in 2012. Beforehand, extrusion line parameters to monitor process factors have been identified and are presented Table 5.

Table 5 – Monitored parameters on extrusion line

Factors / Parameters	Feeding regularity	Melting rate	Melt viscosity
Motor load	X		X
Melt temperature			X
Melt pressure	X		X

Feeding regularity affects melt pressure stability whereas plastic melt viscosity affects melt temperature and melt pressure levels. Motor load is equivalent to melt pressure. Melting rate can be monitored only by checking the presence of inclusions on melted pipe at the exit of extrusion machine.

3.4.2. Sizing factors

Process factors that influence pipe essential characteristics during sizing operation are presented Table 6 with their respective influence on pipe characteristics.

Table 6 – Effect of increasing process factors on extruded pipe characteristics

Pipe characteristics (effect) / Factors (+)	External diameter	Thickness	Elongation at break
Cooling rate			-
Draw down		-	-
Friction			-
Vacuum	+	-	-

Pipe external diameter and pipe thickness depend on vacuum inside water tank that draws the material on sleeve inner wall. Pipe thickness depends also on pipe section determined by pipe draw down. Pipe elongation at break depends on material residual stress in pipe external layer which results from a heterogeneous cooling rate in pipe external layer and the variation of material specific volume during solidification process. Pipe elongation at break depends also on the thermo-mechanical load on pipe external layer which results from the combination of a violent cooling rate of pipe external layer and pipe draw down inside sizing sleeve. Lastly, pipe elongation at break depends on friction inside sizing sleeve that can generates some defects on pipe surface which are potential crack initiations.

Machine parameters that can be adjusted on extrusion line to keep process factors matching with pipe required characteristics are presented Table 7 with their respective influence on process factors.

Table 7 – Effect of increasing extrusion line parameters on process factors

Factors (effect) / Parameters (+)	Cooling rate	Draw down	Friction	Vacuum
Screw speed*		-		
Forming tool dimensions		+		
Water temperature	-			
Extrusion line speed**		+	+	
Water flow			-	
Vacuum level			+	+
Sleeve dimensions			-	-

* at constant extrusion line speed
** at constant screw speed

Pipe cooling rate depends on annular chamber water temperature. Pipe draw down depends on the ratio between pipe section at the exit of extrusion machine determined by forming tool dimensions and pipe final section. It depends also on the ratio between pipe speed at the exit of extrusion machine determined by screw speed and extrusion line speed. Vacuum depends on the force required to draw the material on sizing sleeve internal wall and as a consequence, it depends on sleeve internal diameter. Friction depends on the thickness of the lubricating film inside sizing sleeve which decreases along the sleeve as water is dissipated by vacuum effect. Viscous friction in lubricating film depends on extrusion line speed.

It can be noticed that many factors are interdependent. For instance, plastic melt viscosity at the exit of extrusion machine and pipe draw down inside sizing sleeve depend both on forming tool dimensions.

Impact of each parameter on process factors and pipe elongation at break will be studied chapter 5.

4. Pipe essential characteristics monitoring

Before working on extrusion process improvement, it is necessary being able to monitor extruded pipe essential characteristics.

Monitored essential characteristics of a product are identified from specification requirements and are specified into the control plan. Product quality management is presented more in detailed in Appendix 2. For extruded pipes, monitored characteristics and their respective controls are divided in two categories:

- dimensional controls:
    - non destructive controls:
        - ultrasonic system: total thickness
        - laser system: external diameter
    - destructive controls:
        - microscope instrument: internal diameter, external diameter, intermediate layer thickness, total thickness
- structural controls:
    - burst test: burst pressure
    - cold impact test: cold impact resistance
    - tensile test: elongation at break

This chapter presents control method and instrument for each characteristic monitored. It includes also a study to improve current dimensional controls in anticipation of the study on process reliability improvement that will be carried out in 2012 and a study to improve tensile test reliability before working on pipe elongation at break improvement.

4.1. Dimensional controls

Dimensional controls allow characterising pipe dimensional properties. Dimensions and tolerances required for the structure PA9T/PA12 10x1are presented Figure 37.

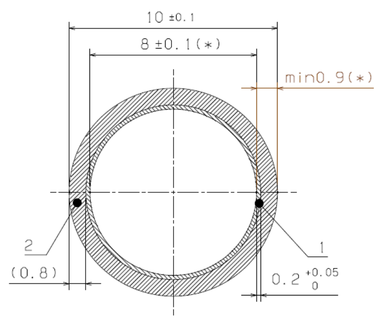


Figure 37 –PA9T/PA12 10x1 structure drawing

There are two categories of dimensional controls:

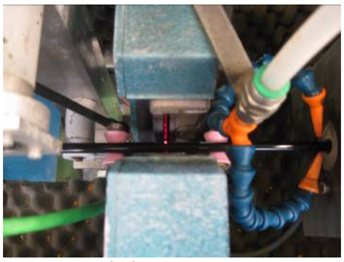
- non destructive controls
- destructive controls

4.1.1. Non destructive controls

Extrusion line is equipped with two monitoring instruments presented Figure 38:

- ultrasonic system: total thickness
- laser system: external diameter

a. Ultrasonic system



b. laser system

Figure 38 – Monitoring instruments on extrusion line

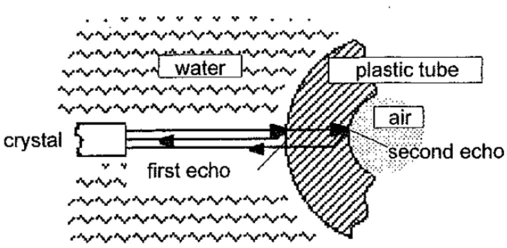
These instruments are used:

- in starting phase to adjust extrusion parameters: set-up time reduction
- in production phase to monitor process deviations: product characteristic control

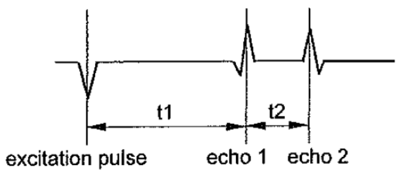
They can only detect broad deviations on pipe dimensions, and not local ones. In fact, control is made according to a given frequency and as a consequence, it is not continuous. Local defects can be controlled using a dedicated mistake-proofing system. Instruments are separated on the line since their operating conditions are incompatible. In fact, ultrasonic system works in liquid environment whereas presence of water droplets on window can distort laser system measurement.

4.1.1.1 Ultrasonic system

The ultrasonic system is positioned around the circumference of the pipe. It is composed of piezoelectric crystals which convert reversibly electrical energy into mechanical energy. Its working principle is presented Figure 39.



a. ultrasonic wave generation and reflection



b. electrical signal measured

Figure 39 – Schematic of ultrasonic system working principle [ZUMB07]

The electronic unit transfers a short electrical pulse to the crystals that are excited and generate sound waves. When sound waves encounter a difference of material density, for instance passing from water to pipe material, part of them are reflected to the crystals that transfer an electrical signal to the electronic unit. When a pipe is measured, its outer wall creates a first echo and its inner wall a second echo. Wall thickness is deduced from the time difference between the two echoes.

Propagation speed of sound waves varies according to material density and as a consequence, according also to material temperature. Thus measurement accuracy of ultrasonic system depends on pipe layer materials and on pipe temperature at the checking point which is difficult to control on the line. As a consequence, this system can be used only to monitor process deviations and not as a measurement instrument like a microscope.

4.1.1.2 Laser system

The laser system is positioned around the circumference of the pipe system. It is composed of two laser diodes that generate laser beams and scan the object measured. Its working principle is presented Figure 40.

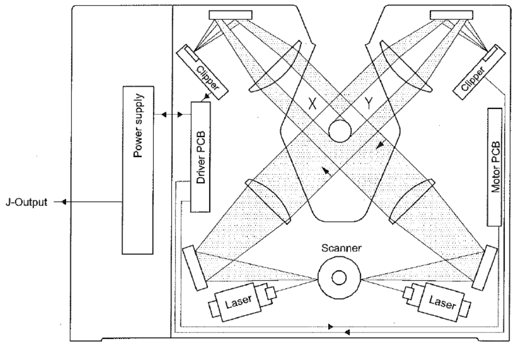


Figure 40 – Schematic of laser system working principle [ZUMB07]

Laser beams are deflected by the rotating mirror. Two deflection mirrors direct the rays into the optic emitters from which they are sent to the measuring field as two absolutely parallel beams at a right angle to each other. Receiver lenses focus the luminous beams onto photodiodes by means of deflection mirrors. The photodiodes do not receive any light when laser beams are outside of the measuring field or if there is an object in their path. Resulting light shadow signals are detected by the photodiodes and converted into electronic signals to be sent to the data acquisition system. The duration of the shadow cast by the object is measured by the data acquisition system which converts it into a dimension.

Next two sections present a feasibility study to substitute partly destructive controls by non destructive controls extending use of ultrasonic and laser systems. First section concerns the measurement of intermediate layer thickness of multilayer pipes using ultrasonic system and second section concerns the calculation of pipe internal diameter from the data of pipe external diameter given by laser system and pipe thickness given by ultrasonic system.

4.1.1.3 Pipe intermediate layer measurement

Measuring intermediate layer thickness of multilayer pipes is not feasible with current system resolution. In fact, densities of layer materials are very close together. Thus reflected signal at the interface between two layers is very low and cannot be detected by the system as shown Figure 41.

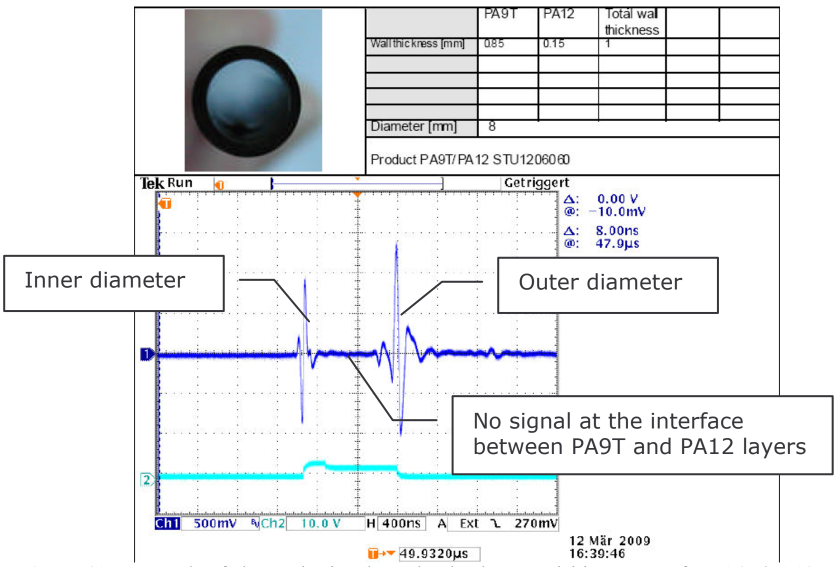
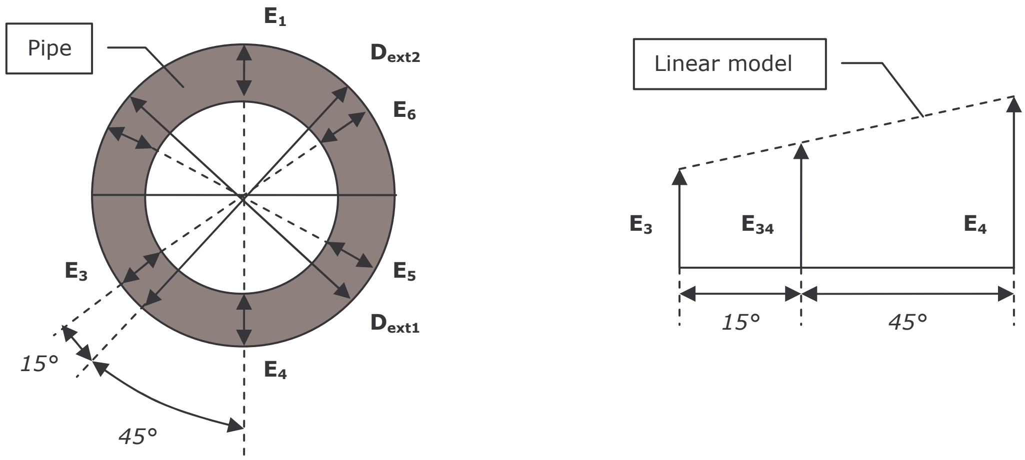


Figure 41 – Example of electronic signal sent by the data acquisition system for PA9T/PA12 structure

4.1.1.4 Pipe internal diameter calculation

Calculation of pipe internal diameter from the data of pipe external diameter given by laser system and pipe total layer given by ultrasonic system can be deduced from the model presented Figure 42.



a. measurement direction of current devices

b. unrolled view: linear model

Figure 42 – Calculation of internal diameter from external diameter and thickness values

Thus pipe internal diameter is calculated from the following formula:

$$D_{\text{int}1} = D_{ext1} - (e_{12} + e_{45})$$
$$D_{\text{int}2} = D_{ext2} - (e_{34} + e_{61})$$

With:

$$E_{12} = \frac{3.E_2 + E_1}{4}$$
$$E_{34} = \frac{3.E_3 + E_4}{4}$$
$$E_{45} = \frac{3.E_5 + E_4}{4}$$
$$E_{61} = \frac{3.E_6 + E_1}{4}$$

This model is based on the assumption that measurement triggering between ultrasonic and laser systems can be synchronized. However, after having consulted the supplier, synchronisation between the two systems is not possible. Thus calculating pipe internal diameter using ultrasonic and laser systems is not feasible.

4.1.2. **Destructive controls**

Destructive controls comprise two steps:

- sample preparation: cutting the pipe
- sample analysis: using an optical instrument

Two types of instruments can be used for dimensional analysis:

- profile projector: internal diameter, external diameter, total thickness
- microscope: internal diameter, external diameter, total thickness, layer thickness

Use of profile projector is limited to monolayer pipes as internal layers cannot be measured. Therefore this study will be interested only in microscope instrument.

4.1.2.1 Current method

Sample preparation consists of cutting by hand the pipe with a pipe-cutter. The sample is positioned in front of a microscope equipped with a video-camera which picture is displayed on a computer screen. Luminosity and sharpness are adjusted and the picture is analysed with a dedicated program. An example of analysis is presented Figure 43.

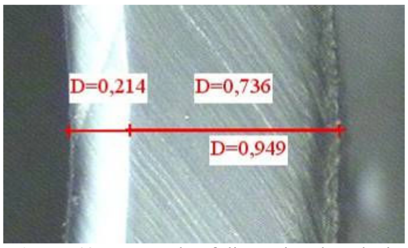


Figure 43 – Example of dimensional analysis

Thus measurement quality depends on the following parameters:

- sample cutting
  - section not altered to be able to distinguish clearly layer borderlines
  - section perpendicular to pipe axis to measure circles and not ellipsis
  - section not deformed to measure circle and not ellipsis
- sample positioning
  - section parallel to optical plane to be able to get a sharp picture
- picture aspect
  - luminosity adjusted to maximize contrast between layers (sample length)
  - sharpness adjusted to maximize contrast gradient between layers
- vision accuracy
  - high discrimination capacity to distinguish precisely layer borderlines

As each operation is performed manually, measurement quality relies on operator skills. As a consequence, it can vary from a controller to another one. According to quality standards, uncertainty on the value measured must be less than ten percent of dimension tolerance otherwise measurement method is considered to be not capable. This criterion is checked carrying out a Repeatability and Reproducibility study (R&R study). In the case of current dimensional control, study result indicates that method is acceptable but can be improved to reduce measurement variability, especially for multilayer structures which tolerances on layer dimensions are smaller than those of monolayer structures. Therefore it is necessary improving this aspect before the study on process reliability improvement that will be carried out in 2012.

Two axes of improvement can be deduced from previous analysis:

- developing a specific cutting machine
- developing a program to automate measures

Thus next sections present a concept of cutting machine then a program to automate measures. However their development is out of the scope of this thesis.

4.1.2.2 Cutting machine

A basic outline of the problem is presented Figure 44.

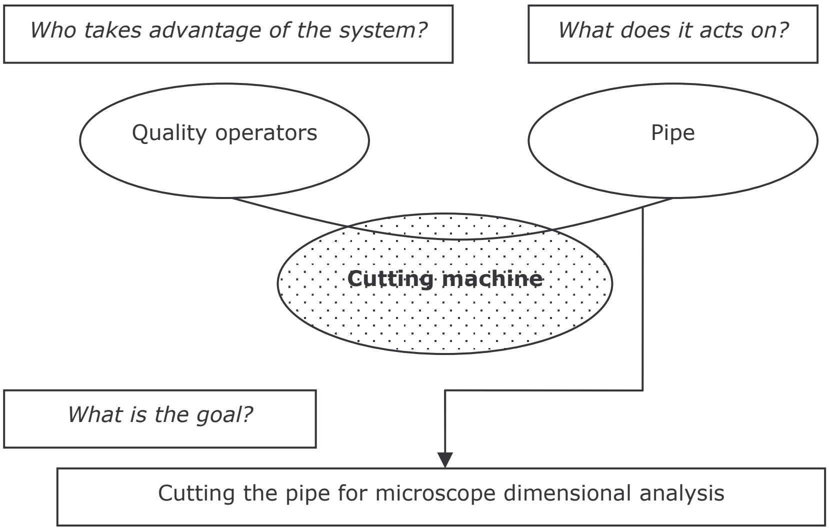


Figure 44 – Basic outline of the problem

Several cutting machines were developed in the past but they did not completely meet the requirements specified in section 4.1.2.1:

- Spring-drive cutting machine:
  - section altered by the cutting depending on material stiffness (blade marks)
- Razor blade cutting machine:
  - section altered locally by the cutting (blade impact zone)
  - section not perpendicular to pipe axis
  - section deformed

Two examples of machines are presented Figure 45.

a. spring-drive cutting machine

b. razor blade cutting machine

Figure 45 – Specific cutting machines developed in the past

Thus it is necessary developing a new machine which design should be based on a detailed functional analysis. Functional analysis then machine design are presented in this section.

First, expected functions of the system have been identified:

- Operating functions (OF)
- Constraining functions (CF)

Interactions of the system with its environment are presented Figure 46. Functions associated are described Table 8.

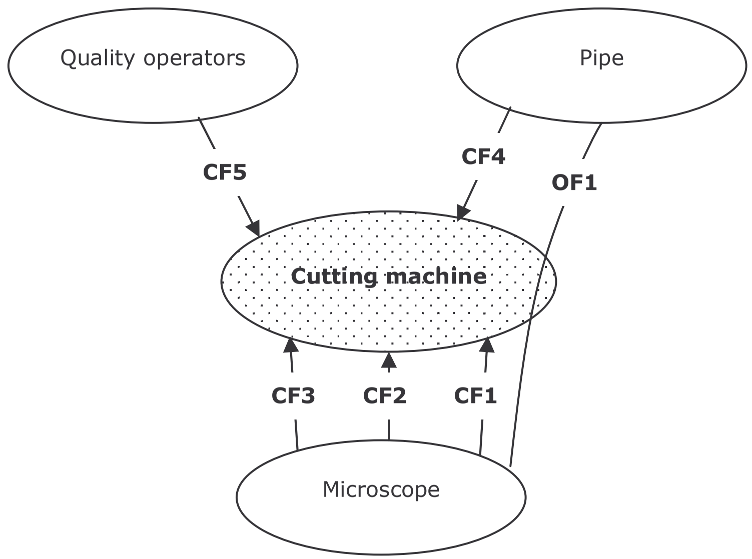
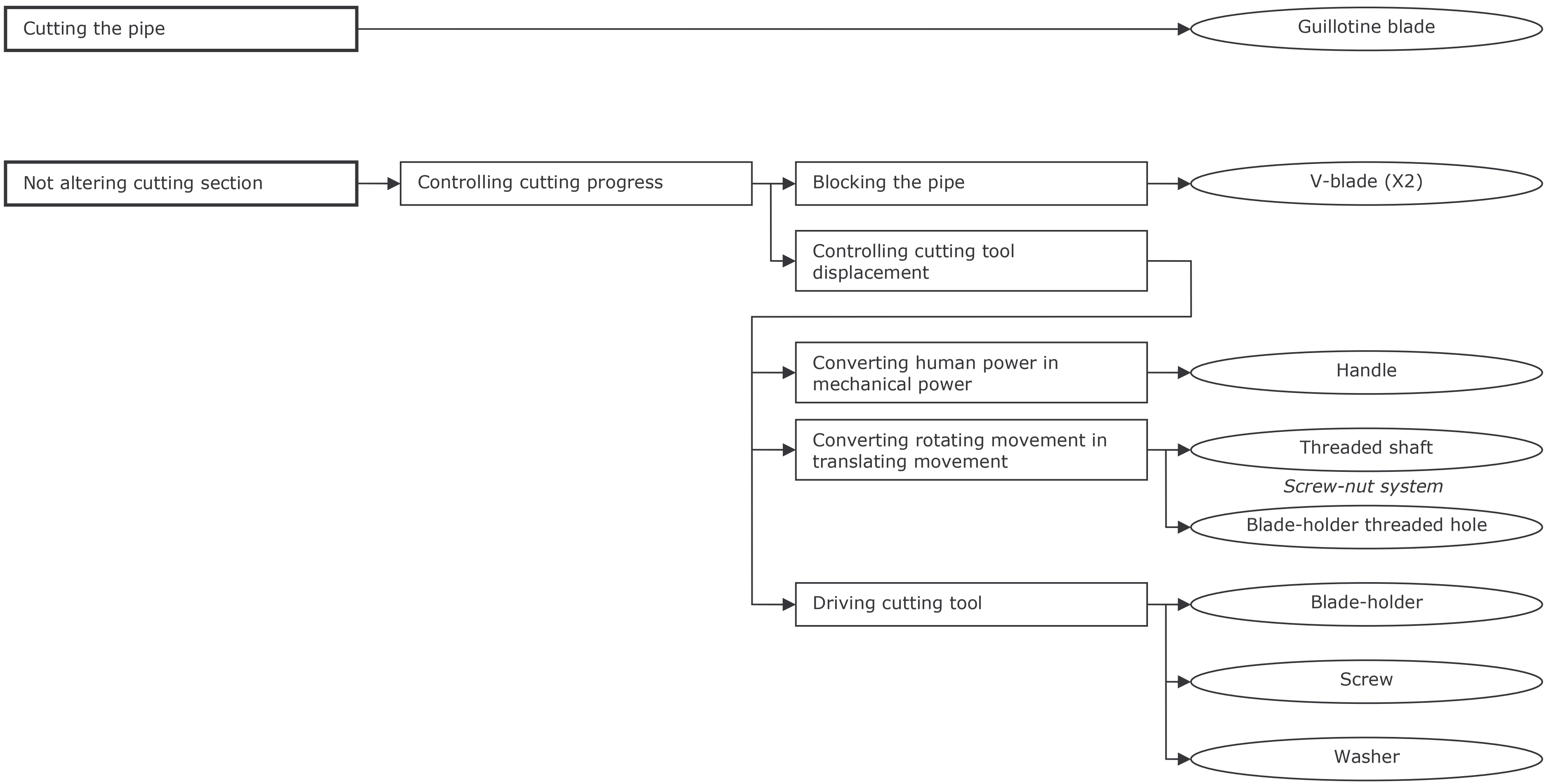


Figure 46 – Interaction diagram

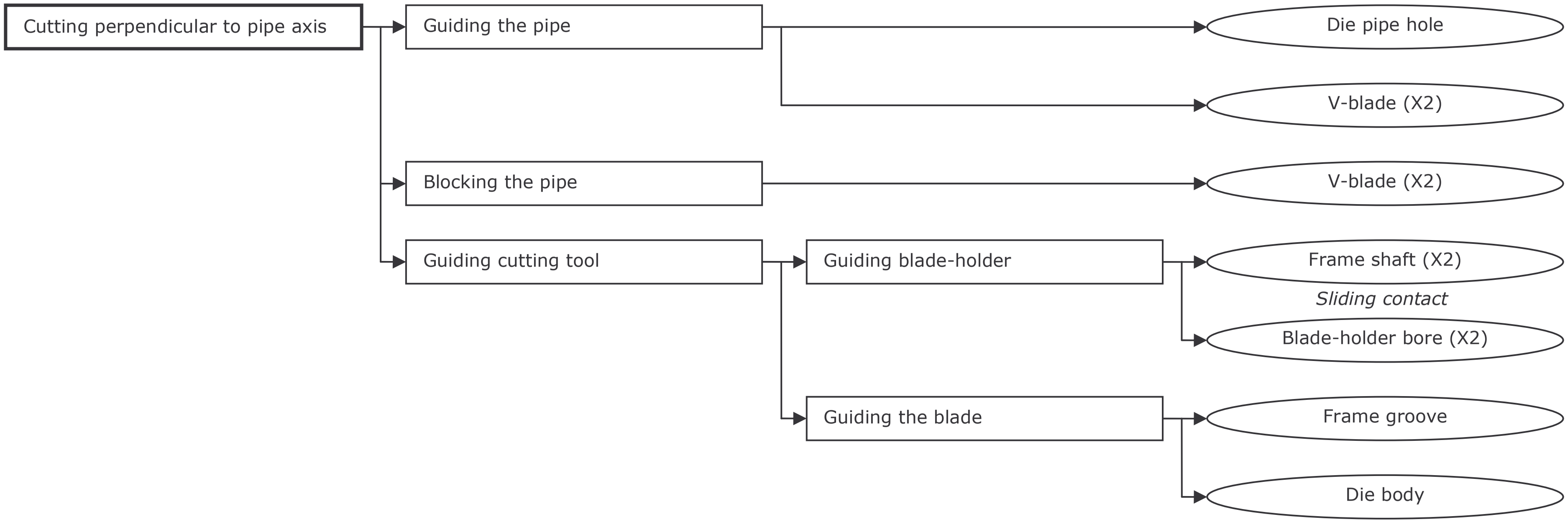
Table 8 –Function description

N°	Description	Criterion	Level
OF1	Cutting the pipe	Two separated parts at operation end	Yes
CF1	Not altering cutting section	Presence of marks that could interfere with layers	No
CF2	Cutting perpendicular to pipe axis	Angle between cutting section and pipe axis (°)	Minimum
CF3	Not deforming the pipe	Internal / external diameter variation (%)	Minimum
CF4	Being compatible with existing pipes	Pipe type	Smooth / corrugated
		Pipe diameter (mm)	4 < diameter <30
		Pipe material	Plastic / rubber
CF5	Being user-friendly	Operation number	Minimum
		Operation complexity	Basic (no specific skill)

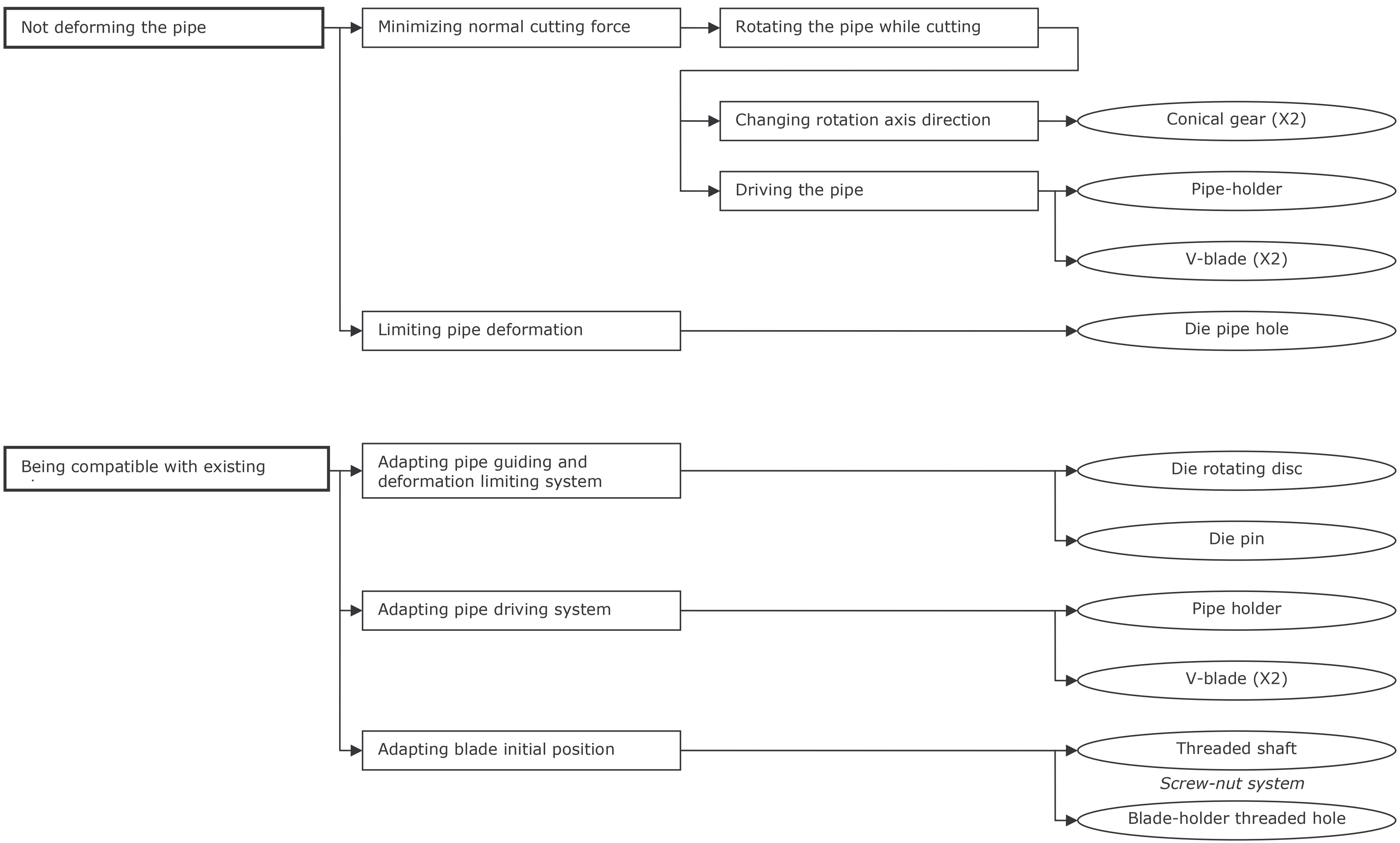
Then, technical solutions for each function have been defined. They are presented Figure 47 and justified below.



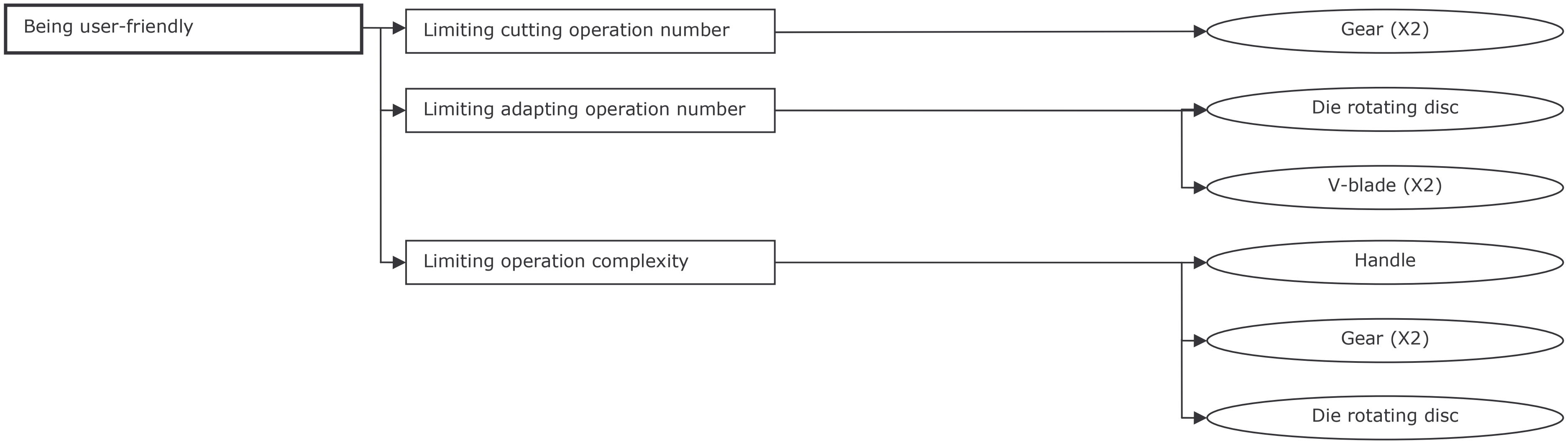
a. Function diagram (OF1 and CF1)



b. Function diagram (CF2)



c. Function diagram (CF3 and CF4)



d. Function diagram (CF5)

Figure 47 – Function diagram

Function:
- Cutting the pipe

Two solutions were considered:
- laser system
- guillotine blade

Laser system was abandoned because cutting section could be altered by melted material. Moreover such system is very expensive compare to a simple guillotine blade.

Function:
- Blocking the pipe
- Driving the pipe
- Guiding the pipe
- Adapting driving system
- Limiting adapting operation number

Two solutions were considered:
- Drill tightening system
- Crossed V-blades

Drill tightening system requires putting a pin inside the pipe otherwise the pipe would collapse. This pin should be changed for each pipe diameter. Crossed V-blade solution is easier to design and manufacture. Moreover its geometry ensures pipe self-centring and flexibility in terms of dimensions. But feasibility has to be confirmed, especially concerning pipe insertion force.

Function:
- Rotating the pipe while cutting

Blade relative movement has been fixed at 1 mm per revolution. In fact, considering that most pipes are 1 mm thick, it is a compromise to limit both normal cutting force which deforms the pipe and blade marks on cutting section (one unique mark on the section) as illustrated Figure 48. However minimum thread for standard screw-nut system of this order of dimension is 4 mm. Therefore diameter ratio between pipe gear and blade gear should be 1:4.

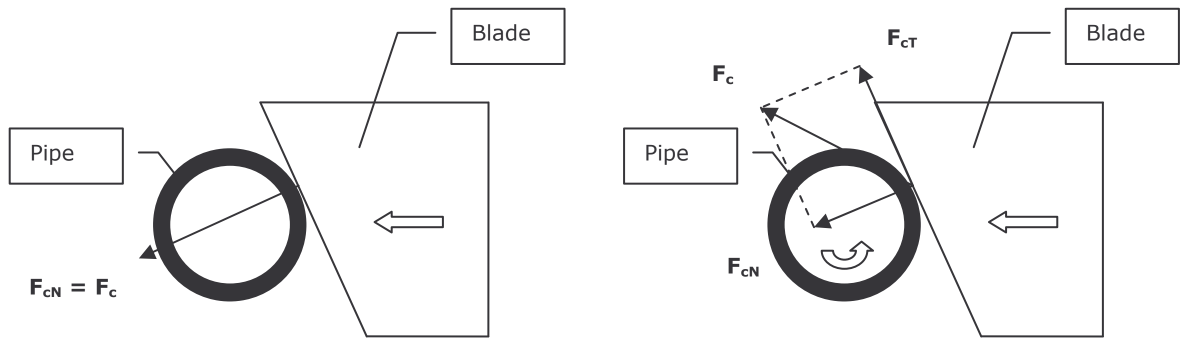


Figure 48 – Pipe cutting force

Function:
- Rotating movement in translating movement
- Adapting blade initial position

Two solutions were considered:
- Cam system
- Screw-nut system

A cam system is more complex to develop than a screw-nut system. In fact, cam geometry should be specifically designed according to rod movement profile. Moreover rod spring which purpose is to maintain a continuous contact between came and rod should be well sized to ensure smooth operating conditions. Lastly, blade initial position cannot be adjusted. Therefore this solution was abandoned.

Function:
- Guiding the pipe

Die thickness has been fixed at 30 mm (maximum pipe diameter) to minimize the angle between cutting section and pipe axis inside pipe hole as illustrated Figure 49: the thicker the die, the smaller the angle.

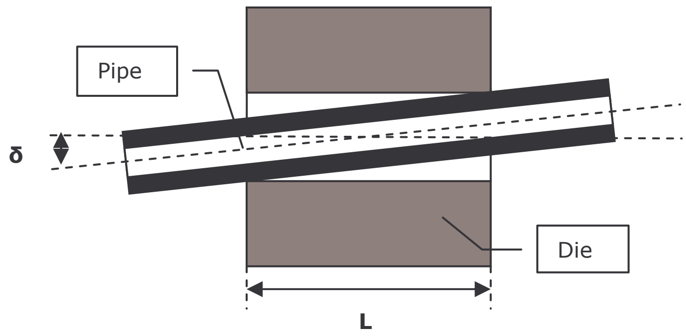


Figure 49 – Influence of die thickness on pipe guiding

Function:
- Guiding the blade

Guiding the blade is necessary to avoid it bending during cutting. This function is performed by die body and a groove machined in the frame.

Function:
- Driving cutting tool

Friction force generated by screw tightening pressure through the washer allows blade holder driving directly the blade. Thus screw works only in traction and not in shearing.

Function:
- Limiting pipe deformation

Pipe shape under cutting stress is an ellipsis. It is assumed that pipe deformation during cutting operation can be limited by stopping radial expansion inside die pipe hole. If necessary, an internal pin can still be added.

Function:
- Adapting pipe guiding and deformation limiting system
- Limiting operation complexity
- Limiting adapting operation number

This function is performed by die rotating disc. It avoids having extra tooling in the case of smooth pipes.

Lastly, cutting machine has been designed using some standard mechanical elements chosen from a catalogue. Machine concept is presented Figure 50.

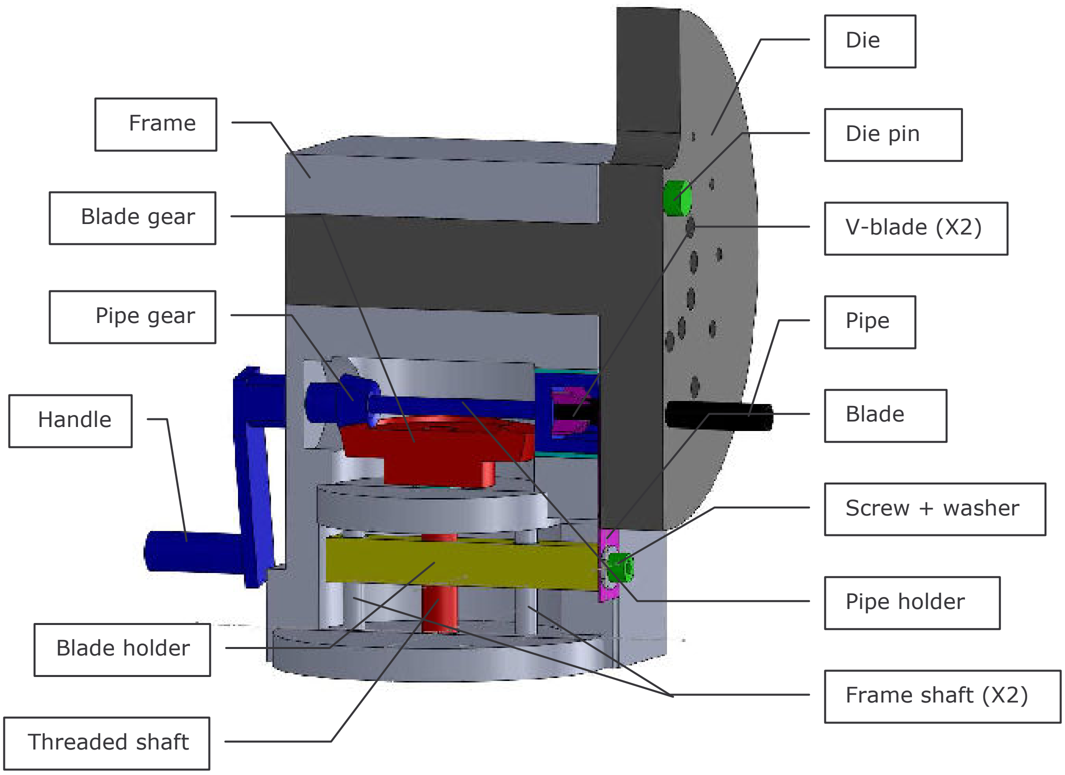
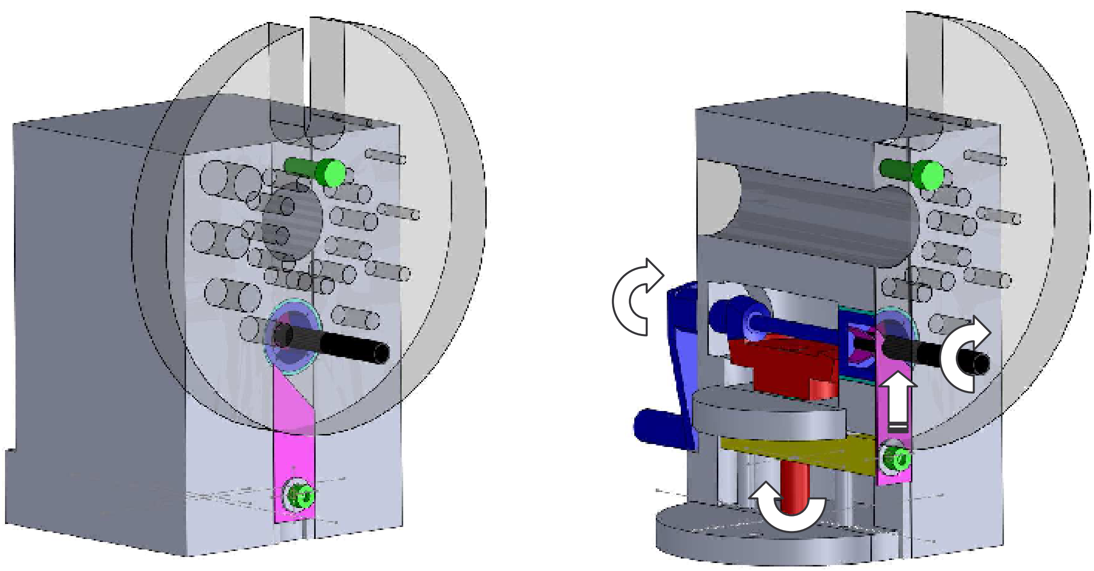


Figure 50 – Cutting machine concept description

Working principle of the machine is presented Figure 51.



a. blade in initial position

b. blade in cutting position

Figure 51 – Cutting machine working principle

Handle rotation drives simultaneously in rotation the pipe and in translation the blade which cuts the pipe.

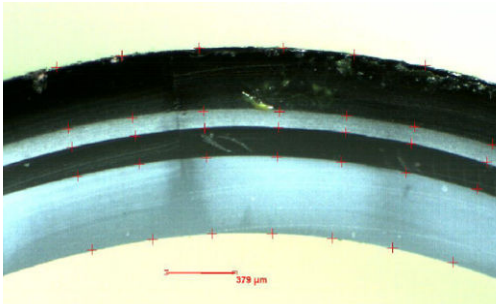
Next step consists of completing the development of the machine then manufacturing it. However it is out of the scope of this thesis. Cutting machine bill of material is presented in Appendix 3.

4.1.2.3 Automatic measurement program

Measurement steps are the following:

- Conversion of the picture in black and white colour
- Measurement of luminosity contrast gradients to identify layer boundaries
- Calculation of layer diameters using mathematical formula

Boundaries between layers can be identified since they are zones of high contrast gradients. An example of measurement is presented Figure 52.



a. picture analysed

Résultats des mesures d'épaisseurs					
Points	0	1	2	3	4
d1 (pixel)	192,760	195,612	195,729	198,597	196,746
d2 (pixel)	76,792	75,251	76,038	75,541	77,216
d3 (pixel)	49,585	47,192	44,239	40,959	42,905
d4 (pixel)	160,276	161,457	162,417	162,679	158,705

b. layer diameters calculated

Figure 52 – Example of measurement with a first version of the program

Result accuracy depends on the requirements specified in section 4.1.2.1, especially on sample cutting. Therefore it is necessary completing cutting machine development before working on program development.

4.2. Structural controls

Structural tests allow characterising pipe mechanical properties. During validation phase, the purpose of these tests is to check that pipe characteristics match with target application. Requirements are based on pipe mounting and operating conditions. In serial production, the purpose of these tests is to monitor raw material and process quality deviations. Tests are carried out in parallel with production activity. Requirements are based on structure typical performance determined during validation phase.

4.2.1. Burst test

Burst test characterizes pipe resistance to radial load. It consists of increasing the pressure in the pipe tested until it bursts. Burst pressure value is recorded. It must be over a given limit, typically four times operating pressure. A picture of Hutchinson burst test bench is presented Figure 53.

Figure 53 – Hutchinson burst test bench

4.2.2. Cold impact test

Cold impact characterizes the ductility of pipe material at high energy level. It consists of cooling down the pipe tested until a given temperature, typically -40°C, then impacting it with a suspended mass with a fixed geometry. At the end of the test, no crack should appear on pipe surface. A picture of Hutchinson impact test apparatus is presented Figure 54.

a. impact machine

b. suspended mass

Figure 54 – Hutchinson impact test apparatus

4.2.3. Tensile test

Tensile test characterizes the ductility of pipe material at low energy level. It is performed on a dynamometer. It consists of elongating the pipe tested until it breaks. Instantaneous values of bench load and sample length are recorded during the test as shown Figure 55. For production control, traction force is orientated in the direction of extrusion and monitored parameter is elongation at break. A picture of Hutchinson dynamometer is presented Figure 56.

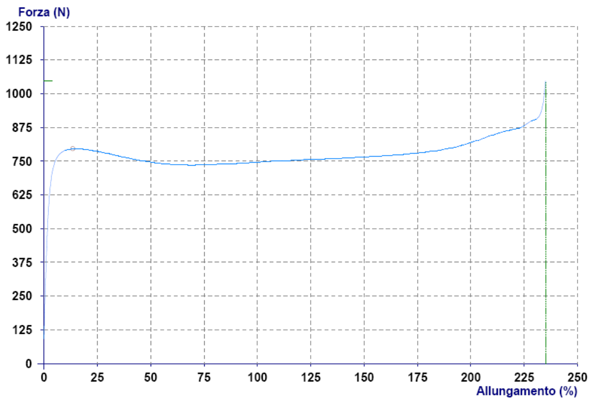


Figure 55 – Tensile curve

Figure 56 – Hutchinson dynamometer

4.2.3.1 Test configuration

Test configuration is defined by customer specifications. Two configurations are possible as presented Figure 57. Gripping zone is by definition where jaws, pipe and pin interfere. Pin role is to avoid pipe collapsing under grip tightening pressure.

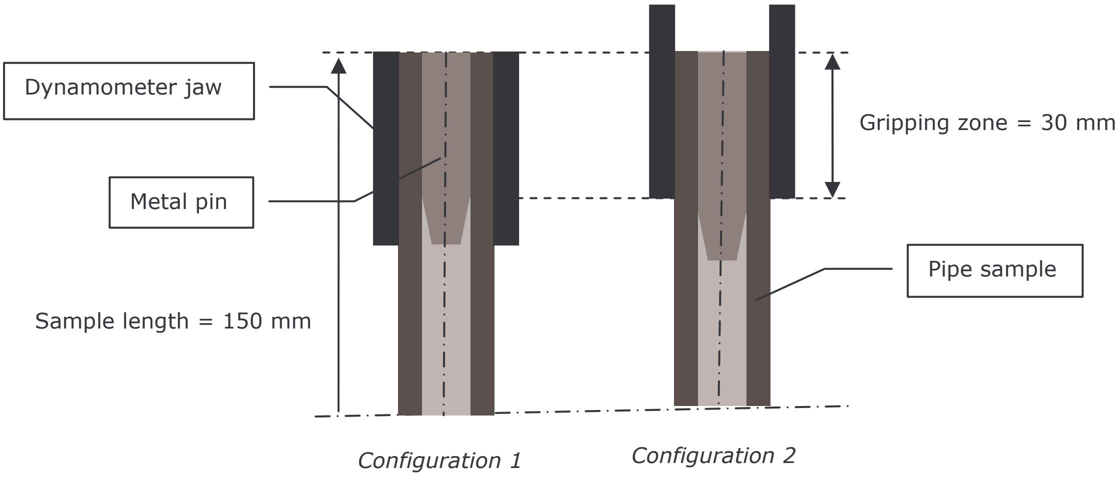


Figure 57 – Basic tensile test configuration

ISO samples (injected or cut) are specifically designed for tensile test. Their section is smaller in the middle than at the extremities to get higher stress level. Thus ISO sample elongation is mostly located in measurement zone contrary to pipe elongation which propagates till jaw area. This phenomenon is unavoidable since pipe geometry is uniform. Therefore gripping system should be able to compensate pipe section diminution otherwise the pipe will slide out of the jaws as tightening pressure will decrease during the test.

4.2.3.2 Self-closure mechanical gripping system

Self-closure mechanical gripping systems, especially those originally designed for non ductile materials like metals, are very sensitive to sample section reduction. Therefore grips are equipped with teeth to reduce sliding phenomenon. In fact, tooth presence increases jaw and sample friction forces because it increases locally contact pressure: the higher the pressure, the higher the forces. However pressure peaks which level depends on tooth geometry generate inevitably stress peaks in pipe material which are also potential crack initiations: the sharper the geometry, the higher the peaks. As a consequence, many test results are rejected due to anticipated breaks caused by the grips. Therefore it is necessary improving test reliability before working on pipe elongation at break improvement. A picture of a self-closure mechanical gripping system is presented Figure 58.

Figure 58 – Self-closure mechanical gripping system

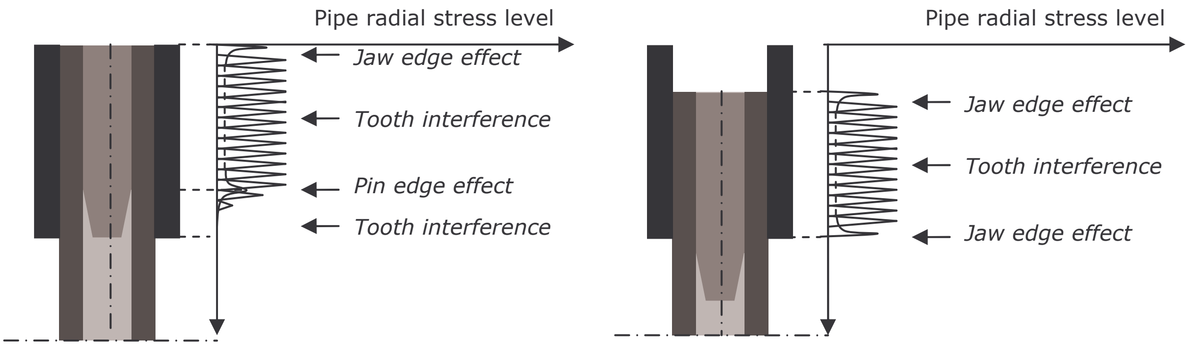
A compromise has to be found between limiting sliding phenomenon to be able to perform the test until the end and avoiding undesirable anticipated breaks as presented Figure 59. It depends mainly on:

- grip and pin relative positions
- jaw teeth pattern
- grip initial tightening

Pipe sliding	Pipe anticipated break caused by the grips	Pipe self break
Rejected	Rejected	Accepted

Figure 59 – Tensile test result validity

Concerning grip and pin positions, quantity of tests rejected is lower in configuration 2 than in configuration 1. In fact, last tooth marks on sample surface are not free compare to configuration 1 as illustrated Figure 60. Thus anticipated breaks will be postponed and as a consequence, quantity of tests rejected is likely to be lower.



a. configuration 1 b. configuration 2

Figure 60 – Pipe radial stress distribution in gripping zone

However, the problem is not solved, especially with stiffer materials which load at given elongation is higher. In fact, sliding phenomenon is accelerated and as a consequence, or the sample slid out of the grips or the break is anticipated depending on grip initial tightening: the higher the strength, the earlier the anticipated break. Thus two new designs of jaws have been studied. They are presented Figure 61.

a. original jaw b. threaded jaw c. ringed jaw

Figure 61 – Jaw configuration studied

Threaded pattern purpose is to maximize grip and sample interaction to reduce sliding phenomenon. This sharp geometry results systematically in anticipated break. Therefore thread teeth has to be enlarged to reduce material stressing as illustrated Figure 62. However no significant improvement is noticed with ring pattern compared to original one.

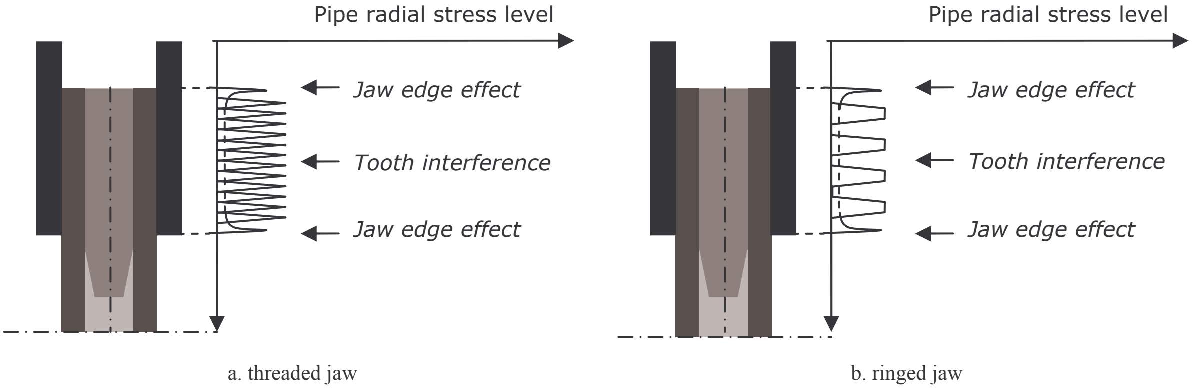


Figure 62 – Pipe stress distribution in gripping zone

All tests performed and their respective results are compiled Table 9.

Table 9 – Test results with mechanical gripping system

	Original jaw	Threaded jaw	Ringed jaw
Configuration 1	- -	- - - -	- -
Configuration 2	-	- - -	-

Success rate: - low, - - - - very low

Finally, use of self-closure mechanical gripping system is not appropriate for plastic pipe samples as quantity of tests rejected is always high whatever adjustments are made. The reason is that a mechanical system is not able to follow large sample section reduction. This issue can be solved using a pneumatic gripping system. A picture of a pneumatic gripping system is presented Figure 63.

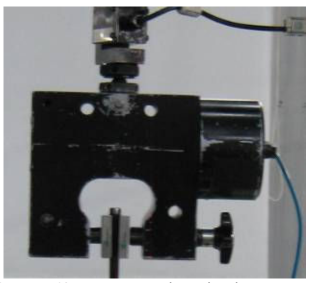
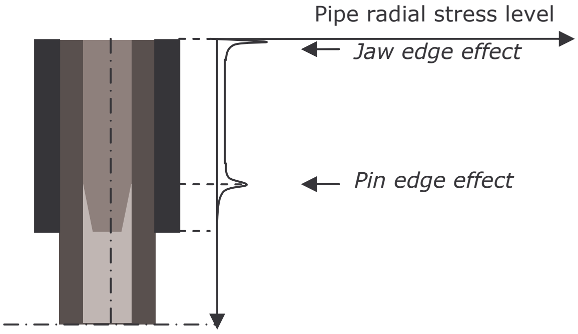


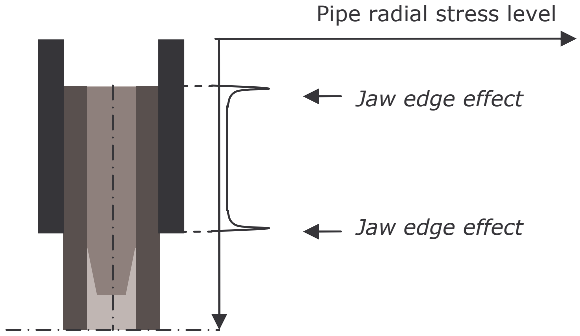
Figure 63 – Pneumatic gripping system

4.2.3.3 Pneumatic gripping system

Contrary to mechanical system, pneumatic system is able to maintain a constant pressure on the sample. When compressed air inlet is equipped with a section reducer, average contact pressure can be significantly higher than with a manual closure. Thus sliding phenomenon is strongly reduced and teeth are not necessary anymore. Pipe stress distribution in gripping zone for smooth jaws is illustrated Figure 64.



a. configuration 1



b. configuration 2

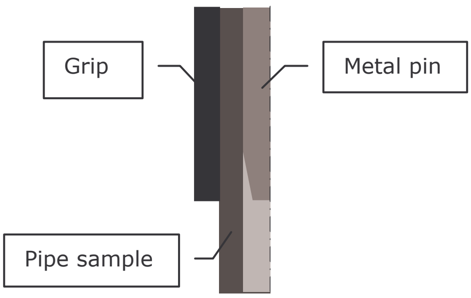
Figure 64 – Pipe radial stress distribution in gripping zone

Quantity of tests rejected is significantly lower than with mechanical system, particularly in configuration 1. In fact, pin edge effect is lower than jaw edge one because the angle is smaller and can be rounded. Jaw edge does not have any impact on test success when located at pipe extremity.

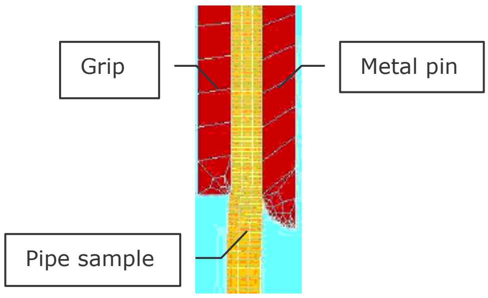
A finite element model analysis has been performed to corroborate previous explanations. Criterion chosen is Von Mises stress calculated from the following formula:

$$\sigma_{Von\_Mises} = \frac{1}{\sqrt{2}}\sqrt{(\sigma_x - \sigma_y)^2 + (\sigma_y - \sigma_z)^2 + (\sigma_z - \sigma_x)^2 + 6.\tau_{yz}^2 + 6.\tau_{zx}^2 + 6.\tau_{xy}^2}$$

Gripping zone model is presented Figure 65.



a. schematic model



b. FEM model

Figure 65 – Gripping zone model

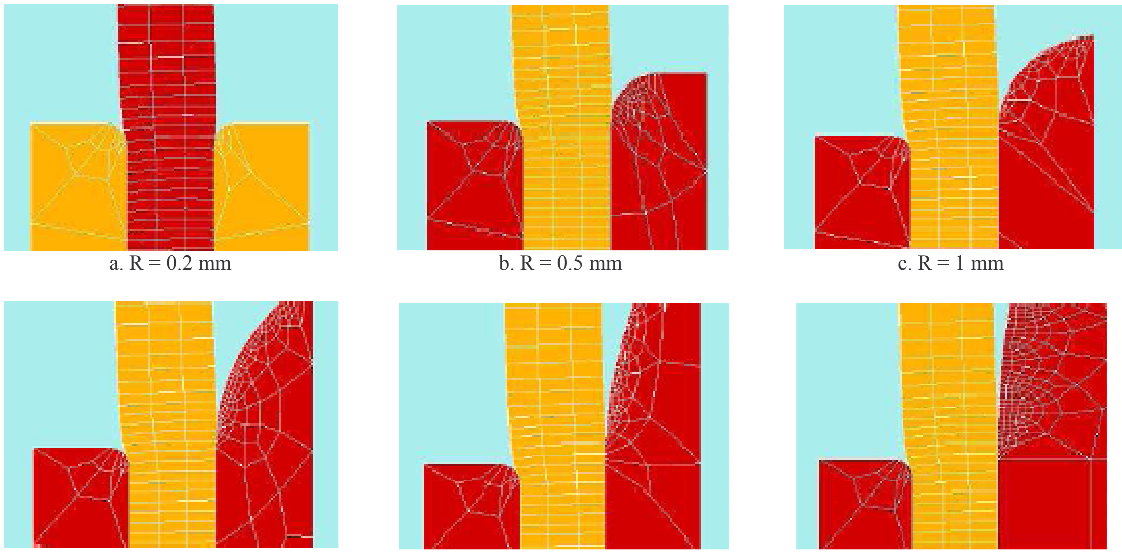
Model parameters are presented Table 10.

Table 10 – Model parameters

element	Behavour law	Young modulus (MPa)	Yield stress (MPa)	Hardening
Pin	Elastic	210000	-	-
Pipe	Elastic-plastic	300	28	100
Grip	Elastic	210000	-	-

Two variables have been considered in this study:

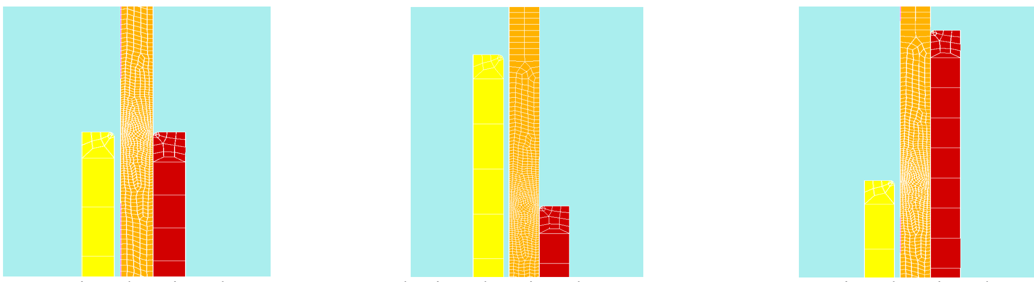
- tip edge rounding radius: see Figure 66
- pin length: see Figure 67



a. R = 0.2 mm b. R = 0.5 mm c. R = 1 mm

d. R = 2 mm e. R = 5 mm f. R = 10 mm

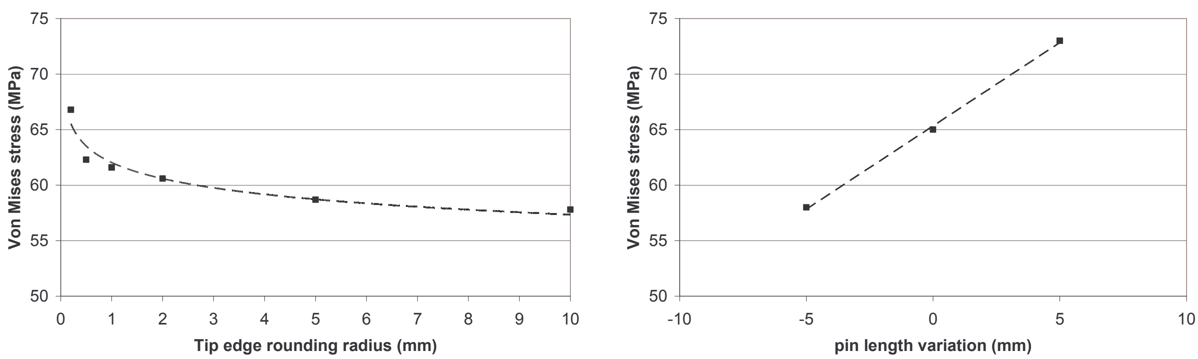
Figure 66 – Tip edge rounding radius variation



a. pin end = grip end b. pin end = grip end – 5 mm c. pin end = grip end + 5

Figure 67 – Pin length variation

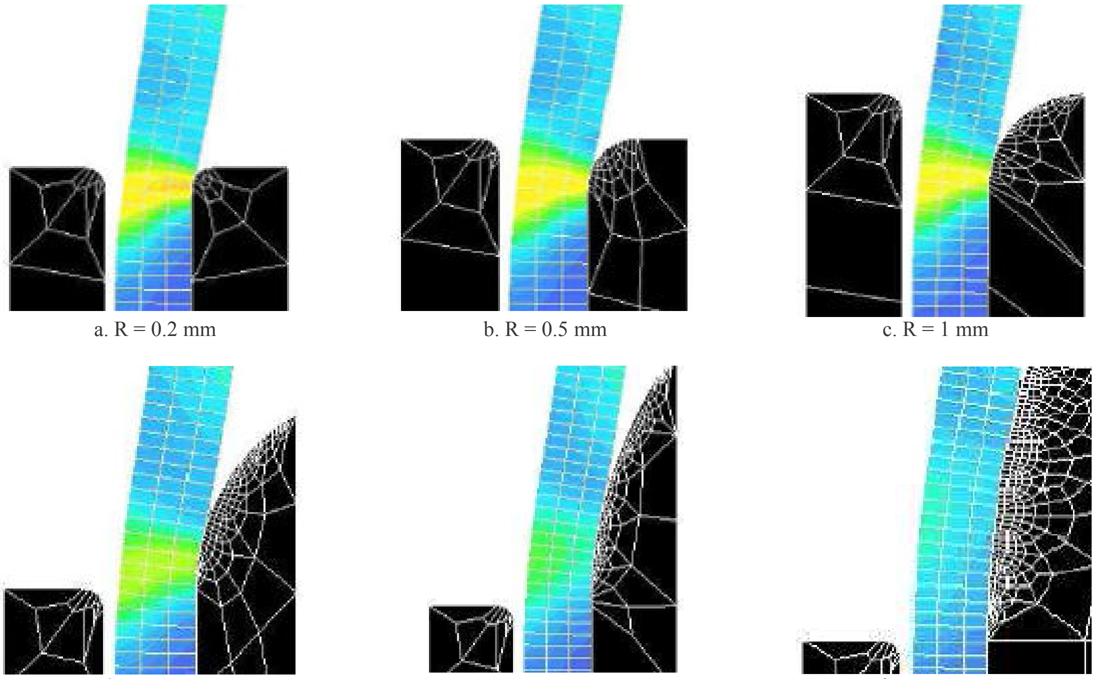
Results are presented in the graphs Figure 68.



a. tip edge rounding radius b. pin length

Figure 68 – Variation of Von Mises stress in function of FEM analysis variables

Maximum Von Mises stress in gripping zone decreases when tip edge rounding radius increases and pin length decreases. These results corroborate previous explanations. Figure 69 shows stress distribution in gripping zone function of tip edge rounding radius.

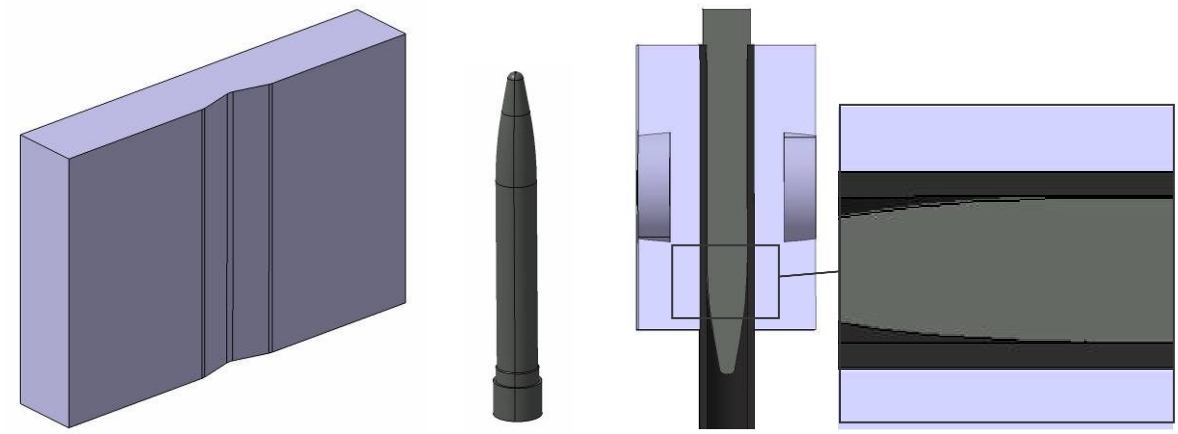


a. R = 0.2 mm b. R = 0.5 mm c. R = 1 mm

d. R = 2 mm e. R = 5 mm f. R = 10 mm

Figure 69 – Stress distribution in gripping zone according to tip edge rounding radius

Finally, jaw and pin designs are presented Figure 70.



a. jaw b. pin c. assembly

Figure 70 – Tensile test tool design

Jaw dimensions are compatible with pipe diameters ranging from 4x6 to 13,5x16 that covers pipe dimensions. As load at given elongation is higher for larger diameters, friction factor between plastic and aluminium is too low to avoid the pipe sliding before the end of the test. A solution could be to change jaw material.

Pin external diameter matches with pipe minimum internal diameter to limit the interference between pin and pipe. Tip edge has a radius of 60 mm to minimize edge effect. Pin base is slightly higher than pipe maximum internal diameter to ensure keeping the pin inside the pipe during test set-up.

4.2.3.4 Measurement method

Elongation is deduced from the following formula:

$$\varepsilon = \frac{l_f - l_i}{l_i} \times 100$$

With:

ε Elongation

l_i Sample actual initial length

l_f Sample actual final length

Pipe elongation at break can be measured using an optical or a mechanical extensometer. Optical extensometer measures the distance between two laser reflecting markings stuck to the sample. Elongation is then calculated automatically by the system. Mechanical extensometer is composed of two mobile forks carried by the sample during the test. Elongation is deduced from the distance between the forks. Contact pressure on the pipe is light enough so that forks are not sensible to material elastic relaxation after the break. Optical extensometer is the most accurate system.

4.2.3.5 Result interpretation

Final test configuration is presented Figure 71.

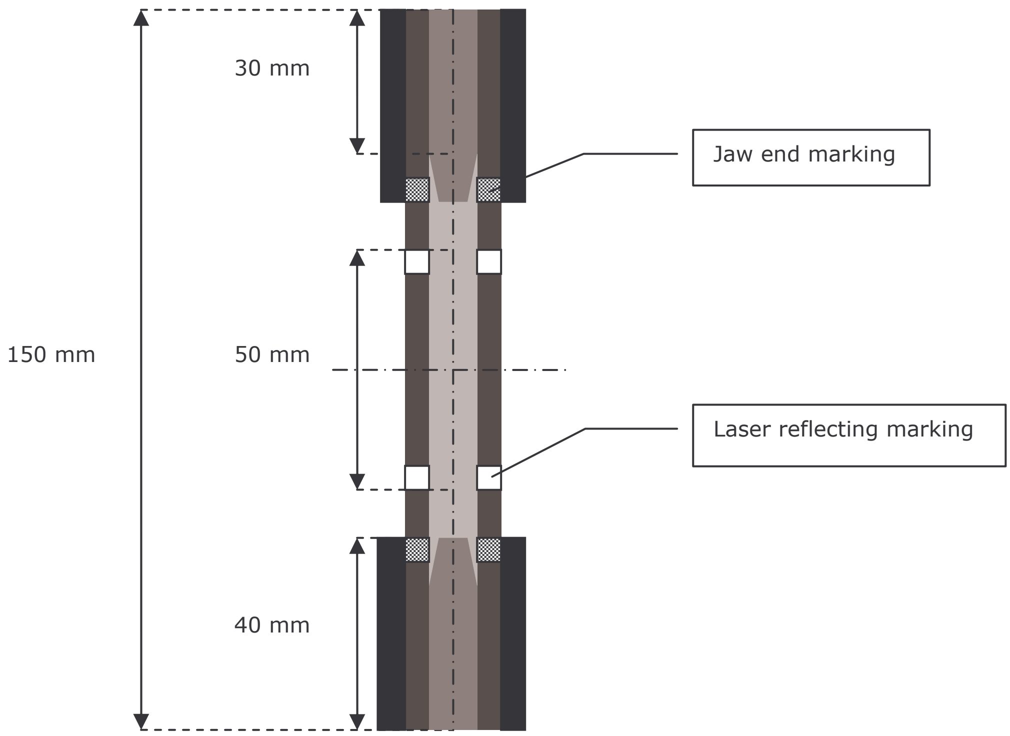


Figure 71 – Final test configuration

Maximum elongation gradient is located around break zone. Moreover measured value is the average of sample local elongations in measurement zone. Thus when a sample breaks out of measurement zone, elongation at break is underestimated and if the break occurs out of grip end initial positions, rupture mechanism is probably influenced by pin and jaw interaction with the pipe. In the last case, the break is anticipated compare to pipe self rupture and as a consequence, elongation at break is highly underestimated. Test result interpretation is presented Figure 72.

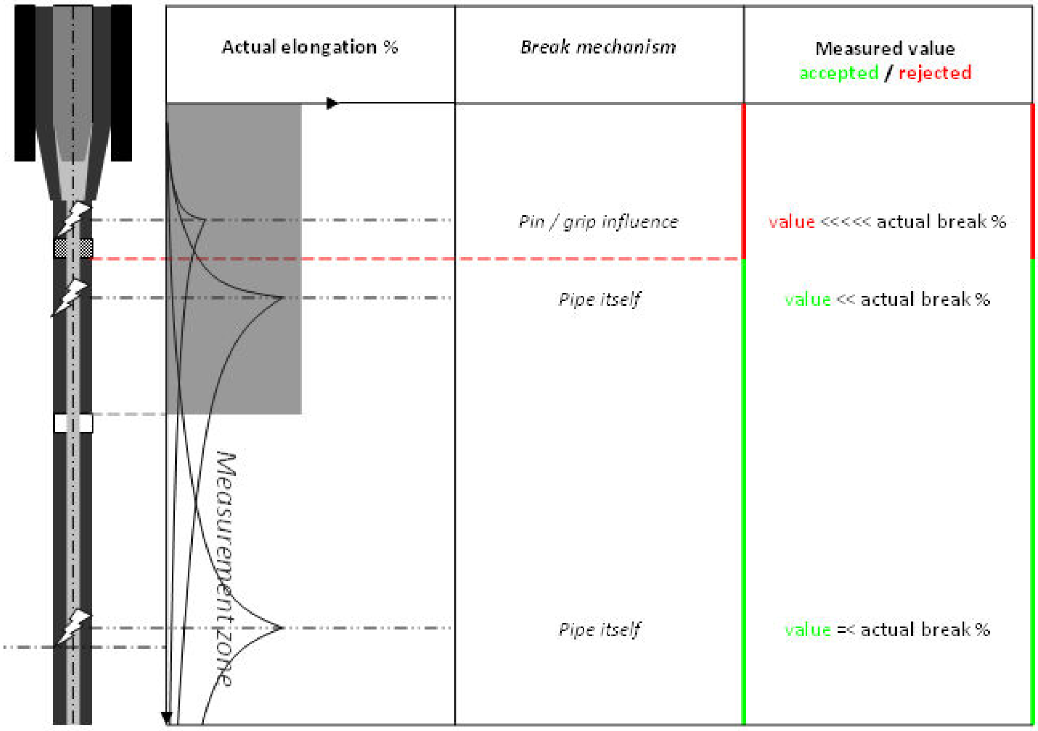


Figure 72 – Test result interpretation (break % = elongation at break)

4.3. Conclusion

Before working on extrusion process improvement, it is necessary being able to monitor extruded pipe essential characteristics.

Monitored essential characteristics of extruded pipes and their respective controls are divided in two categories:

- dimensional controls:
    - non destructive controls:
        - ultrasonic system: total thickness
        - laser system: external diameter
    - destructive controls:
        - microscope instrument: internal diameter, external diameter, intermediate layer thickness, total thickness
- structural controls:
    - burst test: burst pressure
    - cold impact test: cold impact resistance
    - tensile test: elongation at break

4.3.1. Dimensional controls

Feasibility of substituting destructive controls by non destructive controls extending use of ultrasonic and laser systems has been studied in anticipation of the study on process reliability improvement that will be carried out in 2012. However neither intermediate layer thickness of multilayer pipes nor internal diameter can be controlled with current system characteristics. As a consequence, these characteristics can only be controlled by cutting the pipe then analysing its section with a microscope. Current control method was acceptable but could be improved to reduce measurement variability. Thus a concept of cutting machine and a program to automate measures have been presented. However their development was out of the scope of this thesis.

4.3.2. Structural controls

Current tensile test has been improved to have a reliable method to evaluate pipe elongation at break in the next chapter. In fact, many test results were rejected because of anticipated breaks caused by the grips. Thus current mechanical gripping system has been substituted by a pneumatic gripping system and new jaws have been specifically designed to limit anticipated breaking phenomenon. Finally new test configuration allows reducing significantly the quantity of test results rejected.

5. Extrusion performance improvement: results and discussion

A reason for working on process improvement is when some essential characteristics of the product do not meet customer specification requirements. For plastic pipes, elongation at break is an important characteristic. On new projects in development, minimum value required for pipe elongation at break has been increased and is not reached with current process configuration. Thus the purpose of this thesis is to improve this characteristic.

5.1. Current process optimization

A design of experiment has been carried out on PA9T/PA12 and PA6/EVOH/ADH/PA12 structures to improve elongation at break. Target value of elongation at break is 200%. This experiment focuses on sizing operation and parameters have been deduced from chapter 3 (see 3.4.). They are the following:

- forming tool dimensions: D ratio, T ratio, DDR
- water temperature
- extrusion line speed
- sizing sleeve internal diameter: influence vacuum

Parameter combination for each test is presented Table 11.

Table 11 – Test parameter combination

a. PA9T/PA12 structure

Test N°	D-ratio	T-ratio	D sleeve [mm]	Line speed [m/min]
6	2,05	1,45	10,85	20
4	2,05	3,35	10,85	20
7	1,78	2	10,85	20
8	1,78	2	10,95	20
12	1,42	1,45	10,85	20
13	1,42	1,45	10,95	20
9	1,42	2	10,85	20
10	1,42	2	10,95	20
15	1,42	2	10,85	10
16	1,69	1,55	10,85	20
17	1,69	1,55	10,85	10

b. PA6/EVOH/ADH/PA12 structure

Test N°	D-ratio	T-ratio	Water temp. [°C]	Line speed [m/min]
1	1,73	1,8	12	40
2	1,73	1,8	6	40
3	1,51	1,77	12	40
4	1,73	1,8	12	30
5	1,73	1,8	12	20

Vacuum level and water flow have been voluntary excluded from the design of experiment. In fact, vacuum level determines pipe dimensions (see 3.4.2.) that are fixed during the experiment otherwise it would not be possible to compare elongation at break. Therefore vacuum level should be adjusted freely to keep pipe dimensions constant between the different tests. Moreover vacuum level influences lubrication thickness inside sizing sleeve at constant water flow (see 3.3.3.2). If vacuum level increases, lubrication will be scarce at the beginning of the sleeve where material is not solidified yet and as a consequence, the pipe could stick locally on the sleeve affecting process stability. Therefore water flow should be adjusted freely according to vacuum level to keep a stable process as shown Figure 73 where water flow increases with increasing vacuum level.

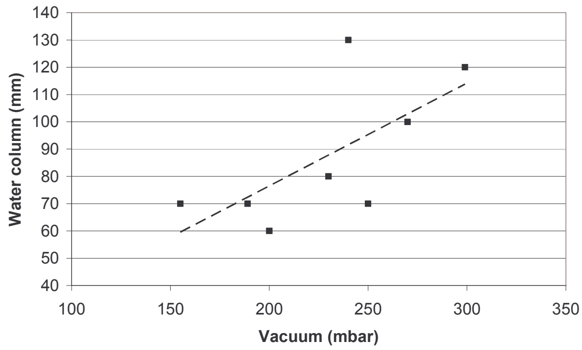
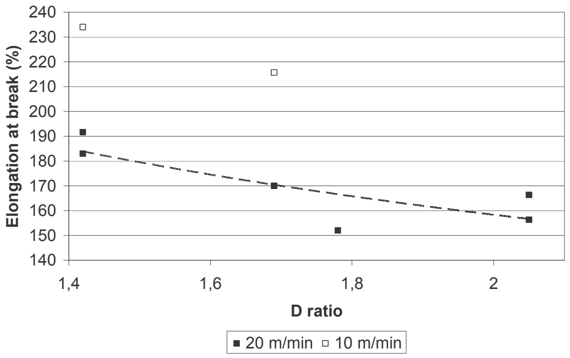
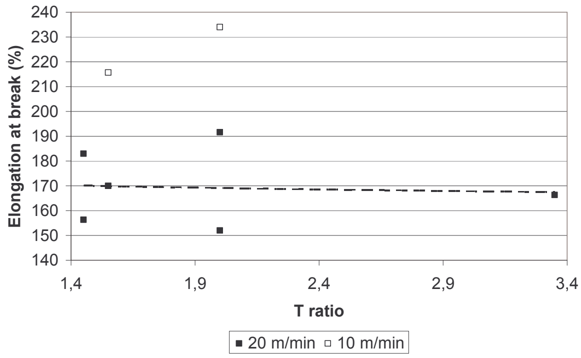


Figure 73 – Influence of vacuum level on water flow

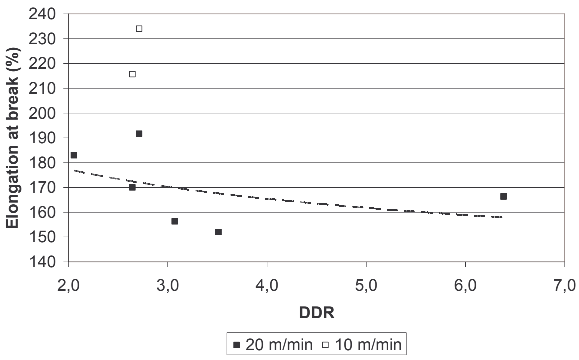
At given vacuum level, large deviations can be observed on water flow because water flow is adjusted according to operator feeling. The effect of water flow adjustment is limited to the beginning of the sleeve since increasing vacuum level reduces lubrication length (see 3.3.3.2). Thus average friction along the sleeve increases when vacuum level increases and as a consequence, pipe elongation at break decreases anyway (see 3.3.3.2). Therefore correlation between water flow and elongation at break is not as obvious as for vacuum level as it can be observed Figure 74 e and Figure 74 f.



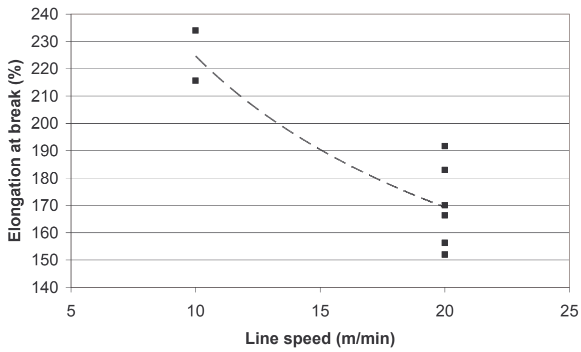
a. D ratio



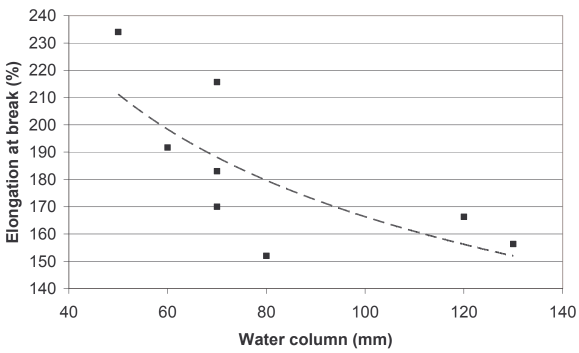
b. T ratio



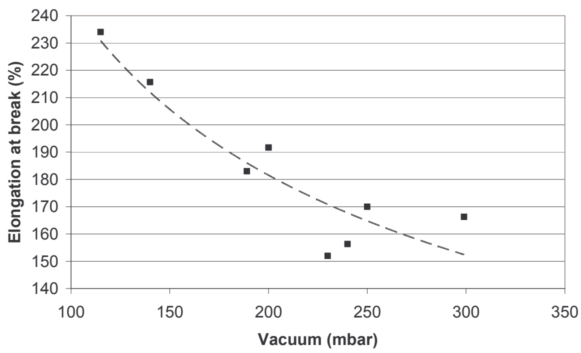
c. DDR



d. extrusion line speed



e. water flow



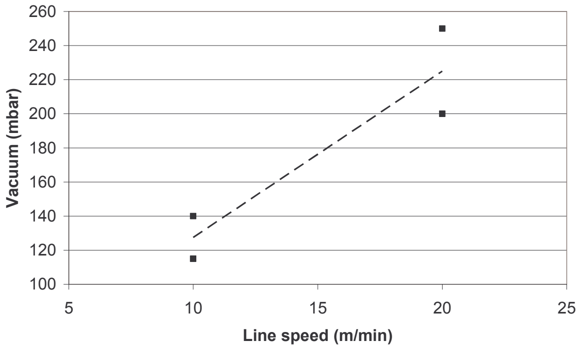
f. vacuum level

Figure 74 – Influence of experiment parameters on pipe elongation at break

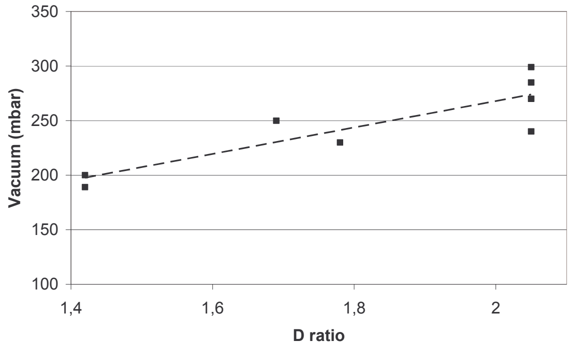
It can be deduced by comparing the different graphs Figure 74 that vacuum is the best indicator when working on pipe elongation at break improvement: the lower the vacuum, the higher the elongation at break.

Vacuum level is influenced by extrusion line speed. In fact, vacuum level required to size the pipe is lower at lower speed as shown Figure 75 a. Therefore friction is lower and pipe elongation at break is higher.

Vacuum level is also influenced by forming tool dimensions, especially D ratio. In fact, vacuum is adjusted to fix pipe external diameter (see 3.3.). Therefore when extruded pipe external diameter is closer to final one, vacuum level required is lower as shown Figure 75 b and pipe elongation at break is higher. Other ratios have a minor influence.



a. extrusion line speed



b. D ratio

Figure 75 – Influence of experiment parameters on vacuum level

Increasing sleeve internal diameter decreases vacuum level as shown Figure 76. However over a given diameter, sizing operation is not stable anymore.

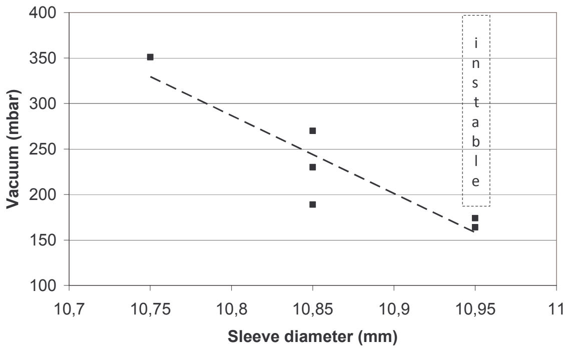
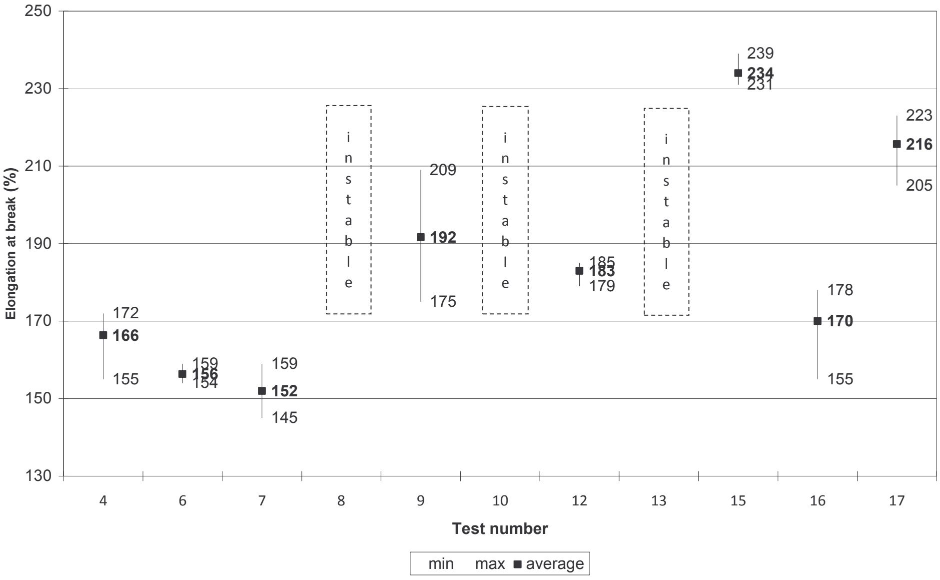


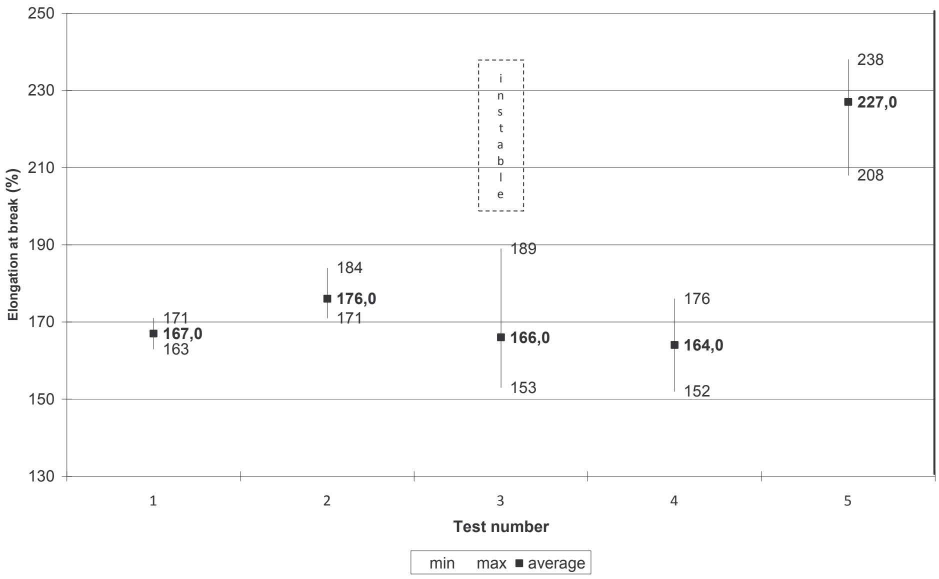
Figure 76 – Influence of sizing sleeve internal diameter on vacuum level

Lastly, water temperature does not seams to have a significant influence on pipe elongation at break.

Results of the experiment are summarized Figure 77.



a. PA9T/PA12 8x1 structure



b. PA6/EVOH/ADH/PA12 8x1 structure

Figure 77 – Results of the experiment

For PA9T/PA12 structure, best results are obtained for a line speed of 10 m/min and a D ratio of 1,42 as it could be expected from previous explanations. Line speed is lower than current one of 40 m/min which would impact process cost. Moreover, even with optimized parameters, some pipes are statistically below 200% of elongation at break that is not acceptable. For PA6/EVOH/ADH/PA12 structure, conclusion is similar but for a line speed of 20 m/min.

Pipe elongation at break decreases when material molecular orientation and quantity and size of defects on pipe surface increase (see 2.9.3.). High molecular orientation is concentrated in pipe external layer, typically 200 µm (see 3.3.2.2). Thus a solution to improve elongation at break would be to remove pipe external layer molecular orientation and defects by melting the surface of the pipe again after sizing operation, in other terms, to equip extrusion line with a flaming treatment.

5.2. Flaming treatment addition

Flaming treatment purpose is to remove pipe external layer molecular orientation and defects by melting the surface of the pipe again after sizing operation as illustrated Figure 78.

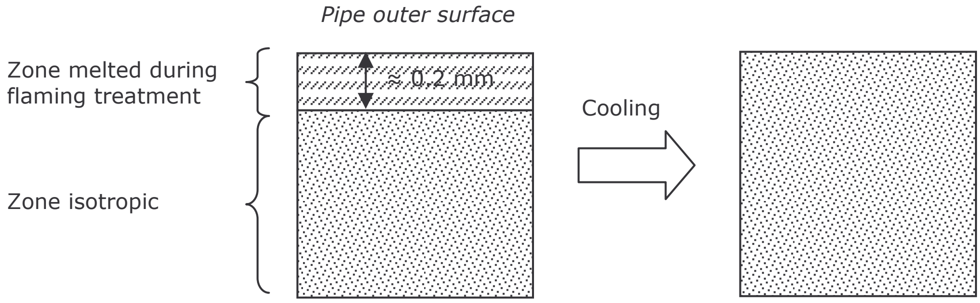


Figure 78 – Schematic of flaming treatment principle

Two gases can be used for this treatment: methane (natural gas) and propane. Heating value of methane gas is higher than that of propane gas. Combustion equation for each gas is given below:

Methane:

$$CH_4 + 2\ O_2 = CO_2 + 2\ H_2O$$

Propane:

$$C_3H_8 + 5\ O_2 = 3\ CO_2 + 4\ H_2O$$

Flaming treatment main parameters are:

- gas nature: treatment intensity
- gas flow: treatment intensity
- air /gas mass ratio: treatment efficiency (see Table 12)

Treatment intensity corresponds to the thermal power provided to the pipe. At given gas flow, treatment intensity depends on extrusion line speed and pipe dimensions.

Table 12 – Air / gas mass ratio

Gas	Stoichiometric	During trial
Propane	15.5	16
Methane	17.2	not defined

Impact of flaming treatment on pipe elongation at break has been evaluated through a new design of experiment always carried out on PA9T/PA12 and PA6/EVOH/ADH/PA12 structures. Experiment parameters are:

- flaming intensity
- extrusion line speed

Parameter combination for each test is presented Table 13.

Table 13 – Test parameter combinations

a. PA9T/PA12 structure

Test N°	Flaming treatment*	Line speed [m/min]
1A	No	20
2.1	++++	20
2A	No	40
2.2	++++	40
3A	No	60
2.3	++++	60
4A	No	100
2.4	++++	100
C1	+++	40
D1	++	40
E1	+	40

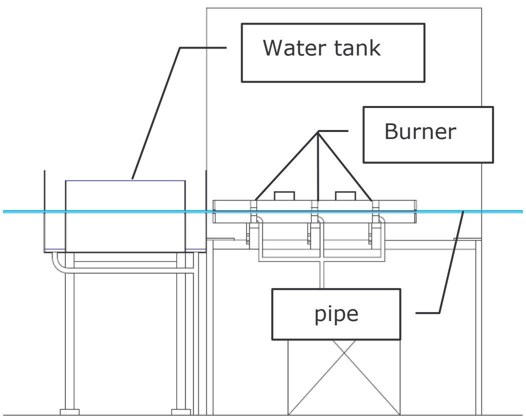
*Flaming intensity: + low, ++ medium, +++ high, ++++ maximum

b. PA6/EVOH/ADH/PA12 structure

Test N°	Flaming treatment*	Line speed [m/min]
1A	No	20
2.1	++++	20
2A	No	40
2.2	++++	40
3A	No	60
2.3	++++	60
4A	No	100
2.4	++++	100

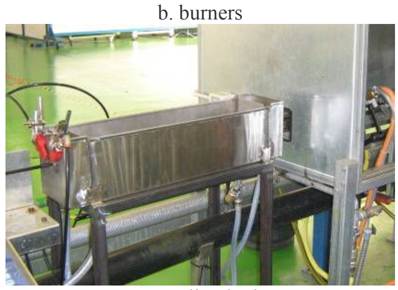
*Flaming intensity: ++++ maximum

Three burners with their control unit have been rent specially for the trial. Flaming unit layout on extrusion line is presented Figure 79.



a. schematic

b. burners



c. cooling bath

Figure 79 – Flaming unit layout on extrusion line, Rivoly, Italy

Flaming treatment efficiency depends by definition on the capacity to heat up pipe surface over material melting point, typically 165°C for polyamide 12. Pipe surface temperature along extrusion line has been measured for different test conditions as presented Figure 80. Measurement has been made with an infrared detector.

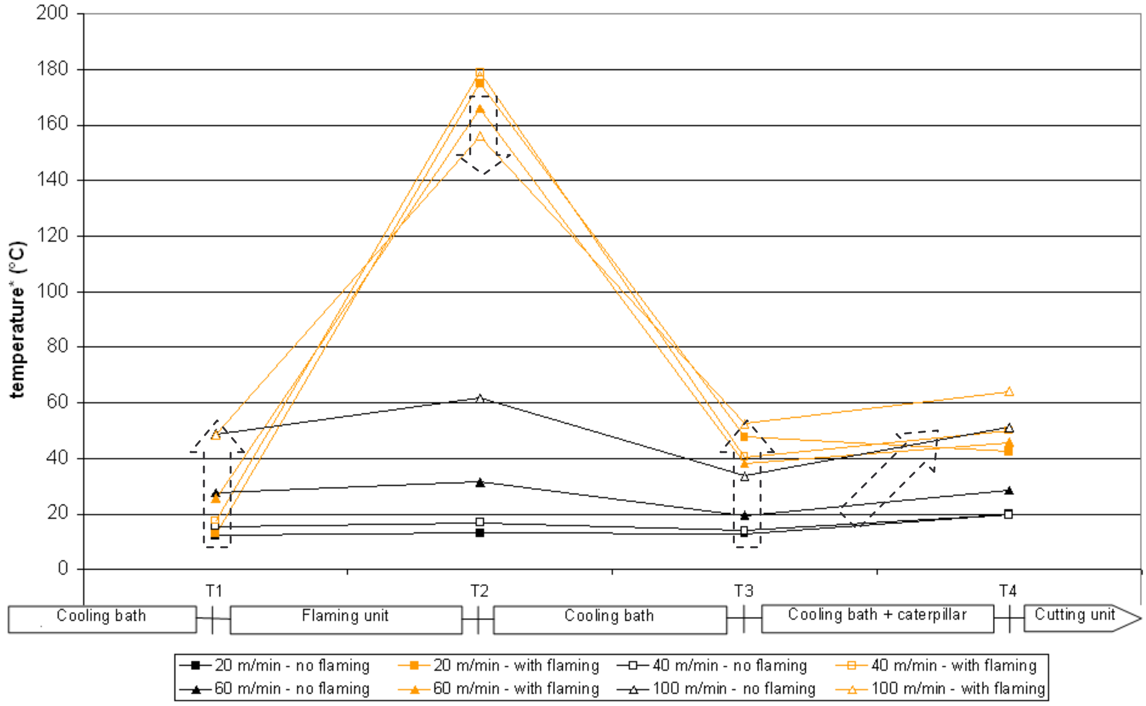


Figure 80 – Pipe surface temperature along extrusion line

Temperature after cooling bath increases when line speed increases because cooling time decreases. Temperature after flaming unit decreases significantly when line speed exceeds 60 m/min. It means that the thermal power provided to the pipe is too low over 60 m/min and as a consequence, that an additional burner should be installed on the line. Temperature after flaming unit cooling bath increases when line speed increases. It shows that the bath could be extended. Lastly temperature between cooling bath and caterpillar increases because external layer of the pipe is heated up by the core of the pipe (see 3.3.1).

Influence of pipe surface temperature after flaming treatment on pipe elongation at break is presented Figure 81.

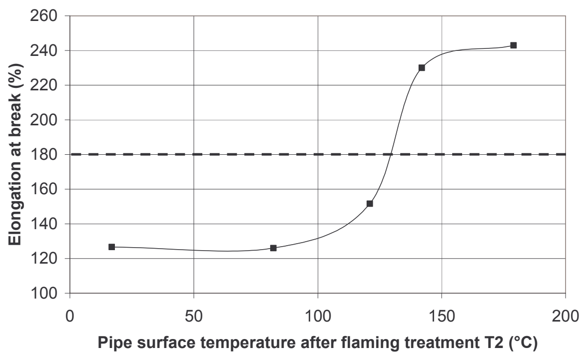
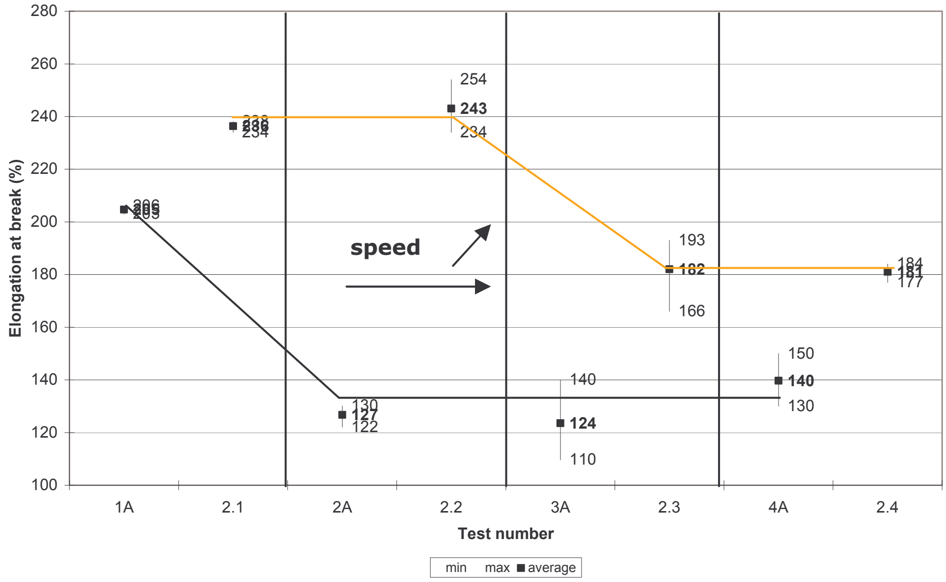


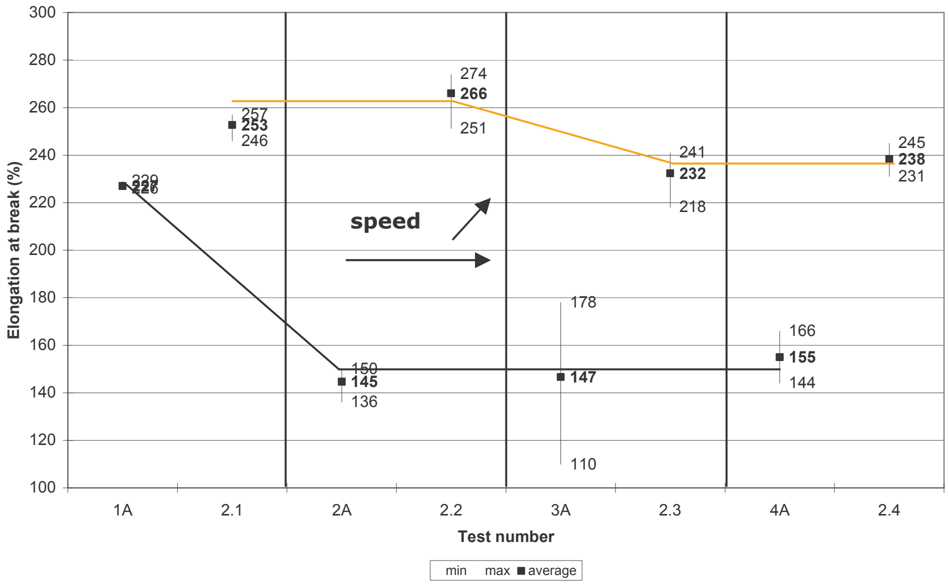
Figure 81 – Influence of pipe surface temperature after flaming treatment on pipe elongation at break

Pipe elongation at break drops down when pipe surface temperature after flaming treatment is under 160°C. This result corroborates previous explanations.

Results of the experiment are summarized Figure 82.



a. PA9T/PA12 8x1 structure



b. PA6/EVOH/ADH/PA12 8x1 structure

Figure 82 – Experiment results

Without flaming treatment, results are similar to those of the first design of experiment: pipe elongation at break decreases strongly over 20 m/min. Adding flaming treatment allows increasing pipe elongation at break up to 100% and achieving target value of 200%. Treatment effect is reduced over 40 m/min which confirms that an additional burner should be installed on the line. However extrusion line equipped with four burners could be run until 80 m/min keeping pipe mechanical properties meeting requirements which would allow in the meantime increasing production rate and as a consequence, improving process performance. Lastly, pipe mechanical properties are less dependent on sizing conditions with a flaming treatment. Thus forming tool dimensions can be chosen independently from sizing operation focussing exclusively on process reliability. This last point is important in anticipation of the study on process reliability improvement that will be carried out in 2012.

6. Conclusion

This thesis was the introductive part of a study on plastic pipe extrusion process improvement. Plastic pipes for automotive application are essentially composed of semi-crystalline polyamides. An important characteristic of semi-crystalline materials is their melting point that corresponds to crystal melting temperature. Polymers behave like viscous fluids above this temperature and like solids below.

6.1. Pipe dimensional properties: improvement of process reliability

The first purpose of working on process improvement is to reduce production cost which depends on process reliability and line speed in the case of extrusion. A long-term study focussed on pipe dimensional properties will be carried out in 2012 to improve process reliability. The first purpose of this thesis was to prepare this study.

The main factors of the process that influence pipe dimensional properties have been identified and justified. They are the following:

- feeding regularity inside the extruder:
  - no fluctuations of pipe dimensions
- melting rate inside the extruder:
  - no inclusions of non melted material in the pipe
  - feeding regularity
- melt viscosity at the exit of extrusion machine:
  - no distortions at pipe layer interface
- draw down inside sizing sleeve:
  - pipe thickness
- vacuum inside water tank:
  - pipe external diameter
  - pipe thickness

Extrusion line parameters that allow controlling above factors have also been identified and are presented Table 14 with their respective influence on process factors.

Table 14 – Effect of increasing extrusion line parameters on process factors

Parameters (+) / Factors (effect)	Feeding regularity	Melting rate	Melt viscosity	Draw down	Vacuum
Screw speed		+	-	-*	
Barrel temperature in feeding zone	-				
Barrel temperature in compression zone		+ / -			
Barrel temperature in metering zone			=		
Head temperature			=		
Forming tool dimensions			+		
Forming tool temperature			+		
Extrusion line speed				+**	
Vacuum level					+

* at constant extrusion line speed
** at constant screw speed

Impact of each parameter on process factors and pipe dimensional properties will be studied in detail during the study on process reliability improvement.

Beforehand, extrusion line parameters to monitor process factors have been identified and are presented Table 5. Moreover essential characteristics to monitor pipe dimensional properties and their respective controls have also been identified and are the following:

- non destructive controls:
  - ultrasonic system: total thickness
  - laser system: external diameter
- destructive controls:
  - microscope instrument: internal diameter, external diameter, intermediate layer thickness, total thickness

A study has been carried out to improve current dimensional controls. It shows that is not possible to substitute destructive controls by non destructive controls with current ultrasonic and laser system characteristics. As a consequence, pipe dimensional properties can only be controlled by cutting the pipe then analysing its section with a microscope. Current control method was acceptable but it could be improved to reduce measurement variability. Thus a concept of cutting machine and a program to automate measures have been presented. Their development will be completed during the study on process reliability improvement.

6.2. Pipe mechanical properties: improvement of pipe elongation at break

Another reason for working on process improvement is when some essential characteristics of the product do not meet customer specification requirements. For plastic pipes, elongation at break is an important characteristic. On new projects in development, minimum value required for pipe elongation at break had been increased and was not reached with current process configuration. The purpose of this thesis was to improve this characteristic.

The main factors of the process that influence pipe mechanical properties, especially elongation at break, during sizing operation have been identified and justified. They are presented Table 6.

Extrusion line parameters that allow controlling above factors have also been identified and are presented Table 15 with their respective influence on process factors.

Table 15 – Effect of increasing extrusion line parameters on process factors

Factors (effect) / Parameters (+)	Cooling rate	Draw down	friction	Vacuum
Forming tool dimensions		+		
Water temperature	-			
Extrusion line speed			+	
Water flow			-	
Vacuum level			+	+
Sizing sleeve dimensions			-	-

It was noticed that many factors are interdependent. For instance, plastic melt viscosity at the exit of extrusion machine and pipe draw down inside sizing sleeve depend both on forming tool dimensions.

A design of experiment has been carried out on PA9T/PA12 and PA6/EVOH/ADH/PA12 structures to improve pipe elongation at break. Target value of elongation at break was 200%. This experiment focused on sizing operation and parameters were deduced from Table 15.

Beforehand, current tensile test has been improved to have a reliable method to evaluate elongation at break. In fact, many test results were rejected because of anticipated breaks caused by the grips. New test configuration allows reducing significantly the quantity of test results rejected.

Best results were obtained for extrusion line speeds lower than current ones which would impact process cost. Moreover, even with optimized parameters, some pipes were statistically below 200% of elongation at break that was not acceptable. A solution was to equip extrusion line with a flaming treatment to remove pipe external layer molecular orientation and defects by melting the surface of the pipe again after sizing operation. Adding flaming treatment allows increasing pipe elongation at break up to 100% and achieving target value of 200%. Moreover extrusion line can be run until 80 m/min keeping pipe mechanical properties meeting requirements which would allow in the meantime increasing production rate and as a consequence, improving process performance. Lastly pipe mechanical properties are less dependent on sizing conditions with a flaming treatment. Thus forming tool dimensions can be chosen independently from sizing operation focussing exclusively on process reliability. This last point is important in anticipation of the study on process reliability improvement that will be carried out in 2012.

Appendix 1: Plastic pipe product presentation

Hutchinson products

The principal function of a pipe is to transfer a fluid. Pipe characteristics depend on vehicle application:

- Assembly conditions:
    - Ergonomics: mounting effort, flexibility…
- Operating conditions:
    - Fluid: nature, temperature, pressure…
    - Environment: temperature, vibrations, aggressive elements…

Hutchinson produces plastic pipes for different applications. Main ones are:

- Fuel transfer: diesel liquid, gasoline liquid, gasoline vapour
- cooling: water liquid, water vapour (degassing)
- valve piloting: air vacuum

Examples of Hutchinson products are presented Figure 83.

a. fuel transfer pipe b. cooling pipe c. valve piloting pipe

Figure 83 – Example of Hutchinson products

A pipe also called structure can be smooth or corrugated as presented Figure 84 and composed of one or several layers of material. On final product, it can be assembled with other components such as:

- Connector
- Rubber
- Protective sleeve
- Tape
- Clip

a. smooth pipe b. corrugated pipe

Figure 84 – Example of pipe configurations

Multilayer structures

Multilayer structures are more complex to extrude than monolayer ones:

- extrusion of several materials which processing conditions can differ significantly
- tolerances on intermediate layer dimensions are smaller than those of monolayer structures

These structures were developed few years ago for two main reasons:

- Technical: new environmental norms
- Economical: cost reduction

New environmental norms constrained car manufacturers to reduce drastically vehicle fuel emissions. However parts of these emissions depend directly on permeability of the materials used for fuel transfer and storage. Thus raw material suppliers had to develop new structures including an intermediate "barrier" layer to stop fuel particle migration. Concerning economical aspect, the possibility to dissociate internal and external functions of the pipe allows selecting the cheapest material for each layer. Thus, for instance, functions of PA9T/PA12 and PA6/EVOH/ADH/PA12 structure layers are presented Table 16.

Table 16 – Structure layer functions

a. PA9T/PA12 structure

Material	Technical function	Cost
PA9T	Compatibility with the fluid of the application (E85, 110°C, 3.5 bars relative…)	++++
PA12	Compatibility with the environment of the application (110°C, engine vibrations…)	++

b. PA6/EVOH/ADH/PA12 structure

Material	Technical function	Cost
PA6	Compatibility with the fluid of the application (E10, 110°C, 3.5 bars relative…)	+
EVOH	Permeability to the fluid of the application: "barrier" layer	+++
Bonding	Adhesion between EVOH and PA12 layers	++
PA12	Compatibility with the environment of the application (110°C, engine vibrations…)	++

Appendix 2: Product quality management

Product quality management procedure is illustrated Figure 85. It is divided in three steps.

Characterisation

Product required characteristics are identified from specifications then they are quantified or qualified depending on their nature. Thus an exhaustive list of parameters is drawn up with all criteria that manufactured product should meet to ensure that each requirement is fulfilled.

Design

Product and manufacturing process are designed in parallel since they are interdependent. Product definition is based on product required characteristics identified in the previous section.

Quality control

A Failure Mode and Effects Analysis (FMEA) is carried out from product definition, process description and product required characteristics. Risks relative to process deviations are preferentially eradicated by mistake-proofing. When it is not possible, product characteristics are controlled during serial production or if it is not possible to measure them directly, equivalent monitored parameters are identified. Lastly, risks depending on product design are removed by carrying out validation tests during development phase. First version of FMEA document is created early in development phase then it is continuously enriched by experience until the end of serial life.

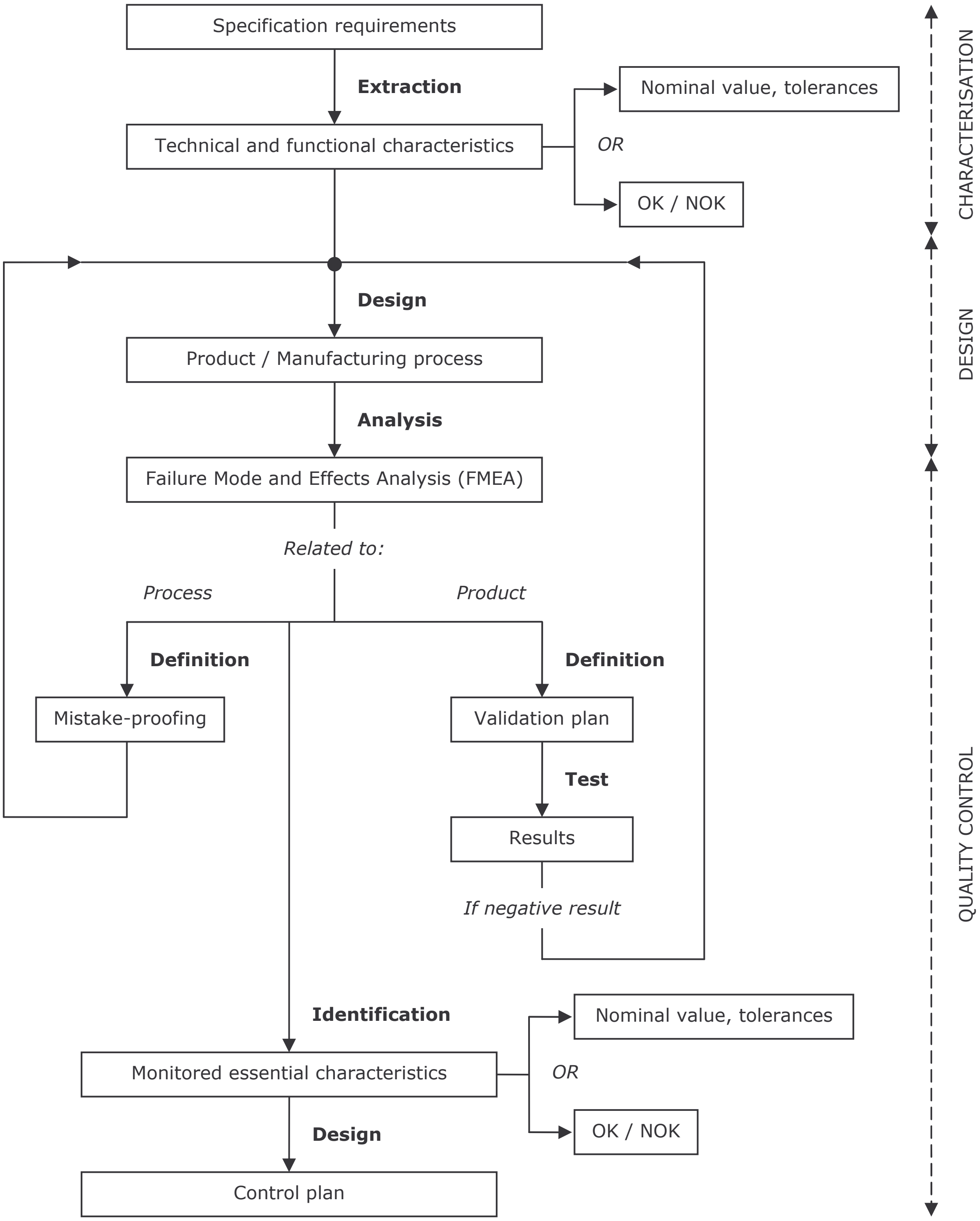


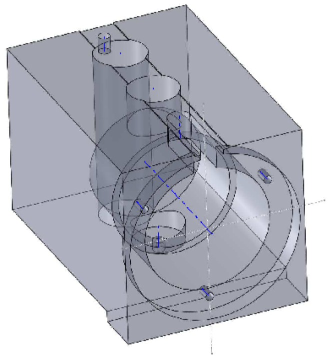
Figure 85 - Product quality management procedure

Monitored parameters, measurement method and control frequency are specified into the control plan. A batch of production is blocked until all items of the control plan are completed.

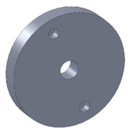
Appendix 3: Cutting machine bill of materials

Cutting machine components are listed below:

Frame

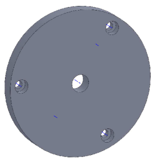


Frame part1



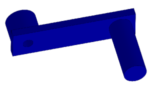
Frame shaft (X2)

Frame part2

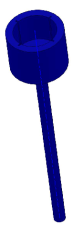


Screw M8x16 (X3)

Handle



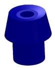
Pipe holder



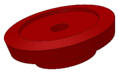
V-blade female

V-blade male

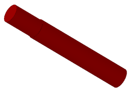
Pipe holder gear



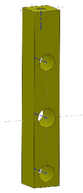
Blade holder gear



Threaded shaft



Blade holder

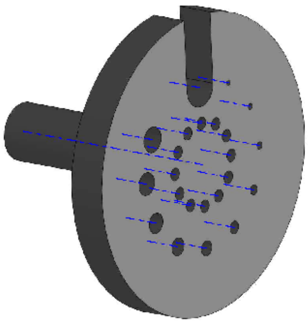


Screw M8x12

Washer

Guillotine blade

Die



Die pin

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