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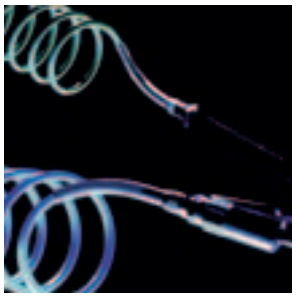
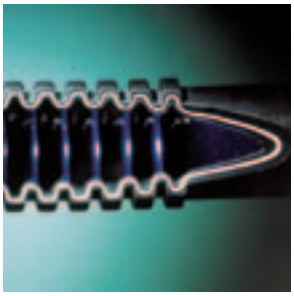
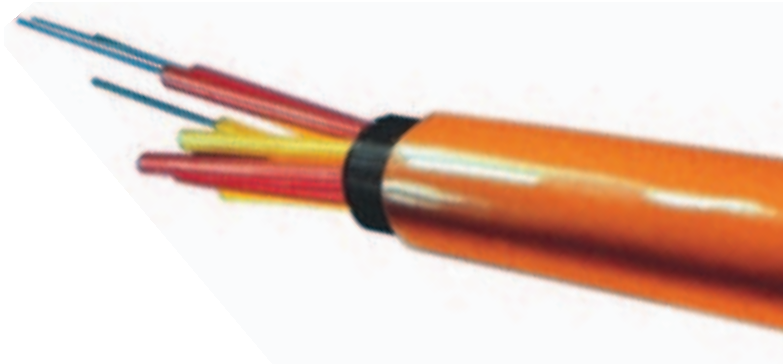
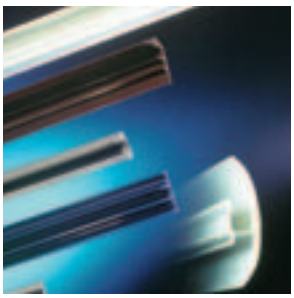








Directory

Handling and Processing of VESTAMID

Introduction

Degussa manufactures a range of speciality polyamide resins which are supplied under the registered trademark VESTAMID.

VESTAMID L-series:
Polyamide 12 (PA 12)
based on laurolactam

VESTAMID E-series:
Polyamide 12 elastomers (PEBA)
based on laurolactam and polyether diols
(polyether block amides)

VESTAMID D-series:
Polyamide 612 (PA 612)
based on hexamethylene diamine and
dodecanedioic acid

The properties of VESTAMID can be modified to suit the requirements of many applications by incorporating various additives such as stabilizers, plasticizers, reinforcements, fillers, etc.

Many of the VESTAMID resins are suitable especially for the injection molding of precision parts; others have been developed specifically for the extrusion process. A detailed description of the product categories is given in the brochures "Polyamide 12", "Polyamide 12 Elastomers", and "Polyamide 612".

This brochure covers the processing, as well as the pre- and post-treatment of the standard VESTAMID resins. Many of the information given here can be applied onto special developed VESTAMID grades, too. Further details are available from our Technical Marketing Department.

1 General information on handling and processing of VESTAMID resins

Supply of VESTAMID resins
VESTAMID resins are supplied as dry granules in 25 kg moisture-proof bags, ready for immediate processing. The shelf life at temperatures up to 30 °C is virtually unlimited if the packaging is not damaged. By mutual agreement, VESTAMID can also be supplied in octabins of 1,000 kg.

Prior to the opening of the bag, the content should be allowed to reach the ambient temperature, in order to avoid moisture condensing on the cold granules. VESTAMID granules will slowly absorb moisture from the air and hence any bags that are opened but not completely used should be closed again as tight as possible. The hoppers of the processing machines should not be filled with more granules than can be processed within two hours and should be closed with a lid.

Drying
Drying of the granules is necessary only if the original packaging has been damaged or been left open for a longer time (more than 2 hours). In these cases, the resins should be dried long enough to reduce the water content to less than 0.1 %. An excessively high moisture content will lead to problems during processing and/or poor parts properties.

The following conditions are recommended if VESTAMID has to be dried:

	Drying temperature [°C]	Drying time [h]		
		Dehumidified air drying oven	Circulating air drying oven	Fresh air drying oven
PA 12	80 – 110	2 – 4	2 – 16	2 – 10
PA 12 – plasticized	80	2 – 4	2 – 12	2 – 8
PA 612	80 – 110	2 – 4	2 – 16	2 – 10
PEBA	80	2 – 4	2 – 10	2 – 6

Table 1: Drying conditions for VESTAMID resins

The most effective way to dry resins is by using a dehumidified air drying oven. The drying process by using circulating air or fresh air drying oven depends on the moisture content of the air. Therefore, these processes may sometimes increase the moisture content of the granules.

After a long drying time, the granules will be discolored (refer to Figure 1). For resins containing plasticizer, it is possible that the plasticizer will be lost during the drying operation.

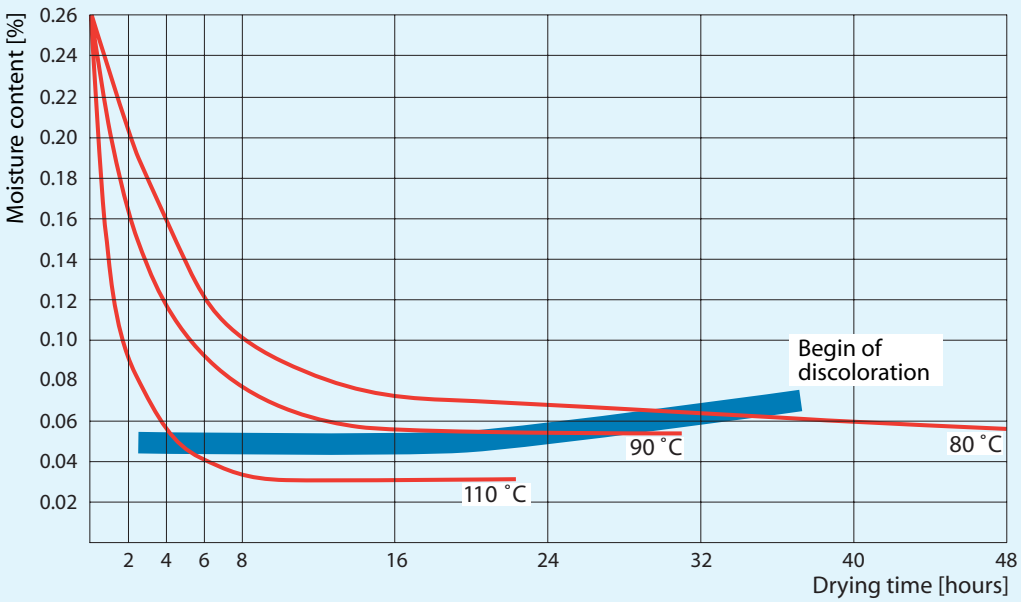


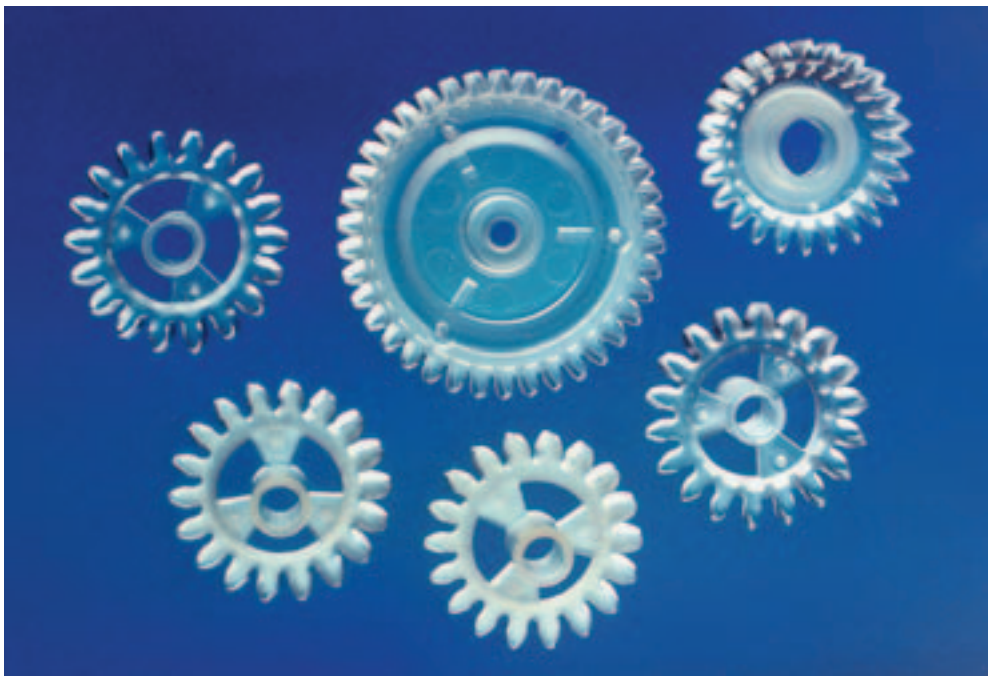
Figure 1: Drying of PA 12

Incompatibility with other thermoplastics
In most cases VESTAMID is not compatible with other thermoplastics. Note that even VESTAMID resins based on PA 12 and PA 612 are not compatible with each other. However PA 12 and PA 12 elastomers possess limited compatibility with each other. Molded parts of VESTAMID which contain traces of foreign polymers will normally embody inferior properties compared with uncontaminated polyamide. The weld lines of the melt behind mandrel carriers in the extrusion or behind cores in injection molding will especially initiate a substantially reduced strength in case of contamination with foreign polymers. Thus, it is extremely important to clean the machine thoroughly before start up of the production.

Coloring of VESTAMID
VESTAMID compounds are supplied in a range of standard colors. Special colors are available for orders of viable commercial quantities.

VESTAMID compounds can also be colored during processing. The preferred method is the application of a pigment concentrate based on PA 12 for PA 12 or PEBA compounds and PA 612 for PA 612 compounds. Dry coloring by tumbling with finely powdered colorants is another possibility but is nonetheless inconvenient. Pneumatic conveyance is then ruled out. The use of color pastes or color concentrates consisting of a "neutral base" (e.g. Polyethylene) can lead to the incompatibility with PA 12 and hence creating poor parts properties (inferior weld line strength or cold impact resistance). Therefore, preliminary testing for the compatibility is absolutely required.

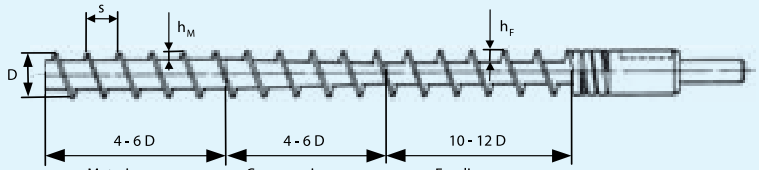
Processing of VESTAMID resins
Generally, all working areas used for thermoplastic processing should be well ventilated especially during the production. It is recommended that an exhaust system should be located over the machine nozzles. This is particularly true for resins containing plasticizers or flame retardant additives.
Under the circumstances of unfavorable conditions, for example given in the processing at excessively elevated temperatures, or during cleaning of the screws by burning off remaining polymer residues, it is possible that small amounts of noxious fumes are produced. Therefore, the cleaning of screws by burning off should always be done under an exhaust hood. Further details can be found in the relevant safety data sheet.



2 Injection molding

General information	
■ Generally, VESTAMID is processable on most of the commercially available injection molding machines. Nevertheless, we recommend to apply the instructions listed below for the injection molding of VESTAMID. ■ A comprehensive application advice is part of our technical service. For example, we employ various Computer Aided Engineering (CAE) programs to solve many problems arising from the mold and part design. Among these are the structural design/analysis programs, flow analysis programs to design the mold or the cooling system, programs to predict shrinkage and warpage. Please contact our Technical Marketing Department for further information.	
Plasticating unit	
Screw and barrel	■ Three zone screw with a length between 18 and 22 D ■ Flight depth ratio shall be ≥ 2 ■ Minimum flight depth: 2 mm in the metering zone, and 4 mm in the feeding zone ■ Screw and barrel diameter should be in the magnitude that a metering stroke between 1 D and 3 D is possible. ■ For more information see Figure 2
Peripheral screw speed	■ In the range of 3 – 12 m/min optimum ■ Larger speeds (e.g. > 18 m/min) can be applied, but might lead to problems in processing.
Nozzle	■ In general free-flow nozzles, for low viscous resins (e.g. VESTAMID L1670, VESTAMID L1723) externally operated shut-off nozzles (e.g. needle valve nozzle) are recommended. ■ Bore of nozzle should be app. 0.5 to 1.0 mm smaller than the gate
Back flow valve	■ Clearance between back flow valve and cylinder shall be ≤ 0.02 mm
Cleaning	■ Light contamination (e.g. cleaning due to changing of resin) (1) Prepare the mixture of high viscous PP and purging resin PLEXIFIX with a mixing ratio of 2:1. (2) Increase the temperature settings of heating zones by 30 to 40 K but not above 300 °C. (3) Plasticize the mixture with a long metering stroke and high dynamic pressure and discharge at high speed. (4) Flush the plasticating unit with the new resin until no purging mixture is contained in the melt ■ Persistent contamination – Persistent contamination can be mostly removed only by mechanical cleaning of screw, cylinder, back-flow valve etc. – Sometimes a cleaning of the cylinder can be avoided by using special purging material such as RAPID PURGE, SUPERNOVA or ASACLEAN.
Clamping unit	
Clamping force	■ Guide number for maximal pressure in cavity with VESTAMID resins: 200 to 600 bar

Mold	
Gate	■ All established types of sprues, runners and gates are possible. ■ Diameter or thickness of pin, submarine and film gates shall be ≥ 0.6 mm.
Hot runner	■ Externally heated hot runners with open sprue nozzle are recommended, nozzle diameter shall be ≤ 0.6 mm
Venting	■ Venting slots in mold parting surface: 0.01 to 0.03 mm deep, 4 to 5 mm wide
Steel	■ Suitable steels: 1.2767 (X45NiCrMo4), 1.2379 (X155CrVMo121), 1.2312 (40CrMnMo58) and 1.2343 (X38CrMo V 51)
Pressure sensor	■ Use of a pressure sensor for the cavity pressure is recommended to facilitate the careful setting of the change-over point.
De-molding	■ Release agent is normally not necessary. ■ Decrease of mold temperature very often eases de-molding ■ Suitable coatings of mold surface: Ni-P-PTFE, TiAlOx
Processing conditions	
Temperatures	■ Refer to Table 2 – 4 for general settings on melt temperatures. ■ Mold temperatures for PA 12 and PA 612 grades 30 -100 °C, for PEBA grades 15 to 40 °C ■ In general temperature of nozzle and heating zone close to nozzle to be set on melt temperature, in case of free-flow nozzles setting of nozzle temperature 10 K below desired melt temperature might be advantageous for non-reinforced resins. ■ Decreasing temperature profile in the heating zones towards hopper in steps of 10 K ■ Cooling of feeding zone to 40 – 80 °C
Interruption of production	■ For short production breaks (e.g. up to 1 hour): discharge of the plasticating unit, moving of screw to ultimate front position and decreasing temperature setting at heating zones down to 150 °C ■ For long production breaks: flushing of the plasticating unit with high viscous PP or PMMA, discharge of the plasticating unit, moving of screw in ultimate front position, switch off of cylinder heating. Remove resins left in the hopper and store in moisture proof packaging.
Trouble shooting	■ For trouble shooting of problems in injection molding guidelines published in the common literature are applicable (e.g. "Guide to surface defects on thermoplastic injection moulded parts" , published by Kunststoff-Institut für die mittelständische Wirtschaft, Karolinenstr. 8, 58507 Lüdenscheid, Germany). ■ For further information please contact our Technical Marketing Department.



Typical figures for PA 12-injection molding:

L_F	L_C	L_M	h_F*	h_M*	h_F/h_M
10 D	5 D	5 D	5 mm	2 mm	2.5

* for 30-mm screws

Figure 2: Design of a three zone screw

PA 12 resins	
VESTAMID (sw = black)	**Melt temperature [°C]**
L1600	190 - 230
L1660 sw	180 - 220
L1670	180 - 220
L1700	190 - 230
L1723	190 - 230
L1800	200 - 240
L1833	240 - 280
L1901	200 - 240
L1930	240 - 280
L1940	200 - 240
L1950 sw	200 - 240
L2128	200 - 240
L-CD22-M sw	240 - 280
L-CF15 sw	230 - 270
L-GB30	230 - 270
L-GF15	230 - 270
L-GF30	240 - 280
L-R1-MHI sw	240 - 280
L-R2-GF25 sw	230 - 270
L-R3-MHI sw	230 - 270
L-R4-MHI sw	230 - 270
L-R7-MHI sw	230 - 270
L-R9-MHI sw	230 - 270
LX9106	240 - 280
LX9107	240 - 280
X3500 sw	240 - 280
X4863	200 - 240
X7000	210 - 250
X7166	200 - 240
X7325	240 - 280
X7373	200 - 240
X7380 sw	240 - 280

Table 2: General melt temperature settings for PA 12 grades

PEBA resins	
VESTAMID (sw = black)	**Melt temperature [°C]**
E40-S1	180 - 220
E40-S3	180 - 220
E47-S1	180 - 220
E47-S3	180 - 220
E50-R2 sw	220 - 260
E55-S1	200 - 240
E55-S3	200 - 240
E62-S1	200 - 240
E62-S3	200 - 240
EX9200	200 - 240
X4442	220 - 260
X7375	200 - 240

Table 3: General melt temperature settings for PEBA grades

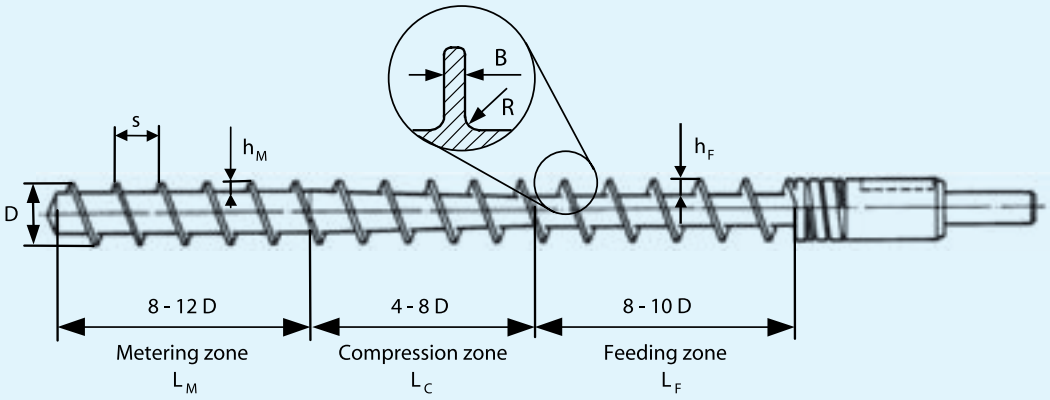
PA 612 resins	
VESTAMID	**Melt temperature [°C]**
D16	230 - 270
D18	230 - 270
D22	250 - 290
DX9300	230 - 270
DX9320	240 - 280
DX9321	240 - 280
DX9322	240 - 280
X7094	230 - 270
X7099	230 - 270

Table 4: General melt temperature settings for PA 612 grades

3 Extrusion

General information	
Screw and extruder design	■ Conventional three zone screw with a length ≥ 24 D ■ Flight depth ratio ranging from 2.5 to 3.5 : 1 ■ Suitable ratios between feeding/compression/metering zone: 2:1:3, 2:2:2, 1:1:1 ■ Radial clearance between screw and barrel: 0.1 - 0.2 mm ■ Mixing and shear elements may be useful to increase the melt homogeneity (e.g. processing films from resins colored with pigment concentrates). ■ For more information see Figure 3 Note: Other screw designs (e.g. barrier screws) may lead to proper processing conditions as well.
Breaker plate	■ Not necessary for the processing of virgin material, recommended only as support for screen packs e.g. when processing regrind.
Gear pump	■ Recommended when processing regrind or if tolerances have to be kept in an absolute narrow range (e.g. for barrier layers in multilayer tubing). ■ Not required with a properly designed screw.
Temperature setting	■ Cooling of the feeding section is mostly required. ■ General temperature setting depends very much on resins to be processed and type of extrudate (tube, film, coating ...), thus a general recommendation can not be given. Temperatures in first heating zone should be set app. 20 K above melting point of resins. Increasing temperature profile from first heating zone to tip of extruder. It might be advantageous to reduce the temperature setting in the adapter and die slightly to increase stiffness of melt leaving the die (e.g. when manufacturing tubing) ■ Optimizing temperature setting by monitoring the temperatures of the heating zones, the melt temperature and the consistency of the melt leaving the die: – high pressure build-up and a dull surface of the melt leaving the die: raise temperature profile – melt with a low stability: lower temperature profile Note: In case of large discrepancies between temperature setting and measured temperature the screw design might not be suitable for the processing of VESTAMID. ■ Temperature setting of heating zones always > 10 K above melting point of resins
Cleaning	(1) Raise temperature of all heater bands about 20 K and purge by using polypropylene (PP with MFR 230/5 = 12 g/10 min). (2) Disassemble the die. (3) Continue to purge extruder with PP and reduce temperature to about 170 °C. (4) At this temperature replace PP by a purging mixture comprising heat stabilized PVC/cleaning fluid/glass fibres (ratio 98:1:1). If the PVC grade is not sufficiently heat stabilized, degradation may easily occur. (5) Pull out the screw and remove plastic residues from screw and barrel. (6) Remove plastic residues from the die before polishing.

General information (continued)	
Trouble shooting	■ Pulsation of melt caused by the extruder – Insufficient cooling of feeding section – Insufficient lubrication of pellets – Shape of pellets inhomogeneous (e.g. when processing regrind) – Pressure build-up of die too low – Improper design of feeding section (grooved/smooth) – Improper screw design – Problems with motor/gear box Note: Pulsation of melt can be caused by the downstream equipment as well, e.g. see trouble shooting when processing tubing ■ Inconsistencies in temperature measurement – Dirt in bore of thermocouple – Tip of thermocouple not in contact with metal surface – Problems with thermocouple and/or recording device (e.g. thermocouple damaged)



Channel depth ratio = $\frac{h_F}{h_M}$ = 2.5 - 3.5 pitch s = D

Typical figures for PA 12-extrusion:

L_F	L_C	L_M	h_F*	h_M*	h_F/h_M	B*	R*
8 D	4 D	12 D	8.4 mm	2.8 mm	3	4.5	3

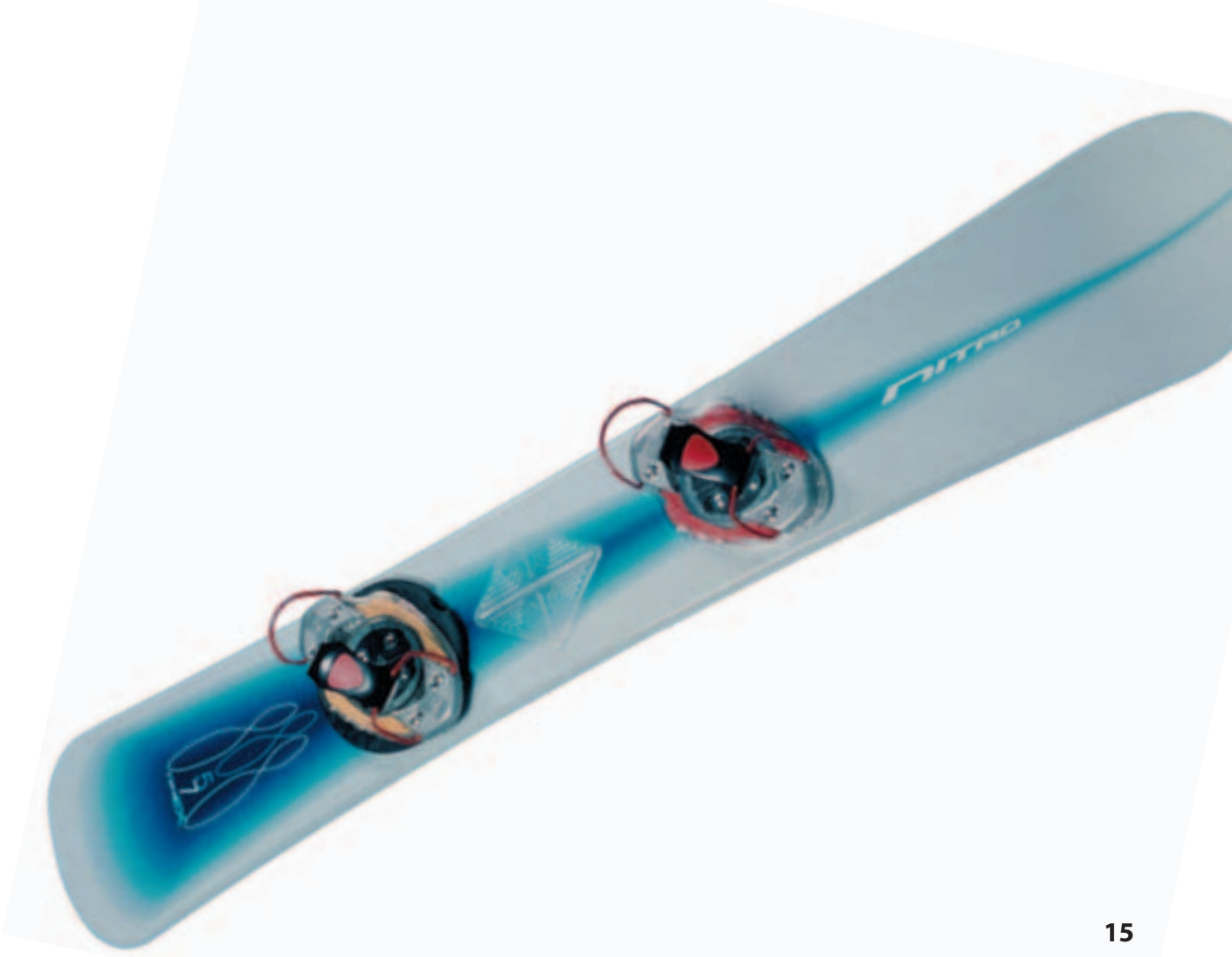
* for 45-mm extruders

Figure 3: Design of a three zone screw

Tubing	
■ Typical tubing outer diameter in the range of 6 to 16 mm ■ Typical resins used: VESTAMID L2140, L2124, X7293	
Die	■ For monowall tubing conventional extrusion dies (mandrel carried by a breaker plate or by spiders) are sufficient. ■ Land length between 20 and 50 mm ■ Draw down ratio (DDR = mean diameter of die gap divided by mean diameter of tubing): 2:1 to 1.7:1 ■ Wall thickness ratio (WTR = die gap width divided by tubing wall thickness): almost equal to DDR ■ Extrusion line should be equipped with an air hood located close to the die to take off vapors evaporating from melt leaving the die.
Calibration unit	■ Calibration by disk or perforated tube sizing ■ Inlet of sizing should be rounded (r = 5 to 6 mm) ■ Inlet of sizing must be covered by a well balanced water film to pre-quench the melt entering the sizing. This will avoid the sticking of melt on the metal surface of sizing. Sandblasting of front plate and inlet of sizing helps to evenly distribute water film around the tubing. ■ Tolerances in water flow must be kept to a minimum, e.g. variation in pressure level of water supply can be eliminated by using a water reservoir generating constant static pressure. ■ Inner diameter of sizing 3 to 6 % larger than nominal tubing outer diameter ■ Vacuum level in the range of 0.1 to 0.3 bar. Vacuum should be used only for fine adjustment of outer diameter. If a higher vacuum level is needed to achieve the nominal tubing outer diameter an other sizing with a slightly larger diameter should be used.
Puller	■ Smooth belt type puller is preferable over block type (caterpillar) puller
Flame treatment	■ Applied to improve adhesion of printing and mechanical properties ■ Positioning of burners around the tubing, not only on one side of the tubing ■ Additional quenching bath behind flame treatment is recommended.
Trouble shooting Surface quality	Note: Many problems from the processing of PA 12 tubing are caused by an improper water flow at the sizing inlet. Thus, the adjustment of a proper water flow is of major importance. ■ Dull surface – Melt temperature too low – Improper water flow at sizing inlet – Contamination of resin with incompatible thermoplastics ■ Grooves and stripes on outer surface – Hole in guiding discs of vacuum tank too small – Improper water flow at sizing inlet – Die damaged – Sizing damaged – Contamination of resin with incompatible thermoplastics

Tubing (continued)	
Tubing geometry	■ Bubbles on tubing surface – Too high moisture content of resins – Vacuum too high – Water splashing on melt due to too large water flow at sizing inlet – Water drops on tubing surface before flame treatment – Large air bubbles on tubing surface in vacuum tank ■ Wave structure on outer or inner surface of tubing – Vibration of components of extrusion line (e.g. cutter, puller) – Hole in guiding discs to small – Line speed too small when using a disk type sizing ■ Uneven tubing surface – Draw down ratio (wall thickness ratio) too small or too high – Water in vacuum tank sloshing ■ Oval tubing – Vacuum too small – Distance between belts of puller too small – Tube too hot when winded to coil ■ Uneven wall thickness – Improper centring of die – Uneven water flow at sizing inlet ■ Twisting of tubing – Uneven water flow at sizing inlet – Improper alignment of tubing and pulled belt ■ Curvature of tubing – Inhomogeneous wall thickness distribution – Uneven water flow at sizing inlet – Flame treatment from one side only – Offset between longitudinal axis of die and sizing – Too hot when winded to coil
Mechanical performance of tubing	■ Ultimate elongation too low – Vacuum level too high – Sharp edges in sizing – Radius of sizing inlet too small – Improper water flow at sizing inlet – Melt temperature too low – Flaming from one side only (or no flaming used at all) – Offset between longitudinal axis of die and sizing – Contamination of resin e.g. with other thermoplastics, dirt, dust etc. – Degradation of resin ■ Insufficient cold impact resistance – Contamination of resin with other thermoplastics, dirt, dust etc. – Melt temperature too low ■ Splitting of tubing – Melt temperature too low – Contamination in weld lines

Wire coating	
■ Maximum line speed up to 2000 m/min ■ Typical resins used: VESTAMID L1670, X7166	
Die	■ Sleeve coating die ■ Draw down ratio (DDR = cross section of die divided by cross section of coating): 15 to 20:1 ■ Vacuum level app. 0.2 bar
Calibration unit	■ Apply cold water cooling
Optical fiber jacketing (loose buffer tubes)	
■ Typical resins used: VESTAMID L1940	
Die	■ Sleeve coating die ■ Draw down ratio (DDR = cross section of die divided by cross section of tube): 9:1 at extrusion speeds > 200 m/min: 12:1 to 15:1 ■ Draw ratio balance (DBR = ratio of die to mandrel diameter divided by ratio of outer to inner diameter of tubing): 1:1 at extrusion speeds > 200 m/min: 1.2 to 1.3
Calibration unit	■ Apply cold water cooling
Cast film and sheet extrusion	
■ Typical resins used: VESTAMID EX9200	
Roll	■ Temperature setting in the range of 45 - 110 °C



4 Fabricating and finishing of molded parts and semi-finished products

Bonding	
General information	■ Adherent surfaces should be kept clean. ■ Avoid use of release agents when manufacturing parts to be bonded.
Adhesives	■ Commercially available adhesives based on: – Epoxies: One- or two-pack adhesives (gap filling) suitable for larger adherent surfaces. Hot setting adhesives reduce clamping time and may increase adhesion strength. – Polyurethane: One- or two-pack and (reactive) hot melt adhesives (gap filling, flexible adhesives mostly with longer pot life and clamping time) suitable for larger adherent surfaces. – Cyanoacrylates: One-pack adhesives (short setting time) suitable for small glue-lines and adherent surfaces. ■ With diffusion bonding adhesives (e.g. m-cresol, phenol with methanol or resorcine with methanol) a good adhesion strength can be reached. But, production with these adhesives is problematic due to reasons of industrial safety and environmental care .
Pre-treatment	■ Improvement of bonding strength by a pre-treatment of the surface to be bonded, e.g. by use of primers, roughening, electrical discharge, chemical or flame treatment. Apply safety instructions especially when using primers and chemical treatment.
Machining	
General information	■ Sawing, turning, milling, drilling, and planing of VESTAMID parts are possible. ■ Use of cooling by fluids or compressed air are recommended to reduce heating up of the VESTAMID parts and hence to avoid problems with sticking.
Printing and painting	
Laser marking	■ Resin modifications for laser printing are available.
Inks and paints	■ For sublimation colors most types of inks are possible. ■ Screen printing colors must be modified for application on PA 12 parts.
Pre- and post-treatment	■ Surface treatment e.g. by electrical discharge, flame treatment or bristling very often lead to an improvement of the adhesion of the ink. ■ After printing the adhesion of the ink can be improved e.g. by heating or flame treatment.
Thermoforming of PA 12-tubing	
General information	■ Data listed are meant only for monowall tubing, with multilayer tubing thermo-forming conditions might be different.
Polyethylene glycol	■ Forming temperature: 150 to 155 °C, forming time: < 5 min
Hot air	■ Forming temperature: 150 to 170 °C, forming time: 15 to 30 min
Steam	■ Forming temperature: 130 to 145 °C (3 to 5 bar pressure), forming time: < 1 min
Others	■ Other thermoforming procedures (e.g. infrared radiation, high frequency) are possible.

Welding	
General information	■ All established types of welding technologies can be applied on VESTAMID. ■ Typical technologies used are: – Hot plate welding: Apply PTFE coated hot plate welding tools to avoid/reduce sticking of resins at temperatures up to 270 °C – Ultrasonic welding: Joint design like a triangular projection (called an “energy director”) or like a shear joint. Welding of resins with a low modulus of elasticity is hardly possible by applying the far field technology. – Friction welding by rotation or vibration – High frequency welding – Laser beam welding

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