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NIKO MERIVIRTA
IMPROVEMENT OF QUALITY CONTROL OF EXTRUDED TUBES WITH ON-LINE OPTICAL MEASURING TECHNIQUE

Master of Science Thesis

Examiners: Assistant Professor Essi Sarlin and doctoral student Ilari Jönkkäri
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ABSTRACT

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Manufacturing of small diameter polymeric medical tubes with high dimensional tolerances is a challenging process that requires high end extrusion equipment to meet the strict quality standards. Microextruders along with controllable die systems are used in clean room environments to assure the uniform product quality and sterility. The tube extrusion process is a continuous process that is affected by the polymer melt temperature, pressure of the melt mass inside the extruder, line speed, cooling rate and other parameters which may greatly influence the quality of the finished product. By measuring the surface roughness continuously and monitoring the surface topography on-line during the manufacturing process, one can see the changes in the product in real time, which give indication on changes in the overall quality of the product. If the surface roughness increases or decreases past set boundaries one can take corrective actions immediately, possibly saving time, material and improving the productivity of the process.

Optical on-line measurement technique was used in this thesis to measure the surface roughness parameters during the production of small diameter plastic tubes in laboratory conditions. Prior to the laboratory measurements, few commercial samples were acquired and surface roughness was measured from the sample tubes using two different techniques: an on-line method and optical profilometry. The comparison of the results showed similar values with some variation due to differences between the techniques. The laboratory measurements were conducted with two thermoplastic materials, linear low density polyethylene, being the reference material, and thermoplastic urethane, being a material commonly used in medical tubing. In addition, to understand medical tube requirements, materials and manufacturing better, an expert interview was conducted and literature references were studied to form the theory basis of the thesis.

Although some challenges were faced, the measurements were conducted successfully and the effect of different processing parameters could be interpreted from the results. The optical on-line measurement technique proved to be a useful tool to control and monitor the quality of the tube during manufacturing, measuring the surface roughness with great precision. In addition, if one wishes to find correct processing parameters for a new material, the technique can provide information when adequate shear rates and material plasticization is achieved. This could be a great benefit in plastic tubing industry where long test runs may be required to find suitable process parameters for new materials.

TIIVISTELMÄ

NIKO MERIVIRTA: EKSTRUUSIOLLA VALMISTETTUJEN LETKUJEN LAADUNVALVONNAN TEHOSTAMINEN OPTISEN ON-LINE MITTATEKNIIKAN AVULLA
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Halkaisijaltaan pienien muovisten medikaaliletkujen valmistus on haastava prosessi erittäin hienojen mittatoleranssien takia. Medikaaliletkujen laatuvaatimukset ovat tiukat, joten ekstruusiolaitteiston on oltava tarkoitukseen soveltuva ja tarkka. Valmistuksessa käytetään pienikokoisia mikroekstruudereita ja tarkkoja letkusuuttimia puhdashuonetiloissa, jolloin tuotteiden jatkuva laatu ja steriilisyys voidaan taata. Letkun valmistus on jatkuva prosessi, jonka onnistumiseen vaikuttavat useat parametrit, kuten muovisulan lämpötila, sulapaine ekstruuderissa, linjanopeus ja letkun jäähtymisnopeus. Pinnan topografiaa voidaan käyttää indikaattorina letkun yleisestä laadusta ja tarkkailemalla pinnankarheutta optisesti ajon aikana, voidaan nähdä mahdolliset laatua heikentävät tekijät reaaliajassa. Mikäli pinnankarheus nousee tai laskee ohi sallittujen rajojen, voidaan korjaavat toimet suorittaa ajon aikana ja mahdollisesti pienentää materiaalikustannuksia, säästää aikaa ja tehostaa prosessia.

Tässä diplomityössä hyödynnettiin optista on-line-mittatekniikka, jolla mitattiin pinnankarheuden parametreja muoviletkun ajon aikana laboratorio-olosuhteissa. Vertailudataa varten hankittiin kaupallisia näytteitä, joista mitattiin pinnankarheus käyttäen kahta eri mittatekniikkaa, edellä mainittua optista on-line tekniikkaa sekä optista profilometriaa. Arvojen vertailu osoitti, että on-line tekniikka antaa vertailukelpoisia ja yhtä tarkkoja arvoja kuin profilometria, joskin pientä hajontaa syntyi tekniikoiden eroista johtuen. Laboratoriomittaukset suoritettiin kahdelle materiaalille, lineaariselle pienitiheyspolyeteenille, joka toimi referenssimateriaalina, sekä termoplastiselle polyuretaanille, jonka tiedettiin olevan yleinen medikaaliletkujen materiaali. Ennen käytännön töitä suoritettiin kirjallisuusselvitystä ja haastateltiin sairaalatarvikkeiden hankinnoista vastaavaa asiantuntijaa, millä pyrittiin selvittämään medikaaliletkujen vaatimuksia, materiaaleja ja valmistusta paremmin. Tiedot muodostavat diplomityön teoriapohjan.

Koeajot suoritettiin hyvällä menestyksellä, vaikka joitakin prosessointiin liittyviä ongelmia ilmeni. Testituloksista voitiin tulkita eri prosessiparametrien vaikutus letkun pinnankarheuteen. On-line mittatekniikka toimi hyvin ajon aikana letkun laadunvalvonnassa ja prosessin kontrolloimisen apuvälineenä, mitaten pinnankarheuden varmasti ja suurella tarkkuudella. Lisäksi tulokset osoittivat, että tekniikan avulla voidaan saavuttaa tietoa myös materiaalin plastisoitumisesta, joka voi osoittautua erittäin hyödylliseksi teollisuuden tuotantolinjoilla, jossa uusien materiaalin käyttöönotossa joudutaan suorittamaan pitkiäkin koeajoja sopivien parametrien löytämiseksi.

PREFACE

This thesis was made with the support of TUT Foundation. The project has been long and challenging but also rewarding and instructive. Working on this thesis has been a good learning experience in project working and a change to use my knowledge gained from the studies and previous work. I hope that working on this project has increased my readiness to work in the industry and I can apply the knowledge and know-how in real life and also develop and learn even more in the future.

This thesis would not have been possible without the support given by numerous people. Therefore, I want to thank all the people who supported and helped me during the project. Many thanks to Kai Syrjälä who has been a mentor and support during the entire process, having faith in me and encouraging me. Second, I want to thank my examiners, doctoral student Ilari Jönkkäri and assistant professor Essi Sarlin for their guidance. Also, thanks to research engineer Tommi Lehtinen, who was a great advisor during the extrusion line assembly for the laboratory measurements, and to doctoral student Olli Orel, who helped with the profilometry imaging. I also want to thank the people in FocalSpec for the chance to work on the thesis, Auli Nummila-Pakarinen in Borealis, who provided material and Kimmo Järventaus in Tuomilogistiikka, who gave an expert insight in medical device materials and acquisitions.

Lastly, I want to thank my family and friends from the support during my studies and this project. Special thanks to my father, who gave me the inspiration to pursue studies in the field of plastics engineering.

Tampere, August 23rd, 2017

Niko Merivirta

CONTENTS

1. INTRODUCTION 1
2. TUBE EXTRUSION PROCESS 3
  - 2.1 Extrusion process in general 3
    - 2.1.1 Single screw extrusion 5
    - 2.1.2 Twin screw extrusion 7
  - 2.2 Tube and pipe extrusion line 9
    - 2.2.1 Die, mandrel and breaker plate 10
    - 2.2.2 Calibration and cooling 11
    - 2.2.3 Puller and cutter 12
    - 2.2.4 Additional equipment 13
  - 2.3 Microextrusion of medical tubes 14
    - 2.3.1 Special characteristics and safety issues 15
  - 2.4 Cable and wire coating 16
3. COMMON POLYMERIC MATERIALS IN MEDICAL TUBE EXTRUSION ... 19
  - 3.1 Plastic and elastomeric materials 21
    - 3.1.1 Types of polymeric materials 21
    - 3.1.2 Isomerism 23
    - 3.1.3 Glass transition temperature and melt temperature 23
    - 3.1.4 Importance of molecular weight 24
  - 3.2 Polyvinyl chloride 24
  - 3.3 Thermoplastic fluoropolymers 25
    - 3.3.1 Polytetrafluoroethylene 26
    - 3.3.2 Fluorinated ethylene propylene 27
    - 3.3.3 Polyvinylidenefluoride 28
    - 3.3.4 Perfluoroalkoxy copolymer 28
  - 3.4 Silicone rubber 29
  - 3.5 Thermoplastic polyurethane 31
  - 3.6 Natural rubber 32
  - 3.7 Other materials 33
    - 3.7.1 Polyolefins 33
    - 3.7.2 Polyamides 35
4. MEDICAL TUBE QUALITY MANAGEMENT 36
  - 4.1 Quality issues in extrusion process 36
    - 4.1.1 Sharkskin, slip and stick and melt fracture 36
    - 4.1.2 Other issues 37
  - 4.2 Medical tube quality management 39
    - 4.2.1 Standards and guidelines 40
    - 4.2.2 Traceability 41
    - 4.2.3 Sterility 41
    - 4.2.4 Dimensional tolerances 43

4.2.5 Surface roughness and friction..44
5. SURFACE ROUGHNESS MEASUREMENT OF POLYMERIC TUBES 46
5.1 Surface roughness parameters...46
5.2 Line Confocal Imaging...48
5.2.1 Previous study...51
5.3 Optical profilometry...52
6. TEST PROCEDURES AND EQUIPMENT FOR SURFACE ROUGHNESS MEASUREMENTS 54
6.1 Reference data from sample tubes 54
6.1.1 Line Confocal Imaging 55
6.1.2 Optical profilometry..55
6.2 On-line test runs 60
6.2.1 Extrusion equipment 61
6.2.2 Materials..64
6.2.3 Test run matrix and shear rate calculations....................................65
7. RESULTS AND DATA ANALYSIS OF SURFACE ROUGHNESS MEASUREMENTS 70
7.1 Reference samples..70
7.2 On-line test runs 73
7.2.1 LLDPE 73
7.2.2 TPU 79
8. DISCUSSION 85
8.1 Benefits of optical on-line surface roughness measurement in industry......88
9. CONCLUSION...91
REFERENCES..94

APPENDIX A: ISO 13485:2016 MEDICAL DEVICES. QUALITY MANAGEMENT SYSTEMS. REQUIREMENTS FOR REGULATORY PURPOSES; CONTENTS AND SCOPE

APPENDIX B: ISO 14644-1:2015 CLEANROOMS AND ASSOCIATED CONTROLLED ENVIRONMENTS. PART 1: CLASSIFICATION OF AIR CLEANLINESS BY PARTICLE CONCENTRATION; ISO CLASSES OF AIR CLEANLINESS BY PARTICLE CONCENTRATION

APPENDIX C: 2D-IMAGES FROM REFERENCE SAMPLES

APPENDIX D: SHEAR RATE CALCULATIONS

LIST OF FIGURES

Figure 1: a) flood-fed extruder b) starve-fed extruder, modified from [2, p. 7] *4*
Figure 2: A schematic presentation of an extruder unit [4] *4*
Figure 3: Screw elements of single flight screw [2, p. 25] *5*
Figure 4: Sections of single screw [2, p. 25] *6*
Figure 5: Melting of polymeric material and solid bed formation [2, p. 54] *6*
Figure 6: Barrier screw transition section, modified from [2, p. 75] *7*
Figure 7: Fully intermeshing co- and counter-rotating parallel twin screws [2, p.125] *8*
Figure 8: Typical twin screw design, modified from [2, p. 153] *8*
Figure 9: Main components of a tube and pipe production line [2, p. 573] *9*
Figure 10: Typical design of breaker plate [5] *10*
Figure 11: Spider die design for tube and pipe extrusion [2, p. 574] *11*
Figure 12: Vacuum calibration tank [2, p. 576] *12*
Figure 13: ESI MD series caterpillar puller [6] *12*
Figure 14: Melt gear pump [2, p. 419] *13*
Figure 15: American Kuhne micro modular extruder [8] *14*
Figure 16: Gimac vacuum calibration unit [9] *15*
Figure 17: A wire coating line [2, p. 556] *17*
Figure 18: Cable and wire coating dies [2, p. 557] *17*
Figure 19: Classification of plastics, modified from [11] *19*
Figure 20: Polymer classification by structure [12, p. 22] *22*
Figure 21: Geometrical isomerism on polybutadiene [12] *23*
Figure 22: Structure of polyvinyl chloride [15] *25*
Figure 23: Structure of polytetrafluoroethylene [12] *26*
Figure 24: Structure of fluorinated ethylene propylene [16] *27*
Figure 25: Structure of polyvinylidenefluoride [17] *28*
Figure 26: Structure of perfluoroalkoxy copolymer [18] *29*
Figure 27: Structures of monomers that typically form polysiloxanes [12] *30*
Figure 28: Structure of thermoplastic polyurethane [12] *31*
Figure 29: Structure of cis-polyisoprene [22] *32*
Figure 30: Structure of polyethylene (left) and polypropylene (right) monomers, modified from [23] *34*
Figure 31: A plastic tube showing: A) sharkskin B) slip and stick C) melt fracture [26] *37*
Figure 32: A silicone tube with microstructured surface finish [33] *45*
Figure 33: Surface roughness R-profile with a mean line [34] *47*
Figure 34: Determination of Rz-value from R-profile [34] *47*
Figure 35: FocalSpec MP900 roughness measurement system [37] *49*
Figure 36: MP900 user interface during a line-mode measurement *51*
Figure 37: Alicone InfiniteFocus G5 optical 3D profilometer *52*

Figure 38: Samples for reference measurements......54
Figure 39: Procedure for static LCI measurements......55
Figure 40: Procedure for 3D profilometry imaging......56
Figure 41: Sample A surface with 20x objective without and with an overlay......57
Figure 42: Sample B surface with 20x objective without and with an overlay......58
Figure 43: Sample D surface with 20x objective without and with an overlay......59
Figure 44: The roughness profile was measured with a line-tool in a zigzag manner to increase the profile length for comparable results with MP900......60
Figure 45: Test line assembly, modified from [2, p. 573]......61
Figure 46: Brabender Plasti-Corder extruder used in the laboratory tests......62
Figure 47: Tubing die disassembled......62
Figure 48: The calibrator and the level of cooling water......63
Figure 49: Caterpillar puller used in laboratory tests......64
Figure 50: Occurrence of sharkskin and slip and stick in LLDPE with increasing shear rate [26]......66
Figure 51: Line speed vs. theoretical shear rates in the die used in laboratory tests showing the theoretical values for sharkskin and slip and stick occurrence......67
Figure 52: Distribution of surface variations from 0-plane in one point in sample A......72
Figure 53: LLDPE at 185 °C......74
Figure 54: LLDPE at 200 °C......75
Figure 55: LLDPE at 210 °C......75
Figure 56: LLDPE at 200 °C with increasing production speed......77
Figure 57: Histogram of Ra-values of LLDPE at 200 °C......78
Figure 58:TPU at 215 °C......79
Figure 59: TPU at 220 °C......80
Figure 60: TPU at 225 °C......80
Figure 61: TPU at 220 °C with increasing production speed......81
Figure 62: Histogram of Ra-values of TPU at 220 °C......82
Figure 63: TPU at 220 °C, line speed of 10 m/min and screw speed of 110 rpm......83
Figure 64: Histogram of Ra-values of TPU at 220 °C, line speed of 10 m/min and screw speed of 110 rpm......83
Figure 65: On the left: a thick defect appearing during TPU processing; on the right: the nozzle used to remove moisture from the tube’s surface......85

LIST OF SYMBOLS AND ABBREVIATIONS

COF	Coefficient of friction
DEHP	di(2-ethylhexyl) phthalate
DP	Degree of polymerization
ePTFE	Expanded polytetrafluoroethylene
EP	Epoxy
EU	European Union
FDA	Federal Drug Administration
FEP	Fluorinated ethylene propylene
GMP	Good Manufacturing Practice
HCl	Hydrochloric acid
HDI	Hexamethylene diisocyanate
HDPE	High density polyethylene
IPDI	Isoprene diisocyanate
ISO	International Organization for Standardization
LCI	Line Confocal Imaging
LCP	Liquid crystal polymer
LDPE	Low density polyethylene
LLDPE	Linear low density polyethylene
MDI	4,4-diisocyanate diphenylmethane
MDPE	Medium density polyethylene
MES	Manufacturing execution system
MFR	Melt flow rate
NR	Natural rubber
OD	Outer diameter
PA	Polyamide
PAI	Polyamide-imide
PC	Polycarbonate
PE	Polyethylene
PEEK	Polyether ketone
PEI	Polyetherimide
PET	Polyethylene terephthalate
PF	Phenol formaldehyde
PFA	Perfluoroalkoxy copolymer
PI	Polyimide
PLA	Polylactic acid
PMMA	Poly(methyl methacrylate)
POM	Polyoxymethylene
PP	Polypropylene
PPS	Polyphenylene sulfide
PPSU	Polyphenylsulfone
PS	Polystyrene
PSU	Polysulfone
PUR	Polyurethane
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl chloride
PVC-E	Emulsion polymerized polyvinyl chloride
PVC-M	Bulk polymerized polyvinyl chloride
PVC-P	Plasticized polyvinyl chloride

PVC-U	Unplasticized polyvinyl chloride
PVC-S	Suspension polymerized polyvinyl chloride
PVDF	Polyvinylidenefluoride
SFS	Finnish Standards Association
SMR	Standardized Malaysian Rubber
TPE	Thermoplastic elastomer
TPU	Thermoplastic polyurethane
TTY	Tampereen teknillinen yliopisto
TUT	Tampere University of Technology
UF	Urea-formaldehyde
UP	Unsaturated polyester
UV	Ultra violet

Cpk	Process capability index
ln	Evaluation lenght
lr_i	Sampling length
N	ISO Class number
Q	Flow rate
Ra	Arithmetical mean surface roughness value
Rt	Total height of the surface roughness profile
Rz	Mean surface roughness depth
Rz_i	Greatest height of the surface roughness profile
Std	Standard deviation
T_g	Glass transition temperature
T_m	Melting temperature
V	Puller speed
$\dot{\gamma}$	Shear rate
λ_c	Cut-off wavelength

1. INTRODUCTION

The purpose of this Master's Thesis was to find out if optical Line Confocal Imaging (LCI) technique can be beneficially used in on-line quality control of small diameter medical tube manufacturing. The aim was to be able to adjust the polymer extrusion process by measuring the tube surface roughness to see possible quality issues in time for corrective actions and thereby increase the productivity of the process. The technique was previously applied in a Master of Science thesis work to monitor plastic film manufacturing process and proven successful by Hautala J. [1].

The objective of the work was to produce reliable and precise quality data from tube manufacturing process on a production line and gain experience on how the roughness sensor fits to the tube extrusion operation environment. Practical laboratory work was conducted in the Laboratory of Materials Science at Tampere University of Technology.

The focus of the thesis was chosen to be in the field of plastic medical tubing because it was expected to benefit from accurate on-line surface roughness measurement. Therefore, it was decided that the initial information gathering should include an expert interview to understand the quality requirements of medical tubing applications better. Mr. Kimmo Järventaus from Tuomilogistiikka Oy was interviewed since he was a person with experience in the field of medical supplies and their purchasing principles.

According to the interview most of the small dimension tubing are used in different catheter applications. The choice of the material depends on the individual person, and the body part where the tube is inserted. Typical catheter sizes vary from 3 mm outer diameter to 7 mm outer diameter. Generally, tubing of softer materials, such as silicone and natural rubber, are used in urinal and rectal catheter applications whereas tubing of more rigid material, such as polyvinyl chloride and thermoplastic polyurethane, are used in intubation tubes and central venous catheters. The key factors are the size of the lumen inside the tube to ensure proper fluid flow, the effect of body heat to the properties of the tube and the softness of the material to reduce patient discomfort.

The materials used in medical tubes are well established since new materials require thorough clinical tests to meet the requirements of ISO and FDA standards and achieve CE-mark to enter the European markets. The most common material is polyvinyl chloride followed by elastomeric latex and silicone rubber. Thermoplastic polyurethane is also an established medical tubing material used e.g. in intubation tubing. Polyamides and polyolefins can be used in some extent but the hardness of the material limits their use. More expensive engineering and high-temperature plastics such as fluoroplastics are used in coating applications.

The acquisition principles for medical tubes are according to the needs and requirements of hospitals. The manufacturer are responsible for the quality and sterility of the product that should meet the requirements set by the international standards. The sterility is vital in medical applications and the method for sterilization depends on the material. However, the manufacturing of medical devices in general requires a clean room conditions which sets yet more requirements for the manufacturing environment.

In general, the surface of the plastic tube inserted into human tissue should be smooth to prevent tearing the mucous membranes. However, some applications, such as suction tubes, exist where the tube surface is profiled into micro-grooves to reduce friction and tackiness.

The changes in surface roughness indicate how stable the extrusion process is and hence how the quality of the product remains at wanted level. The technique for on-line surface roughness measurement, Line Confocal Imaging, was successfully used in this thesis work to observe the changes in surface roughness values caused by altering the extrusion process parameters. In addition, it was verified by comparing LCI with optical profilometry that the technique is not only highly accurate but fast and user friendly as well.

2. TUBE EXTRUSION PROCESS

Tube and pipe extrusion is a process where melt polymeric material is extruded through a die into a shape of tube or pipe. A tube extrusion line consists of several units that need to function together in synchronized manner to produce high quality products. The production line can be quite long and an incorrect setting or wrong process parameters may greatly affect the quality of the finished product. [2, pp. 3-11]

This chapter explains the basic principles and some theoretical background of the polymer extrusion process. Operation of a single and twin screw extruders are explained and polymer melt behavior in the extruder is covered briefly. Tube/pipe extrusion line equipment and operation are explained before advancing to small diameter medical tube extrusion by the means of microextrusion. In addition, the basic operations of wire and cable coating process are covered briefly.

2.1 Extrusion process in general

Extrusion process utilizes a rotating screw inside a heated barrel to homogenize and melt thermoplastic polymer granulates into a dense mass. The melting occurs with the help of heat and friction. The melt mass travels through a die which gives the material its final shape. Extrusion process can be used in manufacturing of different continuous products, such as tubes, pipes, films and coatings. In addition, extrusion can also be used in blending of different polymers together with additives and color agents and in manufacturing of polymer granulates. [3]

The extrusion process begins with feeding the polymeric material into the extruder. In the extrusion of polymeric materials, the raw material is usually in form of granulates or powder. There are few different methods to implement material feeding from which the two most common ways are

- Flood feeding and
- Starve feeding. [2, pp. 3-11]

The simplest way is flood feeding where material is placed into a hopper which is located directly over the extruder feed throat. The material is fed into the extruder with the help of gravity and screw rotation. In flood feed method, the throughput of the extruder is directly proportional to the speed of the screw. In starve feeding the throughput rate is not proportional to the screw rotation speed because the material is deposited in feeders above the feed throat. The screw removes the material faster than it is fed into the extruder so the throughput depends on the feed rate. Flood feeding and starve feeding are illustrated in Figure 1. [2, pp. 3-11]

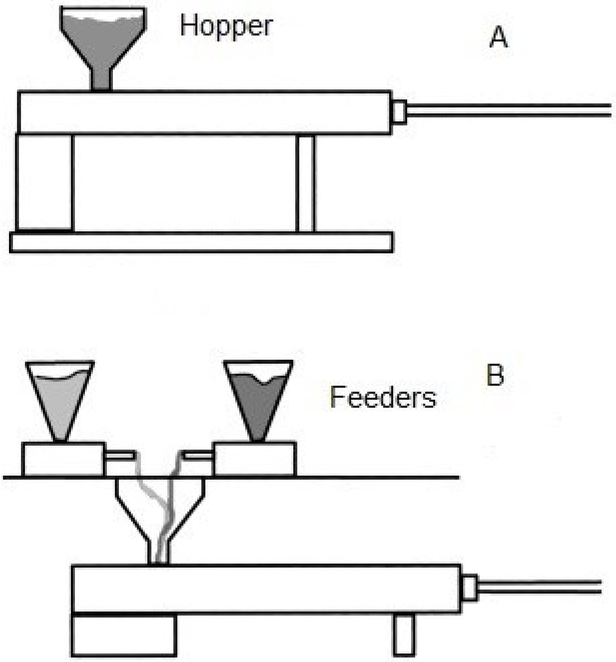


***Figure 1**: a) flood-fed extruder b) starve-fed extruder, modified from [2, p. 7]*

From feeding unit, the material is conveyed to a plasticizing unit. The plasticizing unit consists of a barrel and a screw. The screw inside the barrel is fitted to the diameter of the barrel so that there are few tenths of millimeters' space between the barrel wall and the screw flight. The barrel is surrounded by heating and cooling elements which are used to control the temperature profile of the barrel. After plasticizing and mixing the material is conveyed to die assembly. If provided, pressure gauge for measuring the melt pressure is located at the end of the barrel right before the die. Process parameters such as temperature profile and screw speed can be changed from the control unit's interface. [2, pp. 3-11] A schematic presentation of an extruder is presented in Figure 2.

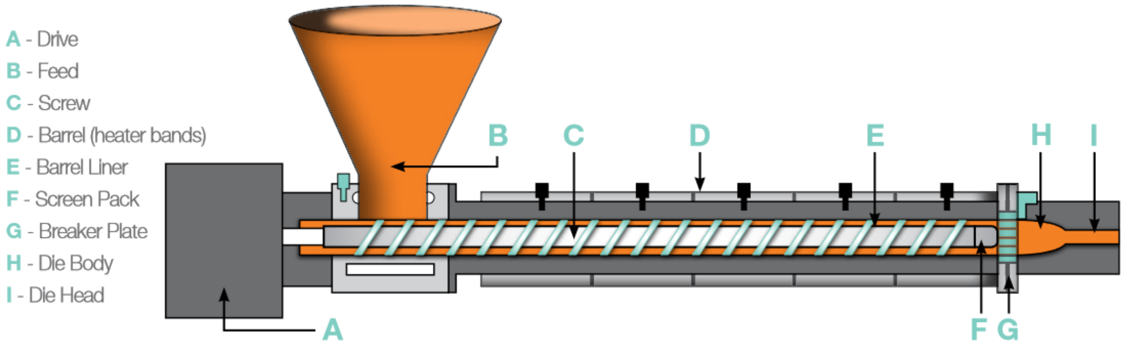


***Figure 2**: A schematic presentation of an extruder unit [4]*

Cooling of the screw is usually implemented with circulating water. Extruder may have one or two screws inside the barrel depending on the usage purpose. Most of the extruders have one screw inside the barrel and they are called single screw extruders. However, extruders that contain two screws, twin screw extruders, are more common when dealing with materials that are not in granular form. Twin screw extruder is typically used in compounding polymers and additives together and when working powder like materials such as polyvinyl chloride (PVC). [3]

2.1.1 Single screw extrusion

The most important part of the extruder is the screw. The screw elements of a single flight screw are depicted in Figure 3. The helix angle is the angle between the screw flight and the center axis and the diameter is the distance between the furthest flights. [2, pp. 17-46]

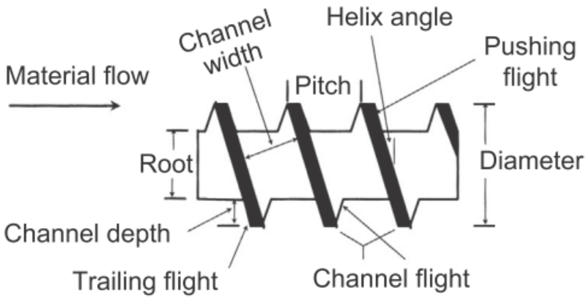


***Figure 3**: Screw elements of single flight screw [2, p. 25]*

The purpose of the screw is to

- Melt,
- Convey and
- Homogenize

the polymeric material before conveying it to the die. The plastic behavior of the polymeric material inside the barrel depends on the processing conditions like temperature, screw rotations per minute and the design of the screw itself. [2, pp. 17-46] The screws are designed so that the different sections of the screw have different effect to the process; by changing the geometry of the screw one can affect to, for example pressure, thermal degradation, material getting stuck to the barrel wall and devolatilization of gasses. [3]

In single screw extrusion, the extruder can be divided into three functional sections (Figure 4):

- Feeding section,
- Transition section and
- Metering section.

In feeding section, the friction between the plastic granulates, friction between the granulates and the screw and friction between the granulates and the barrel wall causes solid material to flow forward. The feeding becomes more efficient when the coefficient of friction between the barrel and the material is high and the coefficient of friction between the screw and the material is low. [3]

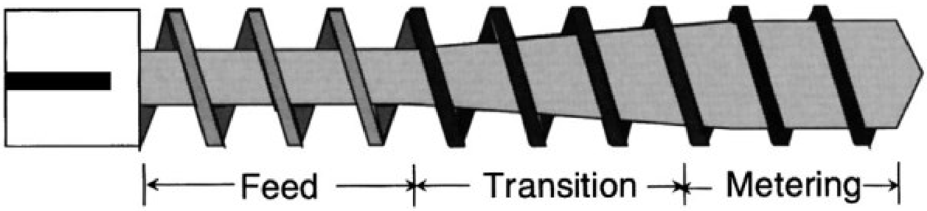


Figure 4*: Sections of single screw [2, p. 25]*

Polymeric materials start their initial melting and plastication in the feeding section. Semicrystalline materials like polyethylene (PE) and polypropylene (PP) show a clear melting point T_m where material starts to melt. Amorphous polymers such as polystyrene (PS) however soften until their viscosity becomes low enough for processing. The softening of amorphous materials begins to occur when the temperature exceeds the glass transition temperature Tg of the material. Heat required for melting and softening to occur is generated by shear heating due to friction between the material, screw and barrel. About 80 to 90% of the heat required for melting is caused by shear heating and the rest 10 to 20% is generated by the barrel heaters. [2, pp. 47-70]

The melting of semicrystalline materials and softening of amorphous materials continue when the material is conveyed from feeding zone to transition section. As seen in Figure 4, the screw channel becomes narrower in the transition section causing compression to increase due to solid material being pushed forward inside the barrel. In transition section, polymeric material forms a thin melt film between the barrel wall and not yet melt solid bed (Figure 5). Further melting and formation of the thin film occurs due to shear heating and barrel heaters while the pushing flight gathers melt material in front of itself and pushes the solid bed against the trailing flight. The size of the melt pool increases when the material travels through the transition section due to solid bed being melted. [2, pp. 47-70]

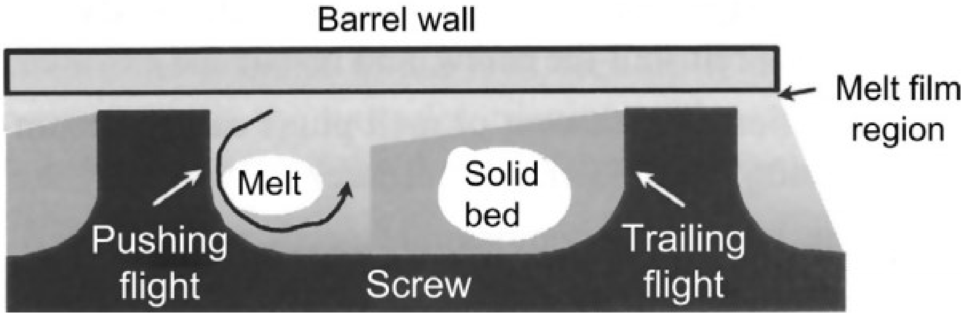


Figure 5*: Melting of polymeric material and solid bed formation [2, p. 54]*

After the material is completely melt in transition section it is conveyed to metering section. The purpose of the metering section is to convey the melt material to the die to form the extrudate. In addition, metering section may include a mixing zone to achieve uniform melt temperature and homogenous structure. Mixing can either be

- Distributive or
- Dispersive

depending on the shear stress. Distributive mixing utilizes the separation and rearrangement of the melt flow in low shear rates and is used to mix reinforcing fillers to the polymer melt. Dispersive mixing is high shear rate mixing where particles such as pigments in a masterbatch are broken up and mixed with a polymer matrix. [2, pp. 47-70]

Single screw extrusion may also utilize a special type of screw, a barrier screw, in cases where screw speed and the homogeneity of the material must be high. A barrier screw is used to improve the melting of the material and prevent solid bed breakup. In solid bed break up unmelt polymer granulates float in the melt pool causing nonuniform structure, poor mixing and lowered properties. [2, pp. 71-87]

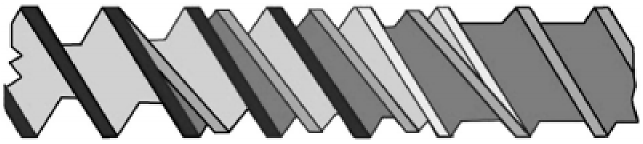


***Figure 6**: Barrier screw transition section, modified from [2, p. 75]*

As seen in Figure 6, barrier screw has an additional secondary flight along with the primary flight. The diameter of the barrier flight is smaller than the diameter of the primary flight which allows the molten polymer to be collected in separate melt pool in the barrier channel. Solid bed breakup cannot occur since the solid material must go through the barrier channel to exit the transition section. In the metering section, the barrier flight becomes the only channel as the primary flight disappears at the end of the transition section. [2, pp. 71-87]

2.1.2 Twin screw extrusion

As mentioned before, twin screw extruders are used in mixing and homogenizing of polymer resins and additives. Material feeding method to twin extruders vary depending on the design of the extruder and the material under process. The most typical mode is volumetric or gravimetric starve-fed feeding with single or multiple resin and additives feed streams. Further additives and reinforcement can also be fed into the melt with side feed extruders. Liquid components can be added to the melt downstream with liquid injection pump systems. [2, pp. 125-148]

A twin screw extruder consists of two parallel screws inside a barrel which has a number eight like cross section [3]. The typical case is that the flights of the screws intermesh one another. However, partially intermeshing and non-intermeshing builds where the distance between the screw shafts is higher are also possible. [2, pp. 125-148] In addition, a common case is that the screws rotate in different direction being called counter-rotating screws, but also co-rotating design is used [3]. Intermeshing co-rotating and counter-rotating screws are presented in Figure 7.

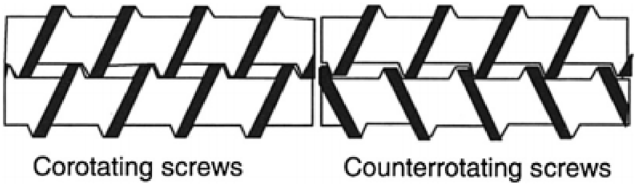


***Figure** 7: Fully intermeshing co- and counter-rotating parallel twin screws [2, p.125]*

Co-rotating twin screw extruders with high screw rpm are used in compounding polymer resins with additives, solvent removal by devolatilization and in situ chemical reactions during the process. Co-rotating and counter-rotating intermeshing screws with lower screw rpm are used in profile and pipe manufacturing. Counter-rotating extruders are especially used in PVC compounding. Non-intermeshing co-rotating extruders are not used in practice but counter-rotating non-intermeshing extruders have use in performing devolatilization and chemical reactions. [2, pp. 125-148]

Twin screw extruder screws and barrels are both modular, meaning that different sections can be added to improve compounding effectiveness, devolatilization and material feeding for optimal process conditions. Twin screw extruder can also be divided into functional sections like single screw extruder but due to modularity and flexibility of the process the sections may overlap and are not as explicit as in single screw extrusion. The screws typically consist of feeding section, melting section, melt conveying section, mixing section, downstream feeding section (to add fillers liquids etc.), devolatilization section and metering section. [2, pp. 125-148] Figure 8 shows a typical twin screw design with reducing flight pitch in the feeding zone section and kneading blocks in the melt conveying section.

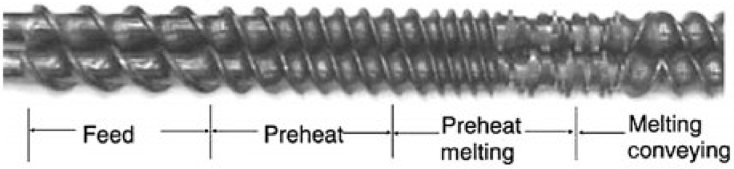


***Figure 8**: Typical twin screw design, modified from [2, p. 153]*

Although it is possible to manufacture profile and sheet products with twin screw extrusion, one limitation is rather poor melt pressure especially in the case of parallel co-rotating designs. Because of that twin extruder lines often utilize a melt gear pump to produce uniform melt flow, pressure and temperature before the material is conveyed to the die. [2, pp. 149-169]

2.2 Tube and pipe extrusion line

Profile extrusion that uses a die which produces round cross-section is called pipe and tube extrusion. The line is like any profile extrusion line except it usually utilizes vacuum calibration tank to adjust the pipe/tube diameter into correct dimensions. Products vary from small few millimeter size class diameter medical tubes to over meter size class diameter water carrying pipes depending on the design of the extruder used. In addition, different coextrusion procedures, corrugation and surface microstructure finishes are possible. Finished products are cut into predefined length packed in stacks or wound into coils as continuous product. [2, pp. 573-583]

The main components of a tube/pipe production line in order they appear in the line are:

- Single screw or twin screw extruder,
- Die and mandrel for piping/tubing,
- Vacuum calibration and cooling tank(s),
- Puller,
- Cutting unit and
- Packaging unit.

Figure 9 presents the main equipment schematically. In addition, some additional components may be found in industrial production lines such as mechanical or laser printers for marking and laser gauges for diameter and ovality monitoring. [2, pp. 573-583]

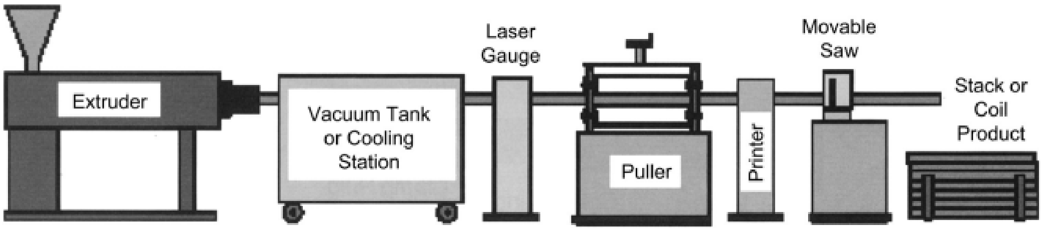


Figure 9*: Main components of a tube and pipe production line [2, p. 573]*

Since the functions of single and twin extruders were already described in previous sections the following sections will briefly describe the principles behind the other main components of tube/pipe production line.

2.2.1 Die, mandrel and breaker plate

An important part called a breaker plate is located after the barrel exit right before the die. The purpose of a breaker plate is to filter impurities, increase the melt pressure and stop turbulent flow of the melt mass [3]. In addition, breaker plate functions as a seal between the extruder and the die preventing polymer mass leaking around the die. Typical design of a breaker plate is a disk containing holes in symmetrical manner as seen in Figure 10. Breaker plate work in combination with a screen pack located before the plate. Screen pack is a metal wire screen which filters contaminations and creates back pressure. However, use of screen pack is limited since it cannot be used when processing materials containing fillers or reinforcements. [2, pp. 17-46]

***Figure 10**: Typical design of breaker plate [5]*

The die gives shape to melt polymer mass. An important aspect for even quality products is that the flow of the melt mass inside the die is constant in every part of the die. In addition, no sharp edges are allowed since they may form dead spots in the melt flow causing structural defects on the product. [3] Wanted product dimensions, diameter and wall thickness, affect to the selection of the die in tubing applications [2, pp. 573-583].

A common design of a pipe or tubing die is called a spider die (Figure 11). The center section, the mandrel (or torpedo), is held in place by three or more supporting spokes. These spokes are called spider legs since they support the weight of the mandrel. The polymer mass enters the die from the extruder through the breaker plate and the entrance cone distributes the melt mass around the mandrel. Larger dies require additional support such as dedicated stand or cart to support the weight of the whole die to avoid damaging the extruder barrel. Spider support design’s downside is structurally weaker weld lines that appear in the final product as seen in Figure 11. [2, pp. 573-583]

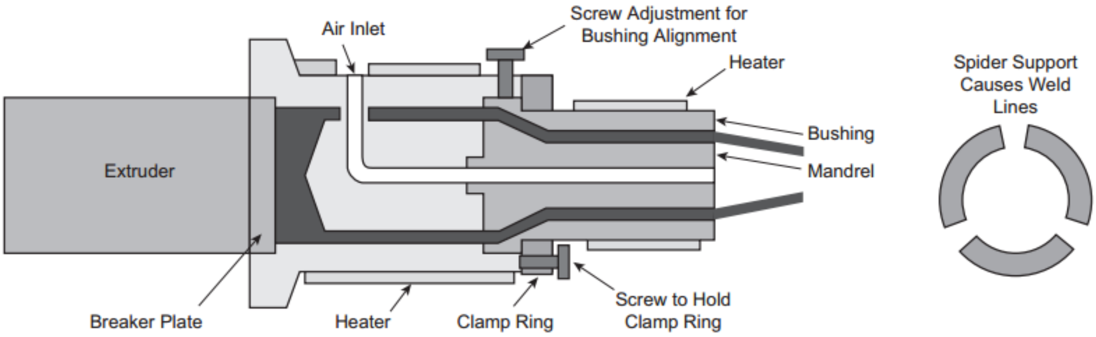


Figure 11*: Spider die design for tube and pipe extrusion [2, p. 574]*

The part around the mandrel is called bushing and to have the correct wall thicknesses in the final product the centering can be adjusted with adjustment bolts. Air inlet allows constant atmospheric pressure inside the tube or pipe to obtain the correct diameter with vacuum calibration. The area where the melt channel is constant in diameter forming the material into the shape of tube is called the die land area. The length of the area affects how well the material settles into uniform orientation. [2, pp. 573-583]

Spider die represents a typical construction for pipe extrusion which fits well for pipes with large diameters. However, small diameter pipes and tubes such as medical tubes can be produced with smaller dies which allows the design to be different from conventional spider dies since the weight of the mandrel is much lower. One possible design is to expand the flow channel after the spider legs to create a melt pool and then again restrict the channel to force the polymer chains to re-entangle which allows the elimination of weld lines in the final product [2, pp. 573-583].

2.2.2 Calibration and cooling

Calibration of a small diameter tube or pipe outer diameter (OD) is usually done with vacuum sizing. When the tube exits the die it first travels a small distance in the air and enters a vacuum sizing tank (Figure 12). The tube enters the tank through a vacuum sizing tube, also known as calibrator, which is a cylinder-shaped piece of metal that has holes for vacuum and for cooling water to reach the tube surface [2, pp. 573-583]. The tube can either be fully submerged in water or water can be sprayed around it. The calibration of the tube OD is achieved with the vacuum inside the tank. Atmospheric air pressure inside and vacuum outside of the tube forces the tube to expand against the inner walls of the sizing tube.

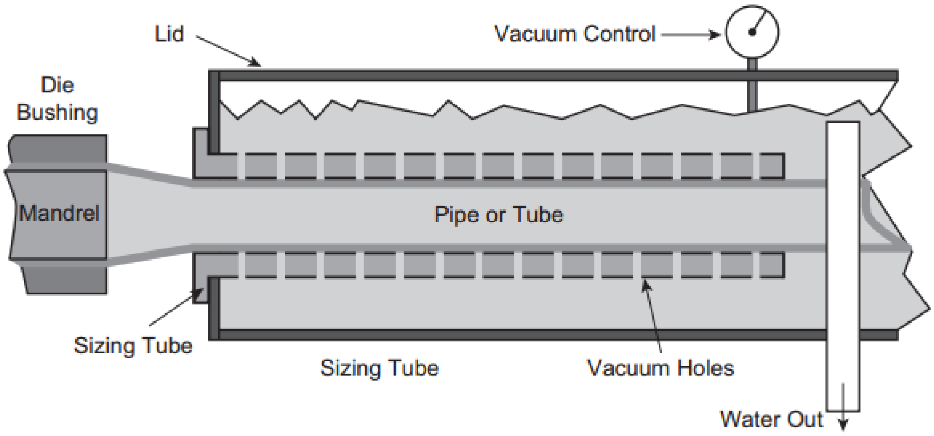


***Figure 12**: Vacuum calibration tank [2, p. 576]*

As seen in Figure 12 the diameter of the extrudate must be larger than the size of the sizing tube to form a proper vacuum seal. The ratio between the extrudate dimensions right after exiting the die and the dimensions of the tube is usually referred as the draw down ratio. Vacuum levels and water temperatures inside the tank can be controlled to achieve the wanted dimensions for the product and control the cooling rate. The cooling rate affects the crystallization of semicrystalline materials and shrinkages in both semi-crystalline and amorphous materials. [2, pp. 573-583] With larger pipe diameters one cooling unit is usually not sufficient and several cooling units are used in succession.

2.2.3 Puller and cutter

A puller is used to pull the tube or pipe from the die to the cutter and control the line speed. The puller and the cutter can be constructed in one unit or they can be separate units. A caterpillar design of a puller unit is common and presented in Figure 13.

***Figure 13**: ESI MD series caterpillar puller [6]*

The speed of the puller affects the wall thicknesses of the tube; if the walls are too thin the puller speed is decreased and increased if the walls are too thick. In addition, the diameter of the product can be controlled with the puller speed; if the diameter is too large the pulling speed can be increased or extruder screw speed decreased. The production line usually has a length counter that signals the cutting unit when to cut the product. [2, pp. 573-583]

2.2.4 Additional equipment

Packaging station is the final part of the extrusion line. The products can be coiled or stacked together. Coilers wind the tube or pipe into the reel in layers. When a reel is full or in fixed length the coiler pushes the full reel away and automatically starts winding a new one. The speed of the winder must be adjusted according to the speed of the puller delivering the tube or pipe. Tube and pipe extrusion line may also include on-line laser measurement of the product dimensions and printing equipment to mark the product. The printing may require surface treatment with corona to make the surface more polar especially with polyolefins to improve the quality of the print. [2, pp. 573-583]

In some cases, use of a melt gear pump is necessary to produce high melt pressure output. The gear pump provides consistent melt flow and enables higher throughput with lower screw speed with reduced shear heat. [2, pp. 417-424] A schematic presentation of a gear pump is presented in Figure 14.

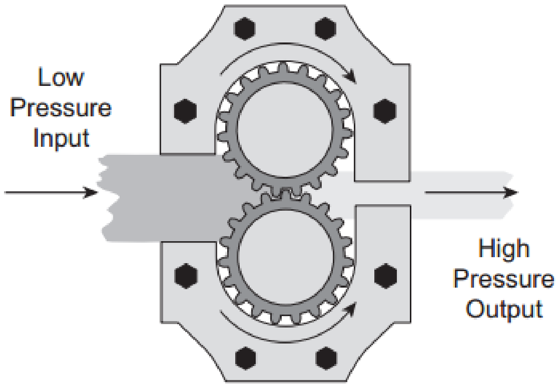


***Figure 14**: Melt gear pump [2, p. 419]*

Most extrusion based processes benefit from using a gear pump but especially microextruded products with small dimensions and high dimensional tolerances gain great advantage from the reduced pressure variations.

2.3 Microextrusion of medical tubes

Microextrusion is a technique used to produce thin walled small diameter tubes such as medical tubes. The technique is similar to normal tube and pipe extrusion but the equipment and the line length is in smaller scale. However, just scaling down a normal pipe line is not enough, the equipment must be specifically designed for production of high precision dimension tubes. [7] In addition, production of tubes for medical applications requires clean room environment.

If a conventional extruder was used to produce microtubing it would require the extruder to run on very low rpm which would increase the residence time of the material in the barrel. Too high residence time leads to decreased physical properties and therefore lower quality so it should be avoided especially in manufacturing of medical tubes. Using small diameter screw extruders that are designed to run at low screw rpm overcome the problem with thermal degradation originating from too long residence time. [7] Figure 15 presents a micro modular extruder from American Kuhne that is suitable for medical tubing applications [8].

Figure 15: *American Kuhne micro modular extruder [8]*

Extruders with screw diameters of 12—19 mm are common in the field of microextrusion [8; 9]. Since the product dimension are very small, both diameter and wall thickness often in sub millimeter range, and the output volume very low, the precision must also be very high. Microextruders may have automatically centering die head to meet the required tolerances in wall thicknesses since it is not possible to accurately adjust the centering by hand with such small product dimension. In addition, the pressure control during the process must be very accurate for uniform consistence of the product. [7]

Calibration of small diameter tubes also requires high precision from the vacuum calibration unit. A solid structure to minimize any mechanical vibration during the process, high level of cooling stability and precise control of vacuum inside the tank are standard requirements for a proper calibration unit in microextrusion [9]. Figure 16 presents a calibration unit from Gimac which is suitable for tubes with diameters between 0.4 mm and 6 mm [9].

***Figure 16**: Gimac vacuum calibration unit [9]*

Pullers used in microextrusion also need to be specially designed for tubes with very small diameters and wall thicknesses. It is essential that the puller does not harm the product by deforming it or scratching the surface. The parts that are in contact with the tube must be controlled with highly sensitive pneumatic system to avoid damage to the product. [9]

2.3.1 Special characteristics and safety issues

Some special characteristics exist in polymer melt processing that must be considered for operator safety and process functionality. Especially fumes caused by material degradation should be removed properly to avoid operator's exposure to harmful byproducts.

Some hygroscopic polymers absorb moisture from the surrounding air which means that certain materials may require drying prior to feeding to the extruder to remove excess moisture from the granulates. If moisture is not removed from the material it may vaporize and turn into steam inside the barrel and cause harm to the polymer structure and properties. Typical procedures to remove moisture from granulates are oven drying systems, hopper dryers and central drying systems. Polymers, such as polyamides, polycarbonate and polyesters, absorb moisture well and must be dried before extrusion. Non-hygroscopic polymers that do not absorb moisture are commonly used in medical tubing applications. These include polyethylene, polypropylene, polyvinyl chloride and fluorinated polymers. [2, pp. 399-408]

When considering medical tube extrusion, there are few commonly used materials that must be processed in well ventilated conditions due to operator health safety. Melt processing of polymers can cause material to degrade in the hot barrel releasing harmful gases into the air. Polyvinyl chloride is one of the materials that should be processed with care since degradation reaction produces hydrochloric acid (HCl) which is harmful to the operator but also corrosive to the machinery. Careful temperature profile management and knowledge about the upper processing limit is required in addition to well ventilated processing environment. [2, pp. 13-16]

In addition to polyvinyl chloride, fluoropolymer processing also requires well ventilated environment due to gaseous emissions released in material degradation. If the fumes are inhaled by the operator, it usually results in flu-like conditions known as polymer fume fever. The symptoms are fever, chills and coughs. The symptoms are temporary and disappear in one to two days. [10]

Although medical tubes are mainly produced from thermoplastic materials, there are tubing applications that are produced from elastomeric rubbers, natural and silicone rubber, requiring different processing conditions. Silicone rubber comes in two grades, liquid and solid, solid grade being more suitable for extrusion applications. Due to elastomeric structure, silicone rubber requires curing of the cross-linked polymer network after it exits the extrusion die. There are microextruders constructed specifically for silicone rubber extrusion where curing is performed in ventilated heating tunnel, often with peroxide gas. A curing tunnel is also used similarly when processing natural rubber by extrusion.

2.4 Cable and wire coating

Cable and wire coating is required in some medical applications, such as guidewires for stents which are inserted into the vein to keep it open, to modify the surface properties. Cable and wire coating is an extrusion based process but differs from the normal tube extrusion with changes in the production line. Figure 17 presents a wire coating line schematically.

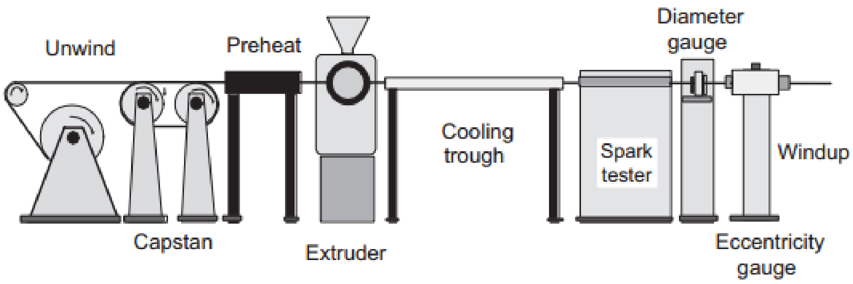


***Figure 17**: A wire coating line [2, p. 556]*

Coating lines usually utilize a single screw extruder. The purpose is to melt the polymer material and coat a wire with the resin uniformly. The line typically runs in high throughput and high melt pressure. The line consists of:

- Unwind stations from which the cable is fed to,
- Capstan that sets the wire or cable into wanted pretension,
- Preheating station for better adhesion,
- Extruder and crosshead die,
- Cooling trough,
- Spark tester, diameter and eccentricity gauge to monitor quality and
- Winding station. [2, pp. 555-560]

As seen in Figure 17 the extruder operates at an angle of 90 ° compared to the rest of the production line. Coating of multiple wires or cables is possible when extra extruders are added on the sides and the extrudate runs in the center of the line. Figure 18 shows a traditional design of the crosshead die used in coating. In pressure die the wire is coated inside the die and in tubing die the coating happens outside the die. The mandrel or core tube prevents the polymer material from flowing back to the point where the wire enters the die. [2, pp. 555-560]

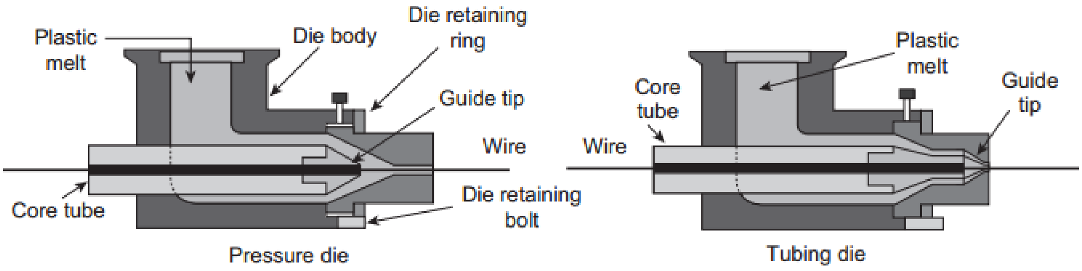


***Figure 18**: Cable and wire coating dies [2, p. 557]*

Cooling of the coated wire or cable happens in cooling through. Water is used to cool the product uniformly to avoid uneven shrinkages. Shrinkage is unavoidable but controllable through proper preheating, melt temperature control, cooling rate and cooling temperature control and drawdown ratio control. Diameter-, eccentricity and spark gauges are typically used in quality control after cooling to monitor the diameter, wall thicknesses and possible pin holes. [2, pp. 555-560]

3. COMMON POLYMERIC MATERIALS IN MEDICAL TUBE EXTRUSION

Polymeric materials, plastics and elastomers, are ideal choice of material for medical tubing applications. Among the different material categories, following attributes give plastic and elastomeric materials the edge compared to other materials:

- Light weight,
- Low cost,
- Easy processability,
- High performance mechanical, thermal, optical and electrical properties and
- Easy disposability.

High quality standards for medical tubing also set requirements for high quality medical grade materials and the end use purpose has high effect on the material selection. There are several materials that may be used in medical tubing applications processable by the means of microextrusion. The materials vary from common standard plastics to more expensive and less frequently used engineering and high-performance plastics. Figure 19 presents the classification of plastic materials as well as guidelines to their prices.

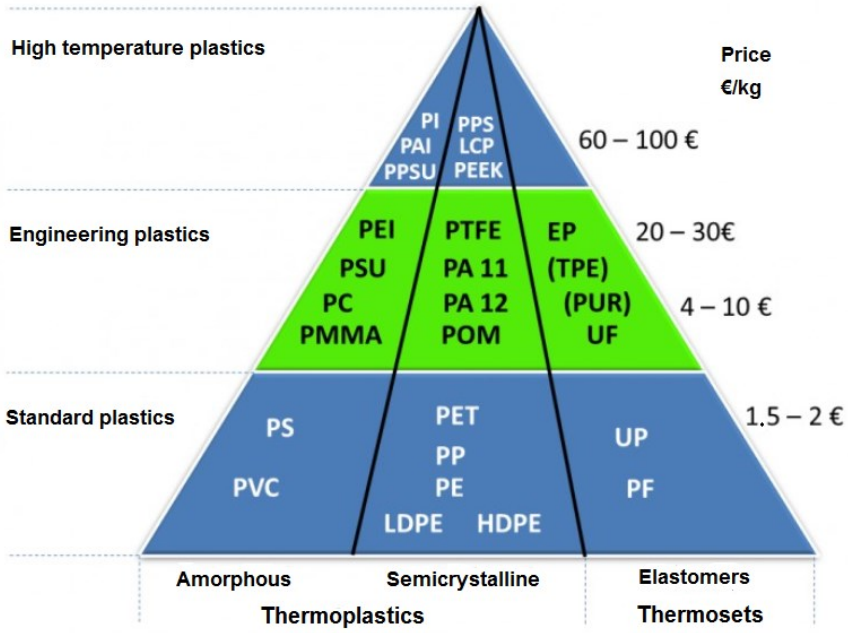


***Figure 19**: Classification of plastics, modified from [11]*

Tubes of varied materials are required in various purposes in the field of medical devices. Both thermoplastic polymers and elastomeric materials can be used in different medical tubing applications. The most common applications are various catheters and intubation tubes. The selection of material is based on the target usage, price, quality and other factors, such as:

- The usage time of the catheter (permanent/temporary),
- Individual (male, female, children): different tube sizes required,
- Patient allergies,
- Lumen size: the larger lumen the better fluid flow,
- Structural rigidity: to prevent blockage of flows and
- Surface properties: less hard and rough tube is less traumatizing for patients.

The common trend is that urinary catheters are softer material such as silicone rubber or natural rubber whereas temporary catheters and peripheral venous catheters are harder and more rigid material such as PVC. The sizing of the tubes depends on the use purpose and the patient body size. Common medical tube applications, materials and tube sizes are collected in Table 1.

***Table 1**: Medical tube applications and tube sizes*

Application	Material	Tube size
Urinary catheters	Silicone rubber Natural rubber PVC	~10—14 Fr
Peripheral venous catheters	PVC TPU Fluoroplastics	~5 Fr
Intubation tubes	PVC	~6—7.5 Ch
Rectal tubes	Silicone rubber PVC	~16—30 Fr
Central venous catheters	TPU	~5—7 Fr

Fr=1/3 mm
Ch=1 mm

Some of the most common polymeric medical tube materials, their properties, structure and additives are presented in following sections starting with general introduction to polymeric materials and their characteristics.

3.1 Plastic and elastomeric materials

Plastic and elastomeric materials consist of polymers that are large molecules formed by smaller units called monomers joined together with chemical bonds in a long chain like structure. Polymer molecules are organic compounds with carbon and hydrogen atoms forming the basis of the chain structure. When a polymer material is blended with additives it can be referred as a plastic material. There are numbers of additives for different purposes such as

- Fillers and reinforcements (fibers, particles etc. to increase strength),
- Release agents (internal/external),
- Friction reducing and lubricative additives,
- Catalysts,
- Tougheners (impact resistance increase),
- UV-stabilizers (prevent degradation by sunlight),
- Optical brighteners,
- Plasticizers (to increase flexibility and decrease viscosity),
- Pigments, dyes,
- Coupling agents (to improve filler adhesion),
- Thermal stabilizers (flame retardants etc.) and
- Antistats (to modify conductivity). [12]

The basic polymer material may have hundreds of different grades depending on the structure and size of the polymer chain or blended additives. The geometry and the molecular weight of the polymer chain also have great effects on the properties and performance of the material. [12]

3.1.1 Types of polymeric materials

The most familiar way for consumers is to divide polymer materials into natural, synthetic and biodegradable polymers. The basic division of plastic materials in the field of engineering is usually following:

- Thermoplastics,
- Thermosets and
- Elastomers.

The division originates from the structural differences of the plastic types. Thermoplastic material is a polymer material that is re-processable and re-meltable without the chemical chains being destroyed in the process. Thermosetting materials are the opposite: they cannot be re-processed thermally since the cross-linked chemical bonds between the molecules will be destroyed if done so. [13] Elastomeric materials also consist of crosslinked chemical bonds able to withstand heavy deformation which are however destroyed if the material is re-melted similarly to thermosets. [14]

Thermoplastic materials may have a certain degree of order in their structure and form crystalline regions within the disordered structure. The regions with disordered structure where the chains are obstructed from sliding against each other are called amorphous regions. Thermosetting polymers and elastomers are always fully amorphous and crystalline polymers always have certain number of amorphous regions in the structure. [14] Therefore, polymers with crystalline structure are referred as semicrystalline materials. In short, plastic and elastomeric materials are either

- Semicrystalline or
- Amorphous.

Amorphous polymers are optically more transparent than crystalline polymers since the unordered structure enables better passage for light rays. Amorphous polymers can be as clear as glass but their ability to resist chemicals is poorer compared to crystalline polymers. Crystalline polymers withstand wear and chemicals well but they have larger shrinkage compared to the amorphous polymers. [13]

Polymer materials may also be classified by their chain structure to

- Linear,
- Branched and
- Crosslinked polymers as seen in Figure 20.

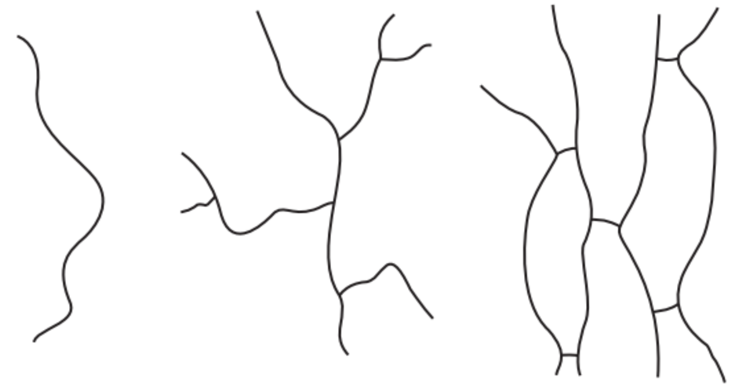


***Figure 20**: Polymer classification by structure [12, p. 22]*

Polymer chains that align themselves in long linear chains are called linear polymers. If there are side chains of the same polymer as the main chain attached to the linear backbone chain, the polymer is a called branched polymer. The secondary chains do not attach to other polymer chains by any chemical structure. The third type is a crosslinked polymer where the chains and side chains are connected to other chains on their ends. [12]

3.1.2 Isomerism

The term isomerism refers to polymer compounds that share the same molecular formula but differ from their atom arrangement. Isomerism may have an effect to the material properties of the polymer or it may affect very little. Isomerism can be divided into following categories:

- Structural isomerism,
- Geometric isomerism and
- Stereoisomerism. [12]

Structural isomerism means that the atoms in the polymer chain are arranged in completely separate way. Geometric isomers occur when there is a carbon double bond present in the molecule. The side groups attached to the double bonds can be arranged in two different ways due to nature of the bond. As seen in Figure 21, the hydrogen atoms on the left-hand side are on the opposite sides and the referred as *trans*-isomer. On the right-hand side, the hydrogens are on the same side and referred as *cis*-isomer. [12]

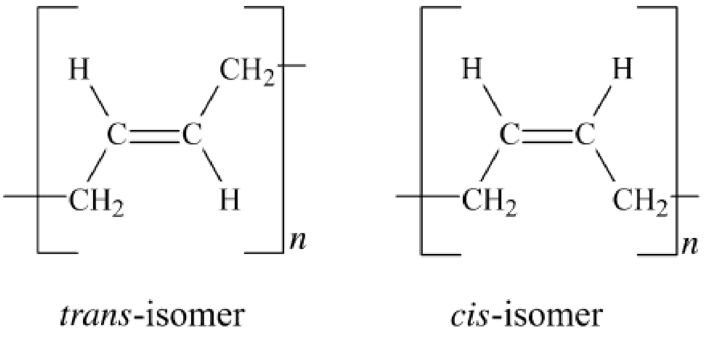


***Figure 21**: Geometrical isomerism on polybutadiene [12]*

Stereoisomers share the same molecular formula and the atoms are arranged in same way but the spatial arrangement of the atoms is different. Depending on the arrangement, polymers with stereoisomerism may be referred as atactic isotactic or syndiotactic. [12]

3.1.3 Glass transition temperature and melt temperature

When designing applications for polymeric materials the limiting factor is often the operating temperature range of the material. Polymer materials operate in a quite narrow temperature range, however there are few high temperature plastics available and more of them are developed. At low temperatures polymer materials become hard and brittle and at high temperatures their mechanical properties suffer from softening and creeping may occur. [3]

Amorphous polymers and the amorphous regions of semicrystalline polymers are glass-like and stiff when under glass transition temperature T_g. When the temperature is elevated above T_g the polymer chains in amorphous regions gain movability and the material becomes fully (amorphous) or partly (semicrystalline) flexible. Depending on the material, polymer based applications can be designed to be used in either under T_g range or above it. [3]

Melting temperature T_m indicates the temperature range where crystalline regions of semicrystalline polymers start to melt. The melting does not break the molecular bonds but the chains can move more freely and slide past each other. Melting temperature occurs only with semicrystalline materials. [3]

3.1.4 Importance of molecular weight

The molecular weight of the polymer indicates an average length of the polymer chains in the material bulk. However, not all the molecules share the same molecular weight which means that there is a distribution of different molecular weights in the material bulk. [12] It is favorable in most cases to use polymers with narrow molecular weight distribution [3]. A common guideline is that polymers with lower molecular weight have lower viscosity and flow more easily. Although lower viscosity means easier processability, also the properties and performance of the material is lower compared to higher molecular weight compound. [12]

A term that closely relates to molecular weight is degree of polymerization (DP). Degree of polymerization indicates the average number of monomers in the polymer chain. [12] For example, if ethylene molecule, with ethylene molecular weight of 28 g/mol, would consist of 100 ethylene monomers it would have a DP of 100 and the molecular weight would be 2800 g/mol. The degree of polymerization affects the nature of the polymer; high DP polyethylene is a hard plastic but as the molecular weight lowers the polymer turns wax-like like and eventually into liquid and gas. In addition, as the DP rises also the mechanical properties greatly increase. Above molecular weight of 10 000—50 000 g/mol polymers usually reach the desired mechanical properties for technical application purposes. [3]

3.2 Polyvinyl chloride

Polyvinyl chloride (PVC) is one of the most common plastic materials in the world and the most common plastic in medical devices [12]. PVC belongs to the category of standard plastics. PVC is a heavily compounded plastic and requires different kinds of additives for it to be processable and suitable for different applications. It has an amorphous structure and can be either rigid or soft depending on the compounding. [13; 14] The structure of PVC is illustrated in Figure 22.

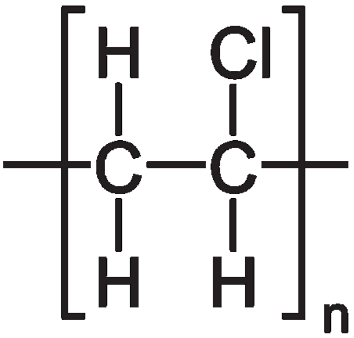


Figure 22: Structure of polyvinyl chloride [15]

Vinyl chloride monomer is mainly produced by adding chlorine to ethylene through free radical polymerization. To produce polyvinyl chloride from monomers, three different processes may be used and the basic grades can be differentiated according the polymerization process used. PVC-E is produced through emulsion polymerization and PVC-S through suspension polymerization. Bulk polymerization produces PVC-M. [14]

PVC materials can also be divided by the usage of plasticizers. PVC-U is an unplasticized grade that has high strength, rigidity and hardness but low cold impact strength, good electrical properties, high chemical resistance and self-extinguishing properties. Plasticized grade, PVC-P, has high flexibility, temperature dependent toughness, good electrical properties and low molecular weight. Both grades can be manufactured translucent to transparent. [14]

The most common PVC additives are plasticizers to make the material less rigid and more flexible. Phthalates are common plasticizers added to the base polymer, especially di(2-ethylhexyl) phthalate (DEHP). In addition, heat stabilizers are added to PVC grades to increase stability when the materials are exposed to high temperatures during processing and sterilization procedure. Calcium-zinc based additives can be used as heat stabilizers with medical PVC grades. [12]

PVC has been used in medical device manufacturing for several decades due to low cost, high performance, processability and wide range of customization possibilities for different applications. Good transparency of PVC-U allows observing the fluid flow inside the tube and high flexibility versatility in use. PVC shows good chemical resistance and is nontoxic and compatible with human body. In addition, low cost makes PVC ideal material for other disposable medical applications besides tubes, such as blood bags and surgical gloves. [12]

3.3 Thermoplastic fluoropolymers

Thermoplastic fluoropolymers are a group of polymeric materials based on fluorine compounds and belong to the category of engineering and high temperature plastics. They share similar properties, most notably

- High temperature resistance,
- Inflammability,
- High chemical resistance,
- Low coefficient of friction (COF) and
- Low wear resistance. [13]

The common problem with fluoropolymer processing is that they require very high melt temperature for the viscosity to be low enough for processing. In addition, they may cause corrosion in the extruder and the gasses that are formed during the process must be ventilated. The most common fluoropolymer is polytetrafluoroethylene (PTFE) better known with the trade name Teflon. Other well-established fluoropolymers are FEP, PVDF and PFA. [13]

3.3.1 Polytetrafluoroethylene

PTFE is the most common of fluoropolymers and approximately 90% of fluoropolymer production is PTFE. PTFE can be polymerized from tetrafluoroethylene monomer through pressure polymerization with added inhibitors or through suspension or emulsion polymerization for better heat control. PTFE is an unbranched linear polymer with very strong fluorine-carbon bonds presented in Figure 23. [14]

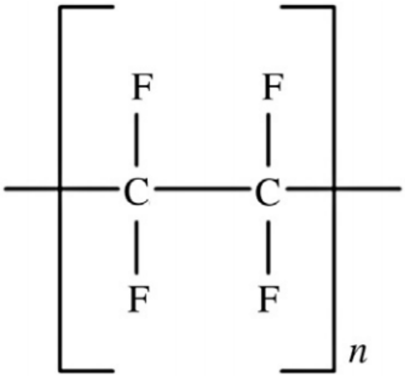


***Figure 23**: Structure of polytetrafluoroethylene [12]*

PTFE also has high degree of crystallinity, reaching up to 94%. With high crystallinity, PTFE's melting point usually lies around 320—340 °C. Due to strong covalent bonds in the polymer backbone, PTFE is also resistant to all known solvents. Although the mechanical strength and stiffness are not as good as PVC's, the thermal, electrical and chemical properties show high performance in elevated temperatures. [14] The properties of PTFE are:

- Chemical resistance,
- Insolubility (all solvents under 300 °C),
- Service temperature range of -270 to 260 °C,

- Low COF and tackiness,
- Very high electric and dielectric properties,
- Stress cracking and weathering resistance and
- Low elastic modulus. [14]

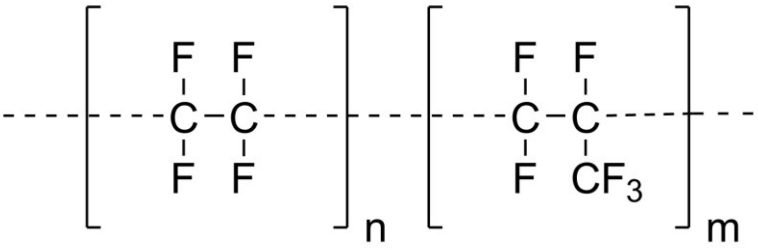
PTFE generally requires very little additives but the additive content may vary between 5 and 40%. PTFE does not require any stabilizers but especially inorganic additives such as graphite, coal, bronze and molybdenum disulfide can be used to improve properties. Graphite is usable in increasing abrasion resistance, coal and coke in increasing wear resistance, bronze in creep and wear resistance and molybdenum sulfide as a dry lubricant. Chopped glass fiber particles can also be used to increase creep strength. [14]

PTFE is processable through ram extrusion but not with conventional or microextrusion [14]. PTFE can be used in medical applications but the usability is limited due to physical properties of the material. One common application is to utilize low coefficient of friction of PTFE in medical tube or wire coatings. [12] Because PTFE is not processable with extrusion, other fluoropolymer materials were developed after the discovery of PTFE to overcome the short coming.

Expanded PTFE (ePTFE) is one of the materials derived from PTFE. The polymer matrix of PTFE is expanded with physical methods to produce large number of pores into the structure. The porous structure results in a very light weight structure and material savings without the use of chemical foaming agents or other additives. [12]

3.3.2 Fluorinated ethylene propylene

Fluorinated ethylene propylene (FEP) or tetrafluoroethylene/hexafluoropropylene copolymer is a fluoropolymer related to PTFE although its properties are little different. Most notably, FEP is processable by the means of extrusion like standard thermoplastic materials. The structure of FEP is presented in Figure 24.



$$-\left[CF_2-CF_2\right]_n-\left[CF_2-CF(CF_3)\right]_m-$$

***Figure 24**: Structure of fluorinated ethylene propylene [16]*

FEP consists of fluorinated ethylene and propylene monomers that form the backbone of the polymer chain. When two or more monomers alternate in the chain the polymer is referred as copolymer. The properties are similar to PTFE but the main differences are:

- Processable by extrusion, injection molding and thermoforming,
- High impact strength,
- Lower service temperature compared PTFE and
- Lower electrical, chemical and weathering resistance compared to PTFE. [14]

Some reinforcing additives, such as graphite and milled glass fiber, and non-ionic wetting agents can be used with FEP. [14] Unlike PTFE, FEP is suitable for medical tubing material since it is melt processable by extrusion.

3.3.3 Polyvinylidenefluoride

Polyvinylidenefluoride (PVDF) is a semicrystalline fluoropolymer that has high fluorine content. The degree of crystallinity depends on the processing parameters: high cooling rate results in reduced degree of crystallinity and transparent product whereas slow cooling rate or annealing leads to highly crystalline product with high stiffness and good creep strength. [14] The structure of PVDF is presented in Figure 25.

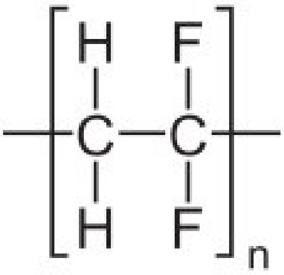


***Figure 25**: Structure of polyvinylidenefluoride [17]*

PVDF is particularly suitable for medical tubing applications because it has

- High strength, stiffness and toughness,
- Good service temperature range (-60 to 150 °C),
- Good chemical resistance,
- Good processability and
- Good UV resistance. [14]

Common additives for PVDF are organic solvents such as dimethyl phthalate and diisobutylketone. PVDF is processable with extrusion at the temperature range of 230—270 °C and suitable for medical tube material. [14]

3.3.4 Perfluoroalkoxy copolymer

Perfluoroalkoxy copolymer (PFA) is a copolymer consisting of perfluoroalkoxy side chains attached to PTFE backbone. The side chains link to the polymer backbone through oxygen bond. Due to long side chains and oxygen bond, the mobility of the molecule is better than PTFE's and melt viscosity much lower which makes processing with extrusion possible. [14] The structure of PFA is seen in Figure 26.

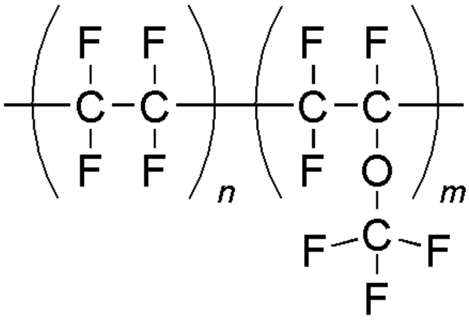


***Figure 26**: Structure of perfluoroalkoxy copolymer [18]*

The properties are similar to other fluoropolymers presented before including

- High thermal properties (melting point of 305 °C),
- High creep strength,
- Low COF,
- Flame resistance,
- High flexural strength,
- Weathering resistance and
- Good chemical resistance. [14]

Above the melting point PFA becomes easily processable due to rapid drop of viscosity. Due to presence of long perfluoroalkoxy side chains, PFA maintains its good mechanical properties even at elevated temperatures. PFA is non-toxic and also suitable for medical tubing applications. [14]

3.4 Silicone rubber

Silicone is a special case in the field of plastics since the backbone of the polymer chain is not composed of carbon atoms but inorganic silicon atoms. Silicon can bond with different elements such as oxygen, aluminum, titanium and phosphorous giving silicone based materials variety of grades. Silicones may be produced to oils, waxes, gels, thermosetting resins and elastomeric rubbers. High molecular weight polysiloxanes are used to produce elastomeric silicone rubber. Silica can be used as a filler and crosslinking of the chain network occurs with the help of peroxides or with a platinum catalyst. Figure 27 presents the typical monomers from which polysiloxanes usually consist of. [14]

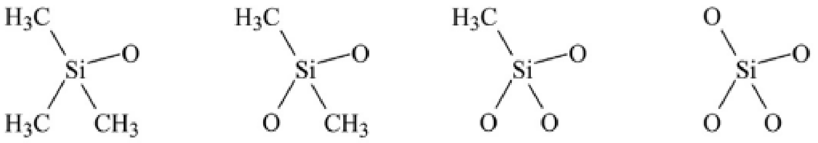


Figure 27: Structures of monomers that typically form polysiloxanes [12]

By having inorganic crosslinked structure, silicon rubber possesses following properties:

- High flexibility,
- Operation temperature -50 °C to 250 °C,
- Stability in low and high temperatures,
- High toughness at low temperatures,
- Good chemical resistance (except against solvents and ozones) and
- Poor tensile and tear strength. [12; 14; 19]

Silicone polymers are usually referred with different abbreviations depending on the organic groups attached to the siloxane backbone. Different side groups have great effect on the properties of the polymer. [19] Typical classification is:

- MQ: polydimethylsiloxane, siloxane backbone + two methyl groups attached
- VMQ: siloxane backbone + methyl groups but some of them are replaced by vinyl groups
- PVMQ: VMQ in which some of the methyl groups are replaced by phenyl groups
- FVMQ: VMQ in which some of the methyl groups are replaced by trifluoropropyl substituents. [19]

Silicone rubbers can also be classified according to the viscosity of the material. The major classes are solid silicone rubber and liquid silicone rubber. [19]

- Solid silicone rubber is a high molecular weight elastomer consisting from long polymer chains. Solid silicone rubber is usually cured with peroxide-crosslinking but addition cured grades are also available. The material is suitable for tubing applications and processable by extrusion. [19]
- Liquid silicone rubber on the contrary contains smaller molecular weight polymer chains with shorter chain length but better flow properties than solid rubber. Liquid silicone rubber is always addition-cured, usually with a platinum catalyst. The liquid material is provided in two components, the other container containing the platinum catalyst and the other containing the crosslinker. Material is suitable for injection molding. [19]

Notable advantage compared to the most common medical plastic PVC is, that silicone rubber requires very few additives, and additives such as curing accelerators, plasticizers and antioxidants are not necessary at all [19]. However, medical grade silicone rubbers usually contain fumed silica as a filler. Silica works as an enforcing filler increasing strength and hardness and reducing tackiness. [20]

Solid silicone rubber can be used in medical tubing manufacturing because its processable by extrusion unlike liquid silicone rubber. However, microextruders that are suitable for thermoplastic materials cannot usually be used as such with silicone materials since they require curing in separate heating tunnel. Extrusion is possible because solid silicone has higher mechanical integrity compared to liquid grade [20]. The curing tunnel must be well ventilated to eliminate peroxide by-products that may affect the quality of the product [20].

3.5 Thermoplastic polyurethane

Thermoplastic polyurethane (TPU) is a thermoplastic elastomer having few major advantages over cross-linked elastomers like silicone rubber: processing is easier and does not require curing tunnel, the waste material can be recycled and the process is faster overall than with thermosetting elastomers. [12]

TPU is a copolymer of polyisocyanate and polyol monomers formed with either block or graft polymerization. The structure of the molecule is segmented into thermally stable hard components and soft components that provide the elastomeric characteristics. The hard segment forms crystalline regions and the soft region is amorphous. The properties of the material depend on the segment lengths and the ratio of monomers. [12] The structure of TPU is presented schematically in Figure 28.

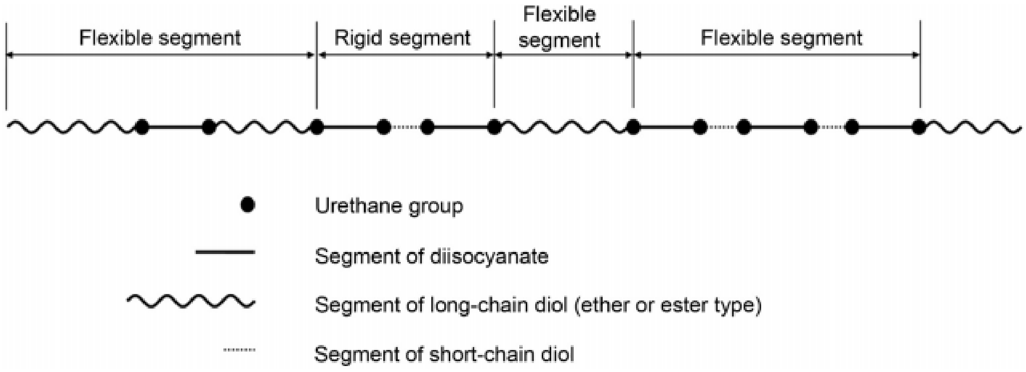


***Figure 28**: Structure of thermoplastic polyurethane [12]*

Common diisocyanates used are MDI (4,4-diisocyanate diphenylmethane), HDI (hexamethylene diisocyanate) and IPDI (isoprene diisocyanate). Short chain diols such as butan-1,4-diol and hexan-1,6-diol are used with polyethers or polyesters to create a reaction with diisocyanate to form polyurethane molecule. Diisocyanate and diol form the hard segment and the polyols form the soft segment. [12; 14] The hard segments affect the processing temperatures, tensile and tear strength and chemical resistance. The soft segments affect the elastomeric properties of the TPU such as lower service temperature limit, hardness, flexibility and compressibility. [12]

TPU is very suitable for medical tubing applications since it is non-toxic and processable like conventional thermoplastics with extrusion. The most notable properties include:

- High tensile strength and elongation at break,
- High flexibility,
- Low permanent deformation in static and dynamic loading,
- High resistance to oils, fats and many solvents,
- No plasticizers and
- Good friction and abrasion properties. [14]

Polyester based TPU shows good oil, solvent, UV, abrasion and heat resistance in addition to high mechanical properties. Polyether based TPUs have better acid and base resistance, hydrolytic stability, low temperature flexibility and fungus resistance. [12]

3.6 Natural rubber

Natural rubber (NR) is an organic compound that is refined from the sap of latex-producing plants, most often from Hevea brasiliensis tree. The plantations require certain conditions for growth and the largest producers are located in eastern Asia. The manufacturers of NR medical supplies also base their production near the raw-material suppliers since the organic material spoils over time if it is in storage too long. [21]

Solid natural rubber is recovered from latex by coagulating the wild latex, drying it and then processing the coagulate further. Different plantations of Hevea trees produce rubber material with variations in processability and cure rate creating a need for classification system for the different grades. The most notable grade is Standardized Malaysian Rubber (SMR) but other grades such as SIR (Indonesia), SLR (Sri Lanka) and CSR (China) are also available. Natural rubber's main polymer chain is built from *cis*-polyisoprene, shown in Figure 29. [21]

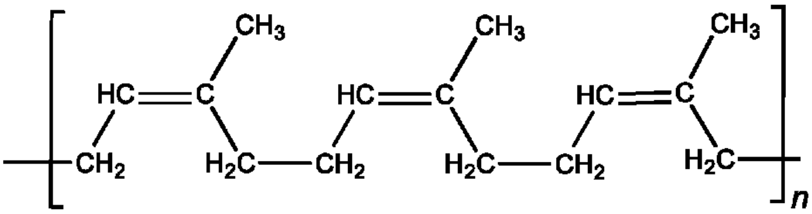


***Figure 29**: Structure of cis-polyisoprene [22]*

Prior to processing by extrusion, NR is usually masticated to lower its viscosity. After mixing and extrusion, a vulcanization process is required to form the cross-linked structure of the elastomer. Vulcanization is performed with sulfur or peroxides and proper accelerators. Metal oxides such as zinc oxide are used to maximize the potential of the accelerators. The use of activators and inhibitors is also common. During compounding, protective agents, fillers and softeners may be added to the rubber material. NR is often compounded with agents, such as aromatic amines, protecting the material from heat and ozone and with reinforcing fillers to improve abrasion and tear resistance. Common softeners for natural rubber are mineral oils but animal and vegetable oils may be used as well. [21]

Although NR is very common material for medical tube applications, it may cause allergic reaction in some people which limits its usability. The properties of vulcanized NR include:

- High tensile strength,
- Very good tear resistance,
- High elasticity,
- Good low temperature flexibility,
- Excellent dynamic properties,
- Insufficient heat, aging and ozone resistance and
- Very low heat build-up. [21]

Despite few downsides, NR is very versatile and cost-efficient material for medical tube applications. The material can also be coated with other polymeric materials, such as silicone rubber, to prevent tissue contact with the material and overcome the problem with allergic reactions.

3.7 Other materials

There are other possible polymer materials that can be used in medical tube manufacturing besides the abovementioned main materials. Polymers such as polyolefins and polyamides are common medical materials but not very often used in tubing applications as such. Some biopolymers, especially polylactic acid (PLA), are also getting more common in medical applications.

3.7.1 Polyolefins

Polyolefins such as polyethylene (PE) and polypropylene (PP) are polymerized from simple hydrocarbon molecules to create two of the most commonly used plastic materials in the world. The structure of PE and PP monomers are depicted in Figure 30.

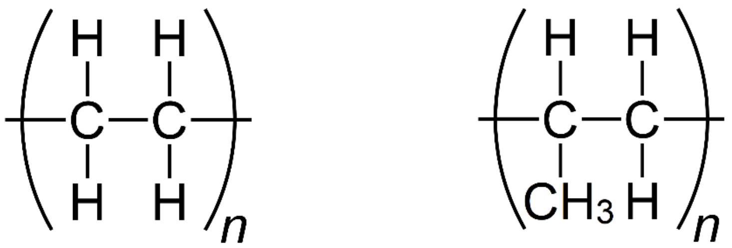


***Figure 30**: Structure of polyethylene (left) and polypropylene (right) monomers, modified from [23]*

PE can be produced in various grades that have different properties depending on the density, molecular weight and molecule structure. The grades are:

- LDPE: Low density PE; branched molecules with side chains of varying length, degree of crystallinity 40—50%, density 0.915—0.935 g/cm^3
- LLDPE: Linear low density PE; linear molecules with less and shorter branches, density 0.917—0.939 g/cm^3
- MDPE: Medium density PE; formed by mixing LDPE and HDPE, density 0.926—0.940 g/cm^3
- HDPE: High density PE; very few branches, linear molecules, degree of crystallinity 60—80%, density 0.942—0.965 g/cm^3. [12-14]

PE can be produced in homo- or copolymers, low to ultra-high molecular weight grades and with wide or narrow molecular weight distribution. PE in general has the following properties:

- Low density compared to other plastics,
- High toughness and elongation,
- Very good electrical and dielectrical properties,
- Very low water absorption,
- Low permeability to vapors,
- High chemical resistance and
- Easy processability. [14]

PE requires antioxidants and additives against UV radiation and heat since the material is prone to aging. In addition, different functional fillers and reinforcements are commonly used in PE grades. [14]

PP is most often used in its semicrystalline, isotactic grade. The greater the isotactic content is the higher the degree of crystallinity becomes, resulting in better tensile strength, stiffness and hardness. [14] PP shares similar properties with PE with some differences:

- PP has lower density,
- Higher T_g,

- Higher melting range,
- Becomes brittle at around 0° C,
- Does not suffer from stress cracking and has
- Worse oxidation resistance. [14]

The brittleness of PP can be reduced by reducing the T_g by block copolymerization with PE. PP requires same type of additives against environmental aging as PE. [14] PE and PP as such are not often used in medical tube applications as such due to the hardness of the materials, but can be utilized in different polymer blends or in copolymer structures.

3.7.2 Polyamides

Polyamides (PA) are group of different polymers that are differentiated from one another by the number of carbon atoms in the repeating unit of the polymer chain. The most commonly used polyamides are PA 6 and PA 66, both being semicrystalline materials. Polyamides are commonly used in textile applications and better known as nylon. [13]

Polyamides show good impact and chemical resistance, toughness and barrier properties against gases. They however absorb moisture very well which limits the usability and sets requirement for drying prior to processing. [13] In addition, polyamides are hard, non-toxic, wear resistant and easy to process. Some aromatic polyamides are clear and transparent, whereas semicrystalline aliphatic polyamides are opaque. [14] The hardness of the material however limits its usability as a medical tubing material.

4. MEDICAL TUBE QUALITY MANAGEMENT

Following chapter focuses on the common quality issues concerning the extrusion process of small diameter medical tubes and the quality management of the process and product. The aim is to understand what parameters affect the quality of the product and how the production line equipment affects the effectiveness of the process.

Manufacturers traditionally specify the material properties and dimensional tolerances of their medical tube products since the regulations of medical devices are strict and the diameters and wall thicknesses of medical tubes must be very precise [24]. However, an equally important quality issue is the surface roughness of the tube. The surface roughness has impact on how well lubricants are wetted onto conventional tube materials' surfaces and on reduction of friction during the insertion of the tube into the human tissue. Surface roughness also gives indications about the overall quality of the tube. Since on-line measurement of surface topography during the extrusion process is not yet in wide use, the surface properties of the contemporary products may vary greatly if processing conditions are changed.

This thesis work suggests that with LCI technique the manufacturers of medical tubes could be able to observe the changes in product quality in real time with on-line measuring equipment and acquire reliable and precise quality data by measuring the surface roughness. The data would help to optimize and monitor the extrusion process making quality control faster and resulting in reduced costs. The technique will be introduced later in Chapter 5.

4.1 Quality issues in extrusion process

There are few process related problems that may occur during the extrusion process and affect the overall quality of the produced tube. These problems include thermal degradation, melt fracture, slip and stick and wavy "sharkskin". The problems arise from nonuniform melt flow velocity and pressure fluctuations, contamination accumulation in the extrusion die head, weld line weaknesses, improper wall thicknesses, crystallization speed of semicrystalline polymers etc. Recognizing the quality issue and the reason behind the problem is the key factor in increasing process productivity.

4.1.1 Sharkskin, slip and stick and melt fracture

Instable melt flow may cause surface waviness also known as sharkskin to the surface of the product (Figure 31A). Sharkskin is a surface distortion that appears because of too high velocity and excessive shear rate of the surface layer of the tube extrudate in the die land area. To prevent sharkskin related issues, the melt flow can be made uniform by increasing the die temperature or reducing the extrusion speed. Polymers with high viscosity and narrow molecular weight such as high density polyethylene, linear low density polyethylene, fluoropolymers and thermoplastic polyurethanes tend to be more prone to sharkskin effect. [25] Slip and stick is caused by material sticking onto the barrel walls, blocking the flow and reducing throughput. Throughput increases when the stuck material is suddenly released from the barrel wall. The alteration of slipping and sticking causes irregular feeding and causes the surface to alter between wavy and smooth surface seen in (Figure 31B). [2, pp. 329-344]

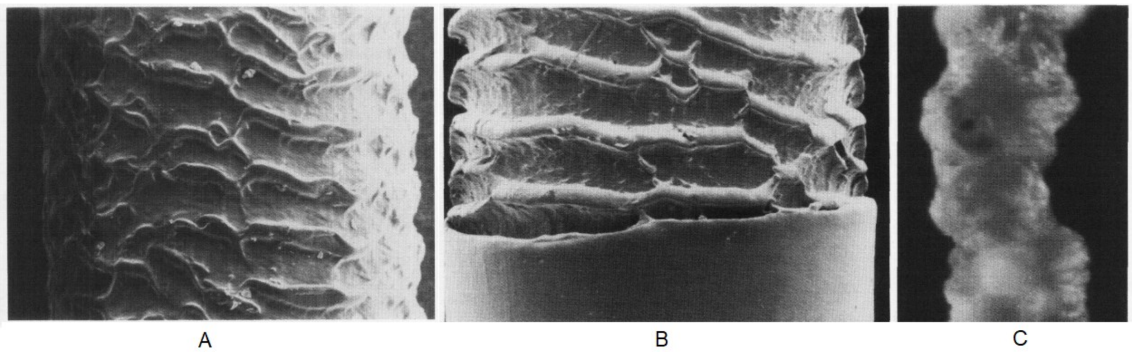


Figure 31*: A plastic tube showing: A) sharkskin B) slip and stick C) melt fracture [26]*

More severe melt flow problem than sharkskin is melt fracture seen in Figure 31C. It is caused by fluctuations of the die pressure but, unlike sharkskin, it is not a surface phenomenon but changes the structure of the whole extrudate into highly distorted form. Melt fracture occurrence varies between different polymers but it always involves exceeding the critical wall shear stress. Die design changes, extrusion rate, melt viscosity reduction and increase in die land temperature are the procedures that help to correct melt fracture if it occurs. [2, pp. 47-70]

The die geometry should be carefully designed for the right purpose, to eliminate unbalanced die flow and temperature fluctuations causing nonuniform melt flow. Too long residence time in the barrel and in the die in addition to wrong temperature profiles cause thermal degradation of the material and leads to broken polymer chains causing decreased properties. Wrong settings also affect the melt viscosity and pressure fluctuations in the die that may lead to occurrence of melt fracture. [2, , pp. 47-70]

4.1.2 Other issues

The problems with too thick or too thin wall thicknesses relate to incorrect die lip opening, wrong processing conditions, die centering or slippage in the puller. The throughput and puller speed must be adjusted according to each die lip opening in order to achieve correct wall dimensions. If the puller speed compared to the extruder is too low the walls will be too thick and vice versa. The tube may also slip in the belt puller due to moisture on the surface which affects the pulling rate. To prevent slippage the pressure of the of the caterpillar puller must be sufficient without causing damage to the product. [2, pp. 371-374]

In some cases, the tube may become too oval due to incorrect processing conditions. A common problem is that the production line is not aligned correctly; the extruder and the calibration tank are not in the same level which causes the tube to flatten when it exits the die. Excess ovality can also occur if the cooling rate is slow and the tube runs through pulleys or puller belts while it is still warm. Ovality can be corrected by proper alignment of the production line, reduction of line speed and increasing the cooling rate.

Problems with inner surface of the tube may also occur during the process. The inside surface may suffer from waviness due to too low melt temperature and high viscosity of the melt, puller slippage causing cyclic distortion due to nonuniform pull or the material can stick or slip on the die lip requiring reduction of friction between the material and the die. [2, pp. 371-374]

In addition, contaminations accumulate on the die head as the process is continued a long time. Contaminations in the die may cause scratches and marks on the surface of the tube and cause uneven flow from the die lip. Process parameters should be optimized properly to avoid excess cleaning pauses during the manufacturing since they reduce the productivity of the process. Table 2 summarizes the above-mentioned problems that are possible during the extrusion process of tubes and displays what corrective actions can be performed.

***Table 2**: Process related problems and corrective actions [2, pp. 371-374; 4]*

Problem	Causes	Actions
Walls off tolerance	Wrong die gap opening	Increase/decrease the gap
	Incorrect die centering	Center the die
	Puller speed not correct Screw speed not correct	Verify settings/increase or decrease speed
	Puller force not correct	Verify settings and ensure that the product is not moist
Wavy inside surface	Too low melt temperature	Increase the temperature in barrel zones and the die
	Puller slippage	Verify settings and ensure that the product is not moist
Uneven melt flow, surging	Die land length not correct	Modify die land length
	Uneven material feeding	Modify feeding system
	Contaminations in the die	Purge the die
	Temperatures fluctuate	Verify functionality and tighten the heater bands
Outer diameter off tolerance	Incorrect vacuum pressure Screw speed not correct	Verify settings
Diameter oval	Incorrect vacuum pressure	Verify settings
	Melt temperature too high	Reduce melt temperature
	Cooling bath too short	Add additional cooling tank
Tube sagging	Too high melt temperature	Reduce melt temperature
Bubbles	High moisture content	Remove moisture from granulates
	Too high melt or die temperature	Reduce die/melt temperature

The most important parameters in tube extrusion that have an effect to the process stability and quality of the final product are melt pressure and melt temperature since they affect how well the material flows out of the die and how uniform the consistency is. In addition, temperature profiles of the extruder barrel and die head, extruder screw speed, proper calibration pressure, cooling rate and the speed and force of the caterpillar puller should be properly adjusted to avoid any process related quality problems.

4.2 Medical tube quality management

In general, the most important quality requirements for medical tubes according to end use purposes are dimensional tolerances, adequate structural rigidity, lumen size, sterility and cleanliness of the product and packaging, biocompatibility, non-toxicity, material and product traceability and appropriate surface properties. The surface finish should be clean and low friction for patient comfortability. General guidelines for the quality management requirements for medical devices are stated in international standards and regulations.

4.2.1 Standards and guidelines

The general guidelines for quality management and production of medical device are stated in International Organization for Standardization (ISO) standard ISO 13485:2016 Medical devices — Quality management systems — Requirements for regulatory purposes. In addition, the manufacturers should follow the guidelines of Good Manufacturing Practice (GMP) guidelines [27].

ISO 13485 consists of sections that set the requirements for quality systems of medical devices primarily focusing on the device manufacturers and risk management. The standard consists of 11 sections (Appendix A):

1. Scope: defines the scope of the standard
2. Normative references: references to ISO 9000:2015
3. Terms and definitions: defines the terms used in the regulation
4. Quality management system: process and procedures to implement quality management, general requirements and documentation
5. Management responsibility: management of the whole process, customer needs, quality policy, planning, responsibility and use of reviews
6. Resource management: the amount of resources (personnel, infrastructure, contamination control) must be sufficient
7. Product realization: product planning, design and development, purchasing, traceability and identification of all production phases
8. Measurement analysis and improvement: section focuses on measurement systems, product and process monitoring, control of nonconforming products, data analysis and corrective and preventive actions
9. Annex A
10. Annex B
11. Bibliography.

Medical device manufacturers must also meet the requirements of European Union (EU) medical device directives in order to acquire the CE mark to bring their product into European markets. CE mark acts as a quality system certification and is granted to medical products that have been properly tested by the manufacturer according to standards and proven to be safe and functional. Products without the CE mark cannot enter the European markets. CE marked products must include a declaration of conformity that includes compliance to the requirements set in the directive, risk analysis, clinical study showing the effectiveness of the product, technical information and toxicity and biocompatibility studies. [28]

In the United States bringing a medical device product into domestic or international markets the product must comply the regulation set by the Federal Drug Administration (FDA). Similar to global ISO standard the FDA regulations set requirements for quality system for monitoring manufacturing, design, storage, sterility etc. The FDA inspects manufacturer's facilities and inspects data from manufacturers and end users to determine the safety and effectiveness of the product. Most countries follow modified versions of ISO 13485 and FDA regulations and manufacturers in those countries must comply the regulations. Raw material suppliers do not have to follow these regulations though they must prove the quality of the material with proper documentation and audits. [28]

4.2.2 Traceability

Medical device products must be traceable to the beginning of the manufacturing process and material batch used in case of possible failure cases or defective products. Manufacturers must have documentation about the material batches, the time and date of the manufacturing, process parameters, machine operator, quality control reports etc. to be able to define the life-cycle of the medical device from raw material to the finished product. Guidelines to the proper documentation are stated in ISO 13485.

ISO 13485 states that the records should at least include document statements of quality policy and objectives, a quality manual, documents of the procedures required by the standard and documents to ensure the effective planning, operation and control of the processes. In addition, for each medical device type or family the company should establish a file for showing that the product conforms with the requirements set by the standard. The records must be maintained for at least the life time of the medical device but not less than two years from the product release.

4.2.3 Sterility

Manufacturing of medical devices requires a clean room environment to ensure the sterility and cleanliness of the product. In clean room environment, the production process happens in a controlled environment, where contaminations that are transmitted through the air can be prevented. Medical tube manufacturers should follow the standard ISO 14644: Cleanrooms and associated controlled environments to meet the requirements set for clean room operations and prevent the product from contaminations during the manufacturing process.

Currently ISO 14644:2015 consists of following parts:

- Part 1: Classification of air cleanliness by particle concentration
- Part 2: Monitoring to provide evidence of cleanroom performance related to air cleanliness by particle concentration
- Part 3: Test methods
- Part 4: Design, construction and start-up
- Part 5: Operations
- Part 6: Vocabulary
- Part 7: Separative devices (clean air hoods, gloveboxes, isolators and mini-environments)
- Part 8: Classification of air cleanliness by chemical concentration (ACC)
- Part 9: Classification of surface cleanliness by particle concentration
- Part 10: Classification of surface cleanliness by chemical concentration

Clean room environments can be classified by ISO Class number, *N*, that is defined by the maximum permitted concentration of particle for each considered particle size. The ISO Classes of air cleanliness by particle concentration are tabled in Appendix B.

The sterilization of medical device packaging can be performed with few different methods such as using steam, ethylene oxide, gamma rays or electron beam. The methods are either physical, chemical or radiation based depending on the design, material and packaging type of the medical device. [29] There are strict international standards that cover the different sterilization methods.

Sterilization by steam is performed in an autoclave. Steam is sprayed into sealed vessel loaded with packaged medical devices where the steam displaces air killing micro-organisms. The effectiveness of the process depends on the time, heat and steam pressure. Sterilization chemically by ethylene oxide happens also in sealed vessel in vacuum conditions. The sterilization occurs with the help of humidity and injection of ethylene oxide which again kills the micro-organism present in the packaging. To remove the ethylene oxide gas, an aeration process is necessary since the gas is both harmful for health and flammable. Both methods require the packing to be porous for diffusion to work. [29]

Radiation methods include sterilization by gamma rays or by electron beam. The gamma ray method is faster than steam and ethylene oxide methods due to gamma rays being able to penetrate the packaging easily but however requires special structure to contain the harmful radiation in control. The packaged medical devices pass through the radiation source on a conveyor and receive a dosage of radiation which kills the micro-organism. The electron beam method works in similar manner but the electrons are not as penetrating as gamma rays and do not produce harmful waste. Due to lower penetrating effect, their utility is more limited compared to gamma rays. [29]

4.2.4 Dimensional tolerances

In the case of single lumen tubing, manufacturers report the nominal outer diameter and wall thicknesses of the product as well as the approved tolerances for the dimensions. If the product is not provided in continuous length, the length of the tube is also included. In addition, information about the cleanliness, sampling procedures and packaging requirements should be included. [24]

In many cases the common assumption is that if the material remains the same and the process parameters are not changed the quality of the product remains acceptable. However, there is a change for process related problems to occur, which could decrease the quality beyond acceptable levels. [24] The decrease in quality may be difficult to see during the manufacturing process and requires on-line measurement equipment.

The dimensional tolerances of medical tubes may be difficult to meet depending on the selected material since the wall thicknesses and diameters of the tubes are so small. Viscosity of certain materials, such as fluoropolymers, is so high that the temperature of the melt must be raised into a level where the material may start to suffer from thermal degradation. [24] Tolerance values with the smallest tubes however seem to be common with different manufacturers ranging approximately ± 0.10 mm for inner diameter and ± 0.05 mm for wall thicknesses. [30; 31]

To overcome problems with the dimensional tolerances the manufacturers often use high draw down ratios; the dimensions of the extrudate from the die are much larger than the dimensions of the finished tube making melt viscosity control easier. High draw down ratio improves the dimensional tolerances and line speed and makes designing the die tools significantly easier. However, the downside is that high draw down ratios cause residual stresses and line direction orientation in the product. If the stresses are released with thermal post processing the tube may shrink significantly in length and increase in wall thicknesses and diameter. [24]

Therefore, it is important that the manufacturers of medical tubes have correct equipment for small diameter thin walled tubing manufacturing. If the extruders are too oversized the residence time of the polymer material inside the barrel becomes too long and results in thermal degradation and loss of properties. [24] Microextruders that are specifically designed to run in low rpm should be used in medical tube application production. However, if production speed must be raised to maximum output the manufacturers should be aware of increase in shear rate and shear stress that may cause shark skin and melt fracture with certain materials [24].

4.2.5 Surface roughness and friction

The surface properties of medical tubes are important in many aspects, though there are no standardized quality requirements for them since the frictional properties depend greatly on the material used. The material may or may not require an external lubricant before insertion into the human tissue. The frictional properties of medical tubes can be tested with standardized methods as a part of quality control routine but on-line measurement of frictional properties is not a viable option. However, on-line measurement of tube surface roughness is much easier to perform and provides information about the changes that may occur during the manufacturing process.

Surface roughness of medical tubes should be low since reduced coefficient of friction (COF) is more comfortable for the patients when the polymer tube is inserted into human tissue [32]. However, if the roughness is too low the patient's movement or pressure caused by blood circulation may cause the tube not to stay in place correctly. In addition, if the tube requires external lubricant, such as saline or silicone oil, the surface roughness must be high enough for the lubricating particles to properly wet into the surface of the tube. It is also a common practice to coat medical tubes with another hydrophilic polymeric material that has a lower COF than the primary tube material [32].

In addition to coatings, lubricants and internal additives that reduce COF, the extrusion process can also be modified to achieve a special surface finish to the product. A matte surface finish can be achieved with lower melt temperatures and result in reduced tackiness. Higher melt temperatures result in glossy and tackier surface. [25] If the material is too tacky the inner walls may stick together under pressure causing blockage and preventing the flow of the fluids inside the tube.

It is also possible to modify the extrusion die so that the mandrel and the bushing have microstructure machined on them. The machined die head produces a microstructure on the surface of the tube that lowers the COF and tackiness of the tube and eliminates the need of external lubricants [25]. A silicone tube with extruded microstructure on the surface manufactured by Hoowaki is presented in Figure 32.

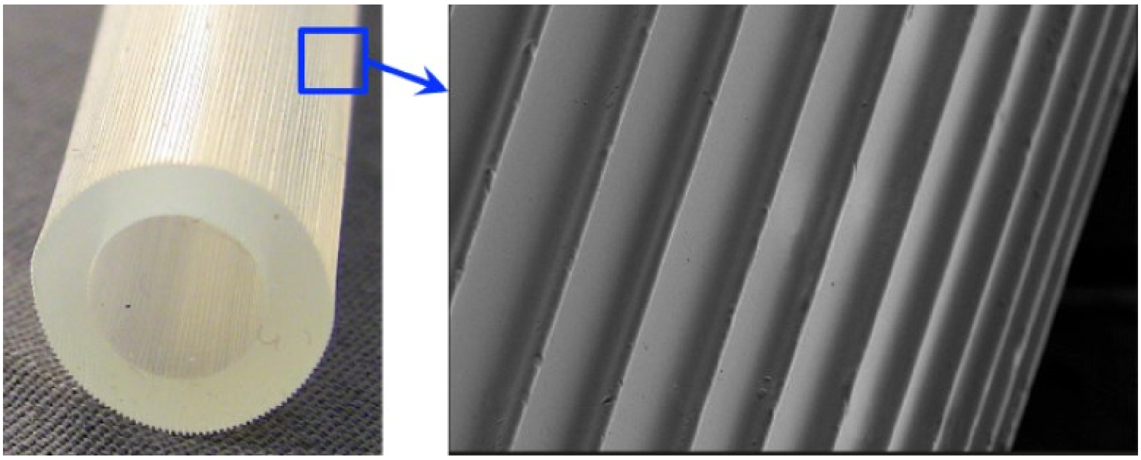


***Figure 32**: A silicone tube with microstructured surface finish [33]*

A study conducted by Hoowaki shows that it is possible to achieve 60% reduction in friction with microstuctured surface finish compared to smooth surface finish in silicone tubing. In addition, the inner surface microstructure reduces the tackiness greatly eliminating the need for partitioning- and friction reduction agents. [33]

Since the surface friction plays major role in the functionality of medical tube it would be important to be able to observe the changes in the surface quality during the manufacturing process. As mentioned, it is difficult to test the frictional properties during the process but with optical measurement techniques monitoring of surface roughness can be performed on-line. Especially in the case of microstructured surface, it would be important to monitor if the micro-ribs on the tube surface start to deviate too much from their tolerance values.

On-line surface roughness measurement is not widely used by the medical tube manufacturers and therefore no reference data was found in literature references or was available from the manufacturers. Therefore, surface roughness measurements were conducted in laboratory conditions for commercial sample tubes and two thermoplastic materials to acquire the reference data. The procedures and results are discussed in further chapters of this thesis. On-line measurement of surface roughness would be a good asset for the manufacturers since the stability of the process can be observed in real time by gathering constant quality data during the extrusion process. In addition, the speed of the entire process can be optimized if on-line measurement device can detect surface defects like shark skin before it is visible to the naked eye.

5. SURFACE ROUGHNESS MEASUREMENT OF POLYMERIC TUBES

Several techniques for surface roughness measurement exist but not many of them is capable of measuring roughness on-line with high precision and reliability. Most of the techniques are based on optical methods where light source produces a beam that reflects from the surface of the object and returns to the sensor. Other methods such as x-ray scanning, atomic force microscopy and contact profilometer can be used for surface texture measurement in post-process quality control in laboratories but not in fast paced on-line measurement on a production line. In addition, optical methods are more cost efficient and easy to integrate into industrial environments.

Two optical surface roughness measurement techniques were used in this thesis work. The focus was in Line Confocal Imaging (LCI) technique and all the on-line measurements were conducted with the technique. Optical profilometry was used in laboratory conditions to produce reference data from sample tubes which was compared with the values acquired with static measurements with LCI. The values were comparable since the techniques use the same principles to determine the surface roughness parameters from the measured roughness profile. The parameters and calculation methods are standardized in ISO 4287: Geometrical product specifications (GPS). Surface texture — Profile method. Terms, definitions and surface texture parameters.

5.1 Surface roughness parameters

Surface texture profile can be used to determine several useful parameters that give knowledge about the surface properties. The measured profile is filtered to remove irrelevant parts of the profile:

- Primary profile or P-profile is a profile where the shortest wavelength components are removed by electronic low-pass filtering since they are not relevant to roughness measurement. [34]
- Roughness profile or R-profile results from primary profile that is filtered with high electronic pass filtering. Longest wavelength components are removed from the profile with a cut-off wavelength λ_c resulting in a profile presented in Figure 33. R- profile is used to determine the roughness parameters. [34]

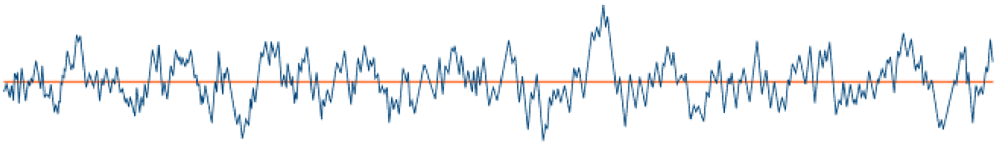


***Figure 33**: Surface roughness R-profile with a mean line [34]*

Parameters that can be derived from the R-profile data are marked with the letter *R* and subscript notifying the meaning of the parameter. The most relevant parameters for this thesis are:

- *Ra*: arithmetical mean roughness value. This parameter tells the arithmetical mean of the values of the deviations (Z_i) on the R-profile. [34]
- *Rt*: total height of the roughness profile. Tells the difference between the highest peak (*Zp*) and the deepest valley (*Zy*). [34]
- Rz_i: greatest height of the R-profile: Tells the sum of the height of the highest peak and the depth of the deepest valley in the profile. [34]
- *Rz*: mean roughness depth. This is the mean value of the five Rz_i values from five sampling lengths (lr_i) within the evaluation length (*ln*). [34]

Ra tells the arithmetical mean value of the peaks and valleys in the sampling length and is a useful parameter for evaluation of the tube surface roughness in general. However, it cuts out individual peaks that may be important in some cases. Parameter that does not cut the individual peaks is *Rz*-value whose derivation from the R-profile is presented in Figure 34. [34]

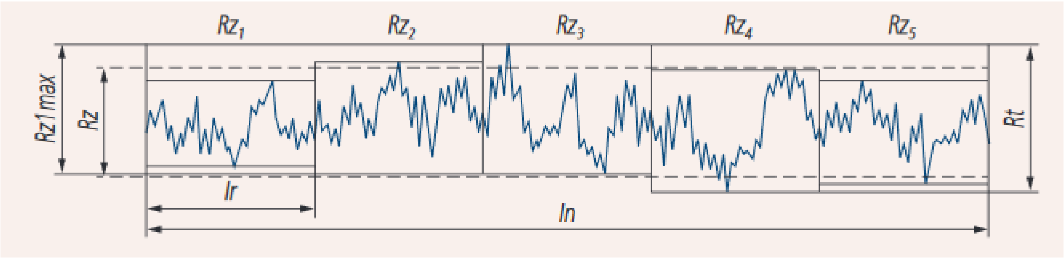


***Figure 34**: Determination of Rz-value from R-profile [34]*

Rz-value describes the minor changes in surface roughness better than *Ra*-value. In an on-line measurement however, the purpose is to be able to adjust the process by seeing how the changes in process parameters affect the surface roughness. Therefore, *Ra*-value is more suitable for process monitoring purpose since it gives better overall impression of the surface quality and was therefore used as the standard roughness parameter in this thesis when surface roughness measurements were conducted on polymeric tubes.

5.2 Line Confocal Imaging

Line Confocal Imaging (LCI) is an optical surface topography measuring technology patented by FocalSpec. The technology was developed to overcome the short comings of existing surface topography measurement techniques. It enables high-speed measurement of surface topography on-line during the manufacturing process and provides constant data about the surface quality. The technology can be applied on all surface types, even transparent and mirror surfaces. LCI technology can be applied in multiple industries to measure roughness, thickness, step height diameter etc. of the product. [35]

The operation of the technology is based on advanced optics and white light emission from the transmitter. The sensor produces a measurement profile line with 2048 individual raw measurement data points which allows measurement of moving surfaces at high resolution. White light is split into continuous spectrum of multiple wavelengths and each wavelength from the white light spectrum is focused on a plane with certain distance from the sensor. The dominating wavelength for each data point is reflected to the receiver. [35]

The key features of the LCI-1200 sensor used in this thesis are listed in Table 3.

Table 3*: Key features of LCI-1200 sensor [36]*

Feature	Value	Unit
Number of points/profile	2048	
Pixel size X	5.5	µm
Pixel size Y	25	µm
Z resolution	0.55	µm
Stand-off distance	16.16	mm
Z-range	2.8	mm
Max. measurement speed	2.5	kHz
Measurement speed at full Z-range	0.5	kHz
Max. slope of objects	20	deg

The on-line tests in this thesis were conducted with MicroProfiler MP900 designed to be used on tube extrusion and cable coating production lines for on-line roughness measurement. Technology can be easily integrated into existing manufacturing environments and remains relatively maintenance free. It works equally on plastic and metal surfaces up to line speed of 150 m/min [37]. The device is depicted in Figure 35.

***Figure 35**: FocalSpec MP900 roughness measurement system [37]*

The sensor is attached to a rigid aluminum frame which stands on four adjustable feet. The tube is guided past the sensor on pulleys which prevent vibration of the tube and measures length with a pulse encoder. The position of the tube can be adjusted in y- and z-direction for precise measurement. Measurement data is presented on touch screen PC's interface and stored in integrated database which can be exported to spreadsheet software for analysis. [38] The key features of MP900 are listed in Table 4.

***Table 4**: MP900 key features [39]*

Feature	**Value**	**Unit**
Min. tube diameter	1	mm
Max. Line speed	150	m/min
Measurement speed	250	Hz
Working distance to surface	16	mm
Parameters	*Ra*	
	Rz	
	Std	
	Rsm	
	Cpk	
Ra measurement range	0.5—20	µm
Ra measurement precision	Better than 0.03	µm
Tube color	Any	
Surface material	Plastic, metal	
Connection to LAN	Ethernet	

The measurement head measures a profile of 11.26 mm in length in an angle of 5 ° to the tube axis. Tilting the measurement head helps to eliminate situation where the tube is out of position and ensures that the reading comes from the top of the profile under measurement. With a step length of 11.17 mm the device can record 89 profiles (which equals 89 *Ra*-values) in one meter's distance. It can be operated in free run mode for static measurements with a constant 100 Hz measurement speed or in line-mode in which the pulse encoder controls the measurement speed frequency according to the production speed. [38]

The data is presented in the user interface on the PC display. Different tabs in the interface allow access to measurement profile, trendlines, histogram, diagnostics and settings. MP900 displays *Ra*-value, standard deviation *Std* of the *Ra*-value and *Cpk*-value. The *Cpk*-value refers to process capability index and shows how well the process can follow the expected output set by user defined specifications. Cut-off wavelength λ_c as well as the upper and lower limits for *Ra*-values can be set by the operator. [38] Figure 36 displays a screen from a typical operator view during a measurement.

Current Ra 0.617 µm

Focalspec on 04.05.2017 at 11:14

Speed [m/min]	4.9
Step [mm]	11.17
Freq [Hz]	7.3
Intensity	80.49
Pulse Width	6.2
Int. level	0.17
Loss Ratio [%]	0.0

Line
Free Run

USL [µm]: 4.5
LSL [µm]: 0.5
Cutoff [mm]: 0.8

Spool: 001 / 04.05.2017
Run Started: 10:25:18
Distance [m]: 135.67 m
Elapsed Time: 00:48:54

Ra [µm]: 0.60
Std [µm]: 0.03
Cpk Value: 1.22

***Figure 36**: MP900 user interface during a line-mode measurement*

The data is stored into a database in PC's internal SSD-drive according to the date and time of the measurement. Individual measurements, referred as spools, can be accessed for brief viewing on the touch-screen display or imported into a spreadsheet program for further analysis. [38]

5.2.1 Previous study

LCI technique has been previously applied in plastic film manufacturing process monitoring by Hautala J. [1]. The technique was used to survey the effect of process parameters to the surface roughness of the film, see possible surface defects, especially sharkskin effect, and optimize the plastic film extrusion process. [1]

Two different sheet-grade thermoplastic materials were used in sheet manufacturing. Sheets were manufactured both transparent and colored using a masterbatch. The effect of different process parameters was studied by changing the screw speed, extruder's temperature profile and rolling speed. LCI was able to measure the surface roughness accurately from the film surface and the capabilities of the technique in process monitoring were verified. [1]

The results showed that an increase in screw speed causes the surface roughness to increase if rolling speed is constant. Therefore, a decrease in screw rpm will result in decreased surface roughness. Knowledge over the proper processing windows allows controlling the surface roughness to the wanted direction. The thickness of the plastic sheet affected the accuracy of the measurements and must be taken into consideration when changing the process parameters by focusing the measurement head correctly onto the surface of the film. [1]

5.3 Optical profilometry

Optical profilometry was used to produce reference data for comparison to LCI. The profilometer used was Alicona InfiniteFocus G5 3D optical profilometer. The profilometer can produce a high resolution 3D-image from surface topography which can be analyzed in comprehensive ways with an analyzing software. The device is presented in Figure 37.

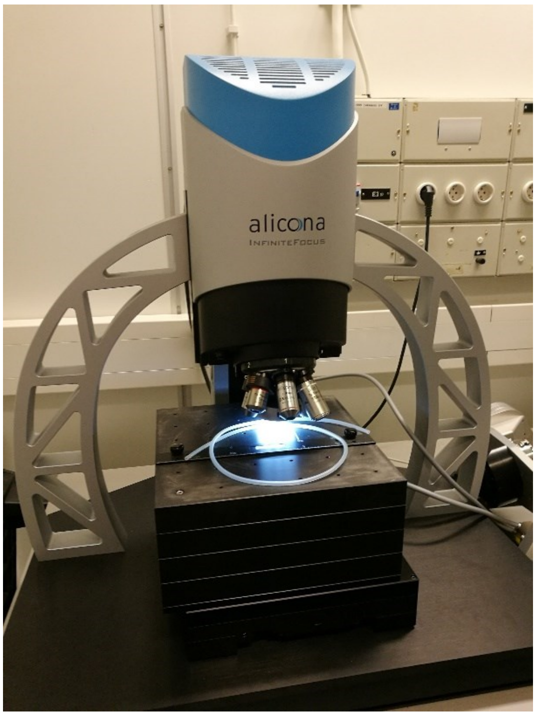


***Figure 37**: Alicone InfiniteFocus G5 optical 3D profilometer*

The device operates within the range of visible light wavelengths and different objectives from 2.5x to 100x magnifications can be used. Coaxial lighting is also possible. Few technical specifications of the device are presented in Table 5. [40]

***Table 5**: Alicona InfiniteFocus G5 key features [40]*

Objective magnification	**5x**	**10x**	**20x**	**50x**
Working distance	23.5 mm	17.5 mm	19 mm	11 mm
Min. measurable *Ra* roughness	1.2 µm	0.3 µm	0.15 µm	0.06 µm
Min. measurable radius	10 µm	5 µm	3 µm	2 µm
Vertical resolution	410 nm	100 nm	50 nm	20 nm

Optical profilometry provides excellent methods for post-production quality control of the surface topography. Suitable also for plastic materials, the sample can be imaged in

3D with different objective magnifications and processed and analyzed using the software. [40] The software provides the means to correct cylindrical shape of the sample tube and form 2D-image with correct coordination system.

6. TEST PROCEDURES AND EQUIPMENT FOR SURFACE ROUGHNESS MEASUREMENTS

The following chapter will describe the procedures for surface roughness measurements in the laboratory as well as the equipment and materials used in testing. Surface roughness was measured statically from commercial samples in order to require reference data and compare the LCI data with optical profilometry data. Afterwards, on-line measurements were conducted with two thermoplastic materials with varying process parameters.

The on-line measurements and the static profilometry tests were conducted in the Laboratory of Materials Science at Tampere University of Technology. The on-line tests were run with an equipment that would simulate actual production environment as accurately as the resources made it possible. Clean room environment was not available in the laboratory facilities.

6.1 Reference data from sample tubes

To acquire reference data and compare LCI technique with other surface roughness measurement device, samples were acquired from a company specializing in small diameter tube extrusion including medical tube manufacturing. The samples acquired were varying in material, dimensions, length and optical properties but only three elastomeric and one harder sample were selected for testing. The samples named A—D are shown in Figure 38.

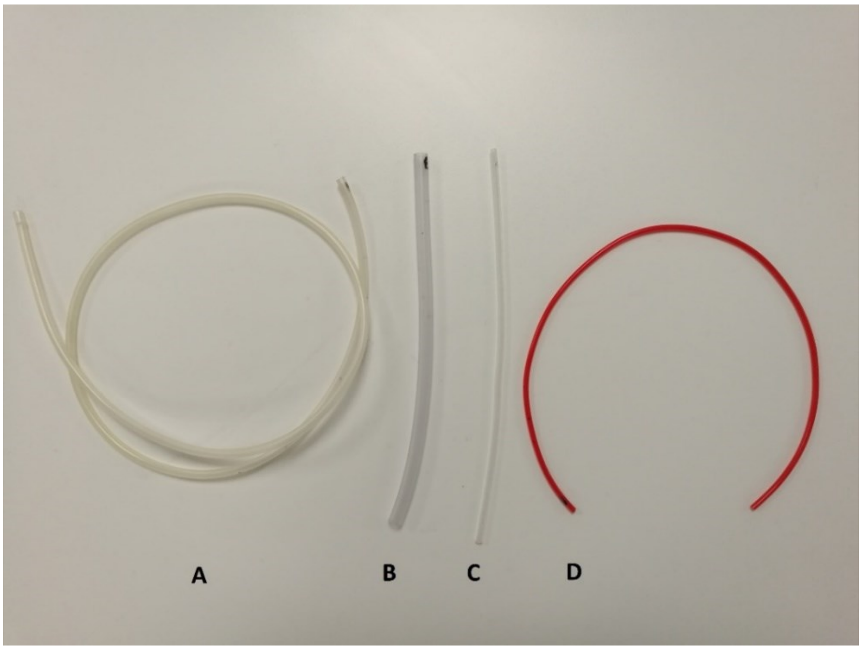


***Figure 38**: Samples for reference measurements*

Selecting only four samples was due to some of the samples being too short in length to be measured with MP900 since the position of the tube in the center of the measurement profile is very essential. In addition, some of the samples were too hard and wavy which made it impossible to acquire accurate measurement with MP900. Elastomeric samples A, B and D with adequate length were easy to measure statically with MP900 since they could be tensioned between the device pulleys easily. Sample C however was harder material and measurement was not very accurate due to sample waviness.

6.1.1 Line Confocal Imaging

The measurements with LCI technique were conducted with MP900 device introduced in section 5.2. The measurements were conducted statically while the device was set to free-run mode. In this way, the amount of data acquired in few minutes was vast and allowed measuring the roughness quickly from different points along the tube diameter.

The samples were positioned on the device pulleys and the sensor was focused properly onto the surface. By applying enough tension by hands the tube remained well in the position limits where measurement could be conducted. Five different points were selected along the tube length and in each point the tube was rotated around its x-axis to measure the whole diameter in few seconds. This resulted in five measurements around the diameter in different points along the x-axis of the tube. Figure 39 shows schematically how the measurement was conducted for each sample.

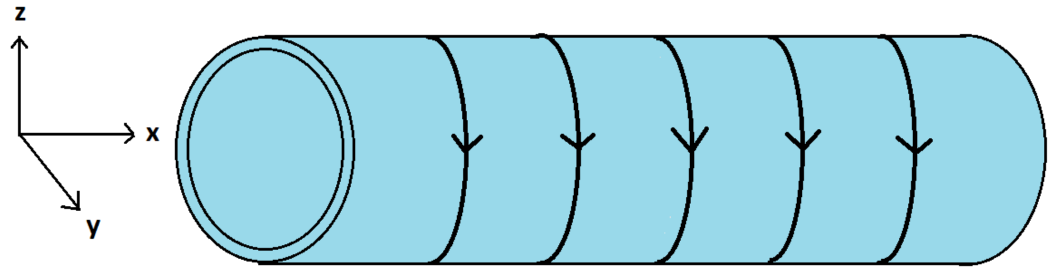


***Figure 39**: Procedure for static LCI measurements*

Measurements could be conducted well with the elastomeric samples. *Ra*- and *Rz*-values were recorded from the samples A, B and D for comparison with the optical profilometry data. The results are displayed in Chapter 7.

6.1.2 Optical profilometry

The samples were imaged with Alicona InfiniteFocus presented in section 5.3. Since 3D-imagin of the whole surface would have been excessively time consuming, the samples were imaged from five different points along the tube diameter. After the first point the tube was rotated around its x-axis and same time changing the spot place further along the x-axis. Five points were measured in appropriate intervals to cover the whole diameter. The procedure is depicted schematically in Figure 40.

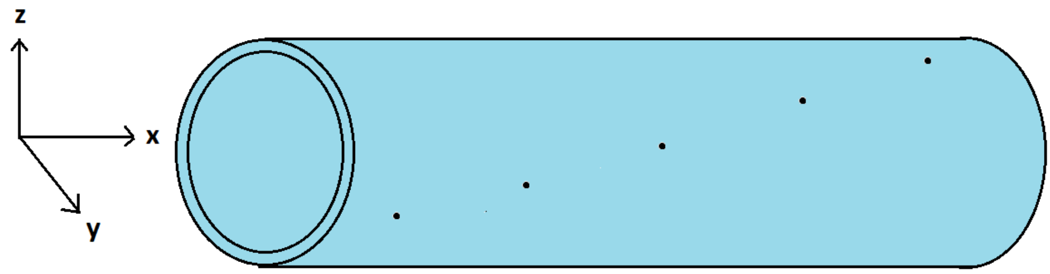


***Figure 40**: Procedure for 3D profilometry imaging*

The procedure resulted in five different 3D-images from every sample. The samples were imaged with an objective of 20x magnification from z-axis direction. Figure 41, Figure 42 and Figure 43 show the images from samples A, B and D as raw 3D-images without any overlay and with an overlay displaying the height changes in the profile.

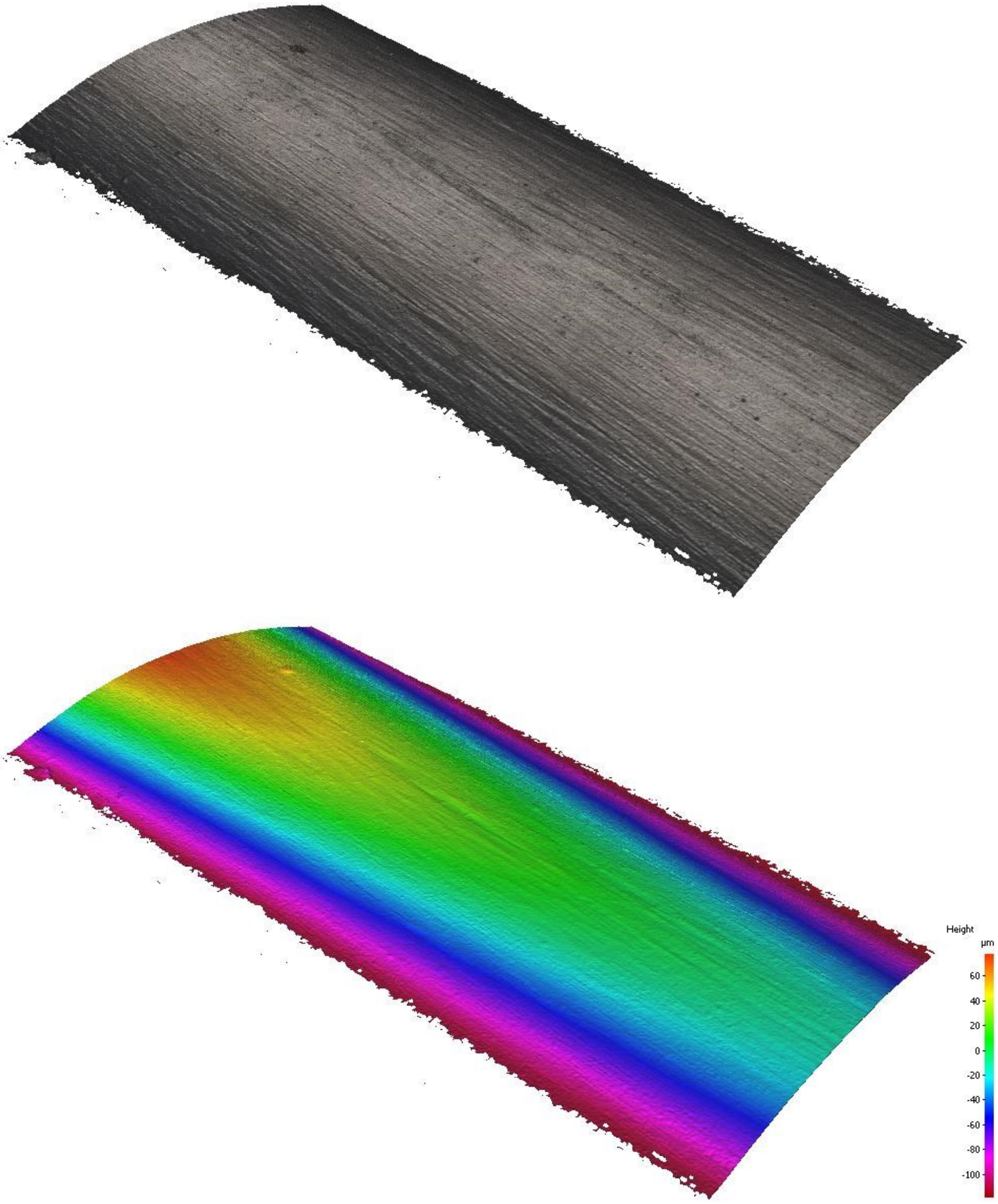


Figure 41*: Sample A surface with 20x objective without and with an overlay*

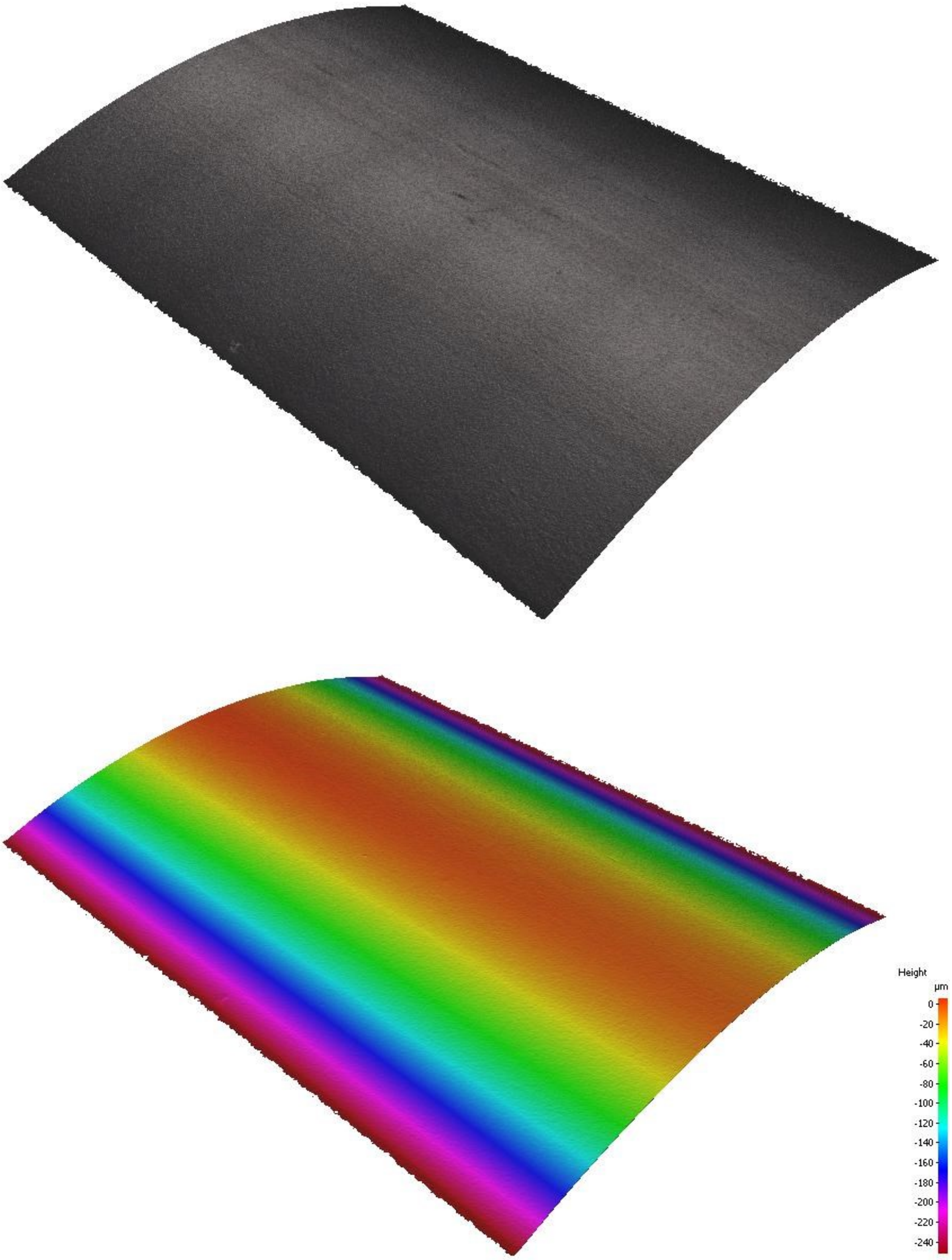


Figure 42: *Sample B surface with 20x objective without and with an overlay*

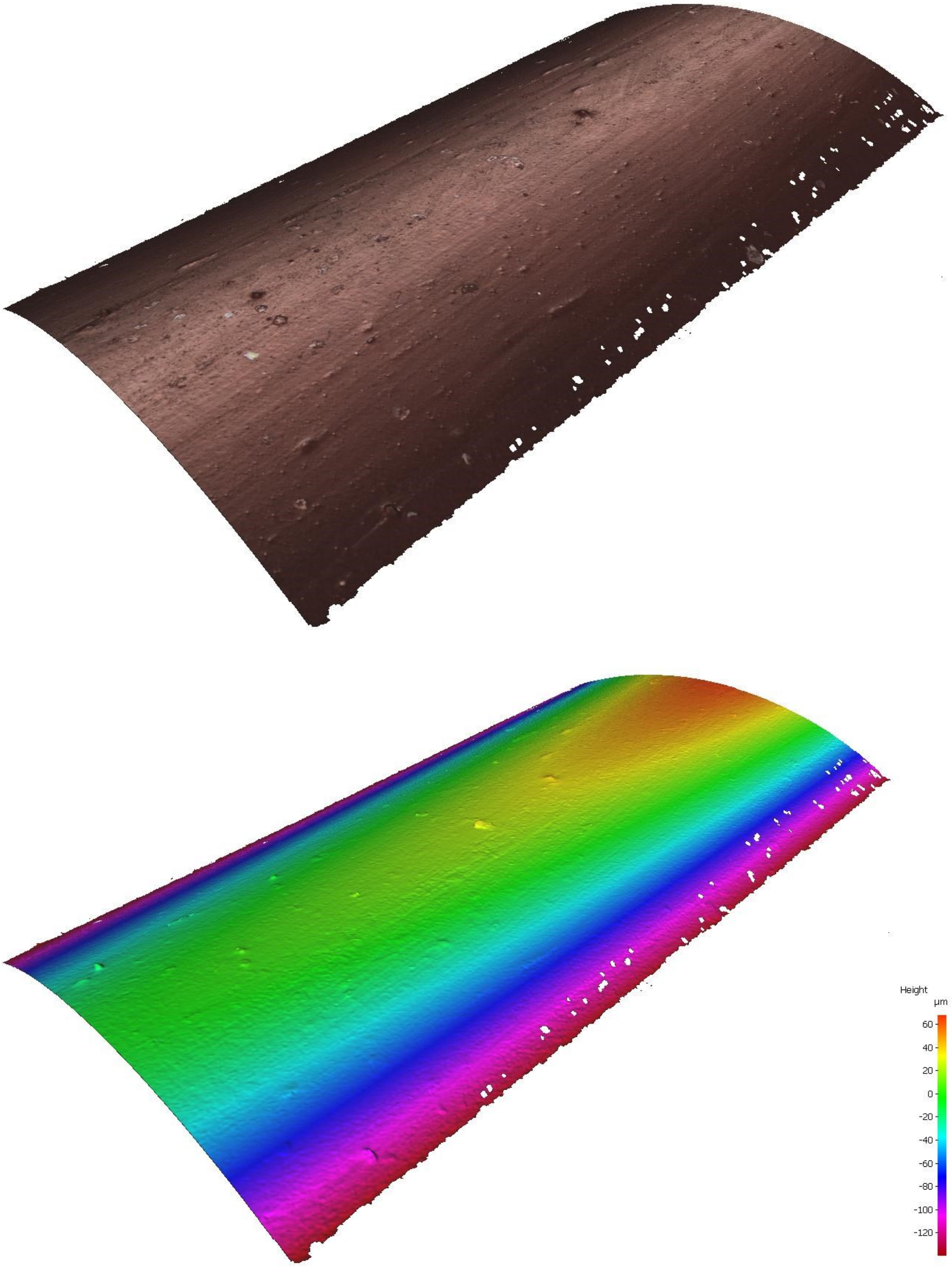


***Figure 43**: Sample D surface with 20x objective without and with an overlay*

After the images were acquired they were analyzed with Alicona IF-MeasureSuite 5.1-software. The images were reduced to 2D- images by using the built-in tool that removes the cylindrical shape of the 3D-image. After cylinder-correction was done the coordination system was set into correct position using the automatic positioning tool. Surface roughness parameters *Ra* and *Rz* were then measured from each 2D-image.

Surface roughness was measured from 2D-images using a line-tool with a width of 5 pixels. The trivial part was how to conduct the measurement since using the line tool in diverse ways one could get very different values for *Ra* and *Rz*. One method was to tilt the line roughly 5 ° and draw a straight line as with LCI sensor but the profile length in this way was much shorter compared to the measurements with MP900 and the amount of data points smaller. The solution was that the profile length was raised by using the line tool in a zigzag manner presented in Figure 44. This way the profile length was raised to match the profile length of MP900 (11.26mm), the distribution of surface deviations became narrower and the results more comparable.

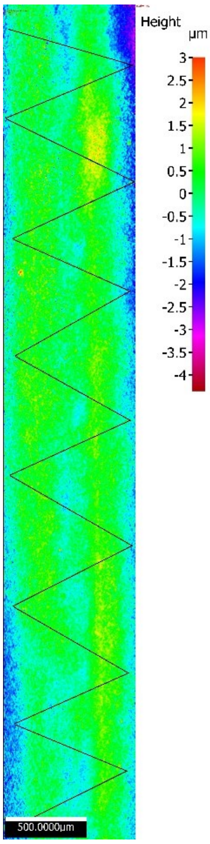


***Figure 44**: The roughness profile was measured with a line-tool in a zigzag manner to increase the profile length for comparable results with MP900*

Measuring the five images from each sample produced very comparable *Ra*- and *Rz*-values. The results are presented and analyzed in Chapter 7. The 2D-images from every measurement can be seen in Appendix C.

6.2 On-line test runs

On-line test runs were conducted with tube extrusion equipment to see how different process parameters affect the surface roughness of the extrudate and how the process can be monitored by observing the changes in surface roughness. The tests were performed with two materials, the initial reference material was linear low density polyethylene and the primary test material was thermoplastic polyurethane. The assembly of the test run line is seen in Figure 45.

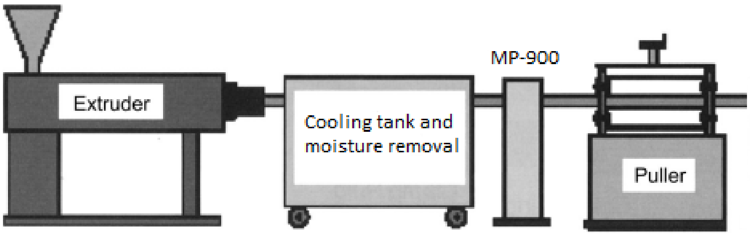


Figure 45: *Test line assembly, modified from [2, p. 573]*

Parameters that were altered during the process were:

- Temperature *T*
- Screw speed *rpm*
- Puller speed *V*.

Some test runs were conducted prior to actual measurements to find suitable processing window for both materials. The conclusion was that the test runs would be performed in two steps. In Step 1, the effect of process parameters was studied with low line speeds by altering the parameters in systematic way. In Step 2, the line speed was raised to the level where sharkskin effect and stick and slip could presumably begin to appear resulting in more severe surface deformations.

6.2.1 Extrusion equipment

The extruder used in the laboratory tests in this thesis was Brabender Plasti-corder single-screw extruder. The L/D ratio of the extruder was 25 and the material was fed through a hopper. The barrel had three heating zones in addition to temperature controlled die assembly. The maximum rpm of the extruder used during the tests was 110 rpm and the processing temperature range varied from 185 to 225 °C. The extruder is seen in Figure 46.

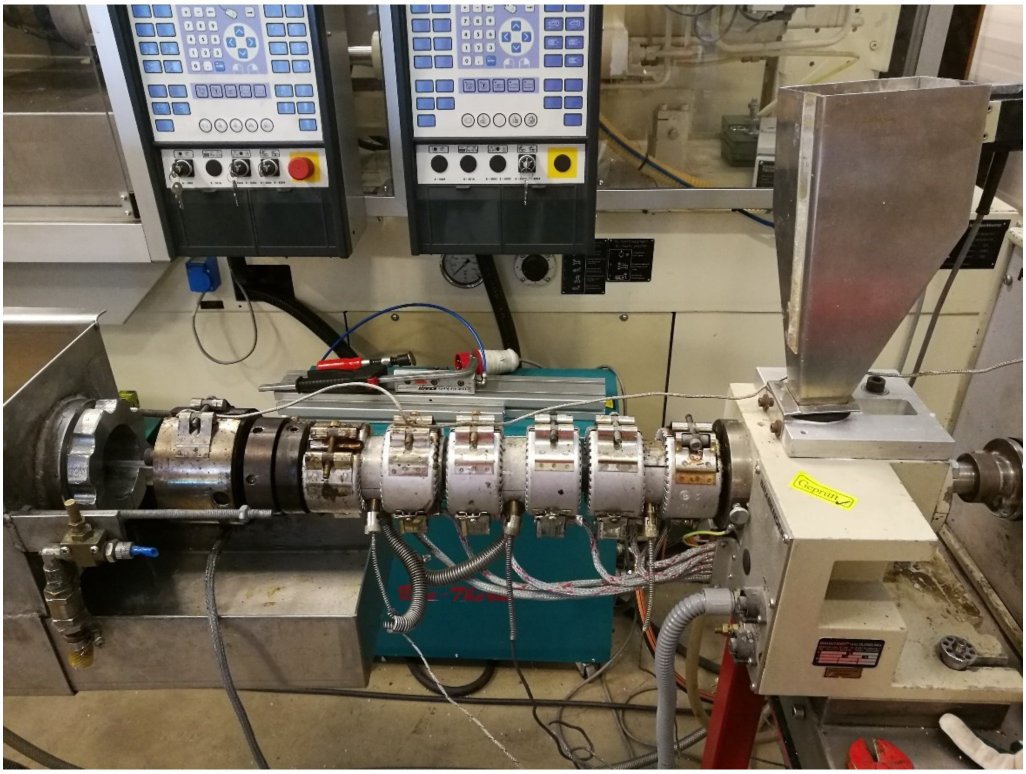


Figure 46*: Brabender Plasti-Corder extruder used in the laboratory tests*

The tubing die consisted of four tool-grade steel pieces that together form the spider-leg supported mandrel and the die gap. The die land length was 13 mm, the outer diameter of the gap 8 mm and the inner diameter 4.05 mm leaving the gap width to 1.975mm. The disassembled die is seen in Figure 47.

Figure 47*: Tubing die disassembled*

During the on-line measurements, the tube was calibrated and cooled in a cooling tank filled with standing water. The tube entered the tank through a cylinder-shaped brass calibrator. The diameter was not calibrated in vacuum because it was not possible with the equipment. However, the cooling water was kept on a level were the calibrator was fully submerged under water allowing rapid cooling after entering the tank. Rapid cooling made it possible for the tube to maintain its circular shape better.

Initial tests with lower water level showed that the tube diameter became oval if it did not cool fast enough after entering the tank, which caused position errors in measurements. In addition, more errors were caused by the leaking water from the rear end of the cooling tank the if the tube was excessively oval. Maintaining rapid cooling by keeping the calibrator submerged helped solving these problems. The calibrator and the level of cooling water are presented in Figure 48.

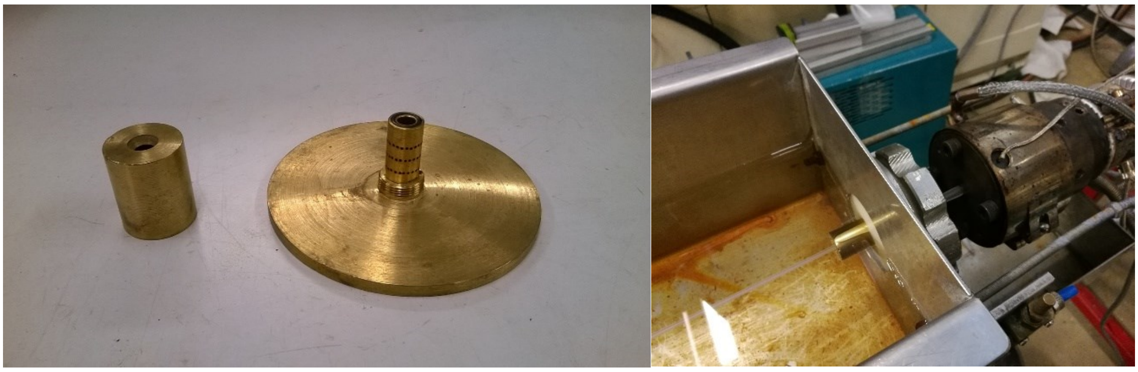


Figure 48*: The calibrator and the level of cooling water*

The MP900 roughness measurement device was situated right after the cooling tank. The silicone seal at end of the tank kept the surface free from moisture if the tube diameter remained circular. However, for keeping the surface entirely moisture free, a compressed air nozzle was installed at the end of the tank blowing the moisture droplets of the surface. The final component of the test run assembly was a Wayne Machine & Die Co. caterpillar puller. The puller is shown in Figure 49.

***Figure 49**: Caterpillar puller used in laboratory tests*

The puller speed could be adjusted from the control unit. The actual production speed measured with MP900's pulse encoder showed roughly 0.5 m/min slower speed when compared to the values of the puller's gauge and had to be taken into consideration during the measurements. The maximum speed of the puller was 10 m/min.

6.2.2 Materials

Two materials were used in the on-line measurement. The initial material was Borealis' Borstar FB4230 granulate which was used to set the equipment properly, to stabilize the process and find suitable processing window for conducting the measurements. FB4230 is designed for thin film extrusion but it was currently available in the laboratory and was found to be also suitable for thin walled tube extrusion. FB4230 is a thermoplastic linear low density polyethylene with added antioxidants.

The second material, which represented medical tube materials better, was Coim's Laripur LPR4525 thermoplastic polyurethane. It was selected since normal elastomers require curing after extrusion which was not possible in the laboratory. TPU was an appropriate alternative because it is a common medical tube material and has elastomeric properties also. The grade was polyester based designed for tubing applications. The material required drying prior to processing for 3 hours at 80—90 °C. The technical information about the two materials is presented in Table 6.

***Table 6**: Technical information of the materials used in test runs [41; 42]*

Property	LLDPE	TPU
Manufacturer	Borealis	Coim
Trade name	Borstar FB4230	Laripur LPR4525
Type	Thermoplastic	Thermoplastic elastomer
Density	923 kg/m3	1220 kg/m3
Melt Flow Rate	2.0 g/10min (190 °C/5.0 kg)	1.4 g/10min (200 °C/5.0 kg) *
Melting temperature	124 °C	Not available
Recommended processing temperature	180—210 °C	185—205 °C

*Incomplete plasticization

The data sheet of LPR4525 did not include a value for melt flow rate (MFR) so the value was measured in the laboratory according to the ISO 1133-1 standard. The test was performed at 200 °C with 5 kg weight with one-minute sample cut interval. It was observed that the material did not plasticize well without proper load, not even with 5 kg load. This lead to assumption that the extrusion of the material required adequate shear forces (high rpm) to work properly. In addition, melting temperature of the TPU was not included in the data sheet and therefore the value is not included in the table above.

6.2.3 Test run matrix and shear rate calculations

To estimate how great shear rates $\dot{\gamma}$ it would require observing the sharkskin effect on the tube surface in Step 2-measurements, theoretical values were calculated and compared to literature references. Shear forces were calculated using an Equation (1) for cylinder geometry

$$\dot{\gamma} = \frac{6Q}{\pi(R_o+R_i)(R_o-R_i)^2}\,, \tag{1}$$

where Q is the flow rate in the cylinder, R_o is the outer diameter of the cylinder and R_i is the inner diameter of the cylinder. The flow rate Q was calculated using Equation (2)

$$Q = VA\,, \tag{2}$$

where V is the puller speed in meters per seconds and A is the surface area of the extrusion die cylinder cross section.

Figure 50 presents a graph from an article by M. Denn [26] where the sharkskin effect and die slip was studied with LLDPE material using a capillary rheometer.

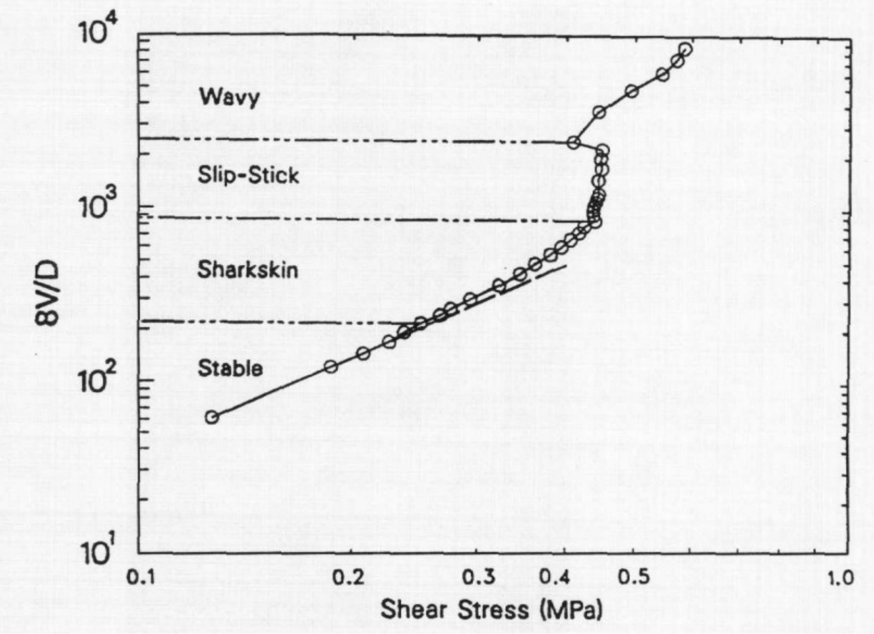


Figure 50*: Occurrence of sharkskin and slip and stick in LLDPE with increasing shear rate [26]*

According to the article, the expected shear rates for sharkskin to occur in LLDPE would be after reaching 200 1/s shear rates. Slip and stick would appear after reaching roughly 800 1/s shear rates.

With this knowledge, shear rates were calculated for different puller speeds to gain information how great line speeds it would require for sharkskin effect to occur on the tube surface (see Appendix D). As Equation (1) shows, the shear rate is directly proportional to the flow rate resulting in a liner graph presented in Figure 51.

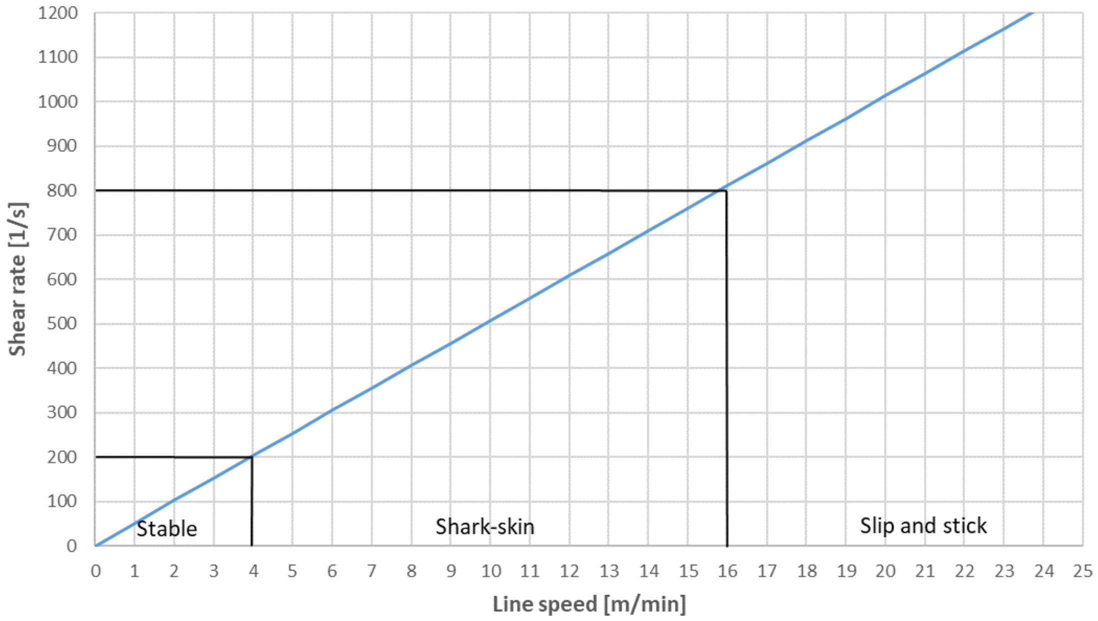


Figure 51*: Line speed vs. theoretical shear rates in the die used in laboratory tests showing the theoretical values for sharkskin and slip and stick occurrence*

According to the calculations for the die used in testing, the sharkskin effect would presumably start to occur after exceeding the line speed of 4 m/min and slip and stick when exceeding the line speed of 16 m/min. However, according to data gained from initial test runs, in reality sharkskin occurs with higher line speeds than the calculations predicts and it was not clear whether it was possible to see the sharkskin effect in LLDPE with the laboratory equipment due to pullers maximum speed being limited to 10 m/min.

Even if it was not possible to observe sharkskin effect on the tube, following test matrix (Table 7) was formed according to the presumptions and preliminary test runs to observe the effect of process parameters to the surface roughness of the tube. In these Step 1-measurements, the puller speeds of 2—4 m/min were used for LLDPE and puller speeds of 5—7 m/min for TPU. With each puller speed setting the screw rpm was raised in 5 rpm intervals after 20 m of data gathering. Each measurement’s cumulative length was 100 m. Three measurements of 100 m were performed in three different temperatures for both materials resulting in nine separate measurements with both materials.

***Table** 7: On-line test run matrix for Step 1*

Material	Temperature [°C]	Puller speed [m/min]	Length [m]
LLDPE (FB4230)	185	2	100
		3	100
		4	100
	200	2	100
		3	100
		4	100
	210	2	100
		3	100
		4	100
TPU (LPR 4525)	215	5	100
		6	100
		7	100
	220	5	100
		6	100
		7	100
	225	5	100
		6	100
		7	100

With each puller speed value rpm is raised in 5 rpm steps after 20 m of data gathering.

Interval [m]	RPM
0-20	Initial
20-40	Initial + 5
40-60	Initial + 10
60-80	Initial + 15
80-100	Initial + 20

After conducting the Step 1-measurements, the line speed was raised by increasing screw rpm and puller speed in response to rpm trying to maintain constant product dimensions. This Step 2-measurement (Table 8) was used to simulate the situation in real industrial production line, where dimensions must remain within tolerances when line speed is raised. The test was also conducted in order to see sharkskin on the tube surface which would indicate that the maximum production rate had been reached. The values for rpm and puller speed were set according to the preliminary test runs. The nominal OD of the tube was maintained at around 3.0 mm. The test run consisted of 100-meter segments with each puller speed and corresponding screw rpm. The temperature profile for LLDPE was set to accustomed processing temperature of 200 °C and 220 °C for TPU.

Table 8: *On-line test run matrix for Step 2*

Material	Temperature [°C]	RPM	Puller speed [m/min]	Length [m]
LLDPE	200	15	2	100
		23	3	100
		32	4	100
		42	5	100
		51	6	100
		60	7	100
		69	8	100
		78	9	100
		88	10	100
TPU	220	50	5	100
		61	6	100
		70	7	100
		80	8	100
		90	9	100
		100	10	100

In total, Step 2-measurements for LLDPE took over three hours to perform due to low production rates in the beginning of the measurement. Measurements with TPU were performed faster in 1.5h because the measurement was started from 5 m/min speed. The cumulative length of the measurement with LLDPE was 900 m and 600 m with TPU.

7. RESULTS AND DATA ANALYSIS OF SURFACE ROUGHNESS MEASUREMENTS

Following chapter displays the results acquired from the static measurements and on-line test runs in the laboratory. The first section 7.1 displays the results from static measurements with the MP900 compared to the results from optical profilometry measurements performed to the reference samples. The second section 7.2 displays the results acquired from the on-line measurements performed with the MP900 for two different thermoplastic materials, LLDPE and TPU.

7.1 Reference samples

Reference samples were measured with the MP900 and Alicona optical profilometer with the procedures described in Chapter 6. Due to MP900's profile length being 11.26 mm the same profile length was also used in profilometry measurements. With this method, the distribution of surface variations from the 0-plane became narrower due to larger amount of measurement points. The distribution of one measurement with optical profilometer from sample A is presented in Figure 52. The results from measurements are presented in Table 9.

***Table 9**: Results from the measurements with the reference samples*

	MP900 in free run mode		**Alicona InfiniteFocus**	
Sample A:	*Ra* [µm]	*Rz* [µm]	*Ra* [um]	*Rz* [µm]
	0.66	3.98	0.63	4.34
	0.58	3.49	0.58	4.96
	0.65	3.93	0.68	5.42
	0.62	3.76	0.58	4.55
	0.64	3.69	0.55	4.23
Average	0.63	3.77	0.60	4.70
Deviation	0.03	0.18	0.04	0.44
Sample B:	0.36	2.47	0.32	2.35
	0.28	1.91	0.24	1.86
	0.25	1.70	0.30	2.42
	0.23	1.55	0.26	2.06
	0.25	1.71	0.28	2.00
Average	0.27	1.87	0.28	2.14
Deviation	0.05	0.32	0.03	0.21
Sample D:	0.37	2.40	0.29	2.91
	0.35	2.36	0.38	3.03
	0.30	2.09	0.35	3.28
	0.29	2.12	0.90	6.92
	0.43	2.66	0.42	5.11
Average	0.35	2.33	0.47	4.25
Deviation	0.05	0.21	0.22	1.56

The results acquired from the reference sample verify that the two measurements technique show comparable results that are well in line with each other. Since the *Ra*-value tells the arithmetical mean value of peaks and valleys in the sampling length, the *Std*-values of *Ra* are similar. The minor differences can be explained with differences in the measurement methods described in Chapter 6 section 6.1: the data in LCI measurement comes from a larger area and the amount of data points is higher. The difference in measurement methods also explain why *Rz*-values show more deviation with profilometry imaging.

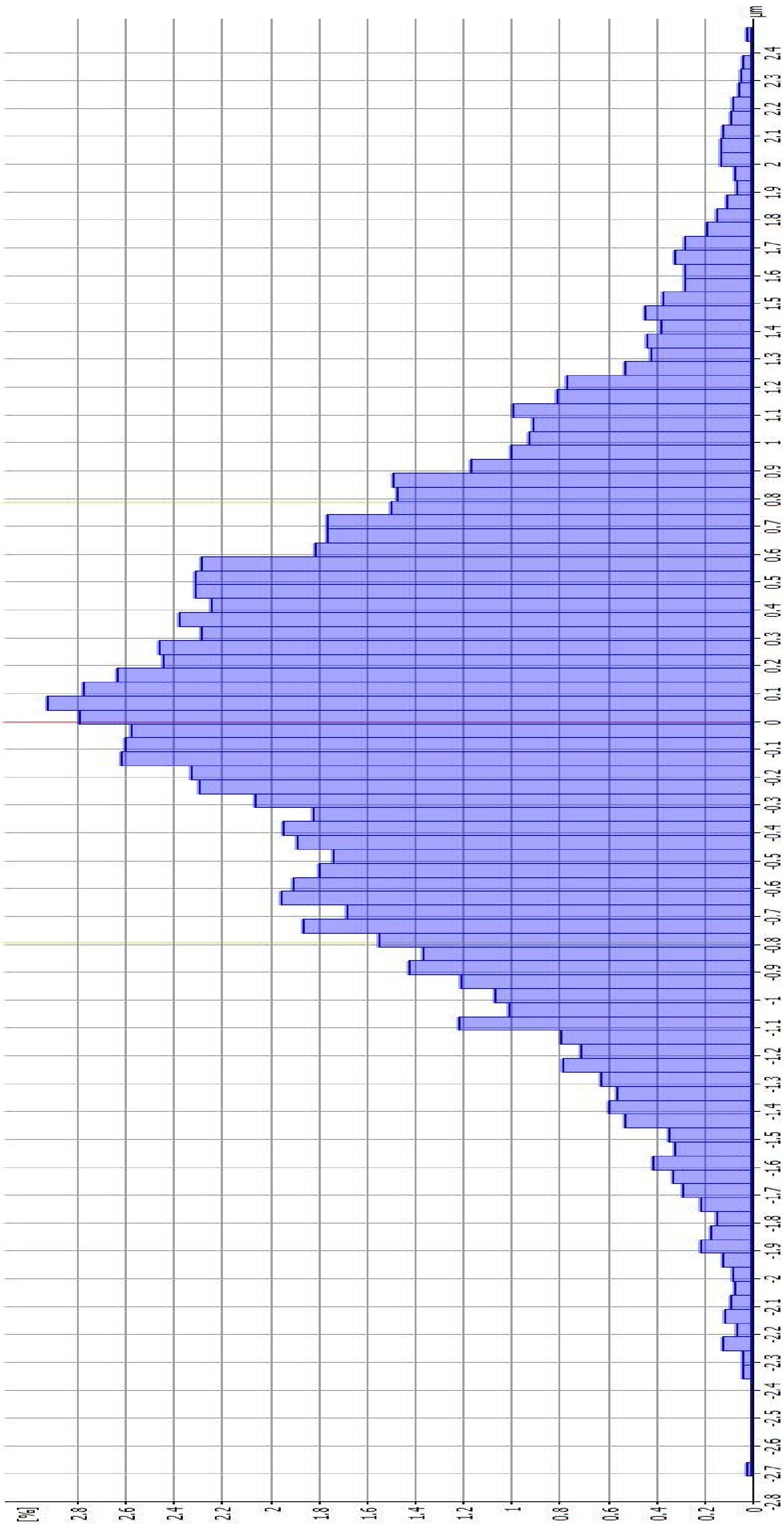


Figure 52*: Distribution of surface variations from 0-plane in one point in sample A*

The results confirm that LCI technique gives highly accurate and reliable information of the surface roughness of extruded plastic tubes. In addition, the measurements with MP900 were much faster to conduct than with optical profilometry. However, static measurement requires the samples to be long enough to form enough tension to keep the sample between the pulleys in the correct position.

7.2 On-line test runs

On-line test runs were performed according to the test run-matrix presented in Chapter 6 section 6.2. The tests were run with two materials, LLDPE and TPU, in three different temperatures with varying puller and screw speed. In addition, one longer test-run with increasing production speed and constant tube OD was performed for the both materials.

The results are presented in sections below numerically and graphically along with verbal explanations and observations. Results for LLDPE are shown first since it was used as a reference material before processing more expensive TPU.

7.2.1 LLDPE

LLDPE was used as a reference material for the on-line measurements due to its easy processability. It withstood different temperature settings well and was plasticized well even with low screw rpm-values. Table 10 summarizes the numerical values acquired from measurements in Step 1 where three different puller speeds were tested at one temperature setting while steadily increasing the screw rpm in 5 rpm steps in low production output range.

***Table 10**: Numerical results for Step 1 measurements with LLDPE*

T [°C]	185			200			210		
V [m/min]	2	3	4	2	3	4	2	3	4
Ra [µm]	0.90	0.66	0.62	0.99	0.84	0.73	0.93	0.73	0.66
Ra Std [µm]	0.04	0.03	0.05	0.05	0.03	0.03	0.05	0.03	0.03
Rz [µm]	5.33	3.86	3.57	5.80	4.88	4.32	5.50	4.25	3.90
Rz Std [µm]	0.55	0.49	0.81	0.77	0.52	0.49	0.70	0.69	1.00

The results display the changes in Ra- and Rz-values as well as the standard deviation of the two parameters. It is evident from the results that with low production speed values the increase of puller speed leads to reduced surface roughness, in both Ra- and Rz-values. The temperature also affects the values, interestingly the highest values for roughness parameters were acquired at 200 °C which was the optimal processing temperature of the material. Figure 53, Figure 54 and Figure 55 present the data from each Step 1-measurement graphically.

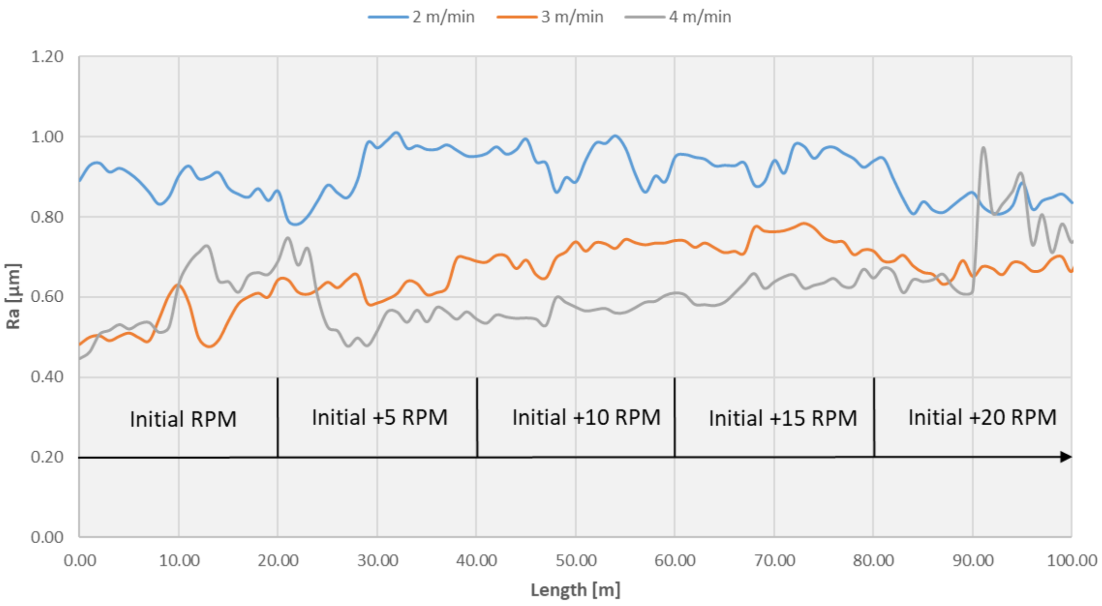


Figure 53*: LLDPE at 185 °C*

Figure 53 presents the test results from the run at 185 °C. The temperature is on 5 degrees higher level than the lowest recommended processing temperature of the material. Screw rpm in 2 m/min and 3 m/min puller speed settings was raised from 20 rpm to 40 rpm in 5 rpm steps after 20 meters of data collecting. For 4 m/min speed the rpm was raised from 25 to 45 rpm due to 20 rpm being too low causing the tube to be inconsistent in diameter.

It can be seen from the graph that *Ra*-values at 3 and 4 m/min speed settings increase with increasing screw speed. However, the value decreases at 2 m/min speed setting when reaching higher rpms. In addition, *Ra*-values are higher the lower the puller speed is. In general, it seems that after exceeding certain rpm rate the values are plateauing towards a same value between 0.7—0.8 µm.

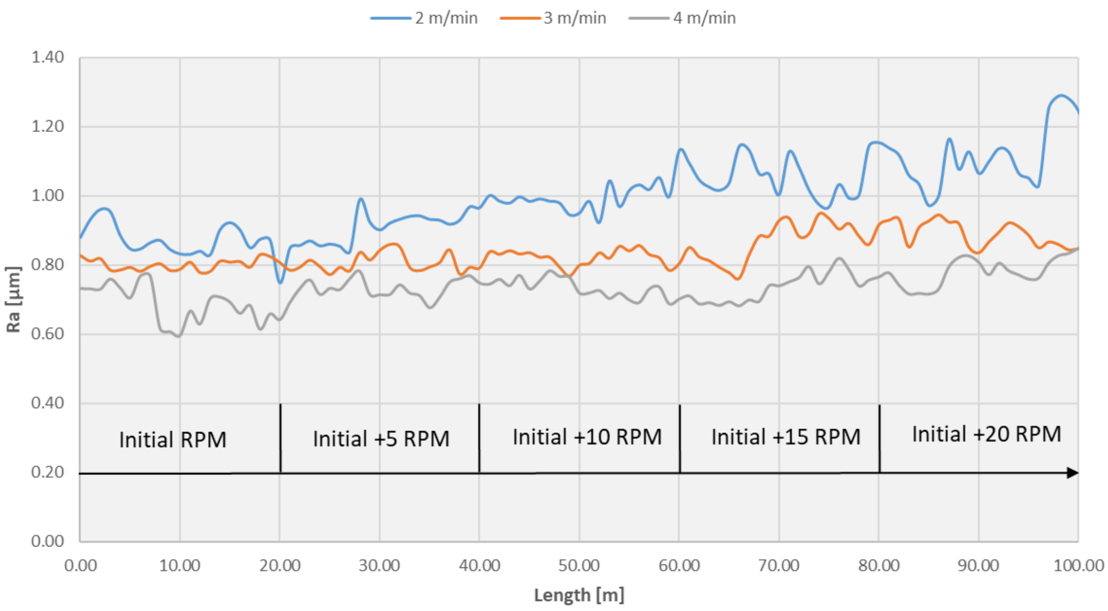


***Figure 54**: LLDPE at 200 °C*

Figure 54 presents the results from the run at 200 °C. The change in process parameters was the same as previously but the temperature was set to the level which was found to be the optimal for LLDPE. The graph shows that the *Ra*-values are now higher than at 185 °C temperature and the 3 m/min and 4 m/min graphs are similarly plateauing. 2 m/min graph shows deviation from the trend but is was likely caused by inconsistency in material feeding.

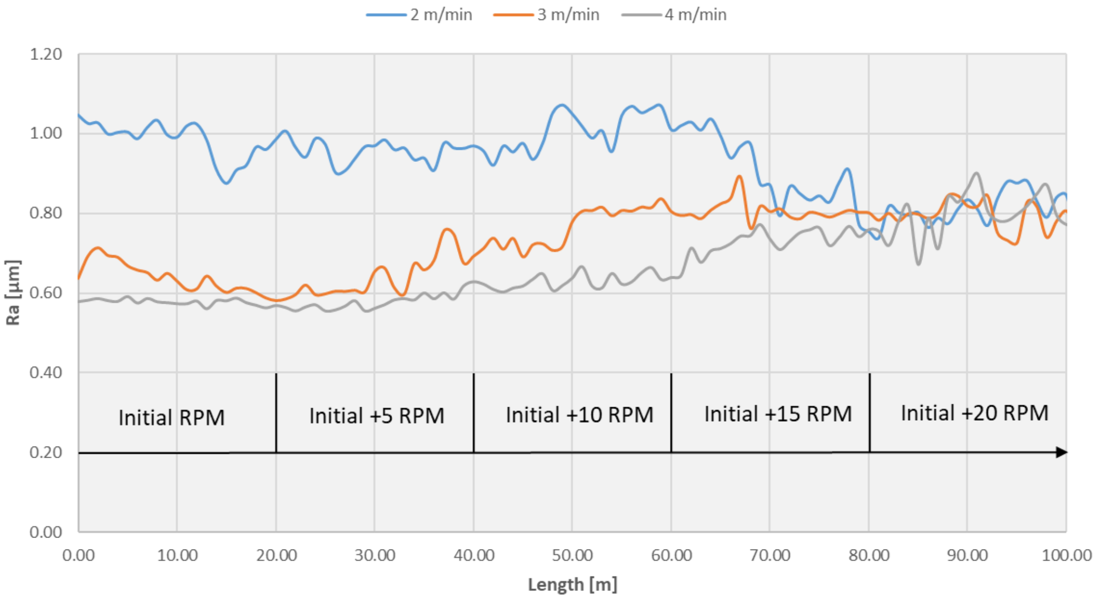


***Figure 55**: LLDPE at 210 °C*

Figure 55 presents the results from the run at 210 °C. This time the temperature was set to the highest recommended processing temperature. Now there is a quite high drop in

Ra-values at low rpm rates when the line from 2 m/min measurement is compared to the values at 3 and 4 m/min measurements.

The lines however plateau again at certain rpm rates which this time happened earlier than in previous measurements at lower temperatures. This was probably due to material being plasticized better at the higher temperature which reduces the need of shear heating. This is supported by that the process was easier to control due to lower viscosity of the material compared to the measurement at 185 °C where the initial screw speed had to be 5 rpm higher than at 200 ° and 210 °C measurements.

The effect of process parameters for the tube diameter was also observed during the measurements. A sample was cut and measured every time the screw rpm was increased. The OD-values are presented in Table 11. Measurement was performed with digital calipers which was used to find the thickest and thinnest spots around the tube diameter in one point along the x-axis of the sample and the values presented in the table are the average of those two values.

***Table 11**: The effect of process parameters to the outer diameter of the tube*

T [°C]		185 °C	200 °C	210 °C
V [m/min]	RPM	OD [mm]	OD [mm]	OD [mm]
2	20	3.2	3.5	3.4
	25	3.8	3.9	3.9
	30	4.2	4.2	4.3
	35	4.5	4.6	4.7
	40	4.8	5.1	5.0
3	20	2.8	2.9	2.7
	25	3.1	3.3	3.1
	30	3.4	3.6	3.4
	35	3.7	3.9	3.7
	40	3.9	4.1	4.0
4	25	2.7	2.8	2.7
	30	2.9	3.1	2.9
	35	3.2	3.3	3.3
	40	3.4	3.5	3.5
	45	3.6	3.8	3.7

As it should, the increase of rpm causes the tube to expand in outer diameter if the puller speed remains constant. Temperature in this case can affect the diameter since no vacuum calibration was available and standing water was used to submerge the calibrator. However, the variations in diameter are 0.1—0.3 mm, which can also be caused by the inaccuracy of the calipers used to measure the diameter. Measuring the diameter was done because it gave guidelines for proper settings for Step 2-measurement where the OD was kept at constant 3.0 mm while the production speed was steadily raised.

Step 2-measurement was conducted to observe how the surface roughness values change in long run with increasing production speed and possibly to observe sharkskin effect. The puller speed was raised from the initial 2 m/min up to the maximum speed of 10 m/min after every measured 100 m length. The shear rates near 10 m/min speed was expected to show possible signs of sharkskin effect occurrence. Each speed was compensated with proper screw rpm (Table 8) to maintain the tube OD at around 3.0 mm. The test was run at 200 °C for optimal processing conditions.

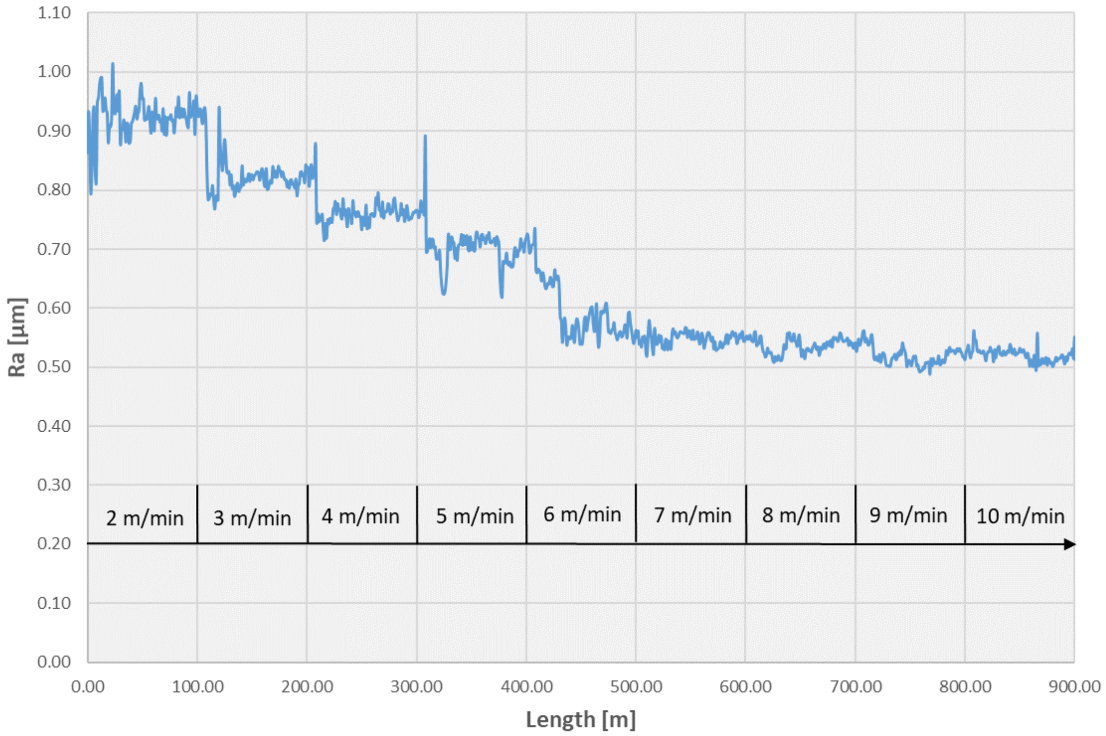


Figure 56: *LLDPE at 200 °C with increasing production speed*

Similar to previous measurements at low production rates the graph in Figure 56 seems to decline when the puller speed is raised. *Ra*-values drop gradually from ~0.9 to ~0.55 µm until the graph starts to plateau at 6 m/min puller speed, the screw rpm at this point being 51 rpm. This seems to be the threshold value for shear stress to plasticize the material uniformly which enables smoother surface finish and the smallest surface roughness value. The overall *Ra*-value for the entire measurement was 0.66 µm *Std* being 0.02 µm. *Rz*-value was 3.99 µm with *Std* of 0.83 µm.

Explanation for the observed behavior may relate to the micro-level interactions of the polymer chains. Faster shear rates allow stronger chain orientation in the line direction.

When the certain threshold value for shear stress is exceeded the material becomes uniformly plasticized. In addition, the *Ra*-value seems to increase and decrease slightly in cyclic manner after plateauing onto certain level. This could be an indication from the cyclic variation in extruder throughput that occurs with single screw extruders.

Figure 57 presents the distribution of *Ra*-values from the Step 2-measurement. The values are depicted as a histogram which displays the frequency of the measured *Ra*-values in 0.01 µm intervals.

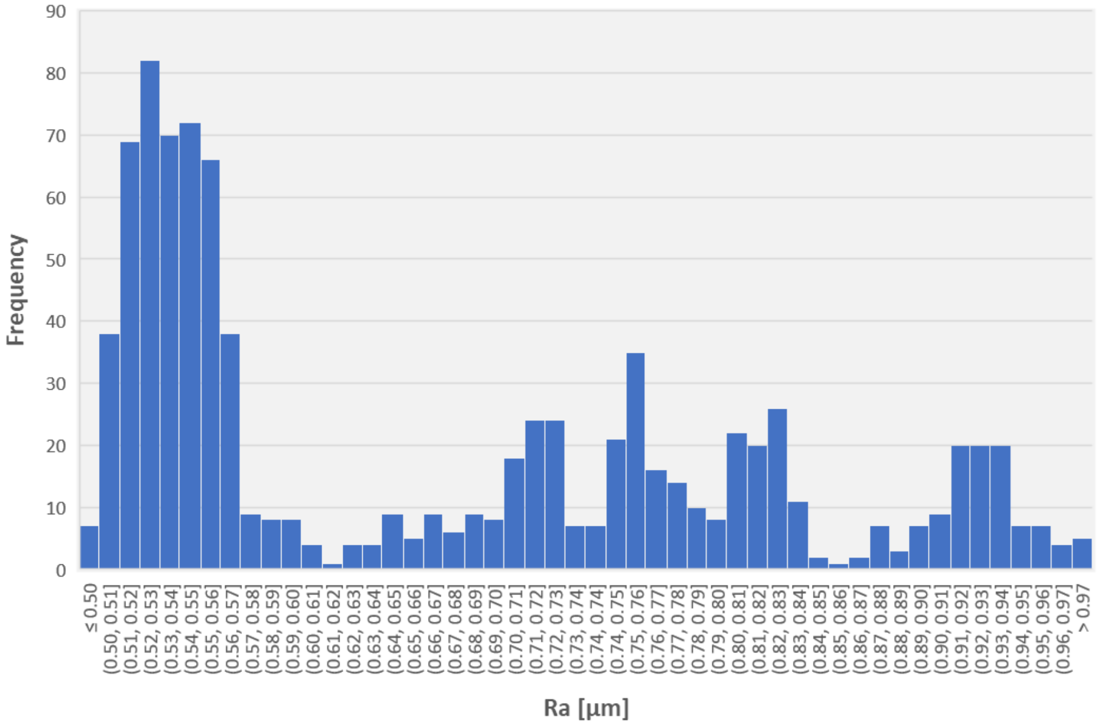


***Figure 57**: Histogram of Ra-values of LLDPE at 200 °C*

The histogram shows how the distribution of *Ra*-values change when line speed is raised. There are multiple peaks because the line speed was raised in the test run and data was collected from 100 m length with each speed setting. The peak shifts from initial 0.93 µm towards the highest peak at 0.55 µm where the *Ra*-values started to plateau in Figure 56.

The test was run up to the maximum puller speed of 10 m/min. However, sharkskin effect was not observed during the measurement even though the theoretical calculations of shear rates suggested that it may have been possible. This could be due to different equipment and possibly differences in material grades used in the reference study.

7.2.2 TPU

Results acquired from the tests with LLDPE were used as guidelines when measurements with TPU were performed. Similarly, three measurements were performed at three different temperatures with different puller speeds and increasing the rpm of the screw. The numerical results from the measurements are shown in Table 12.

***Table 12**: Numerical results for Step 1 measurements with TPU*

T [°C]	215			220			225		
V [m/min]	5	6	7	5	6	7	5	6	7
Ra [μm]	1.18	1.06	1.00	0.98	0.74	0.64	1.11	0.91	0.89
Ra Std [μm]	0.15	0.11	0.12	0.12	0.08	0.08	0.42	0.13	0.15
Rz [μm]	7.13	6.50	6.24	6.08	4.59	3.89	6.53	5.63	5.72
Rz Std [μm]	2.40	2.36	2.57	2.47	1.62	1.18	4.88	2.16	3.13

The results show that the value of both *Ra* and *Rz* become smaller when the puller speed is increased. Surface roughness seems to be more temperature dependent than in the case of LLDPE, which was expected due to elastomeric nature of the material. Due to difficulties during processing the standard deviations are relatively large compared to the measurements with LLDPE. However, at the temperature of 220 °C with puller speeds of 6 and 7 m/min the deviations are smaller than 1.0 μm. This seemed to be the optimum operation temperature with the laboratory equipment and was therefore used in Step 2-measurement. Figure 58, Figure 59 and Figure 60 show the results from measurements graphically.

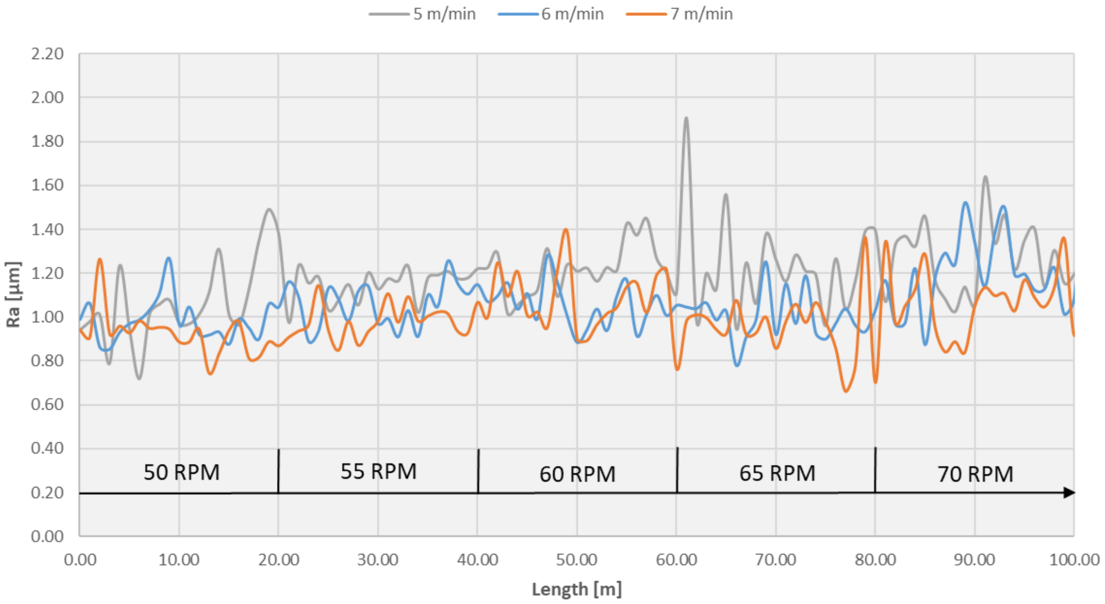


***Figure 58**:TPU at 215 °C*

The screw speed was raised in 5 rpm steps in the range of 50—70 rpm. Within this range the material was already plasticized fully and the *Ra*-values are roughly on the same level. Larger deviation makes it harder to compare the graphs compared to LLDPE-measurements.

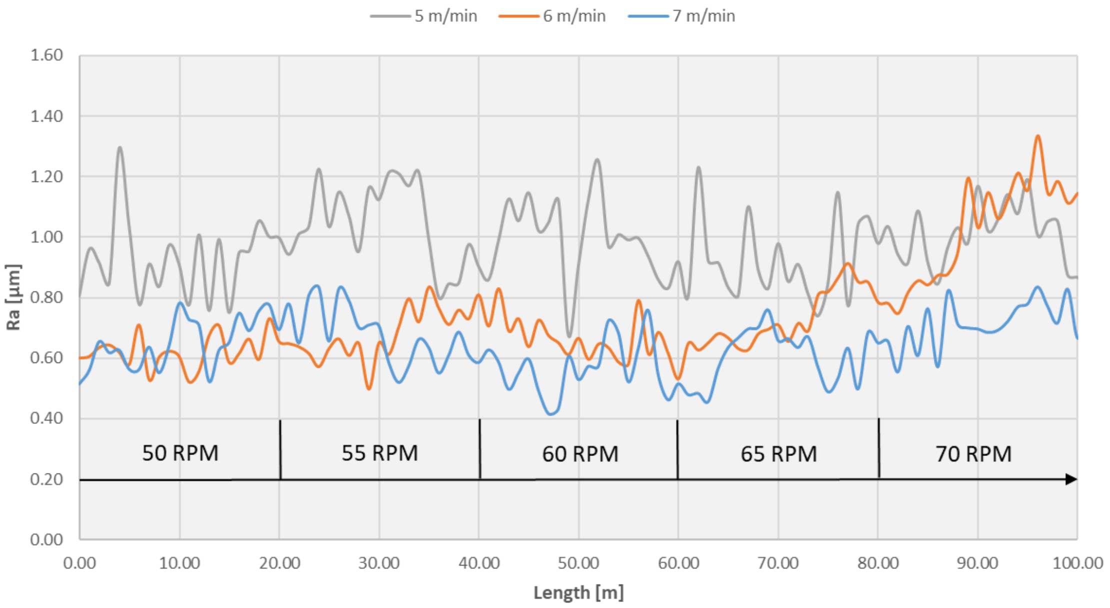


Figure 59*: TPU at 220 °C*

220 °C was observed to be the optimum temperature for processing. Measurement with the puller speed of 5 m/min produced more deviating graph in Figure 59 than faster speed settings due to lower shear forces created by the screw. The rise in the end with 70 rpm and 6 m/min speed was due to defects on the tube surface.

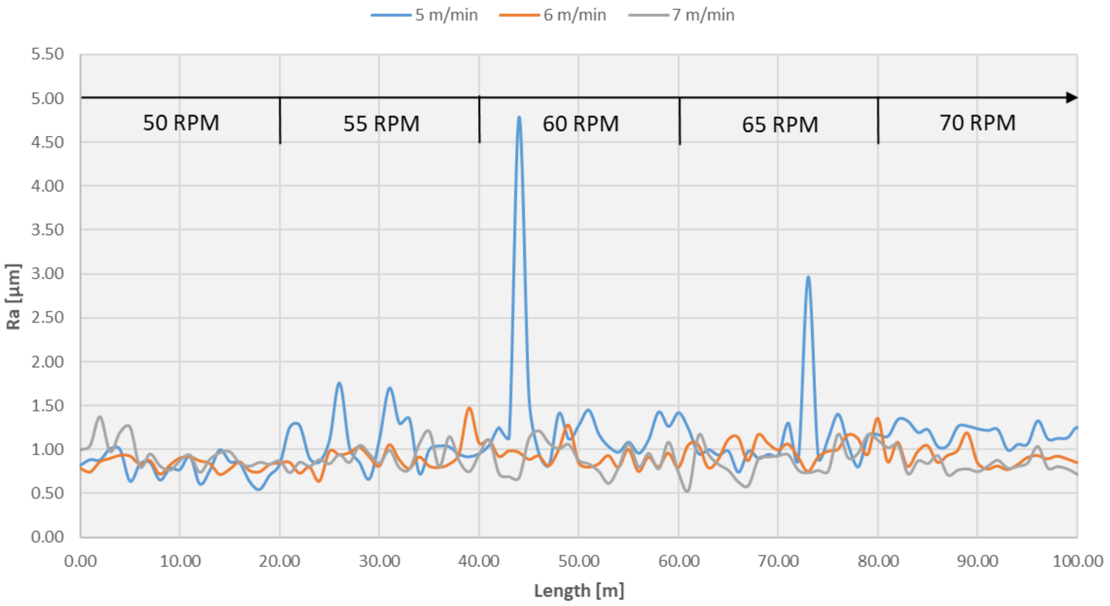


Figure 60*: TPU at 225 °C*

The graphs from the measurements at 225 °C in Figure 60 follow each other quite well with the exception of two sharp defect spikes. These are due to inconsistencies appearing due to uneven material feeding and could not be fully eliminated from the profile with the laboratory equipment. It is also possible that some moisture drops cause the sharp spikes.

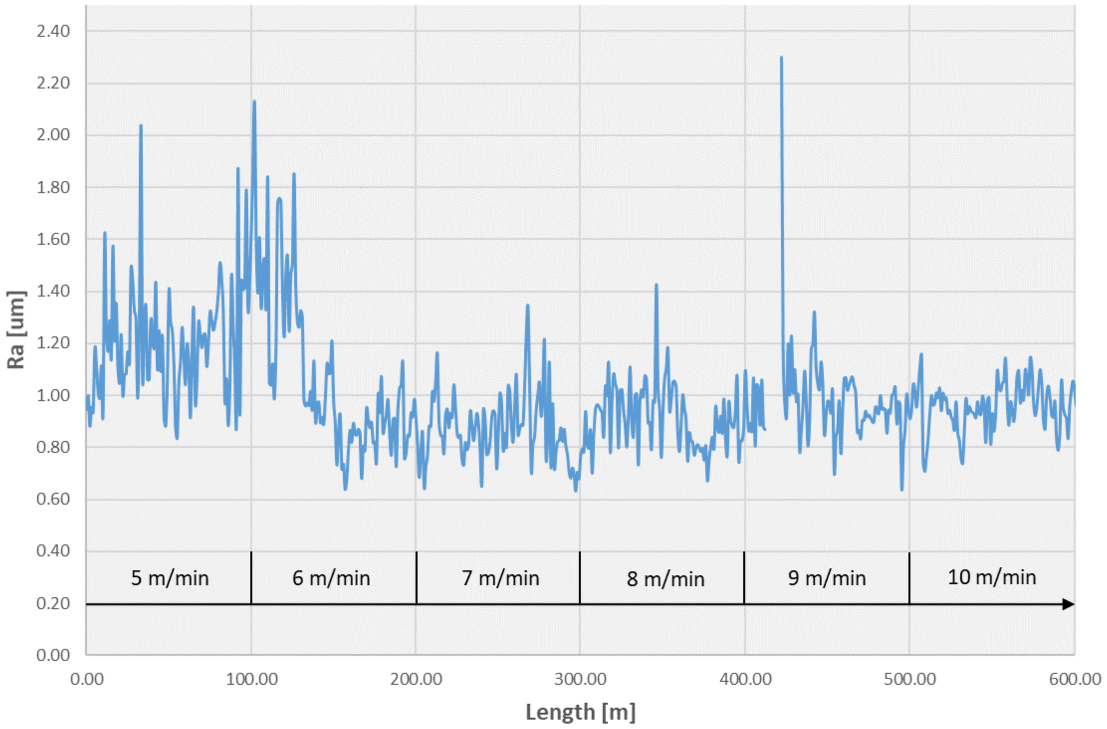


***Figure 61**: TPU at 220 °C with increasing production speed*

Figure 61 shows the data from the Step 2-measurement at 220 °C with increasing line speed. The screw speed was adjusted when the speed was raised to maintain the tube OD at around 3.0 mm. Although the deviation is greater than with LLDPE-measurement the trend seems to be the same. Lower rpm and puller speed with 5 m/min pull produce larger *Ra*-values than with faster line speeds. The values plateau at certain level similarly to LLDPE (Figure 56). The run was discontinued twice after 22 m and 83 m measured due to tube getting stuck in the calibrator which is seen as a defect spikes in the graph. The run was continued in both occasions when the process had stabilized once more. A sharp defect spike and discontinuity are also visible in this measurement at 9 m/min speed rate due to appearance of random thick spots or moisture on the tube surface. The *Ra*-value for the measurement was 1.0 µm with 0.14 µm deviation. The *Rz*-value was 5.72 µm with a deviation of 1.87 µm.

The distribution of *Ra*-values is seen as a histogram in Figure 62 similar to Step 2-measurement with LLDPE. The histogram displays the *Ra*-values in 0.03 µm intervals as the function of frequency.

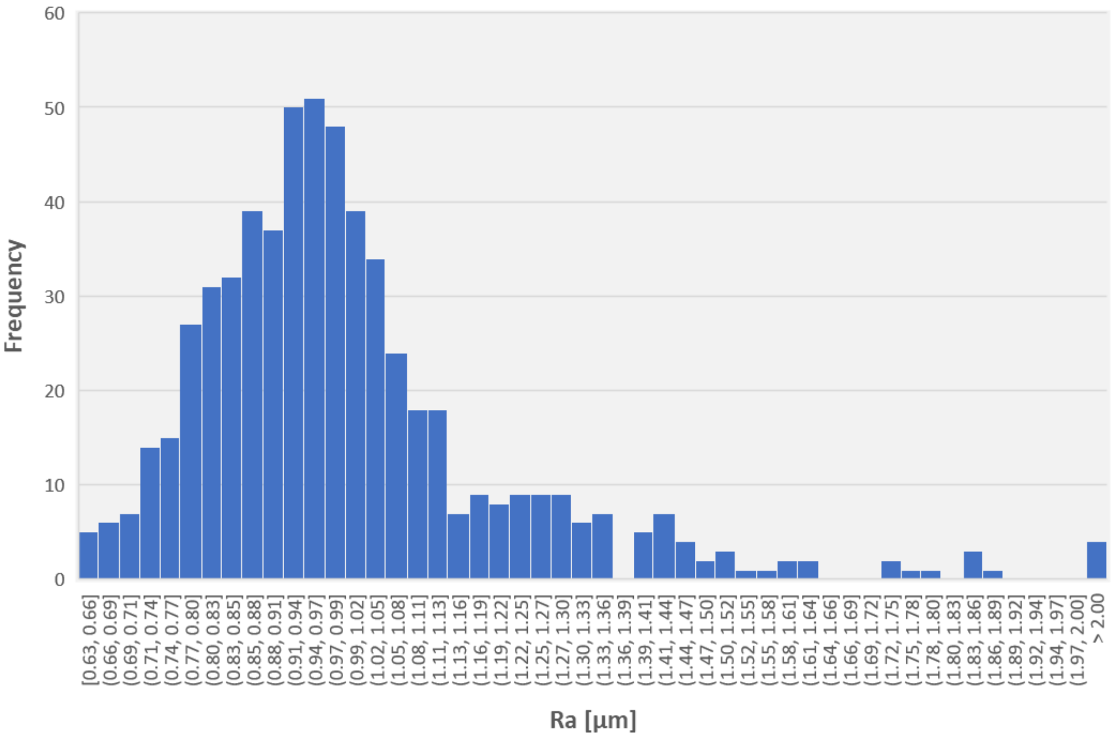


Figure 62*: Histogram of Ra-values of TPU at 220 °C*

Figure 61 from Step 2-measurement with TPU showed a decrease in *Ra*-value when the line speed was raised from 5 m/min to 6 m/min and this is seen in the histogram as a low peak at 1.25—1.27 µm. The graph plateaued on the level of ~1.0 µm after reaching 6 m/min speed and the values form a distribution that peaks at 0.97—0.99 µm. There are values that deviate much from the mean value and are caused by defects appearing during the process.

After Step 2-measurement one more additional measurement was performed in continuation to Step 2 run. The run was performed at 220 °C with puller speed of 10 m/min and screw speed of 110 rpm. The settings approach the maximum capacity of the laboratory test line. The measured length was again 100 m. The changes in *Ra*-values are presented graphically in Figure 63 and the deviation as a histogram in Figure 64.

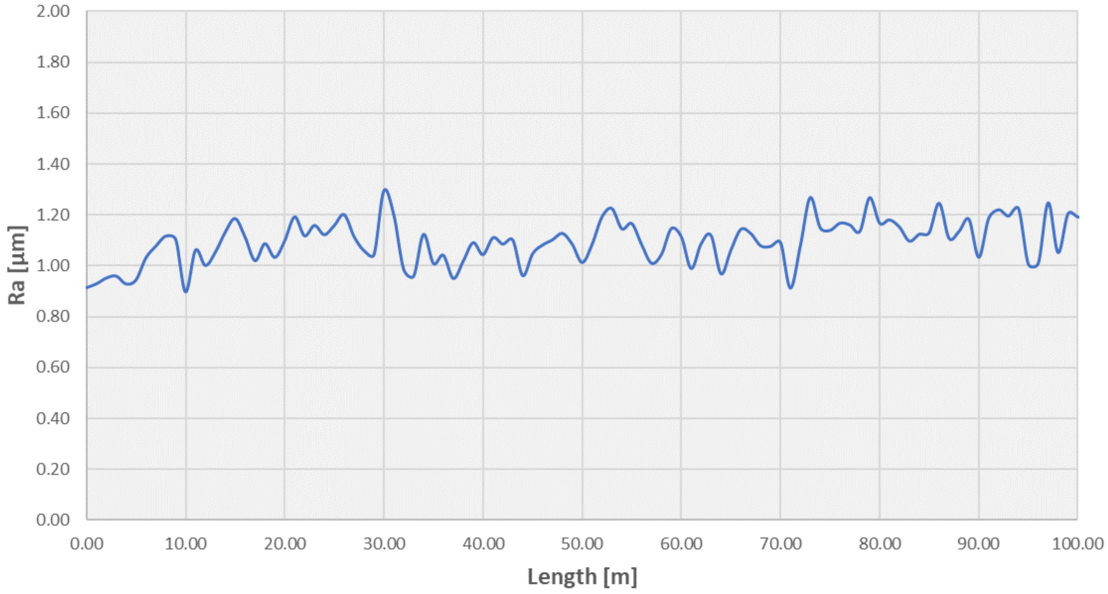


***Figure 63**: TPU at 220 °C, line speed of 10 m/min and screw speed of 110 rpm*

The average *Ra*-value of the run was 1.10 µm with a standard deviation of 0.07 µm. The average *Rz*-value was 6.63 µm with an *Std* of 1.30 µm.

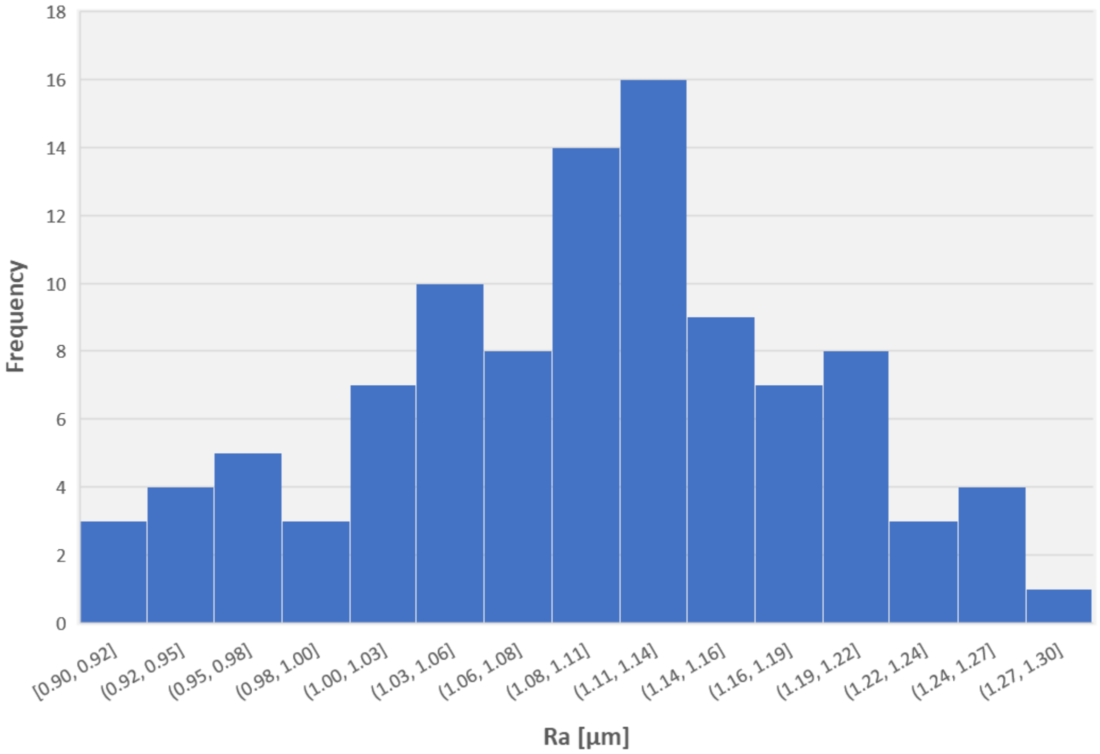


***Figure 64**: Histogram of Ra-values of TPU at 220 °C, line speed of 10 m/min and screw speed of 110 rpm*

The graph in Figure 63 shows a plateauing behavior which was expected according to the previous results. However, the *Std* value seems to get a little smaller compared to the previous runs. The run was also very stable, no sharkskin effect was observed in this test run and no significant defect spikes were recorded either. The very high rpm setting seemed to stabilize the process decreasing the interval of defects appearing on the tubing. The histogram of the *Ra*-values in Figure 64 shows evenly distributed values peaking at around 1.11 µm.

8. DISCUSSION

The results gained from the measurements with both LLDPE and TPU show how well the LCI technique can detect the changes in surface topography and indicate how the process parameters and line speed affect the surface roughness. The test runs were performed successfully as planned with valid results, although some difficulties were confronted.

The measurements were relatively easy to conduct on LLDPE since it was known to be easy to process and therefore selected to be the reference material. TPU on the other hand was much more difficult to process with the straightforward equipment available in the laboratory. The biggest problem was to maintain the tube diameter circular since too much ovality caused miss of data in the measurement. Excess ovality was easy to correct in LLDPE by controlling the cooling water level, but due to higher melt temperature and elastomeric structure, TPU was hard to maintain fully circular. The die had to be brought as close to the cooling tank as possible to cool the tube as early as possible.

Low production rates below 45 rpm screw speeds could not be performed with TPU since the material did not plasticize properly and got stuck in the die causing maintenance work. In addition, due to uneven melt flow, some defects appeared randomly during TPU processing. These thicker lumps depicted in Figure 65 could suddenly get stuck in the calibrator causing the process to discontinue, possibly due to material slipping and sticking in the extruder feed section.

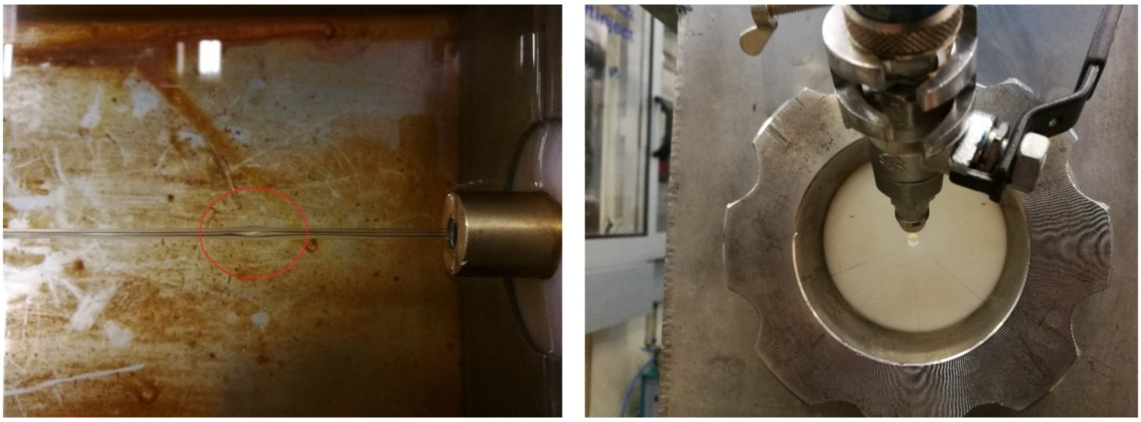


Figure 65*: On the left: a thick defect appearing during TPU processing; on the right: the nozzle used to remove moisture from the tube's surface*

In addition, TPU tubing seemed to suffer from periodical increase and decrease in tube OD. The changes were not apparent with naked eye but could be felt when sliding the tube between two fingers. The variation in OD was periodical and the interval decreased with increasing line speed. The behavior may relate to the screw geometry that causes uneven feeding to appear at certain phase of the screw turning inside the barrel. The interval becomes smaller when the screw rpm is raised since the screw is turning faster. Data in Figure 63 supports this by displaying more stable and less deviating measurement compared to the tests at lower production speeds.

The process would have benefited from using a breaker plat and/or melt gear pump to create more backpressure and a uniform melt flow. In addition, tube diameter calibration would have been easier if a vacuum calibration tank had been available. The moisture on the surface of the tube exiting the cooling tank was initially a problem with TPU but was overcome with compressed air nozzle dryer (Figure 65). If the excess moisture had not been removed from the surface the data acquired would not have been valid.

The measurements with the commercial reference samples showed that the results gained with LCI were well in line with the measurements performed with optical profilometry. In static measurements LCI displayed its advantage, speed and ease of use, well over the profilometry measurements that took hours to perform whereas the measurements with LCI took only minutes.

The results from on-line measurements showed how change in process parameters affect the *Ra*- and *Rz*-values. The measurements with LLDPE in Step 1 were more stable due to easier processability of the material showing less deviation in *Ra*- and *Rz*-values. It seems that the *Ra*-value first tends to decrease on low production speed rates when the puller speed is increased. Interestingly, the values of graphs displaying different puller speeds at certain temperature seem to plateau at the same level after reaching certain screw rpm-value. This was probably due to reaching a point where material became plasticized uniformly due to larger shear heating in the extruder barrel. This was confirmed in Step 2 were both rpm and puller speed were raised simultaneously to maintain the tube OD at 3.0 mm. Figure 56 showed a graph that was decreasing gradually until plateauing after reaching production speed of 6 m/min. Although TPU showed more deviation in surface roughness values, the same phenomenon was also visible in Figure 61. This observation can be useful when studying the plastic behavior of new materials in industrial production lines where finding optimal processing parameters may require time consuming test runs.

Sharkskin effect was not observed on the extrudate even at high production rates. The limiting factor was the puller speed which reached its maximum at 10 m/min. If pulling capacity would have been greater sharkskin could have been observed on the tubing and seen the effect on the graphs. If Step 2-measurements with LLDPE and TPU could have been continued past 10 m/min line speeds the graphs would have first shown similar plateauing behavior than in Figure 56 and Figure 61 but after reaching critical shear rate presumably turned into rising trend with more deviation in *Ra*-values. However, further studies in production lines capable of faster line speeds, possibly in industrial environment, are needed to confirm this.

The temperature dependency of the surface roughness values was also studied and can be interpreted from the results. Interestingly, at the optimal processing temperature of 200 °C, LLDPE showed the highest *Ra*-values. However, TPU at optimal processing temperature of 220 °C, showed the smallest *Ra*-values. This was most probably caused by the fact that the measurement at 200 °C was the first one performed and prior to that there where multiple test runs without purging the die. This may cause the values to be higher than in the other two measurements with LLDPE since after the measurement test runs were performed with TPU and the die was purged after continuing the actual measurements. If so, this indicates that the technique can also be used to detect contamination accumulation in the die. According to the result with TPU it seems that the optimum processing temperature with a given extrusion equipment results in the lowest *Ra*-values.

The results also display good data about *Ra*- alue distribution in both materials. The distributions seem to follow normal distribution quite well but longer test runs with constant production speeds and more data would be needed to verify this. However, these tests provide some future reference about the surface roughness value distributions of LLDPE and TPU, since this kind of data is not commonly found in literature references or scientific publications.

Table 13 displays the numerical results from the Step-1 measurement with LLDPE compared to the previous study by Hautala J. [1] on plastic film measurement. The LLDPE-material was the same in both measurements.

***Table 13**: Comparison of numerical results with LLDPE to film extrusion, film extrusion data from reference [1]*

Tube extrusion

T [°C]	185			200			210		
V [m/min]	2	3	4	2	3	4	2	3	4
Ra [µm]	0.90	0.66	0.62	0.99	0.84	0.73	0.93	0.73	0.66
Ra Std [µm]	0.04	0.03	0.05	0.05	0.03	0.03	0.05	0.03	0.03
Rz [µm]	5.33	3.86	3.57	5.80	4.88	4.32	5.50	4.25	3.90
Rz Std [µm]	0.55	0.49	0.81	0.77	0.52	0.49	0.70	0.69	1.00

Film extrusion

T [°C]	180			195			210		
V [m/min]	20	25	30	20	25	30	20	25	30
Ra [µm]	1.07	1.64	1.01	1.10	1.22	1.03	1.04	0.92	0.85
Ra Std [µm]	0.26	0.59	0.28	0.42	0.32	0.23	0.51	0.35	0.20
Rz [µm]	8.64	12.97	8.17	9.10	9.87	8.42	8.01	7.05	6.73
Rz Std [µm]	2.37	4.48	1.80	3.50	2.60	1.72	3.83	2.23	1.38

Comparison of the results with LLDPE displays that in film extrusion, where the thickness of the film affects the accuracy of the measurement greatly, the deviations are much higher than in tube extrusion. However, at 210 °C the behavior of the material is similar in both processes: the roughness parameters decrease with increasing pulling speed. The deviations in film extrusion are much higher due to surface defects and lack of accurate control in sensor height control during the measurement. Progress in product design was made between the time gap of the two studies and MP900 was released after the study by Hautala J. and sensor height control was integrated into the device allowing fast and accurate focusing of the measurement head onto the surface.

8.1 Benefits of optical on-line surface roughness measurement in industry

The main objective of this thesis work was to study the potential of on-line surface roughness measurement technique LCI in tube manufacturing process and find out how the industry would benefit from the technique. The technique was successfully applied to tube extrusion process monitoring and quality control and following benefits were observed:

- Material plasticization monitoring,
- Monitoring the effect of temperature, screw rpm and puller speed at low production rates,
- Surface defect detection,
- Die contamination detection,
- Diameter dimension variation detection,
- Roughness value distribution in different materials,
- Sharkskin detection on high production rates and finding the maximum production speed (not verified),
- Easy to operate and interpret data,
- Easy to install on the production line,
- Very low maintenance and calibration interval and
- Fast and accurate measurement of surface roughness parameters.

Table 14 summarizes tube extrusion process problems which were confirmed to be detectable (excluding sharkskin effect) with LCI within this thesis work.

***Table 14**: Tube extrusion process problems detectable with LCI*

Process problem	Causes	How LCI detects the problem
Uneven melt flow or melt pressure	Uneven material feeding	Tube dimensions vary, seen as sharp peaks and increasing deviation
	Contaminations in the die	*Ra* starts to deviate from its accustomed distribution
	Temperatures fluctuate	Increasing deviation
Outer diameter off tolerance	Incorrect vacuum pressure Screw speed not correct	Tube's position in z-direction varies causing missed data
Diameter oval	Incorrect vacuum pressure Melt temperature too high Cooling bath too short	Tube's position in both y- and z-direction vary causing missed data
Shark skin	Excessive shear rate	Defect peaks, increasing deviation, overall increase in *Ra*
Incomplete material plasticization	Too low screw rpm Too low temperature	*Ra*-value has not plateaued on its known minimum level

The technique can be applied when searching correct parameters for homogenous plasticization for new materials in industrial production lines since it can display accurately the decrease that occurs in *Ra*-value when the shear rate raises when increasing the line speed. When the *Ra*-value plateaus on a certain level reaching its lowest value the material in the extruder has plasticized uniformly. Proper plasticization affects the extrudates mechanical properties and processability; being able to detect it is a great benefit.

If the application requires higher surface roughness value, the technique can be utilized to find correct parameters for screw rpm, puller speed and temperature to maintain the *Ra*-value on a certain level. The MP900 can detect if the value exceeds the preset limits and warns the operator with sound and light signal. In addition, any anomalies such as fluctuations in tube OD and contaminations are easily detected and observed as increasing standard deviation. The technique can also be used to collect data about *Ra*-value distributions in different materials and products, which can then be used as a reference database in quality control. For example, if a product is known to have certain *Ra*-value distribution and the values of the current run are deviating much from the reference values, then most likely contaminations in the die are affecting the extrudate surface and a maintenance break for purging the die is needed.

Sharkskin effect was not observed during the measurements conducted in this thesis work due to maximum speed of the puller being too low. However, the technique could be utilized in finding the maximum production line speeds by raising the line speed in similar manner to the Step 2-measurements in this thesis and reaching the point of critical shear rate where sharkskin would start to appear on the tube surface. The last stable speed setting before sharkskin's appearance indicated by a change from a plateauing graph to more deviating and increasing trend would give the maximum line speed for the given material with the given equipment. This can be verified in future studies.

Optical on-line surface roughness measuring technique would be a great asset in tube extrusion industry because of advantages listed above. In addition, on-line quality control decreases the need of post-production quality control and may increase the productivity of a production line greatly by enabling to decrease the amount of scrap and maximize the line speed. The device is easy to integrate into an industrial production line, easy to operate and remains relatively maintenance free after initial installation. Integration to manufacturers' MES (Manufacturing Execution system) systems would allow constant stream of quality data from the production line.

9. CONCLUSION

The objective of the thesis was to produce reliable and accurate quality data from polymeric tube extrusion process in laboratory conditions by measuring the surface roughness with on-line optical measuring technique. In addition, the goal was to chart the benefits of on-line measurement technique for polymeric tube extrusion industry, focusing on the medical tube industry. A tube extrusion line was assembled and FocalSpec MP900 surface roughness measurement unit was installed onto the line to measure surface roughness parameter *Ra* and its *Std* on-line during small diameter tube production. Prior to on-line measurements, the technique was compared to another surface roughness measurements technique, optical profilometry, to verify accuracy and reliability of the device and acquire reference data for the on-line measurements. The results from static measurements with commercial sample tubes displayed comparable results with minor differences in *Std* values.

The on-line measurements conducted in two steps showcased the potential of the measurement technique in industrial use. MP900 equipped with LCI-1200 sensor was able to detect the changes in *Ra*-values caused by altering the process parameters in a controlled manner. Most interestingly, the device could indicate how well the material was plasticized in the extruder barrel that was seen as a gradual decrease in *Ra*-graphs when the line speed was increased gradually. This was clearly seen with the first of the two materials, LLDPE, used in the laboratory measurements. The other material, TPU which was known to be a common medical tube material, displayed similar behavior, though the processing conditions were little different than with LLDPE and the process was not as stable due to elastomeric nature of the material.

Although the size of the lumen and tube’s ability to withstand heat and mechanical stress is usually the main concern, the roughness of the tube is also considered when selecting material for different medical tubing applications. Reduced surface roughness in medical tubes results in reduced friction between the human tissue and polymeric tube if the tube is inserted into the human body, for example in the case of urinary catheters. Reduced friction and soft materials are known to be more comfortable for patients. The ability to measure the surface roughness during production helps to remove random bad products that may appear due to process anomalies and maintain the quality at a constant level. Table 15 displays the summary of the thesis subjects and the benefits of on-line roughness measurement in tubing industry.

***Table 15**: Summary of the thesis subjects*

Microextrusion		**Medical tube materials**		**Medical tube requirements**		**Roughness measurement**		**LCI benefits**
Single screw extrusion	Twin screw extrusion	Thermoplastics	Elastomers	FDA standards	ISO 13485, CE-mark	On-line	Static	Material plasticization
		PVC	Silicone rubber			LCI	LCI Profilometry	Quality monitoring
Equipment:		PTFE	Natural rubber	Traceability				Finding the optimal process parameters
Tubing die		FEP	TPU	Sterility		*Ra*		
(Gear pump)		PVDF		Roughness and COF		*Ra Std*		Finding the maximum line speed
Vacuum calibration		PFA		Dimensional tolerances		*Rz*		
Cooling tank(s)		PE				*Rz Std*		Measurement speed
Puller		PP						Accuracy
Process issues:		PA						
Melt temperature	Melt pressure							
Shark skin								
Slip and stick								
Melt fracture								

On-line surface roughness measurement was proven to be beneficial in polymeric tube quality control. However, further measurements would have been conducted if time and resources would have been available. For future study, at least the sharkskin effect detection at high production speed rates should be researched and verified. More data about the surface roughness distribution in common tubing materials could be collected into reference database in companies for quality control purposes. The technique has been tested in plastic sheet and tube manufacturing but other industries could also benefit from on-line roughness measurement. Industries, such as packaging industry with developing packaging materials and adhesion methods, or manufacturers of electronics and circuit boards could also utilize the technique with great benefits.

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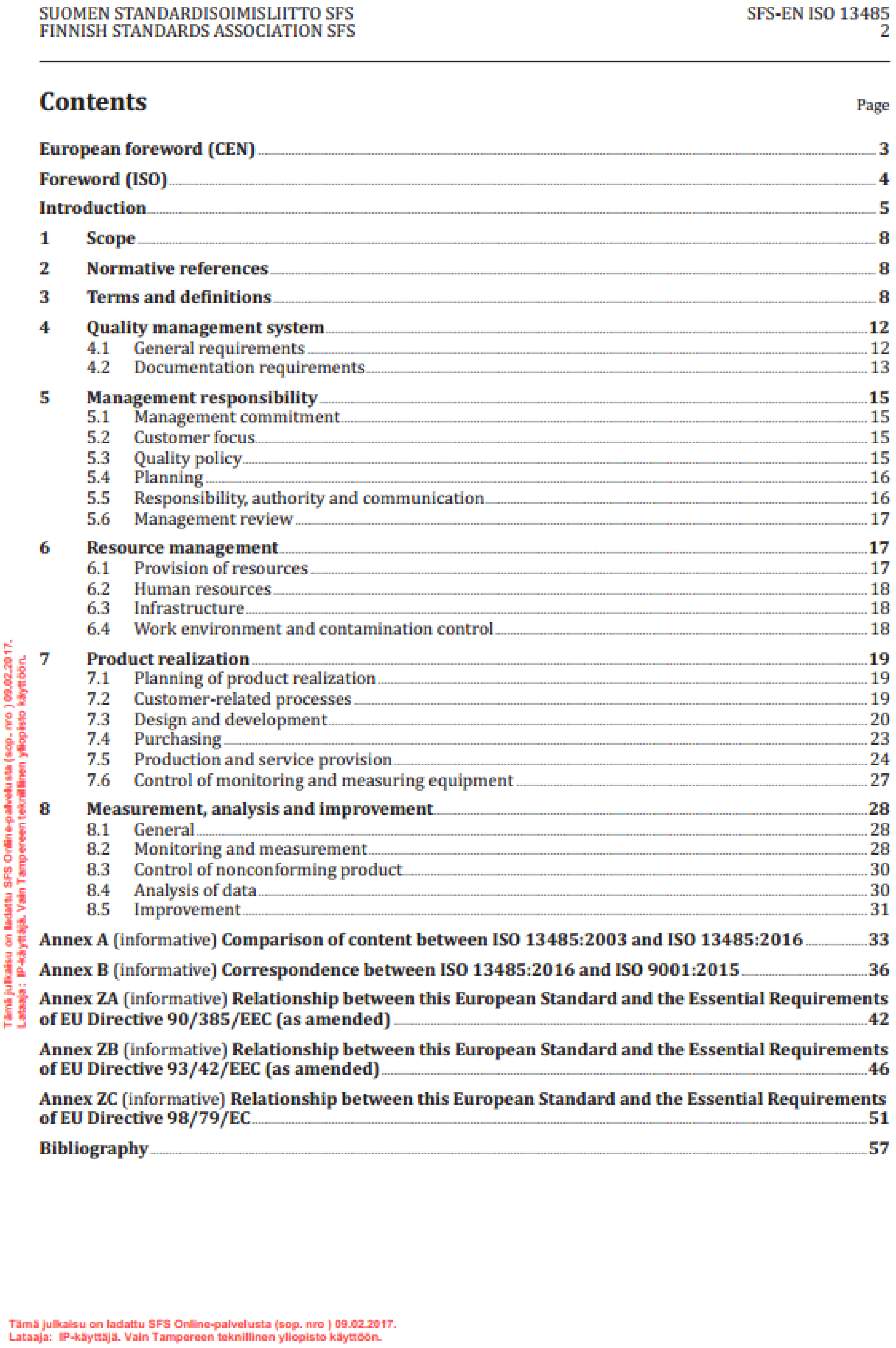
APPENDIX A: ISO 13485:2016 MEDICAL DEVICES. QUALITY MANAGEMENT SYSTEMS. REQUIREMENTS FOR REGULATORY PURPOSES; CONTENTS AND SCOPE



Contents

Page

European foreword (CEN) 3

Foreword (ISO) 4

Introduction 5

1 Scope 8

2 Normative references 8

3 Terms and definitions 8

4 Quality management system 12
- 4.1 General requirements 12
- 4.2 Documentation requirements 13

5 Management responsibility 15
- 5.1 Management commitment 15
- 5.2 Customer focus 15
- 5.3 Quality policy 15
- 5.4 Planning 16
- 5.5 Responsibility, authority and communication 16
- 5.6 Management review 17

6 Resource management 17
- 6.1 Provision of resources 17
- 6.2 Human resources 18
- 6.3 Infrastructure 18
- 6.4 Work environment and contamination control 18

7 Product realization 19
- 7.1 Planning of product realization 19
- 7.2 Customer-related processes 19
- 7.3 Design and development 20
- 7.4 Purchasing 23
- 7.5 Production and service provision 24
- 7.6 Control of monitoring and measuring equipment 27

8 Measurement, analysis and improvement 28
- 8.1 General 28
- 8.2 Monitoring and measurement 28
- 8.3 Control of nonconforming product 30
- 8.4 Analysis of data 30
- 8.5 Improvement 31

Annex A (informative) **Comparison of content between ISO 13485:2003 and ISO 13485:2016** 33

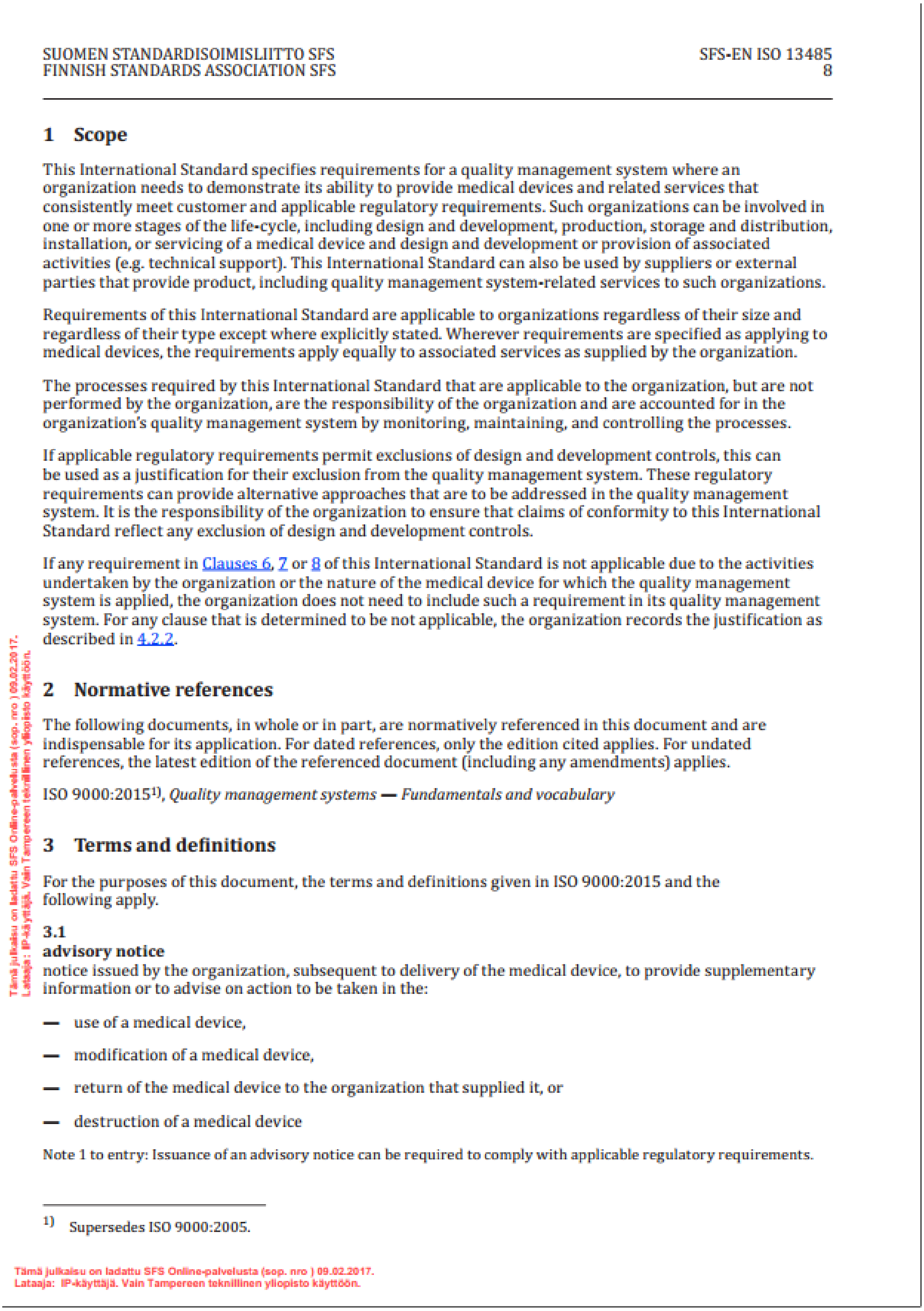
Annex B (informative) **Correspondence between ISO 13485:2016 and ISO 9001:2015** 36

Annex ZA (informative) **Relationship between this European Standard and the Essential Requirements of EU Directive 90/385/EEC (as amended)** 42

Annex ZB (informative) **Relationship between this European Standard and the Essential Requirements of EU Directive 93/42/EEC (as amended)** 46

Annex ZC (informative) **Relationship between this European Standard and the Essential Requirements of EU Directive 98/79/EC** 51

Bibliography 57



1 Scope

This International Standard specifies requirements for a quality management system where an organization needs to demonstrate its ability to provide medical devices and related services that consistently meet customer and applicable regulatory requirements. Such organizations can be involved in one or more stages of the life-cycle, including design and development, production, storage and distribution, installation, or servicing of a medical device and design and development or provision of associated activities (e.g. technical support). This International Standard can also be used by suppliers or external parties that provide product, including quality management system-related services to such organizations.

Requirements of this International Standard are applicable to organizations regardless of their size and regardless of their type except where explicitly stated. Wherever requirements are specified as applying to medical devices, the requirements apply equally to associated services as supplied by the organization.

The processes required by this International Standard that are applicable to the organization, but are not performed by the organization, are the responsibility of the organization and are accounted for in the organization's quality management system by monitoring, maintaining, and controlling the processes.

If applicable regulatory requirements permit exclusions of design and development controls, this can be used as a justification for their exclusion from the quality management system. These regulatory requirements can provide alternative approaches that are to be addressed in the quality management system. It is the responsibility of the organization to ensure that claims of conformity to this International Standard reflect any exclusion of design and development controls.

If any requirement in Clauses 6, 7 or 8 of this International Standard is not applicable due to the activities undertaken by the organization or the nature of the medical device for which the quality management system is applied, the organization does not need to include such a requirement in its quality management system. For any clause that is determined to be not applicable, the organization records the justification as described in 4.2.2.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9000:2015[1], *Quality management systems — Fundamentals and vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 9000:2015 and the following apply.

3.1
advisory notice
notice issued by the organization, subsequent to delivery of the medical device, to provide supplementary information or to advise on action to be taken in the:

— use of a medical device,

— modification of a medical device,

— return of the medical device to the organization that supplied it, or

— destruction of a medical device

Note 1 to entry: Issuance of an advisory notice can be required to comply with applicable regulatory requirements.

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[1] Supersedes ISO 9000:2005.

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APPENDIX B: ISO 14644-1:2015 CLEANSROOMS AND ASSOCIATED CONTROLLED ENVIRONMENTS. PART 1: CLASSIFIATION OF AIR CLEANLINESS BY PARTICLE CONCENTRATION; ISO CLASSES OF AIR CLEANLINESS BY PARTICLE CONCENTRATION

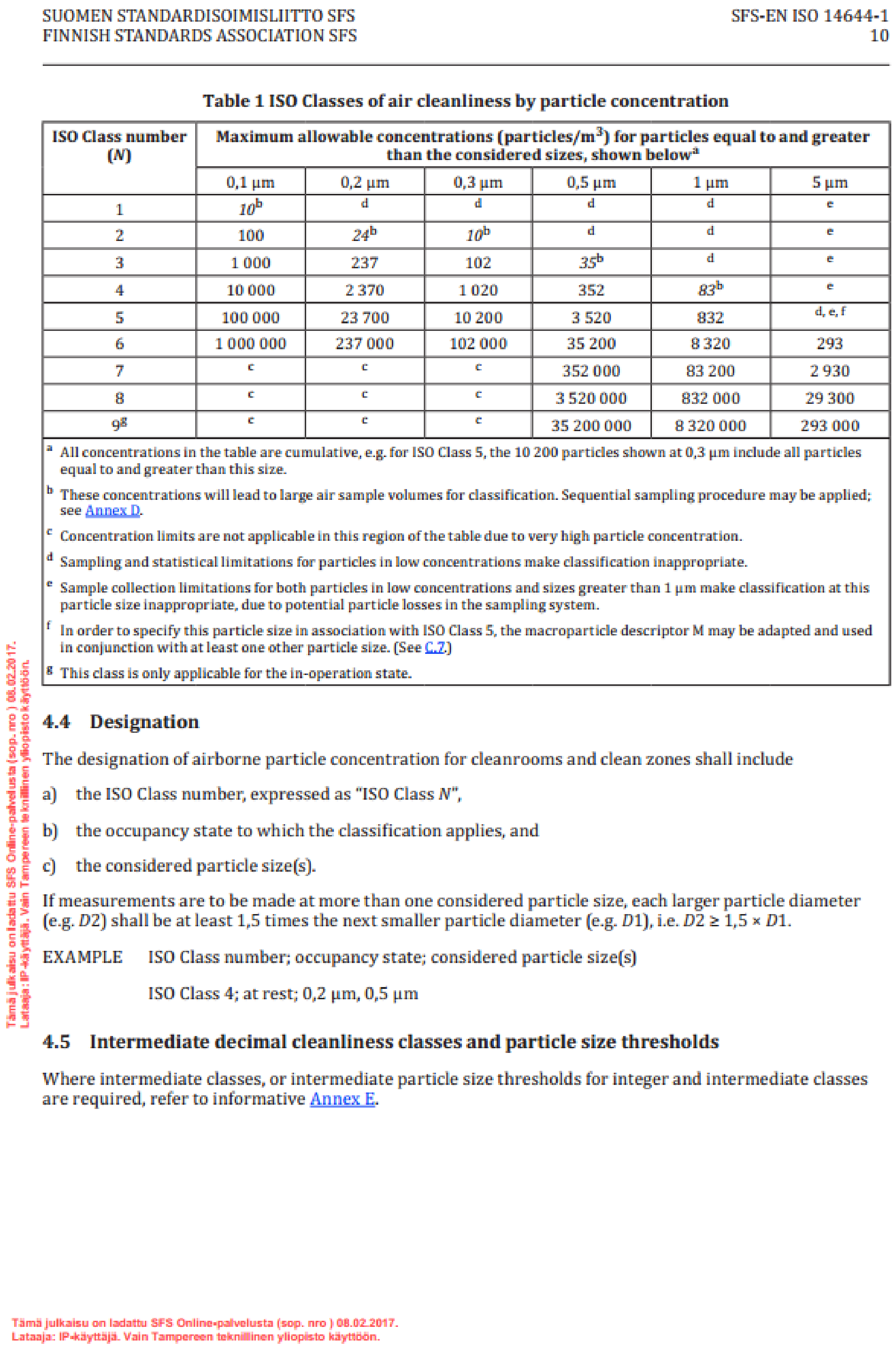

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Table 1 ISO Classes of air cleanliness by particle concentration

ISO Class number (*N*)	**Maximum allowable concentrations (particles/m^3) for particles equal to and greater than the considered sizes, shown below**[a]					
	0,1 µm	0,2 µm	0,3 µm	0,5 µm	1 µm	5 µm
1	*10*[b]	[d]	[d]	[d]	[d]	[e]
2	100	*24*[b]	*10*[b]	[d]	[d]	[e]
3	1 000	237	102	*35*[b]	[d]	[e]
4	10 000	2 370	1 020	352	*83*[b]	[e]
5	100 000	23 700	10 200	3 520	832	[d, e, f]
6	1 000 000	237 000	102 000	35 200	8 320	293
7	[c]	[c]	[c]	352 000	83 200	2 930
8	[c]	[c]	[c]	3 520 000	832 000	29 300
9[g]	[c]	[c]	[c]	35 200 000	8 320 000	293 000

[a] All concentrations in the table are cumulative, e.g. for ISO Class 5, the 10 200 particles shown at 0,3 µm include all particles equal to and greater than this size.

[b] These concentrations will lead to large air sample volumes for classification. Sequential sampling procedure may be applied; see Annex D.

[c] Concentration limits are not applicable in this region of the table due to very high particle concentration.

[d] Sampling and statistical limitations for particles in low concentrations make classification inappropriate.

[e] Sample collection limitations for both particles in low concentrations and sizes greater than 1 µm make classification at this particle size inappropriate, due to potential particle losses in the sampling system.

[f] In order to specify this particle size in association with ISO Class 5, the macroparticle descriptor M may be adapted and used in conjunction with at least one other particle size. (See C.7.)

[g] This class is only applicable for the in-operation state.

4.4 Designation

The designation of airborne particle concentration for cleanrooms and clean zones shall include

a) the ISO Class number, expressed as "ISO Class *N*",

b) the occupancy state to which the classification applies, and

c) the considered particle size(s).

If measurements are to be made at more than one considered particle size, each larger particle diameter (e.g. *D*2) shall be at least 1,5 times the next smaller particle diameter (e.g. *D*1), i.e. $D2 \geq 1,5 \times D1$.

EXAMPLE ISO Class number; occupancy state; considered particle size(s)

ISO Class 4; at rest; 0,2 µm, 0,5 µm

4.5 Intermediate decimal cleanliness classes and particle size thresholds

Where intermediate classes, or intermediate particle size thresholds for integer and intermediate classes are required, refer to informative Annex E.

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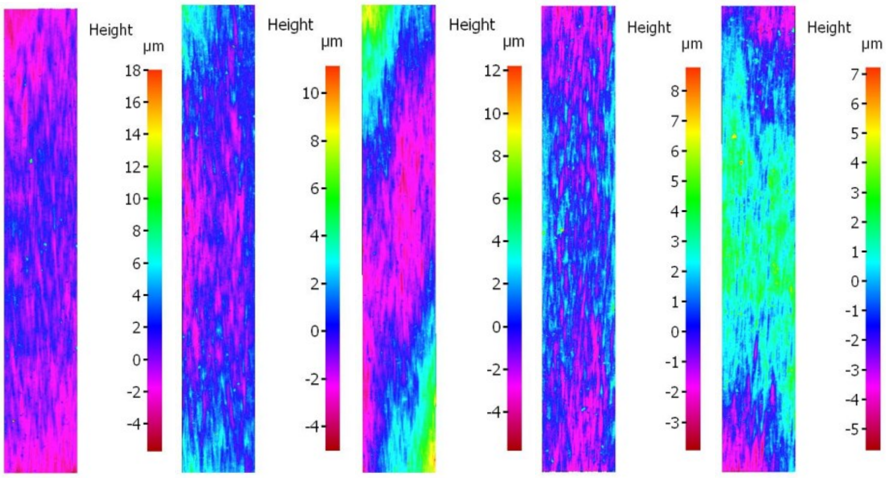
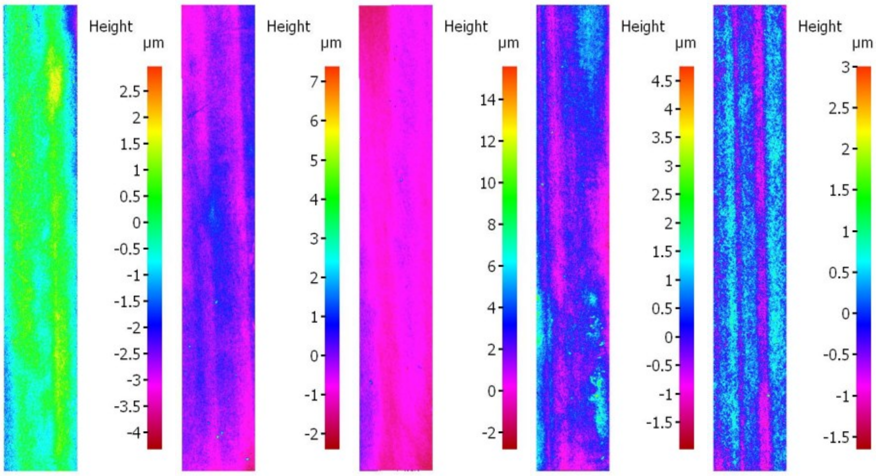
APPENDIX C: 2D-IMAGES FROM REFERENCE SAMPLES

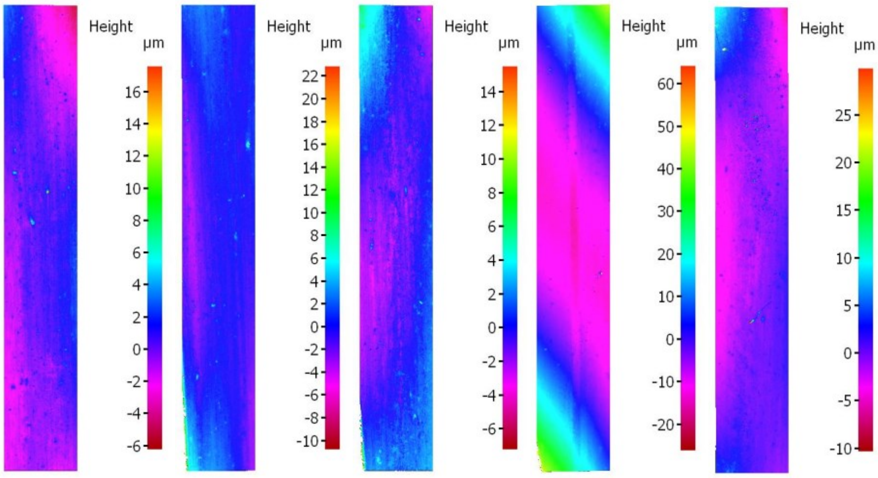
Sample A:



Sample B:



Sample D:



APPENDIX D: SHEAR RATE CALCULATIONS

Ro [m]	Ri [m]	A [m2]	(Ro+Ri)	(Ro-Ri)
0.004	0.002025	3.7383E-05	0.006025	0.001975

V [m/min]	V [m/s]	Q	γ [1/s]
0	0	0	0
1	0.016667	6.2305E-07	50.6329114
2	0.033333	1.2461E-06	101.265823
3	0.05	1.86915E-06	151.898734
4	0.066667	2.4922E-06	202.531646
5	0.083333	3.11525E-06	253.164557
6	0.1	3.7383E-06	303.797468
7	0.116667	4.36135E-06	354.43038
8	0.133333	4.9844E-06	405.063291
9	0.15	5.60745E-06	455.696203
10	0.166667	6.2305E-06	506.329114
11	0.183333	6.85355E-06	556.962025
12	0.2	7.4766E-06	607.594937
13	0.216667	8.09965E-06	658.227848
14	0.233333	8.7227E-06	708.860759
15	0.25	9.34575E-06	759.493671
16	0.266667	9.9688E-06	810.126582
17	0.283333	1.05918E-05	860.759494
18	0.3	1.12149E-05	911.392405
19	0.316667	1.18379E-05	962.025316
20	0.333333	1.2461E-05	1012.65823
21	0.35	1.3084E-05	1063.29114
22	0.366667	1.37071E-05	1113.92405
23	0.383333	1.43301E-05	1164.55696
24	0.4	1.49532E-05	1215.18987
25	0.416667	1.55762E-05	1265.82278