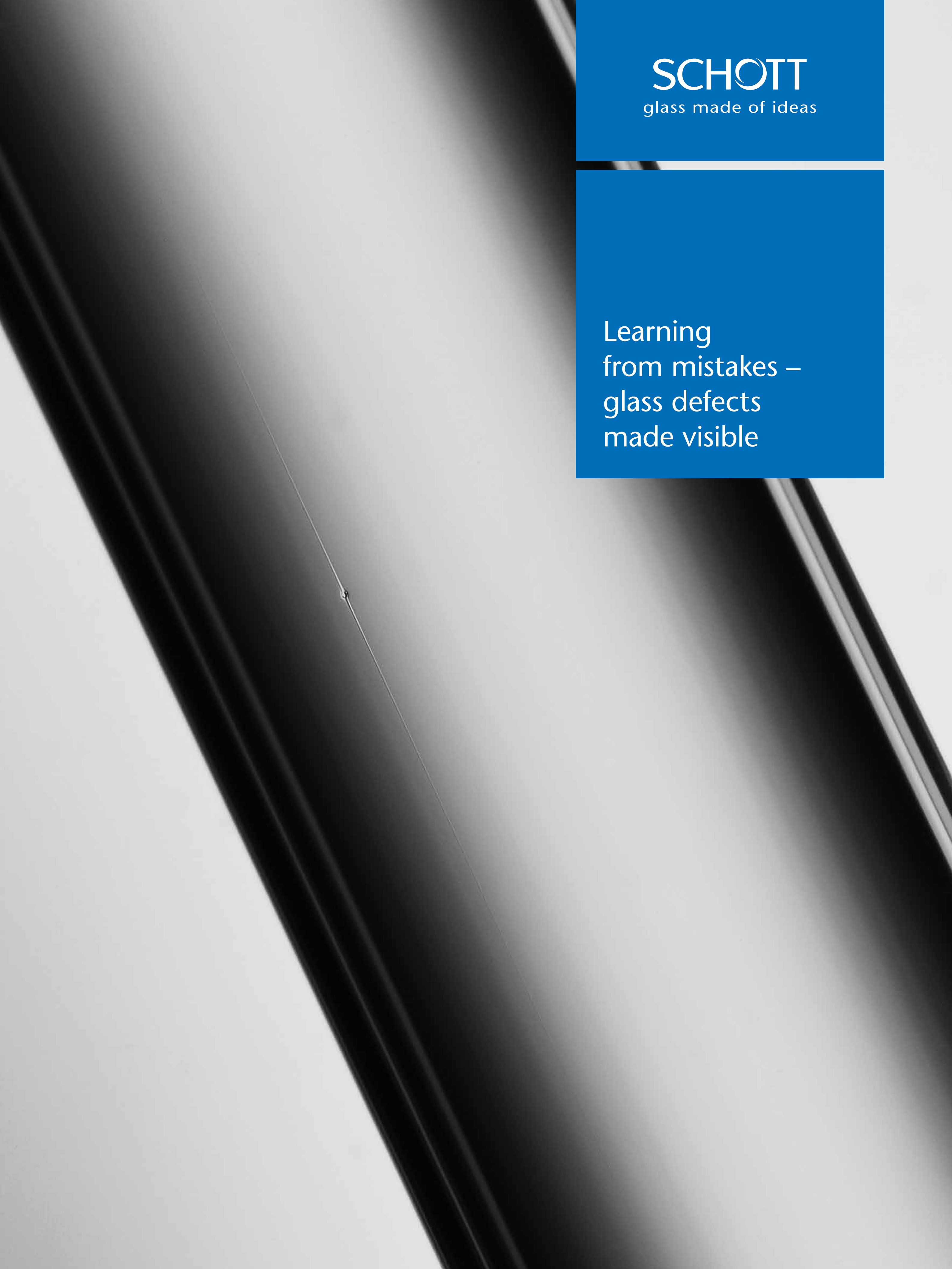
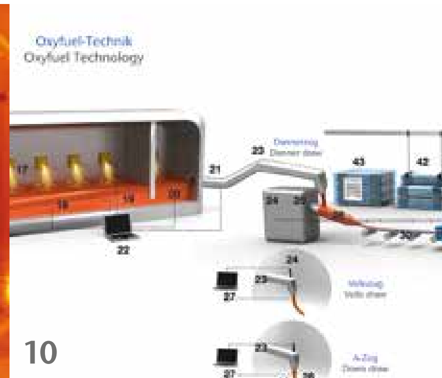
SCHOTT
glass made of ideas

Learning from mistakes – glass defects made visible



SCHOTT is a leading global technology company in the fields of special glass and glass ceramic. Backed by the expertise gained in over 130 years of experience in development, materials and technology, we offer a wide-ranging portfolio of high quality products and intelligent solutions to contribute to the success of your customers. With production capacity of more than 140,000 tons and production sites in Europe, South America and Asia, SCHOTT Tubing is one of the leading manufacturers of glass tubing, rods and profiles in the world. About 60 types of glass in a large variety of outside diameters and lengths are produced on the basis of development, production, and quality-assurance strategies that span all locations. SCHOTT Tubing furnishes custom-tailored products and services for international growth markets such as pharmaceutics, electronics, and industrial and environmental engineering.



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Glass is a millennia-old material that will help shape the future.

What is glass?

Glass is an inorganic molten product that solidifies when cooled without crystallizing. It can also be described as a frozen, superercooled liquid. Analysis of glass at room temperature using X-ray diffraction shows a curve similar to that of a liquid. In structural terms, glass thus has a short-range order but no long-range order. However, in its macroscopic properties glass conforms to a solid in every respect.

How old is glass?

Under very specific geological conditions, glass also forms in nature. In mineralogy, these types of glass are known as pitchstone, perlite and obsidian. An ancient formula, carved on clay tablets in cuneiform script, has captivated mankind for 7,000 years:
Take 60 parts sand, 180 parts ash from seaweed, 5 parts chalk, combine and heat and the result is glass.

Glass was highly prized in the ancient world – the Roman emperor Nero paid 6,000 sesterces – about 2,000 euros nowadays, for almost transparent glass. Glass has, of course, become considerably cheaper today as a result of technological development.

A brief history of glass:

ca. 7000 BC:

In Upper Egypt, glass was produced as a colored glaze while firing pottery due to the chance presence of calciferous sand combined with soda.

ca. 1000 BC:

A ceramic core was used as a negative mold. Craftsmen dipped the mold, which was attached to a rod, into the liquid glass, resulting in the first usable glassware.

ca. 200 BC:

Syrian craftsmen invented the blowpipe.

ca. 1800 AD:

Major progress was made when Fraunhofer first succeeded in producing fairly large, homogeneous glass sheets.

19th century:

Otto Schott laid the foundation for modern glass technology. Using scientific methods, he invented countless new types of glass with previously unknown properties. It was characteristic and typical of Schott and his work to demonstrate an instinctive feel for the right type of glass, as if he could “see into its heart”, as someone later put it. Glass is indeed alive.

The Manufacture of Special Glass Tubing
The history of technical glass

Chemical laboratories have long demanded sturdy glass for beakers, flasks and the like, which would withstand leaching water attack, especially of boiling liquids and thermal shock.

An essential inroad was made by using vitrifying boric acid. A test melt ended successfully when glass was produced with B_2O_3 and SiO_2 in just about any blend with other cations. A new type known as BOROSILICATE GLASS was born. These test melts led to the development of glass such as SCHOTT DURAN® and FIOLAX® clear. Optimization of glass composition made it possible to manufacture such glass commercially.

Production of tubular glass

Nature has produced tubular structures since the dawn of time. Imagine a storm breaking over a desert. The high energy of a flash of lightning striking the sand (which contains sufficient silica) produces structures made of quartz glass.

The oldest type of glass tubing production: hand drawing.

The tube drawer collects a gob of several kilograms of glass on a blowpipe and works it into the shape of a hollow cone by blowing air into it, rolling it and marvering it. Using a tool known as a pontil, another craftsman takes a gather just large enough to form a disc with a diameter matching the size of the tubular gather. The disc maker moves away from the blowpipe on the drawing line. At the same time, the desired tube dimensions are produced by blowing air into the blowpipe and by accurate annealing.

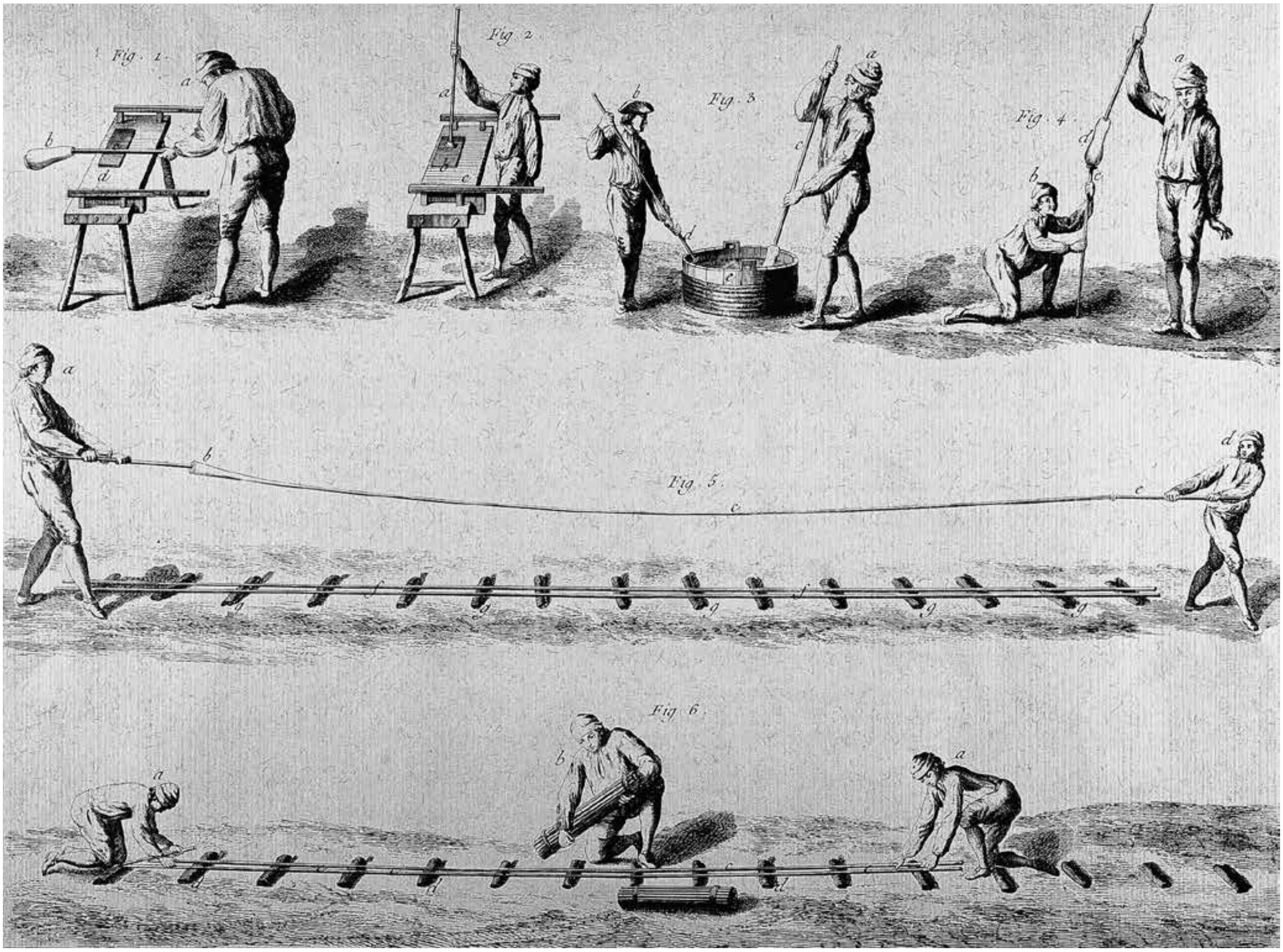
The usable tubular sections are cut out of the annealed tube. DANNER replaced this discontinuous method by continuous machine processing.

The Danner process:

E. Danner developed the first mechanized tube-drawing method, based on the hand drawing method, in 1912, and patented it on January 1, 1917.

An adjustable quantity of glass flows out of the nozzle onto the outer surface of a downwards-inclined rotating ceramic mandrel. The glass tube formed is continuously drawn towards the pipe axis as air is blown into it. The dimensions of the tubes depend on mandrel diameter, glass temperature, the pressure of the air blown and the pull rate.

Tubes with outside diameters of 1 – 130 mm and wall thicknesses of 0.1 – 10 mm can be manufactured using the Danner process.



Hand drawing

Another method for producing tubing is Vello:

The Vello method is named after its inventor, L. Sanchez-Vello, who first applied for a patent in June 1929 in France. The Vello method is a vertical drawing method in which the tube is drawn downwards from a nozzle. The outlet of the drawing head contains a cylindrical opening with a nozzle through which the glass can flow over a vertically adjustable reamer extending downwards like a funnel. The reamer is hollow, and its extension is connected to the blown air source. In the Vello method the drawn tubing is shifted from vertical to horizontal. Tubing with an outside diameter of 0.8 – 70 mm and wall thicknesses of 0.4 – 5 mm can be produced using this method.

The Manufacture of Special Glass Tubing

Production flow of pharmaceutical tubing at SCHOTT

SUB-STEPS OF THE PRODUCTION PROCESS:

- batch preparation
- melting
- tubing production
- quality assurance
- inventory

SCHOTT STANDARD GLASS:

- FIOLAX® clear
- FIOLAX® amber
- DURAN®
- BORO-8330™
- AR-GLAS®
- ILLAX®

Batch preparation:

Selected raw materials are used to produce special glass tubing, forming the basis for tube quality.

In the batch house, batches are prepared fully automatically in accordance with specified formulas. All data collected in the batch house are recorded and documented. Once ready, batches are conveyed, usually pneumatically, upon request to the storage bins of the individual melting furnaces.

Melting:

Melting is carried out in tanks of varying designs and sizes. The production of neutral glass is a major challenge in melting operations due to the specified composition of this glass type. The use of measuring and control instruments as well as electronic aids are indispensable for the control needed to attain high quality. Glass for pharmaceutics, electronics, industrial and environmental engineering are melted.

The melting process:

THE MELTING PROCESS IS DIVIDED INTO THE FOLLOWING STAGES:

- initial melting
- refining
- homogenization
- preparation for processing

Continuously operating tanks are primarily built for large-scale melts. They are so well equipped and their operation is so advanced that they could be referred to as melting machines.

Fully automatic feeders load the batches.

The amounts used must always correspond o the amount of glass being processed on the drawing lines. The two quantities are linked by carefully controlling the level of glass in the melting furnace.

The first stage of glass melting is silicate formation, followed by vitrification when the remaining sand has dissolved.
The term “initial melting” applies to both processes.

After initial melting, glass still contains gas inclusions and inhomogeneities, which are eliminated by refining and homogenizing the glass. This phase of the melting process is termed “refining”. After refining, glass melting is essentially complete. The melt must now be cooled until its viscosity is favorable to further processing.

Tubing production:

The tube drawing process requires considerable effort to meet tight production tolerances. Continuous measuring and testing of the endless tube, together with electronic evaluation, are essential for optimum process control and on-line quality control.

On-line measuring instruments detect (down to the last centimeter of the endless tube) deviations from the specified dimensional tolerances and any exceeding of admissible values such as bubbles, knots/stones (specified in the Technical Terms of Supply), store these readings and reject unacceptable tubes after they have been cut to length. Only tubes within the agreed quality limits continue on to the next processing stage.

Rejected tubes are crushed in the batch house and fed back into the melting process. Every type of glass has coded cullet trolleys, making it impossible to mix glass types.

In the next process stage the tubes are vibrated and filtered air is used to blow out large splinters that form during the cutting process on the drawing machine. The tubing ends are then processed. This procedure – patented by SCHOTT – is called the DENSOCAN® process. In a hot separation process (in which splinters cannot form), the tuning end is shaped to form a closed base and to protect it against further environmental influences.

Once the tube ends are closed, the tubes pass through more inspection systems:

Vision system:

In this system cameras check every single tube (100% control) for surface defects such as contamination (dirt, scratches), check for geometric deviations (bowing), and are rejected if admissible limits are exceeded. Afterwards the tubes pass through the testing machines.

Testing equipment:

These testing machines subject tubes to very high test frequency (1 tube/min.), and all relevant geometric characteristics are inspected. The readings are displayed for employees on on-site terminals. Operators are thus able to follow their processes via SPC (Statistical Process Control) and to intervene with any corrections in the event of deviations.

All characteristic data are stored in a database and compared with set values. If the set values are exceeded, the system blocks itself and flags the pallet certificate. The testing machine rejects tubes that do not meet quality specifications. This makes it possible to create a certificate for the customer that contains all statistical details such as mean value, statistical deviation and more.

After exiting the testing machines, the tubes are conveyed along the line to the automatic packing machine. Here tubes are packed as tightly as possible.

DENSOPACK® packing method:

Until packing, the tubes remain extremely clean, low in particles and absolutely sterile. The role of packing is now to minimize or prevent hazards in transit until the product reaches the customer. The packing material, polyethylene, does not abrade like paper or cardboard and has minimal weight and volume.

Every DENSOPACK® bundle on the pallet is labeled with glass type, barcode, contents, quantity, and also a code number. This code number enables products to be traced back to the minute with relative ease.

After packing, the finished DENSOPACK® bundles are automatically removed from the packing machine and fed to the central palletizing system. In the central palletizing system, which is kept very clean, the incoming bundles are removed and palletized. The pallet is then shrink-wrapped.

At no point in the production stages have human hands touched the glass tubing!

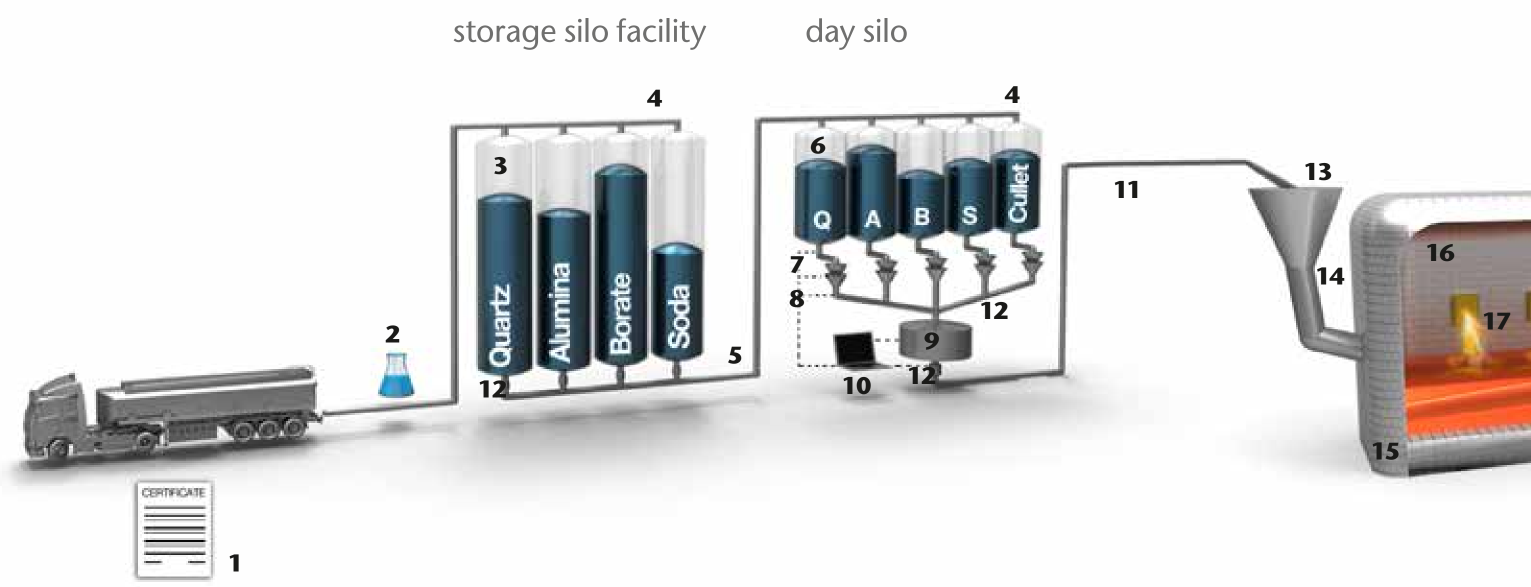
After shrink-wrapping, pallets approved by Production and Quality-Assurance – and only these – are conveyed to the warehouse.

Once at the warehouse, these pallets are moved inside with conveyors; in the warehouse itself the pallets are transported only with electric vehicles.

When stored properly, the physical and chemical properties of DENSOCAN® tubing, which is borosilicate glass with extremely high chemical resistance and thermal shock resistance, does not change, even after several years.

The production of glass tubing is a dynamic process that is improved on an ongoing basis and adapted to customer needs – to the benefit of both our customers and SCHOTT.

Schematic sequence of tubing production



1 RAw material supply

2 Laboratory testing

3 Storage bin

4 Filters

5 Raw material extraction

6 Day bin

7 Dosing element

8 Scale

9 Mixer

10 Process control

11 Batch conveyance

12 Pressure vessel

Quartz

Alumina

Borax

Soda

Cullet

13 Batch bunker

14 Batch feeder

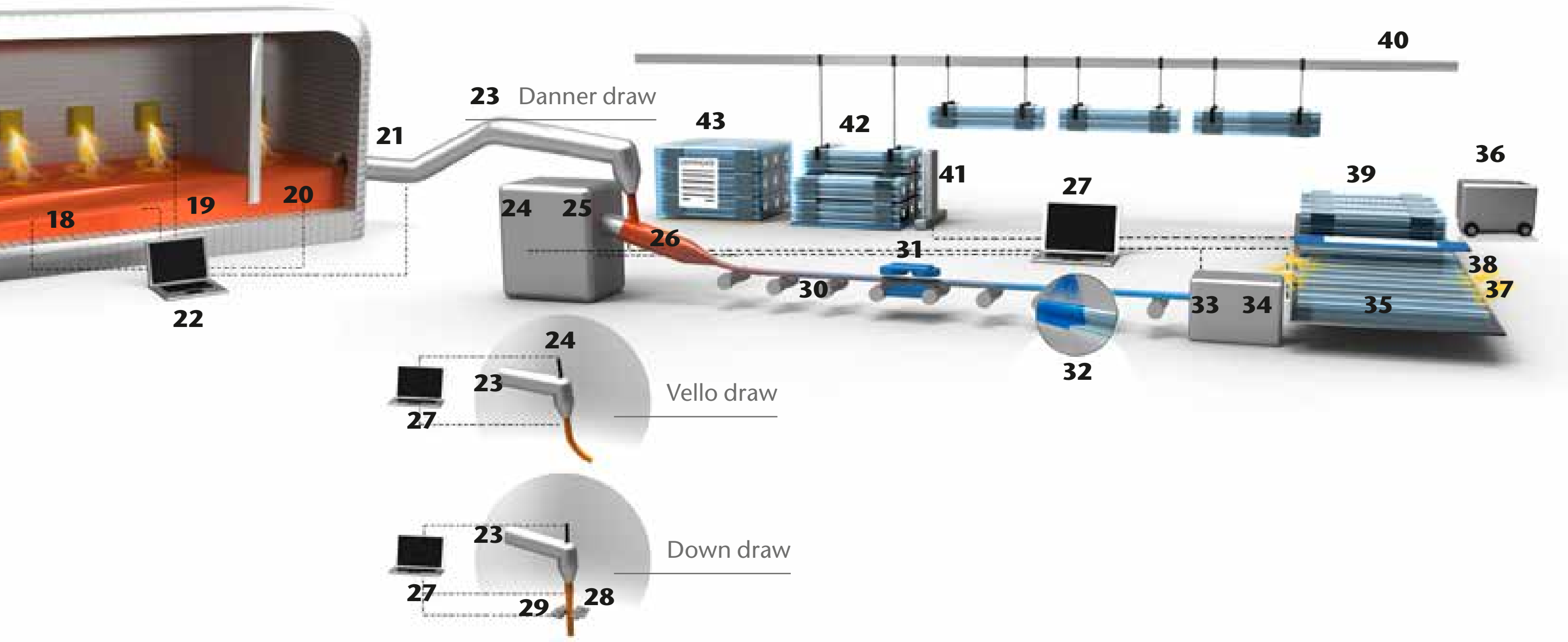
15 Refractory material

16 Combustion chamber

17 Burner

Raw materials

Oxyfuel technology



18 Melting tank	23 Feeder channel	30 Tube drawing channel	36 Cullet recycling
19 Refining tank	24 Air supply	31 Online measuring: glass flaws and geometry	37 Tube-end processing
20 Working tank	25 Pipe drive	32 Anti-scratch coating	38 Inspection system
21 Feeder channel	26 Blowpipe	33 Drawing machine, Danner, Vello	39 DENSOPACK® and labeling
22 Process control	27 Process control	34 Disconnecting device	40 Transport facility
	28 Drawing machine, down draw	35 Sorting	41 Barcode reader
	29 Online measuring: outside diameter		42 Pallet
			43 Pallet with shrink-wrap

Melting

Tubing production

Preface on glass defects

When considering the defects shown on the following pages, please remember that some are only visible under a microscope (e.g. bubbles). Others are deliberately exaggerated or worsened to make them more apparent; i.e. they have been 'produced' to make the list complete. It goes without saying that none of the defects displayed meet our high quality standards. The quality of our products is described in the relevant Technical Terms of Delivery (TLB).

Outside diameter approx. 29 mm
Wall thickness approx. 1.1 mm
Magnification > 5x

Stones

Definition Stones are opaque inclusions.

Cause Unmelted glass mixture causes stones, which occur due to unforeseen disruptions in production, or in rare cases the presence of refractory material.

Outside diameter approx. 20 mm
Wall thickness approx. 1 mm
Magnification approx. 4.5x

Knots

Definition Knots are transparent inclusions.

Cause Knots occur predominately on the surface of melted glass within the melting tank. They are formed by insufficient evaporation of ephemeral glass components.

Outside diameter approx. 16 mm
Wall thickness approx. 1.5 mm
Magnification approx. 3x

Longitudinal airlines

Definition A closed airline is an elongated gaseous inclusion in the tubing, and is not normally visible to the naked eye.

Cause During the melting process, the chemical and thermal reactions of the components of the glass create gases that form round bubbles in the glass. Most of these bubbles are removed during the subsequent refining process. Any remaining round bubbles form into elongated airlines during the drawing process.

Outside diameter approx. 16 mm
Wall thickness approx. 1.2 mm
Magnification approx. 3.5x

Seed

Definition

A seed is a gaseous, elongated inclusion in the tubing with a maximum length of 15 mm and which is not normally visible to the naked eye.

Cause

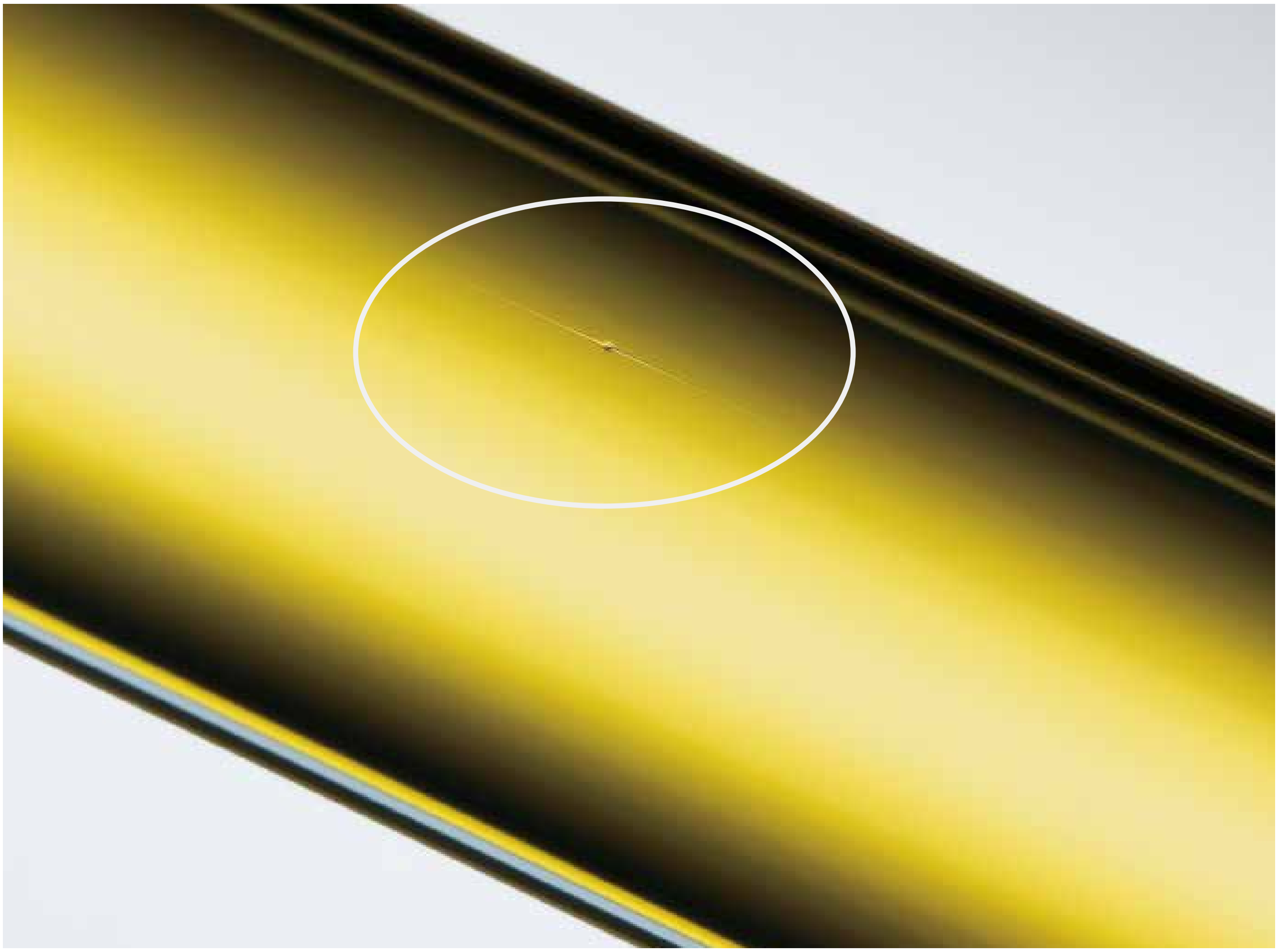
During the melting process, the chemical and thermal reactions of the components of the glass create gases that form round bubbles in the glass. Most of these bubbles are removed during the subsequent refining process. During the drawing process, any remaining round bubbles form into seeds, which are short forms of longitudinal airlines.

Outside diameter approx. 16 mm
Wall thickness approx. 1 mm
Magnification approx. 4.7x

Open airlines

Definition An open airline originates as a closed bubble (see longitudinal airlines).

Cause During the drawing process, a closed bubble on the surface of the glass may open.



Outside diameter approx. 18 mm
Wall thickness approx. 0.9 mm
Magnification approx. 5.5x

Crystals

Definition A crystal is generally an opaque inclusion on the glass surface with a geometric shape.

Cause Glass tends to crystallize at low temperatures during the drawing process.
Crystals may form in sizes of approx 0.05 mm.

Outside diameter approx. 23 mm
Wall thickness approx. 1 mm
Magnification approx. 3.8x

Radial scratches

Definition A scratch, as opposed to a surface crack, is evidence of slight surface damage. It does not penetrate deep into the wall and never perforates it, thus only negligibly affecting the mechanical strength of the structure.

Cause Radial scratches can occur through relative movement during all process stages (tube production, transport, processing).

Outside diameter approx. 30 mm
Wall thickness approx. 1.1 mm
Magnification approx. 2.6x

Longitudinal scratches

Definition A scratch, as opposed to a surface crack, is evidence of slight surface damage. It does not penetrate deep into the wall and never perforates it, thus only negligibly affecting the mechanical strength of the structure.

Cause Scratches are caused by contact with glass or other materials with a harder surface. Precondition: relative movement.

Outside diameter approx. 16.5 mm
Wall thickness approx. 1 mm
Magnification approx. 3x

Axial crack

Definition An axial crack is a planar flaw extending through the glass wall in an axial direction.

Cause Cracks are caused by rapid local heat loss at high temperature differences, e.g. during glass/glass or metal/glass contact.

Outside diameter approx. 17 mm
Wall thickness approx. 2 mm
Magnification approx. 4.5x

Radial crack

Definition A crack is a planar flaw extending through the glass wall in a radial direction.

Cause Cracks are caused in particular by rapid local heat loss at high temperature differences, e.g. during glass/glass or metal/glass contact.

Outside diameter approx. 24 mm
Wall thickness approx. 1.2 mm
Magnification approx. 3.3x

Fissures

Definition Fissures are cracks which occur at the tubing end during the production process.

Cause Fissures occur due to faulty separation of glass tubing (see also cracks).

Outside diameter approx. 16.5 mm
Wall thickness approx. 1 mm
Magnification approx. 4x

Molten fissures

Definition Fissures are cracks which occur at the tubing end during the production process.

Cause Fissures occur due to faulty separation of glass tubing (see also cracks). If the tube ends are subsequently heated, the part of the fissure at the tube end will melt again.

Outside diameter approx. 6 mm
Wall thickness approx. 0.5 mm
Magnification approx. 5.5x

Droppers

Definition A dropper is a glass of different chemical composition that forms on the tube surface as a colorless or brownish craquelé.

Cause A dropper is formed by evaporation and subsequent condensation on the muffle cover, and in rare instances falls onto the glass surface of the mandrel. The dropper is then drawn out in longitudinal direction during the drawing process.

Outside diameter approx. 14.75 mm
Wall thickness approx. 0.55 mm
Magnification approx. 6x

Drawing marks

Definition A drawing mark is a visible longitudinal line on the inner or outer surface caused by mechanical deformation.

Cause Drawing marks can occur during the forming and drawing process while the glass is still in a visco-elastic state.

Note Drawing marks are **not** visible in immersion fluid.

Outside diameter approx. 40 mm
Wall thickness approx. 1.5 mm
Magnification approx. 2.5x

Striae

Definition Longitudinal vitreous lines in tube-length direction, visible due to optical distortion. They are distinguished from the surrounding glassy body by a different chemical composition which refracts light differently.

Cause The cause of striae is inhomogeneous glass from the melting process (see also knots).

Note Drawing marks are clearly visible in immersion fluid.

Outside diameter approx. 23 mm
Wall thickness approx. 1 mm
Magnification approx. 3.7x

Glass particles

Definition Tiny particles of glass.

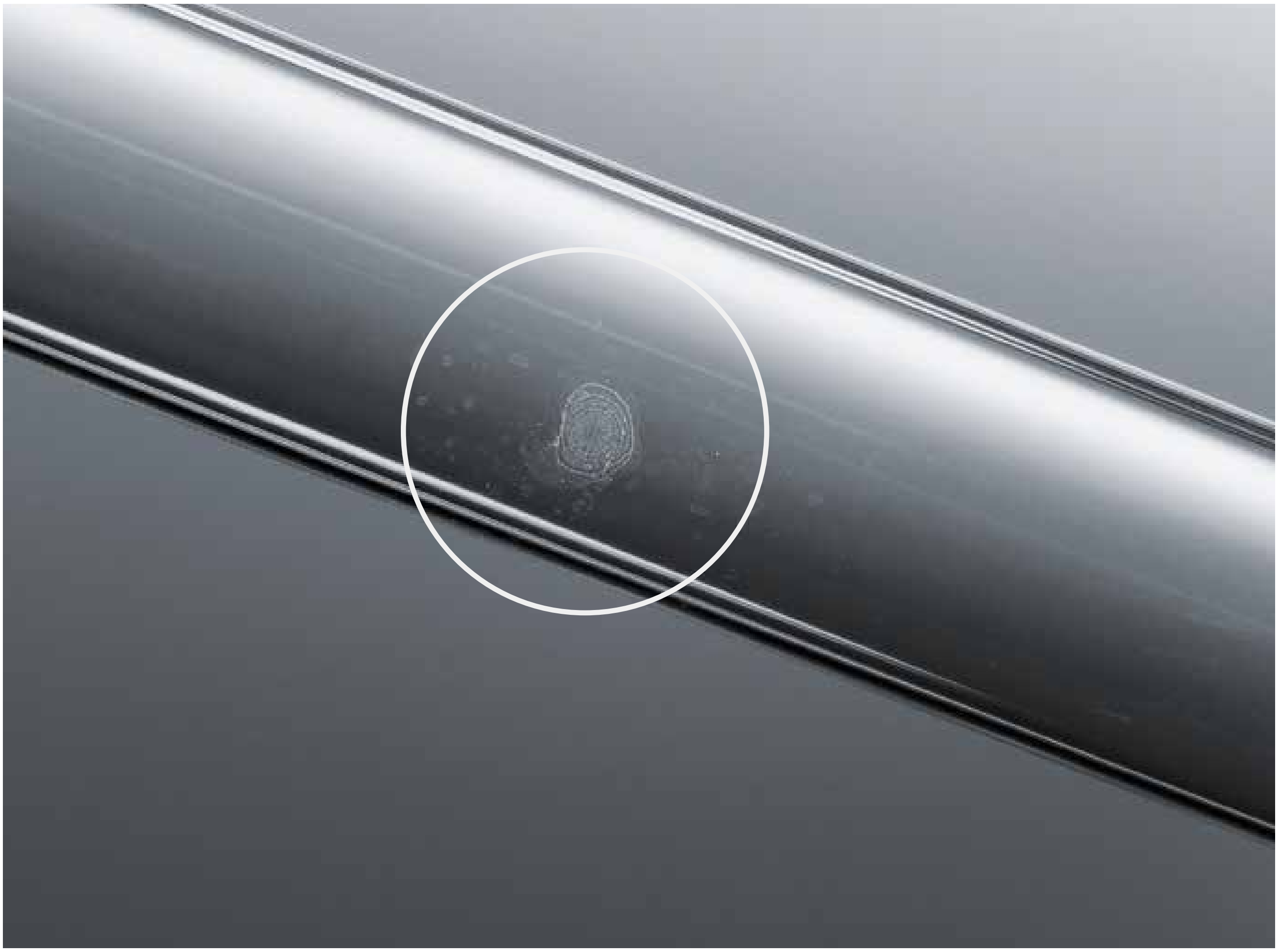
Cause Glass particles may form from unforeseen disturbances in production when tubing is cut. The glass particles in the photo are only visible under a microscope (see last paragraph of page 9). The number of glass particles has been proven lower in closed tubes.

Outside diameter approx. 17.5 mm
Wall thickness approx. 1.1 mm
Magnification approx. 5x

Coating marks

Definition Surface impurities consisting of organic coating.

Cause Drops of spray can form on the walls of the coating chamber and sometimes fall onto the surface of the tube. Coating the tubing surface reduces the formation of scratches by minimizing the friction coefficient of glass with contact materials.

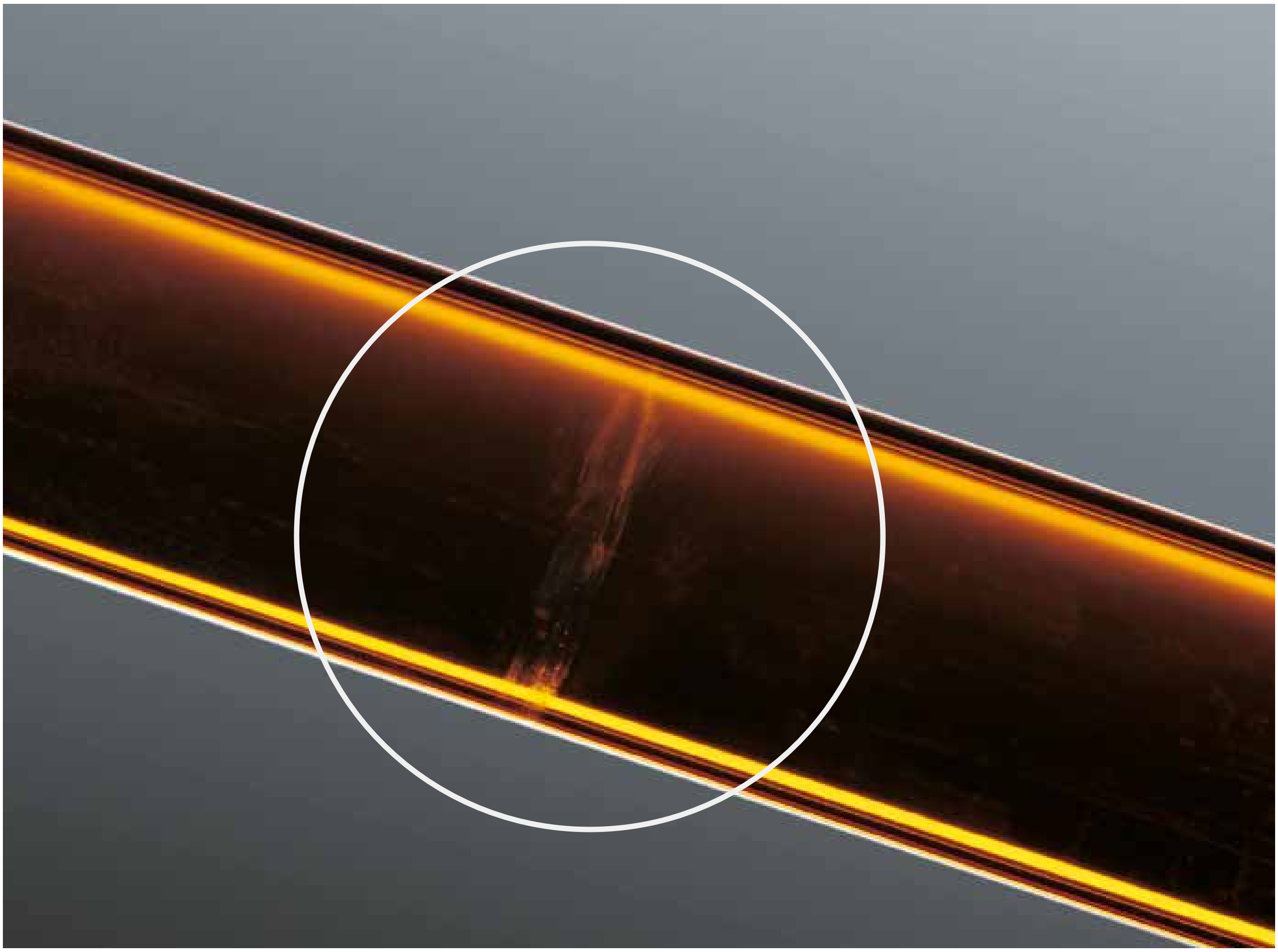


Outside diameter approx. 12.5 mm
Wall thickness approx. 0.5 mm
Magnification approx. 5.4x

Water marks

Definition Surface impurities from water.

Cause Water marks are primarily caused by process disruptions. Water condensation can also form on the inner surface of the tube during the fusing process of tube ends. In certain cases it can be caused on the outer surface by old tube-cutting equipment.



Outside diameter approx. 25 mm
Wall thickness approx. 1.2 mm
Magnification approx. 2.5x

Dust rings

Definition A circular shape made of dust particles.

Cause Dust rings are caused by tubes coming into contact with radial movement during processing. Dust rings of this type are easily wiped off.

Outside diameter approx. 9 mm
Wall thickness approx. 0.5 mm
Magnification approx. 6x

Faulty tubing ends

Definition Tube ends that do not have the specified shape.

Cause Faulty tubing ends are caused by longitudinally cut tubing that remains too long in the heating area.

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